

Technological and chemical properties of chestnut (*Castanea sativa* Mill.) wood after heat treatment

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Abstract

This study was performed to determine the effects 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 chestnut (*Castanea sativa* Mill.) wood. Wood samples were treated to heat at three different temperature (130, 180 and 230°C) and two different time level (2 and 8 h) under atmospheric pressure and presence of air. D_m , D_0 , β , α , FSP, EMC, $\sigma_{c//}$, σ_b and MOE were decreased by heat treatment. Lignin content, 1% NaOH and alcohol solubility values increased with depend on the heating temperature and time. Cellulose crystallinity of the samples was not changed significantly.

Consequently, 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 preferred physical properties. Heat-treated chestnut woods could be utilized by using proper heat treatment techniques in outdoors and indoors applications for several purposes such as musical instruments, kitchen furniture, garden furniture and windows frames.

Keywords: Chestnut, Heat treatment, Physical properties, Mechanical properties, Chemical properties, FT-IR

Introduction

It is well known that wood is a hygroscopic material. The flux of water molecules into the cell wall is exactly balanced by the outward flux into the atmosphere. Nevertheless, wood has been utilized for many applications because of its many excellent material properties such as a good strength to weight ratio, aesthetic appearance, acoustic properties etc. But wood also suffers a number of disadvantages. Many studies have been done in order to ameliorate the properties of wood. The methods brought out based on the results of these studies are commonly named "wood modification methods". "Heat treatment" is one of the wood modification methods, too (HILL [1]).

Heat treatment changes some physical, mechanical and chemical properties of wood i.e. dimensional stability, EMC, color of wood, bending strength, corresponding strength, hardness, amount of wood polymers, biological durability etc. (VIITANIEMI & al. [2]). In this

process, wood is heated to temperatures above 200°C which changes the structure of wood. Hemicelluloses start to decompose, lignin softens, and cellulose and hydrophilic groups modify (BEKHTA & NIEMZ [3] and WEILAND & GUYONNET [4]). As a result, treated wood with high temperatures loses its reabsorbing water capacity contrary to hydrophilic behavior of the conventionally dried wood (KOCAEFE & al. [5]). All these changes are achieved by heat-treatment process without addition of any chemicals. Thus, heat treated wood has been considered as an ecological alternative to chemically treated wood material (KAMDEN & al. [6]).

Chestnut (*Castanea sativa* Mill.), naturally grown in Turkey and covering over 88000 ha, has superior technological properties and high utilization potential in the forest based industry (ANONYMOUS [7]).

Studies on the effects of heat treatment on the properties of Turkish native trees are rather limited. The aim of this study was to determine the effect of heat treatment on some physical, mechanical and chemical properties in chestnut (*Castanea sativa* Mill.) wood, which is one of the most commonly used wood species naturally grown in Turkey.

Materials and methods

Material

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

Preparation of physical and mechanical test samples and application for experiments

Chestnut lumbers were planed and then cut to small clear specimens for determination of D_m , D_0 , MC, EMC (20x20x30 mm), β , α (30x30x15 mm), $\sigma_{c//}$ (20x20x30 mm), σ_b , MOE in bending (20x20x320 mm) and H_B (50x50x50 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. If the MC of the material is too high ($r > 10\%$) before heat treatment, a lot of splitting and color differences may result (SYRJANEN & OY [8]). Therefore all samples were conditioned to 7% MC at $25 \pm 2^\circ\text{C}$ and $35 \pm 5\%$ relative humidity to prevent splitting of woods during heat treatment.

Heat treatment applications were conducted in a temperature controlled small heating unit. Three different temperatures (130, 180 and 230°C) and two different durations (2 and 8h) were applied for chestnut wood species under atmospheric pressure and in the presence of air. After treatments, the temperature is decreased to room temperature taken about 24 hours. The same procedure was run for all experiments. After heat treatments, the MC of samples was measured again.

Preparation of chemical test samples and application of experiments

Before the chemical analyses heat-treated and untreated (control) samples 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 samples according to TAPPI T-212 om-88. We applied the alcohol syclohegzane solution (TAPPI T 204 om-88) 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 Wisés's sodium chlorite method (WISE & al. [9]). Lignin content was determined as acid-insoluble Klason lignin by Runkel method (RUNKEL & WILKE [10] 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 chestnut wood samples were ground in a Wiley mill to homogeneous meal. Control and heat-treated wood powder samples were used for Fourier Transform Infrared (FT-IR) spectroscopy measurement. The dried samples 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 $4000\text{--}400\text{ cm}^{-1}$ 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

Color changes with heat treatment temperature and application time were shown in Figure 1. The color became significantly darker with increasing treatment temperature and time compared with control samples. Increase in lignin content of the treated wood may cause this changing.



