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Full Length Research Paper

Technological and chemical properties of heat-treated Anatolian black pine wood

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In this study the effect of heat treatment on air-dry density (D_m), oven-dry density (D_0), shrinkage (β), swelling (α), fiber saturation point (FSP), compression strength parallel to grain ($\sigma_{c//}$), bending strength (σ_b), modulus of elasticity (MOE) in bending, Brinell-hardness (H_B), equilibrium moisture content (EMC), chemical content and cellulose crystallinity of Anatolian black pine [*Pinus nigra* J.F. Arnold subsp. *nigra* var. *caramenica* (Loudon) Rehder] was evaluated. Specimens were subjected to heat under atmospheric pressure at 3 different temperature (130, 180 and 230°C) and 2 different time levels (2 and 8 h). The value of D_m, D_0, β, α, FSP, EMC, $\sigma_{c//}$, σ_b, MOE, holocellulose, 1% NaOH and alcohol solubility decreased, whereas, lignin content increased depending on the heating temperature and the time. Cellulose crystallinity of the specimens was not changed significantly. 130°C showed minimum effect, on the other hand, 230°C showed maximum effect on all properties of treated wood. Accordingly, for heat treatment process, 130°C for 2 h should be applied in place where mechanical properties are important. However, 230°C for 2 h should be used in place where a physical property is preferred. Consequently, heat treated Anatolian black pine woods could be utilized in applications for several purposes such as kitchen furniture, outdoor furniture and windows frames.

Key words: Anatolian black pine, heat treatment, physical, mechanical, chemical properties, FT-IR, crystallinity.

INTRODUCTION

Wood has been used for many applications because of its many excellent properties (such as a good strength to weight ratio, aesthetic appearance etc). However, wood also suffers a number of disadvantages because wood is a hygroscopic material. Many studies have been done in order to improve the disadvantageous properties of wood. The methods brought out based on the results of these studies are commonly named “wood modification methods”. “Heat treatment” is a wood modification method, too (Hill, 2006).

Heat treatment changes some physical, mechanical and chemical properties of wood, that is, dimensional stability, equilibrium moisture content (EMC), color of wood, bending strength, corresponding strength, hardness, amount of wood polymers, biological durability etc. (Mazela et al., 2004; Yıldız et al., 2006; Gündüz et al., 2008). In this process, hemicelluloses s tart to decompose, lignin softens, cellulose and hydrophilic groups modify (Bekhta and Niemz, 2003). As a result, treated wood with high temperatures loses its reabsorbing water capacity contrary to hydrophilic behavior of the conventionally dried wood (Kocaefe et al., 2007). All these changes are achieved by heat-treatment process without any added chemicals. Thus, heat treated wood has been considered as an ecological alternative material to impregnated wood (Kamdem et al., 2000; Gündüz et al., 2008).

Studies on the effects of heat treatment on properties of Turkish native woods are rather limited. The aim of this study was to determine the effect of heat treatment on some physical, mechanical and chemical properties of Anatolian black pine, which is one of the most common naturally grown wood species in Turkey.

MATERIALS AND METHODS

Material

The Anatolian black pine [*Pinus nigra* J.F. Arnold subsp. *nigra* var. *caramenica* (Loudon) Rehder] trees were randomly selected and

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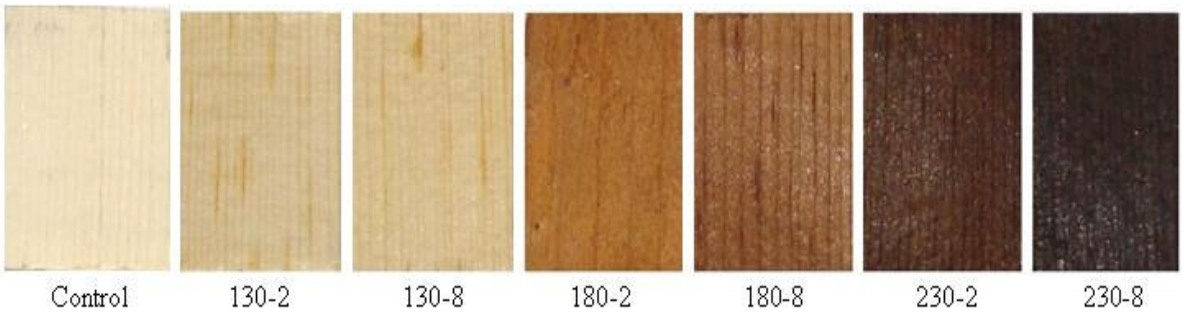

Figure 1. Color change with increasing heat treatment temperature (130, 180 and 230°C) and time (2 and 8 h).

those with a breast height diameter (DBH) of 30 - 40 cm was obtained from obtained from forestlands of the Kastamonu province, Turkey. The lumbers were cut in parallel to grain directions from the logs in sawmill according to the Turkish standard, TS 4176. Afterwards, lumbers were air dried until they reach approximately 12% MC.

Preparation of physical and mechanical test specimens and method of the tests

Anatolian black pine lumbers were planed and then cut in to small clear specimens for determination of air-dry density (D_m), oven-dry density (D_0), moisture content (MC), EMC (20 × 20 × 30 mm), shrinkage (β), swelling (α) (30 × 30 × 15 mm), compression strength parallel to grain ($\sigma_{c//}$) (20 × 2 0× 30 mm), bending strength (σ_b), modulus of elasticity (MOE) in bending (20 × 20 × 320 mm) and Brinell-hardness (H_B) (50 × 50 × 50 mm) according to TS 2470. All the experiments were conducted according to Turkish standards to determine density (TS 2472), MC, EMC (TS 2471), β (TS 4083), α (TS 4084), $\sigma_{c//}$ (TS 2595), σ_b (TS 2474), MOE in bending (TS 2478) and H_B (TS 2479).

Heat treatment

The temperature of the oven is increased to the temperature at which the actual heat treatment occurs. This heating period was taken as 1 h for all trials. Excess amount of splitting and color differences may occur if the moisture content of the material is too high (r > 10%) before heat treatment (Syrjanen, 2001). There-fore, specimens were conditioned to 7% moisture content at 25 ± 2°C and 35 ± 5% relative humidity to minimize such defects.

Heat treatment applications were conducted in a temperature controlled small heating unit. 3 different temperatures (130, 180 and 230°C) and 2 different durations (2 and 8 h) were applied to specimens under atmospheric pressure and in the presence of air. After treatment, the temperature is decreased to room temperature that takes about 24 h. The same procedure was conducted for all experiments. After heat treatment, the MC of specimens was measured.

Preparation of chemical analyses test specimens and method of the tests

Before the chemical analyses, heat-treated and untreated (control) specimens were cut to a length of 1 - 2 cm and ground in a Wiley mill to a homogeneous meal. To remove low molecule weight carbohydrates, degraded celluloses and polyoses, 1% NaOH solution was applied to control and treated wood specimens according to TAPPI T-212 om-88. Alcohol syclohegzane solubility (TAPPI T 204 om-88) was applied to extract oils, waxes, resins, non-volatile compounds, low molecule weight carbohydrates, salts and dissolvable other compounds. Additional alcohol solution (TAPPI T 204 om-88) was applied to remove some other extractives such as tannins, pigments and stilbenes. Holocellulose analysis was made according to Wises's sodium chlorite method (Wise et al., 1946). Lignin content was determined as acid-insoluble Klason lignin by Runkel method (Runkel and Wilke, 1951) using 72% sulfuric acid and 40% hydrobromic acid. In this procedure lignin is left as an insoluble residue and is recovered by filtration and the amount is gravimetrically determined.

FT-IR spectroscopy

Before analyses, heat-treated and control specimens were ground in a Wiley mill to homogeneous meal. Control and heat-treated wood powder specimens were used for Fourier transform infrared (FT-IR) spectroscopy measurement. The dried specimens were embedded in potassium bromide (KBr) pellets and analyzed by using a Nicolet 20SX FT-IR spectrophotometer. They were recorded in the absorption mode in the range of 4000 - 400 cm^{-1} with an accumulation of 64 scans, resolution of 4 cm^{-1}. These spectra were normalized at 2900 cm^{-1} (C-H stretching vibration).

RESULTS AND DISCUSSION

The specimens were visually checked after heat treatment process. The defects were found to be at minimum level. The changes were obtained by calculating the property difference between heat treated wood and untreated same species as a % of untreated wood property.

Figure 1 shows color changes on heat treated specimens with heat treatment. The color became significantly darker with treatment temperature and time compared with control specimens. Increase in lignin content with heat treatment temperature and time justifies these results.

Cellulose and hemicelluloses in untreated wood do not absorb light in the visible region, therefore, they do not contribute to change color. However, colored by products formed during the degradation of hemicelluloses might have a contribution to this change (Kocaefe et al., 2008).

A thermal treatment always results in darkening of the wood (Ayadi et al., 2003; Sundqvist, 2002). Heat treated woods acquire a darker color similar to most tropical

Table 1. Correlation matrix for the parameters characterizing heat treatment temperature and time with the parametric Pearson's correlation.