Figure 1. Color change with increasing heat treatment temperature and time

The color characteristics of untreated wood depend on the chemical components. Cellulose and hemicelluloses in untreated wood don't absorb light in the visible region; therefore, they don't contribute to color change. However, colored byproducts formed during the degradation of hemicelluloses might have a contribution to this change (KOCAEFE & al. [11]).

A thermal treatment always results in darkening of the wood. Plato treated wood had a light brown color, but still retained the appearance of natural wood (AYADI & al. [12] and SUNDQVIST [13]). Heat treated woods acquire a darker color similar to most tropical woods, which is an aesthetical advantage for some applications (BEKHTA & al. [3], MITSUI [14], UNSAL & al. [15]).

As can be seen from Table 1 that, treatment temperature is highly correlated with all physical, mechanical and chemical properties of chestnut wood statistically ($P < 0.01$). However,

the time is only correlated with compression strength and MOE for $P < 0.01$ and correlated with EMC, bending strength, tangential Brinell-hardness and holocellulose for $P < 0.05$.

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)	1,00	0,457*	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Time (2)	0	*	0,647**	0,747**	0,821**	0,843**	0,673**	0,605**	0,846**	0,556**	0,632**	0,420*	0,754**	0,665**	0,922**	0,634**	
D_m (3)		1,000	-0,312	-0,249	-0,324	-0,355*	0,578**	0,455**	-0,423*	-0,344*	-0,285	-0,180	-0,390*	0,274	0,164	0,192	
D_0 (3)			1,000	0,755**	0,756**	0,702**	0,397*	0,757**	0,797**	0,576**	0,716**	0,577*	-	-	-	-	
β_t (4)				1,000	0,970**	0,929**	0,557**	0,710**	0,836**	0,774**	0,873**	0,610*	-	-	-	-	
β_r (4)					1,000	0,953**	0,600**	0,766**	0,893**	0,773**	0,879**	0,665*	-	-	-	-	
β_v (4)						1,000	0,669**	0,669**	0,854**	0,805**	0,868**	0,601*	-	-	-	-	
EMC (6)							1,000	0,554**	0,711**	0,532**	0,531**	0,394*	0,635**	0,527**	0,571**	-0,257	
Compression strength (7)								1,000	0,554**	0,711**	0,532**	0,531**	0,394*	0,635**	0,527**	0,571**	
MOE (8)									1,000	0,877**	0,670**	0,782**	0,821*	-	-	-	-
Bending strength (9)										1,000	0,695**	0,795**	0,714*	-	-	-	-
Tangential Brinell-hardness (10)											1,000	0,828**	0,665*	-	-	-	-
Radial Brinell-hardness (11)												1,000	0,786**	0,741**	0,600**	-0,113	
Cross-section Brinell-hardness (12)													1,000	0,747**	0,724**	0,436**	
Holocellulose (13)														1,000	0,970**	0,751**	
Lignin (14)															1,000	0,690**	
1% NaOH sol. (15)																1,000	
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).

According to temperature and duration of process, all physical properties of specimens is strongly affected negatively by the thermal treatment. Figure 2 displays the decreases on D_m , D_0 , β_t , β_r , β_v , α_t , α_r , α_v , FSP, EMC of heat treated chestnut wood at 130°C, 180°C and 230°C for two different time levels (2 h and 8h). The maximum decreases for all parameters were obtained at 230°C for both 2 h and 8h.

Heat treated wood at high temperature has less hygroscopicity than untreated wood. Because of the decrease in the amount of wood's hydroxyl groups, heat treatment causes less water absorbance of wood cell wall. As a consequence of the reduced number of hydroxyl groups the swelling and shrinking are become lower.