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
Temperature (1)	1.000	0.457**	-0.556**	-0.664**	-0.656**	-0.883**	-0.809**	-0.448**	-0.603**	-0.520**	-0.437**	-0.460**	-0.735**	0.649**	-0.992**	-0.645**
Time (2)		1.000	-0.373*	-0.103	-0.162	-0.370*	-0.517**	-0.207	-0.391*	-0.144	-0.189	-0.361*	-0.340*	0.414*	-0.541**	-0.553**
D_0 (3)			1.000	0.350*	0.542**	0.680**	0.713**	0.407*	0.476**	0.584**	0.692**	0.683**	0.739**	-0.768**	0.545**	0.099
β_v (4)				1.000	0.748**	0.736**	0.597**	0.399*	0.626**	0.625**	0.473**	0.503**	0.760**	-0.643**	0.628**	0.050
α_v (5)					1.000	0.775**	0.628**	0.523**	0.611**	0.665**	0.598**	0.616**	0.801**	-0.703**	0.622**	0.018
EMC (6)						1.000	0.890**	0.483**	0.719**	0.721**	0.683**	0.751**	0.923**	-0.845**	0.863**	0.279
Compression strength (7)							1.000	0.399*	0.717**	0.713**	0.664**	0.739**	0.865**	-0.867**	0.799**	0.319
MOE (8)								1.000	0.511**	0.158	0.497**	0.381*	0.498**	-0.464**	0.438**	0.118
Bending strength (9)									1.000	0.622**	0.648**	0.658**	0.762**	-0.772**	0.587**	0.116
Tangential Brinell-hardness (10)										1.000	0.699**	0.786**	0.832**	-0.816**	0.470**	-0.166
Radial Brinell-hardness (11)											1.000	0.849**	0.817**	-0.850**	0.397*	-0.201
Cross-section Brinell-hardness (12)												1.000	0.882**	-0.910**	0.440**	-0.203
Holocellulose (13)													1.000	-0.962**	0.706**	0.006
Lignin (14)														1.000	-0.518**	0.171
1% NaOH sol. (15)															1.000	0.691**
Alcohol-cyclohexane (16)																1.000

**Correlation is significant at the 0.01 level (2-tailed).
*Correlation is significant at the 0.05 level (2-tailed).

woods, which is an aesthetical advantage for some applications (Bekhta and Niemz, 2003; Mitsui et al., 2001; Mitsui, 2004; Mitsui et al., 2004; Mitsui, 2006).

Treatment temperature is highly correlated with all physical, mechanical and chemical properties of Anatolian black pine wood statistically ($P < 0.01$). However, treatment time is only correlated with compression strength, 1% NaOH solubility and alcohol-cyclohexane solubility ($P < 0.01$). Heat treatment time is also correlated with D_0, EMC, bending strength, cross section Brinell-hardness, lignin and holocellulose ($P < 0.05$). Also, physical, mechanical and chemical properties are strongly correlated with each other ($P < 0.01$) (Table 1).

Figure 2 displays the changes as % on D_m, D_0, β_t, β_r, β_v, α_t, α_r, α_v, fiber saturation point (FSP), EMC of heat treated Anatolian black pine wood at 130, 180 and 230°C for 2 h and 8 h. All values of the physical properties decreased with temperature and duration of process and physical properties are strongly affected negatively by treatment temperature. But, the effects of time were not significantly (Table 1). In this case it can be said that temperature might have greater affect on physical properties than time.

The lowest D_m, D_0 and EMC were obtained at 230°C for 8 h and maximum reductions were obtained as 13.4, 12.7 and 49.7%, respectively. Also, maximum variations for β_t, β_r, β_v, α_t, α_r, α_v and FSP were obtained at 230°C for 2 h as 41.3, 28.0, 37.3, 46.9, 40.2, 44.9 and 36.2, respectively, compared with the control sample.

While the maximum effect of heat treatment was recorded at 230°C, the minimum effect was recorded at 130°C for both time levels. Similarly, Gündüz et al. (2008) studied the effects of heat treatment on physical properties and surface roughness of Camiyanı black pine (*P. nigra* Arn. subsp. *pallasiana* var. *pallasiana*) wood. Their research indicated that reduction in density, swelling, compression strength, Janka-hardness and surface roughness values were observed with the increase in heat treatment time and temperature. Another study showed similar results for Scots pine (*Pinus sylvestris* L.) and beech (*Fagus orientalis* Lipsky) wood (Korkut et al., 2008a). Also, Korkut and Guller (2008) reported a reduction in physical properties, oven-dry density, air-dry density and swelling and surface roughness of wood.

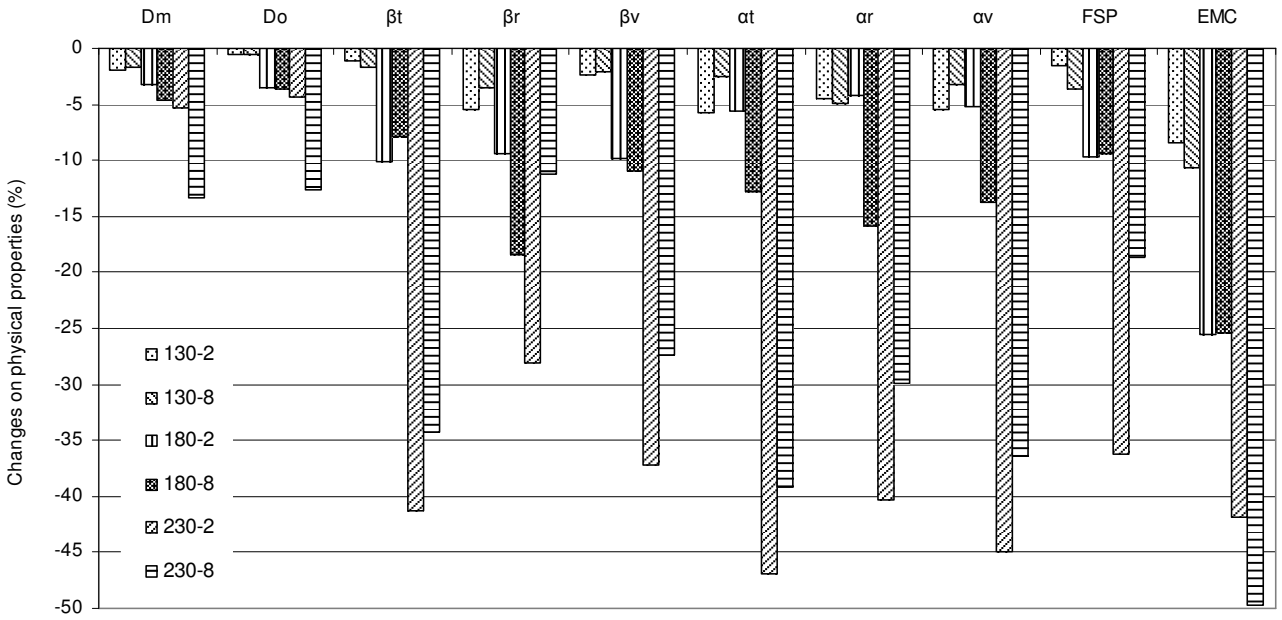

Figure 2. Changes (%) in physical properties as a function of treatment temperature (130, 180 and 230°C) and time (2 and 8 h).

Wood subjected to high temperatures loses its capacity to reabsorb water on the contrary to the hydrophilic behavior of the conventionally dried wood (Kocaefe et al., 2007). An average improvement in hygroscopicity of 40% has been found (Tjeerdsma et al., 1998; Boonstra et al., 1998). Also, Esteves et al. (2007) reported the EMC decrease (46% for pine and 61% for eucalyptus) as well as the dimensional stability increase (maximum anti-shrinking efficiency in the radial direction of 57 and 90% for pine and eucalyptus, respectively).

Heat treated wood at high temperature has lower hygroscopicity than untreated wood. Since decrease of hydroxyl groups on carbohydrate chains, cell wall of heat treated wood absorbs less water. As a consequence of the reduced number of hydroxyl groups, swelling and shrinking are lower.

Figure 3 shows the changes on $\sigma_{c//}$, MOE in bending, σ_b and H_B (tangential-H_{Bt}, radial- H_{Br} and cross section-H_{Bc}) of treated specimens with compared to the control specimens. Also, the Pearson's correlations between mechanical properties and treatment temperature and time were given in Table 1. As can be seen from Table 1, all mechanical properties of Anatolian black pine wood have negatively correlation with treatment time and temperature.

The higher treatment temperature gives the better biological durability. But at the same time, mechanical properties of wood are affected negatively (Jamsa and Viitaniemi, 2001).

The values of all measured mechanical properties decreased except for hardness with increasing temperature and time (Figure 3). This is probably due to the break-up of the hemicelluloses and cellulose polymers. The hardness slightly increases with both temperature and time up to 180°C for 8 h and than, it shows decreasing trend due to the further structural degradation.

For all mechanical properties, maximum decreases were recorded at the treatment of 230°C for both 8 and 2 h. Compared to the control sample, total loss in $\sigma_{c//}$, MOE, σ_b, H_{Bt}, H_{Br} and H_{Bc} were obtained as 57.6, 16.9, 58.0, 41.1, 58.9 and 40.5%, respectively. However, minimum reduction in were obtained at the treatment of 130°C for $\sigma_{c//}$, MOE and σ_b. However, while increasing H_B value at 130 and 180°C for both 8 and 2 h, it decreased at 230°C for both time levels for all sections of the wood specimens. The maximum hardness value was obtained as 4.54 kgf mm^{-2} at 130°C for 2 h.