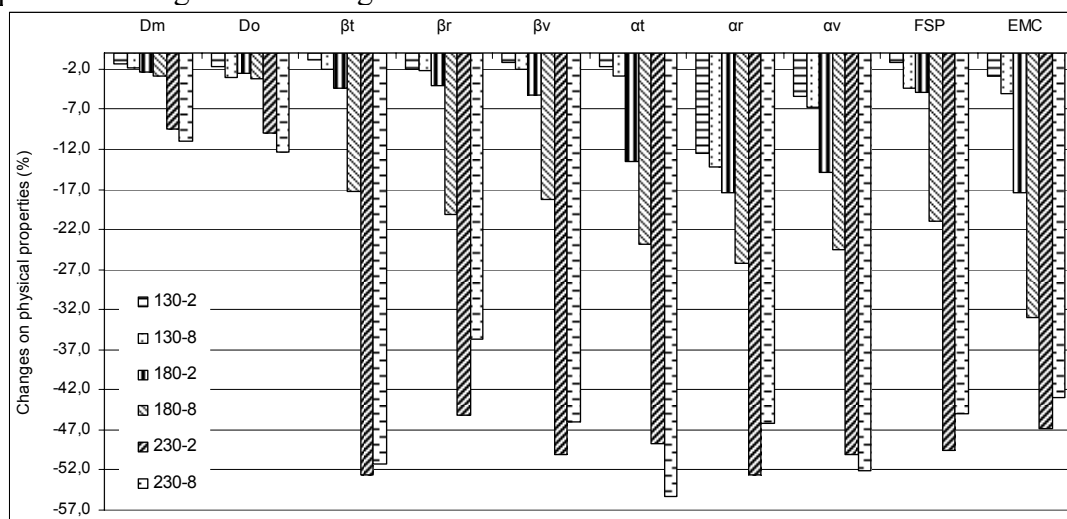


Figure 2. Percentage changes in physical properties as a function of treatment temperature and time

Regardless of the treatment times, maximum decreases on physical characteristics of the samples were obtained at 230°C. However, the minimum effect was recorded at 130°C for both time levels. Similarly, GÜNDÜZ & al. [16] studied the effects of heat treatment on physical properties and surface roughness of Camiyanı Black Pine (*Pinus nigra* Arn. subsp. *pallasiana* var. *pallasiana*) wood. Density, swelling, compression strength, Janka-hardness and surface roughness values were decreased with increasing heat treatment time and temperature. Another study showed the similar results for Scots pine (*Pinus sylvestris* L.) and beech (*Fagus orientalis* Lipsky) wood (KORKUT & al. [17] and YILDIZ & al. [18]). Also, KORKUT & GULLER [19] and KUBOJIMA & al. [20] have reported a reduction in physical properties, oven-dry density, air-dry density and swelling, and surface roughness of wood too.

The investigated properties of thermal treated wood have been found strongly dependant on the applied process types and conditions. However, regardless of the applied process, hygroscopicity, dimensional stability and durability will be improved substantially (HOMAN & al. [21]). Wood, subjected to high temperatures, loses its capacity to reabsorb water on the contrary to the hydrophilic behavior of the conventionally dried wood (KOCAEFE & al. [5]).

Heat treatment reduces certain mechanical properties, but dimensional stability and biological durability of wood increases through with heat treatment (METSÄ & al. [22]). In addition to, Figure 3 shows the changes on $\sigma_{c//}$, σ_b and MOE in bending, H_B (tangential- H_{Bt} , radial- H_{Br} and cross-section- H_{Bc}) of chestnut wood according to the control samples. Also, the Pearson's correlations between mechanical properties and treatment temperature and time were given in Table 1.