These results can be explained with material loses in cell wall and hemicelluloses degradation depend on the applied high temperature after heat treatments. Decrease in strength is mainly due to the depolymerization reactions of wood polymers (Kotilainen, 2000). Furthermore, the wood density can play a role on the mechanical properties.

Similar results for heat treated wood about reductions in mechanical strength properties were reported by Santos (2000), Poncsak et al. (2006), Shi et al. (2007), Korkut (2008), Korkut et al. (2008b) and Unsal and Ayrilmis (2005). Also, Esteves et al. (2007) reported increasing mass losses with treatment time and temperature for pine and eucalyptus woods. Besides, little effect on modulus of elasticity (5% for pine and 15% for eucalyptus) and reducing bending strength (by 40% for pine and 50% for eucalyptus) were recorded by Esteves et al.

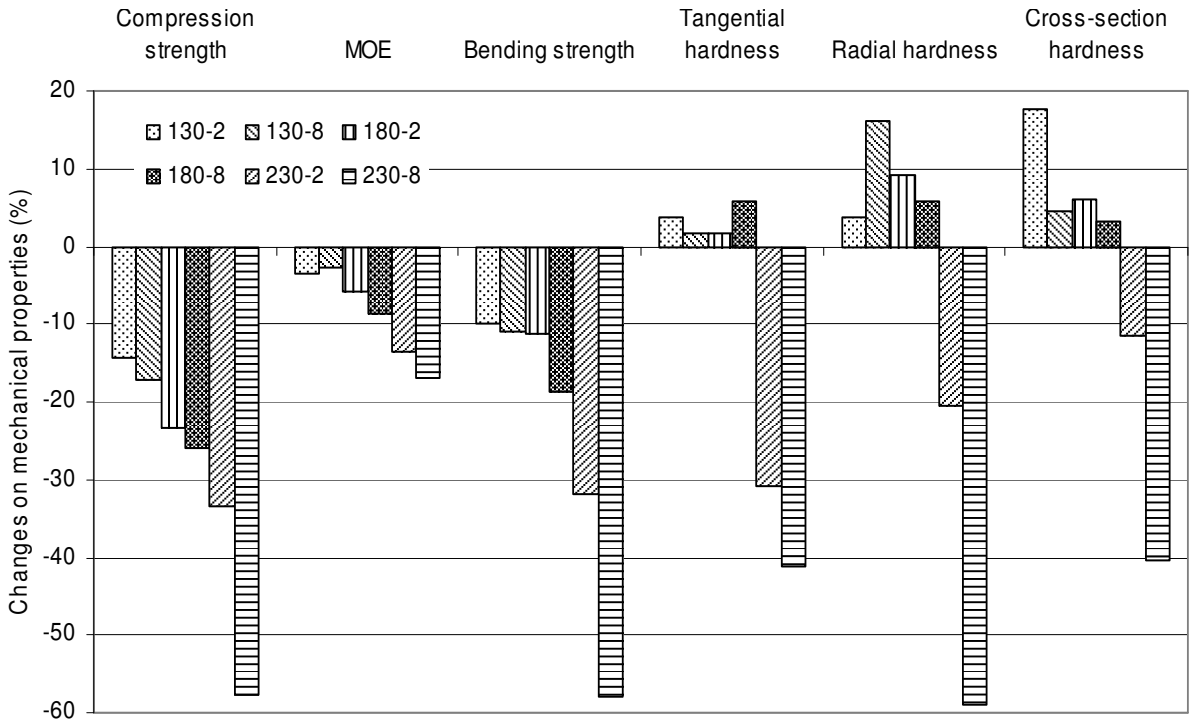

Figure 3. Changes on mechanical properties as a function of treatment temperature (130, 180 and 230°C) and time (2 and 8 h).

(2007).

Changes on some chemical contents of the heat treated Anatolian black pine wood specimens compared to control sample are showed in Figure 4. Holocellulose content changed negatively with increasing temperature and time. Similarly, according to Pearson's correlations results Table 1 holocellulose content is correlated negatively with temperature and time of heat treatments as $r = 0.74$ ($P < 0.01$) and $r = 0.34$ ($P < 0.05$) respectively.

Maximum decrease of holocellulose content was found at 230°C for 8 h as 29.9% and minimum decrease was obtained at 130°C for 2 h as 0.1%. Heat treatment significantly degraded the wood carbohydrate, suggesting depolymerization and alterations through the cleavage of acetic acid from the acetyl side chains (Kartal et al., 2008).

Lignin values increased with rising treatment temperature and time that is justified by Pearson's correlations results (Table 1). Lignin content is correlated positively with temperature and time of heat treatment as $r = 0.65$ ($P < 0.01$) and $r = 0.41$ ($P < 0.05$) respectively. The highest lignin contents were obtained from the variations at 230°C for both 2 h (34.03%) and 8 h (42.7%). This result confirmed most of the conclusions stated in the literature (Kartal et al., 2008; Boonstra and Tjeerdsma, 2006; Kamdem et al., 2002). In other words, increase in lignin content during the thermal treatment was explained that, some of the thermal degradation products of carbohydrates may be retained in the lignin fraction with Klason lignin analyses (Yildiz et al., 2006).

According to Figure 4, 1% NaOH and alcohol – sycloheg zane + alcohol solubility values of the Anatolian black pine wood specimens decreased with rising treatment time and temperature in comparison with control sample. Pearson's correlations results justify this statement (Table 1). It is well known that, extractives are not structural components and most of the compounds evaporate easily during the heat treatment.

Shrinkage and swelling behavior of the heat treated wood was affected positively with increasing of lignin ratio. However, this improvement not only depends on the increasing lignin ratio but also non-damaged carbohydrates with crystalline structure. Besides color darkening occurs with increasing lignin amount in heat treated wood structure. Figure 1 justifies this expression.

FT-IR spectra were recorded for treated specimens at 130, 180 and 230°C for 2 and 8 h and for control sample (Figure 5). For Anatolian black pine wood specimens, the band at 1426 cm^{-1} (CH_2 bending for cellulose) moved up with heat treatment compared to control (Figure 4). Peak shoulder at 1507 cm^{-1} (C=C stretching aromatic ring for lignin) for specimens was not changed by heat treatment, evidently. The band at 1106 cm^{-1} (asym. bridge C-O-C stretching for cellulose) did not change in specimens by heat treatment at 130 and 180°C compared to control wood sample, clearly. However, the band at 1106 cm^{-1} moved up clearly at 230°C (Figure 5). Similar results were found by Akgül et al. (2006). At 1635 cm^{-1}, peak