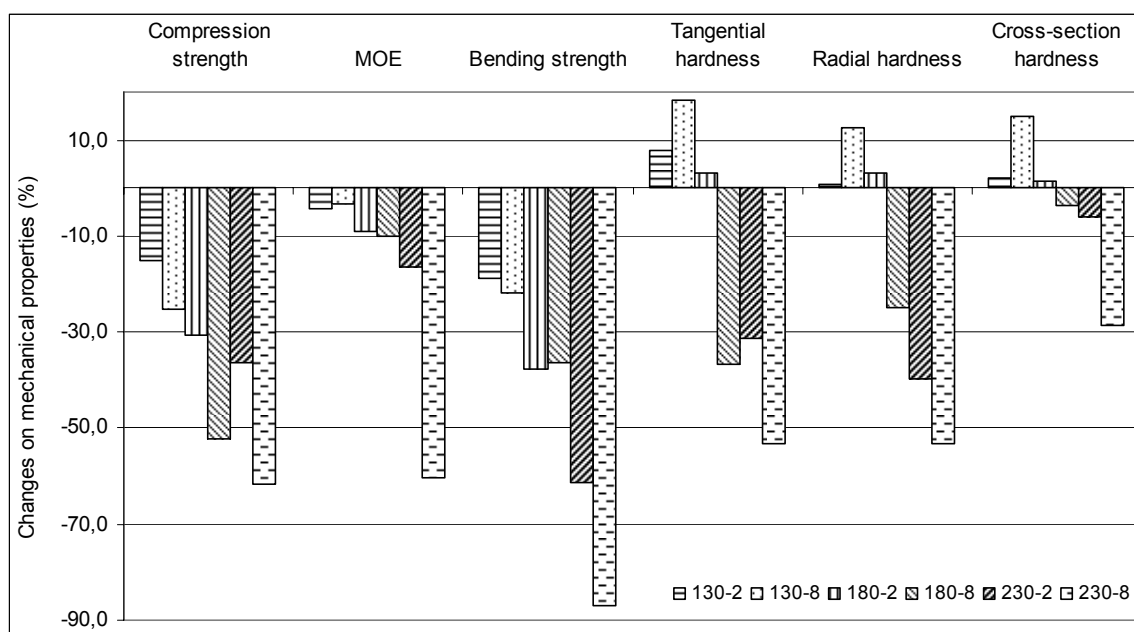


Figure 3. Changes on mechanical properties as a function of treatment temperature and time

All the 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. Although, the hardness increased slightly till 180 °C for 8 h with temperature and time but it reduced with increasing temperature and time probably due to further structural degradation.

The maximum decreases for $\sigma_{c//}$, MOE and σ_b were calculated at the same condition, as 61.6%, 60.3% and 86.9% respectively, at 230°C for 8 h. On the other hand, the minimum losses for $\sigma_{c//}$, MOE and σ_b were calculated at 130°C for 2 h or 8h. as 15.1%, 3.3% and 18.8% respectively. The maximum hardness value was obtained from the parallel to grain direction, surface of cross section as (5.06 kgf mm⁻²). The lowest values were obtained 0.88 kgf mm⁻², 0.97 kgf mm⁻² and 3.15 kgf mm⁻² for H_{Bt} , H_{Br} and H_{Bc} at 230°C for 8 h respectively. The maximum H_B losses for H_{Bt} , H_{Br} were calculated as 53.1% and 53.2% at 230°C for 8h. However, the maximum increases were calculated as 18.4%, 12.6% and 14.8% for H_{Bt} , H_{Br} and H_{Bc} at 130°C for 8h and 2h, respectively. Similar result was reported by PONCSAK & al. [23].

The higher treatment temperature gives the better biological durability. But at the same time it means the more weakened mechanical properties of wood. A negative consequence is that the wood becomes more brittle, and bending and pulling strength decrease by 10% to 30%. No changes were found in compression and impact strengths and surface hardness (JAMSA & VIITANIEMI [24]).

SHI & al. [25] studied on mechanical behavior of some Québec wood species, spruce, pine, fir, aspen and birch by using ThermoWood heat-treatment process (at 200°C or higher for 3 h). They found lower modulus of elasticity and modulus of rupture after treatment. Similarly, reductions in mechanical strength properties were reported by SANTOS [26]; PONCSAK & al. [23] UNSAL & AYRILMIS [27] and ESTEVES & al. [28]. Also HOMAN & al. [21] stated that, thermally treated wood showed an average loss in bending strength varying from 5% to 18% depending on the applied process conditions, which is considerably better than the strength reductions due to other thermal treatments. Similar results were found by KORKUT [29] for mechanical properties of heat treated Uludağ fir (*Abies bornmuelleriana* Mattf.) wood and red-bud maple (*Acer troutvetteri* Madw.).