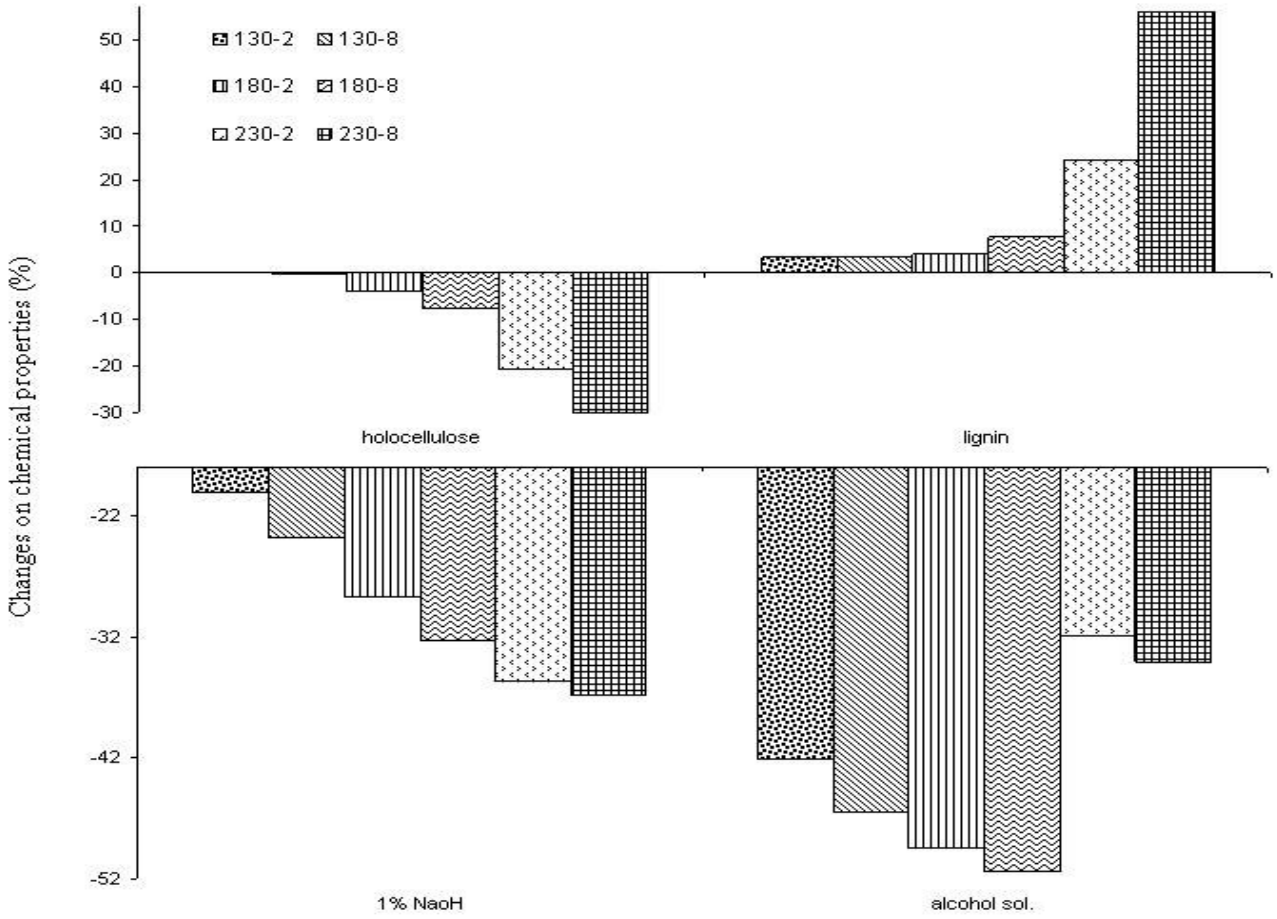

Figure 4. Changes on chemical properties as a function of heat treatment (130, 180 and 230°C) and time (2 and 8 h).

shoulder increased by rising heating temperature and time. Band at 1635 cm^{-1} and band at 1736 cm^{-1} (C=O valance vibration of COOH group) have almost the same absorbance value for all specimens.

The degree of crystallinity is one of the important parameters for polymers. The physical and mechanical properties of polymers depend on the degree of crystallinity (Mo et al., 1994). FT-IR spectroscopy method was used for determination of crystallinity index (Table 2). The absorbance peaks at 1430 cm^{-1} and 897 cm^{-1} were assignned to CH_2 bending mode and deformation of anomeric CH_2, respectively (Kataoka and Kondo, 1998). The rations of the absorbency at A_{1430}/A_{897} and A_{1371}/A_{2900} have been used to measure the relative cellulose crystallinity (Hassan et al., 2000). Meanwhile, the ratios at A_{1371}/A_{690} and A_{1371}/A_{670} can be used as an indicator for of the transformation of cellulose I and cellulose II during alkaline treatment (Hassan et al., 2000; Akerholm et al., 2004). This method actually uses the ratio of the combined areas of the peaks at 1370, 1335 and 1315 cm^{-1} which represented the CH bending, to that of the peak at 670 cm^{-1} (C-OH out of plane bending mode) (Evans et al., 1995).

While cellulose shows crystalline structure, hemicelluloses and lignin show amorphous structure within chemical composition of wood. Heating of wood modify the cell wall components. The most effected compounds in the chemical structure are hemicelluloses, cellulose and lignin. Similarly in this study, significantly decreasing holocellulose ratio and increasing lignin ratio in the wood structure were determined. Carbohydrate compounds with amorphous characteristics are affected negatively by heat treatment. According to the researchers, crystalline structure of cellulose is not changed (Yildiz and Gümüşkaya, 2007) or even can improve up to a certain temperature, which may be as high as 200°C depending on the conditions involved. The results confirm that crystallinity indexes of the celluloses are not affected from different heat treatment times and temperatures (Table 2).

Conclusions

According to our results, air-dry density, oven-dry density, shrinkage (tangential, radial and volumetric), swelling (tangential, radial and volumetric), FSP, MMC and EMC values decreased by increasing temperature and time. Similarly, compression strength parallel to grain, bending

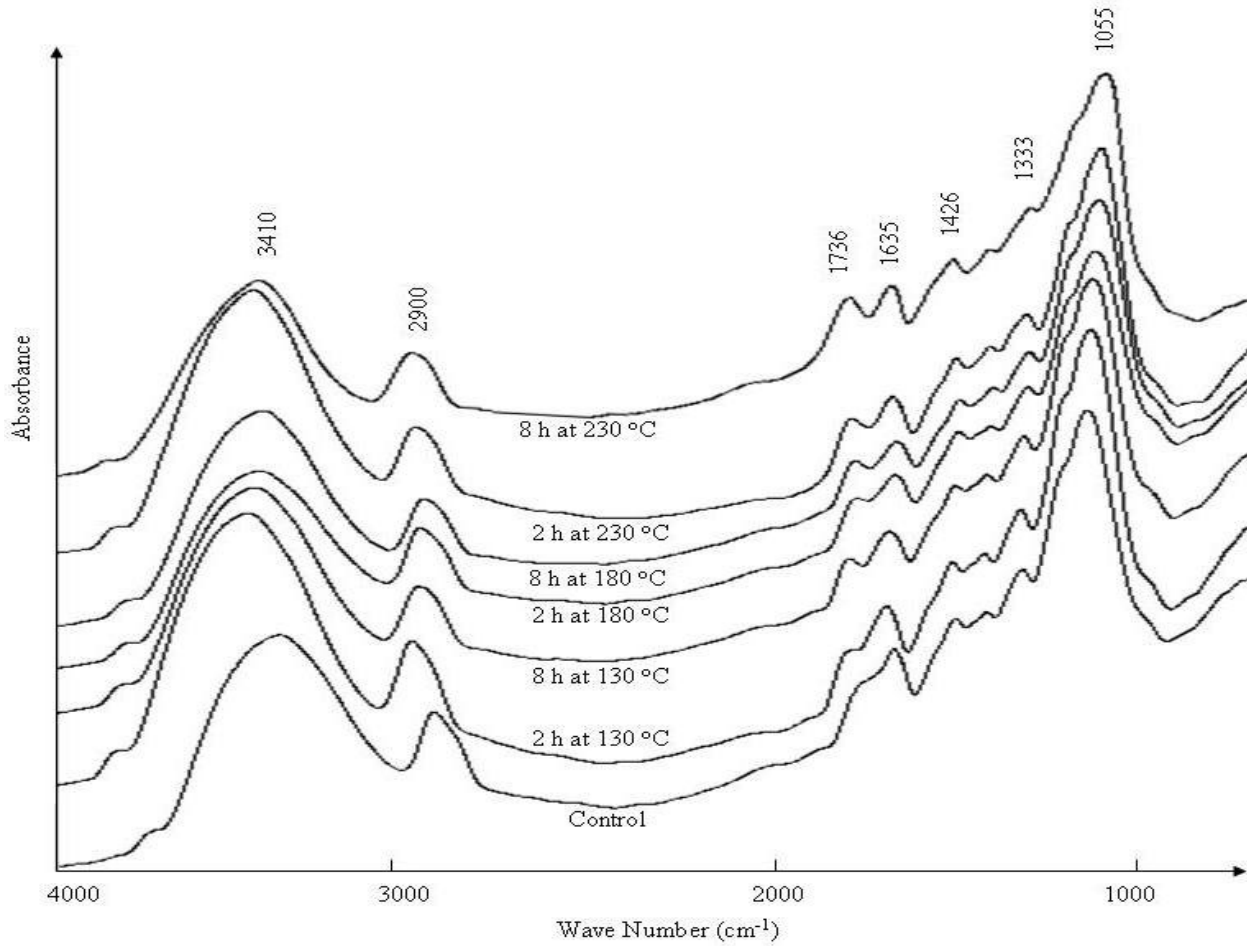

Figure 5. FT-IR spectra of untreated and heat treated Anatolian black pine wood.

Table 2. The crystallinity indexes of heat treated black pine wood.

Absorbency	Control	130°C		180°C		230°C	
		2 h	8 h	2 h	8 h	2 h	8 h
A_{1427}/A_{898}	0.99	1.04	1.01	0.95	1.00	1.07	1.02
A_{1371}/A_{2900}	1.23	1.24	1.41	1.83	1.67	1.29	1.45
A_{1371}/A_{670}	0.97	1.00	0.99	0.91	1.00	1.03	1.08
A_{1371}/A_{690}	0.99	1.02	1.01	0.95	1.04	1.08	1.12

strength and MOE in bending values decreased. But hardness values only decreased at the heat treatment of 230°C for both times (2 and 8 h). However, while holocellulose ratio, 1% NaOH and alcohol solubility of the heat treated wood decreased, lignin content values increased depending on heat treatment temperature and time. Cellulose crystallinity of the specimens was not changed significantly.

The lowest decreases were determined at the heat treatment of 130°C for 2 h. Treatment temperature is highly correlated with all physical, mechanical and chemical properties of Anatolian black pine wood. Heat treatment can be considered as an environmentally friendly technique because no chemicals are involved during the process.

In this case it can be said that temperature has greater effect on strength properties than time. For heat treatment process, 130°C for 2 h should be applied in place where mechanical properties are important. However, 230°C for 2 h should be used in place where physical properties are preferred. As a result, heat treated woods can be utilized (with proper heat treatment time and temperature) without any loses in strength values and chemical characteristics in areas, where woodworking

and stability are important.

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