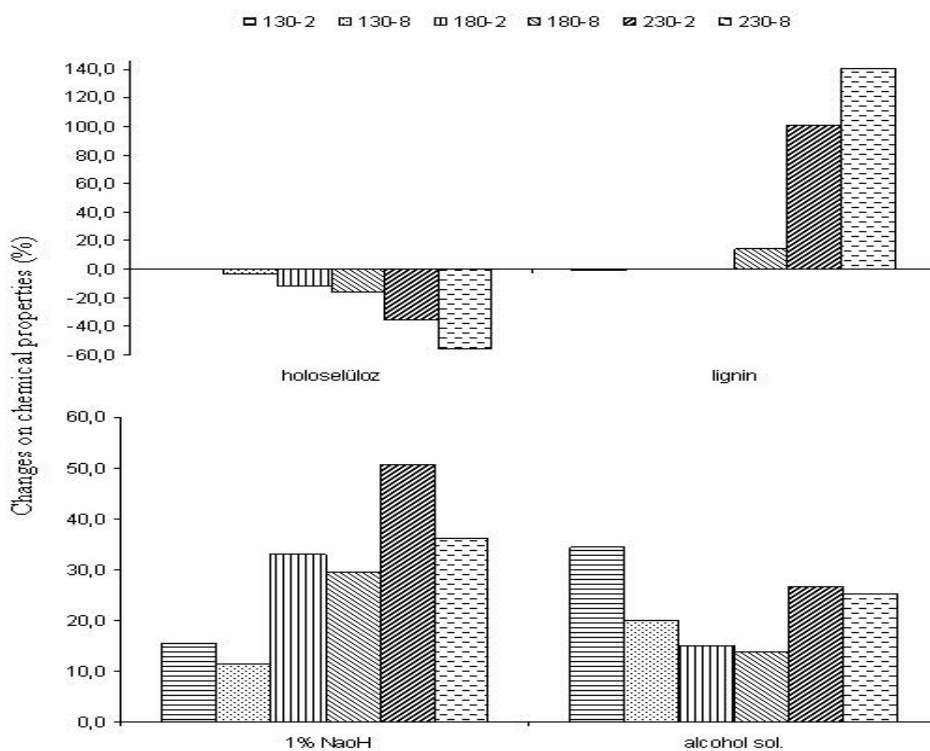


Figure 4. Changes on chemical properties (%) as a function of heat treatment and time

Changes on some chemical contents of the un-treated and heat treated chestnut wood samples were showed in Figure 4. Holocellulose content changed negatively with increasing temperature and time. Pearson's correlations results justify this statement ($P < 0.01$) (Table 1). Maximum decrease of holocellulose content was found at 230°C for 8h as 55.6% and minimum decrease was obtained at 130°C for 2h as 0.2%. Heat treatment significantly degraded the wood carbohydrate, suggesting depolymerization and alterations through the cleavage of acetic acid from the acetyl side chains (KARTAL & al. [30]).

According to Figure 4, 1% NaOH and alcohol-syclohegzane+alcohol solubilities of the chestnut wood increased with rising treatment temperature in comparison with control sample. Since increasing the thermal degradation products of carbohydrates, solubility values were increased by temperature. Table 1 shows that treatment temperature is significantly correlated with both 1% NaOH and alcohol-syclohegzane+alcohol solubilities as $r = 0.92$ and $r = 0.63$ ($P < 0.01$) respectively. It was stated by KOTILAINEN [31] that the heating of carbohydrates probably led to some extent to the formation of unsaturated nonvolatile material (i.e. UV-detectible "lignin-like material"). So, high alcohol-syclohegzane+alcohol solubility values were obtained at high temperature and times. Similar results were found by YILDIZ [32].

Lignin values increased with rising treatment temperature. According to Table 1, heat treatment time is non-significant on lignin content of wood statistically ($P < 0.05$). The highest lignin contents were obtained at 230°C (48.5% for 2h) (58% for 8h). This result confirmed most of the conclusions stated in the literature (KARTAL & al. [30], BOONSTRA & al. [33], KAMDEM & al. [34]. YILDIZ & al. [32]) stated that, some of the thermal degradation products of carbohydrates may be retained in the lignin fraction with Klason lignin analyses.

Shrinkage and swelling behavior of the heat treated wood has been affected positively with increasing of lignin ratio (Table 1). However, this improvement not only depends on the increasing lignin but also undamaged carbohydrates with crystalline structure.

FT-IR spectra were recorded for treated samples at 130, 180 and 230°C for 2 and 8h and for control sample (Figure 5). The absorbance peaks at 1430 cm^{-1} and 897 cm^{-1} were assigned to CH_2 bending mode and deformation of anomeric CH_2 respectively (KATAOKA & KONDA [35]. The bands at 1333 cm^{-1} and 1426 cm^{-1} for chestnut wood samples moved up with heat treatment according to control (Figure 4). Peak shoulder at 1507 cm^{-1} (C=C stretching aromatic ring for lignin) for chestnut wood samples was not changed by heat treatment, evidently. The band at 1106 cm^{-1} (asym. bridge C-O-C stretching for cellulose) did not changed in chestnut wood samples by heat treatment at 130°C and 180°C according 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 & al. [36]. Peak shoulder at 1635 cm^{-1} was 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 samples.

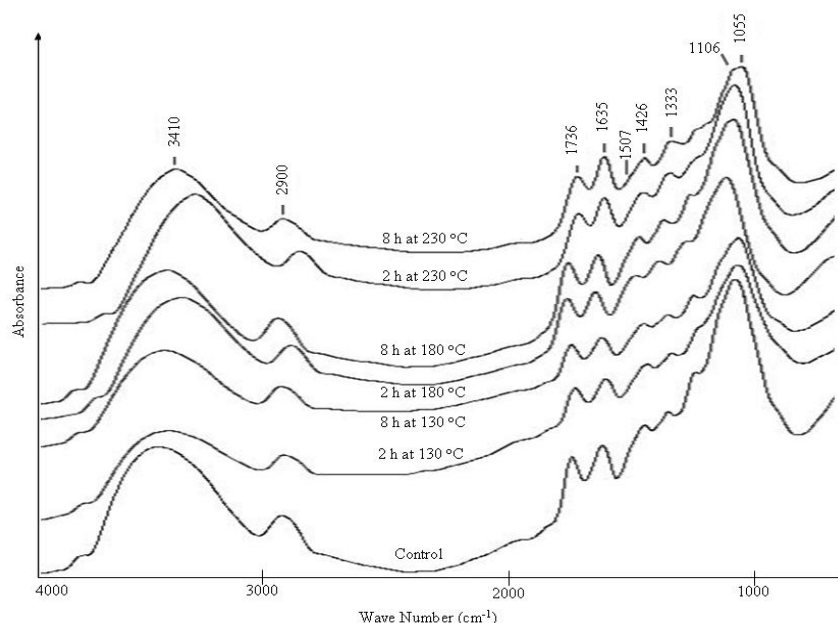


Figure 5. FT-IR spectra of un-treated and heat treated chestnut wood

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 & al. [37]).

The degree of crystallinity is one of the most important parameters for a crystalline polymer. The physical and mechanical properties of polymers are profoundly depending on the degree of crystallinity (MO & al. [38]). FT-IR spectroscopy method was used for determination of crystallinity index (Table 2).

Table 2. The crystallinity indexes of heat treated chestnut wood

	Control	130 °C		180 °C		230 °C	
		2 h	8 h	2 h	8 h	2 h	8 h
A1427/A898	0,95	0,94	0,98	1,00	0,96	1,03	1,03
A1371/A2900	1,48	1,67	2,50	1,43	1,92	1,35	1,35
A1371/A670	0,96	0,96	0,93	1,01	1,00	1,06	1,08
A1371/A690	0,98	0,98	0,98	1,05	1,04	1,09	1,10

The ratios of the absorbency at A_{1430}/A_{897} and A_{1371}/A_{2900} have been used to measure the relative cellulose crystallinity HASSAN & al. [39]. 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 (AKERHOLM & al. [40]). 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 respectively. 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 YILDIZ [32], degraded carbohydrate compounds with amorphous characteristics can be remained in klason lignin. According to the researchers; crystalline structure of cellulose is not changed YILDIZ & GUMUSKAYA [41] 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 obtained results for physical and mechanical properties; air-dry density, oven-dry density, shrinkage (tangential, radial and volumetric), swelling (tangential, radial and volumetric), FSP, MMC and EMC decreased with increasing heat temperature and time. Similarly, compression strength parallel to grain, bending strength and MOE decreased. But, hardness of wood decreased for 180°C for 8h and 230°C for both 2h and 8h due to further structural degradation. However, while decreasing holocellulose ratio of the heat treated wood, lignin content, 1% NaOH and alcohol solubility values were increased. Cellulose crystallinity of the samples was not changed significantly.

There are multiple correlations between density, dimensional stability, strength properties and chemical properties. The decreases in the strength properties can be explained by the rate of thermal degradation and loses of substance after heat treatment.

The smallest effect of the heat treatment was determined at 130°C for 2h. Treatment temperature is highly correlated with all physical, mechanical and chemical properties of chestnut wood.

In this case it can be said that temperature has greater influence on strength properties than time. For heat treatment process, 130°C for 2h should be applied in place where mechanical properties are important. However, 230°C for 2h should be used in place where preferred physical properties.

As a result, heat treated woods can be utilized using proper heat treatment time and temperature without any loses in strength values and chemical characteristics in areas where stability are important. Also, heat treatment can be considered as an environmentally friendly technique because no chemicals are involved during the process.

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