

Effect of heat treatment on the dimensional stability of ash (*Fraxinus angustifolia* Vahl.) wood

Dişbudak (*Fraxinus angustifolia* Vahl.) odununun boyutsal stabilizasyonu üzerine ısı işlemin etkisi

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ABSTRACT

In this study, the effect of heat treatment on dimensional stability of ash (*Fraxinus angustifolia* Vahl.) wood, which naturally grows in Turkey, was investigated. Ash wood samples were subjected to the heat treatment process at different temperatures (120°C, 160°C, 190°C, and 210°C) and for different time periods (3, 6 and 9 h) in water vapor atmosphere. Water absorption, water repellent effectiveness, anti-swell effectiveness (ASE, TS 4084) and Attenuated Total Reflection - Fourier Transform Infrared Spektrofotometre (ATR-FTIR) were performed on the test samples. Generally, although water absorption decreased with an increase in treatment temperature and time period, the mean of water repellent effectiveness and ASE increased. It was determined that decreases in the absorbance value of the peak belong to the -OH stretching vibration depended on heat treatment temperature increases. In particular, ASE increased to >40% with a rapid increase after 3 hours application of heat treatment at 190°C. The highest dimensional stability (58.4%) of ash wood was detected in the highest heat treatment temperature and time period (210°C and 9 h, respectively). Therefore, it was shown that the value and product quality of solid products produced from ash wood with heat treatment can be improved without the use of toxic chemicals.

Keywords: Ash wood, dimensional stability, ATR-FTIR, water absorption, water repellent effectiveness

ÖZ

Bu çalışmada, Türkiye’de doğal olarak yetişen dişbudak (*Fraxinus angustifolia* Vahl.) odununun boyutsal stabilizasyonu üzerine ısı işlemin etkisi araştırılmıştır. Dişbudak örneklerine su buharı ortamında, farklı sıcaklık (120, 160, 190 ve 210°C) ve sürelerde (3, 6 ve 9 saat) ısı işlem uygulanmıştır. Deney örnekleri üzerinde, su alma oranı (SAO), su itici etkinlik (SİE), genişlemeyi önleyici etkinlik (GET) ve ATR-FTIR analizleri gerçekleştirilmiştir. Genel olarak ısı işlem sıcaklık ve süresinin artışı ile SAO’larında azalmalar meydana gelir iken, SİE ve GET değerlerinde artışlar tespit edilmiştir. Isıl işlem sıcaklığının artmasına bağlı olarak, -OH gerilme titreşimine ait piklerin absorbans değerlerinde azalmalar tespit edilmiştir. Özellikle 190°C’de 3 saatlik uygulamada GET değerleri hızlı bir artış ile %40’ın üzerine çıkmıştır. Dişbudak odununun en yüksek boyutsal stabilite değerleri (%58,4), en yüksek ısı işlem sıcaklık ve süresinde (210°C ve 9 saat) elde edilmiştir. Sonuç olarak, ısı işlem uygulaması ile dişbudaktan üretilen masif odun ürünlerinin kalite ve değerlerinin zehirleyici kimyasallar kullanılmadan artırılabilceği görülmüştür.

Anahtar Kelimeler: Dişbudak odunu (*Fraxinus angustifolia* Vahl.), boyutsal stabilizasyon, ATR-FTIR, su alma oranı, su itici etkinlik

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INTRODUCTION

Heat treatment involves holding the wood between 100 and 250°C, either in a standard atmosphere, nitrogen (N), molecular nitrogen (N₂) or any other inert gas atmosphere for a period of time. Heat treatment processes have a significant place in the wood industry and many countries develop these procedures by utilizing different production parameters and methods (Vernois, 2000; Jamsa & Viitaniemi, 2001; Shi et al., 2007; Boonstra, 2008; Esteves & Pereira, 2009).

Heat-treated wood does not exhibit toxicity nor does it produce harmful effects on the environment. Therefore, heat treatment processes are considered as alternative protection methods as opposed to chemical impregnation and other modification methods (Yıldız et al., 2006). Wood modification techniques are designed to improve more than one property with a single treatment. The aim of thermal modification is to reduce the moisture exchange, i.e., to enhance the dimensional stability of wood and increase its resistance against wood decaying organisms (Poncsak et al., 2006; Kocaefe et al., 2008; Rowell et al., 2009; Sinković et al., 2011; Yalcin & Şahin, 2015). Moreover, heat treatment causes the equilibrium moisture content in wood to decrease, while increasing the depth of color and thermal insulation. On the other hand, wood becomes more brittle as a result of heat treatment. After the process, depending on the degree of heat treatment, the bending and tensile strengths of wood decrease (Kamdem et al., 2002; Hakkou et al., 2005; Repellin & Guyonnet, 2005; Şahin, 2014).

Heat treatment alters the chemical properties of wood as well. The deterioration starts with the hemicellulose (Yıldız, 1999). As the hygroscopic (water absorbing) property of hemicellulose is great, the reduction in the amount of hemicellulose content of heat-treated wood increases its dimensional stability (Tuong and Li, 2010; Şahin, 2014). In addition, the lignin softens and changes in the cellulose hydroxyl groups (-OH groups) take place with heat treatment (Bekhta and Niemz, 2003). As a result, in contrast to conventionally dried wood, the water absorption capacity of wood treated at high temperatures is reduced (Kocaefe et al., 2007). In studies carried out with *Pinus pinaster* and acacia wood, it was reported that heat treatment applied at temperatures above 200°C reduced hygroscopic behavior and swelling, while it increased dimensional stability (Tuong and Li, 2010; Surini et al., 2012). A similar result was found with *Eucalyptus camaldulensis* wood by Unsal et al. (2003). Heat treatment applied at different temperatures (120, 150 and 180°C) and duration (2, 6 and 10 h) caused significant decreases in the tangential and radial swelling percentages of the eucalyptus wood. The greatest changes were observed with the highest heat treatment (10 h at 180°C). In another study in which the effect of heat treatment on physical properties of Turkish hazel (*Corylus colurna*) was analyzed, it was found that, depending on temperature increase and duration, there was a statistically significant decrease in the percentage of tangential, radial and longitudinal swelling in the experimental samples compared to control samples (Korkut et al., 2008).

In a study in which heat treatment was applied to hornbeam wood and Uludağ fir wood between 170 and 210°C for 4 to 12 h, all the heat-treated samples revealed lower percentages of water absorption (WA) and swelling when compared to control samples. The WA and anti-swelling efficiency (ASE) values of Uludağ fir wood were found to be higher than those of hornbeam wood. At the end of the application at 210°C for 12 h, it was observed that the WA decrease in the Uludağ fir wood was 44.55%, while the WA decrease in the hornbeam wood was 42.51%. The differences in the percent of swelling were said to be similar to those of the WA values (Aydemir et al., 2011).

Worldwide, the largest forests of narrow-leaved ash (NLA, *Fraxinus angustifolia* Vahl.), one of the important ash subspecies, are located in Turkey. After populus and alder, this is one of the native species which grows fastest (Çiçek & Yılmaz, 2002). In planted stands of NLA in Turkey, the mean annual volume increment (MAI) can reach up to 23 m³/ha; additionally, the current annual volume increment (CAI) can reach up to 33 m³/ha for trees aged 15 to 20 years (Kapucu et al., 1999). NLA wood is classified as having moderate density. Moreover, in terms of tangential and radial shrinkage percentages, it is classified in the surplus trees group (As et al., 2016). Fast-growing tree species exhibit extensive juvenile wood growth with thin heartwood. As a result of rapid growth, the wood displays large annual rings and low density. This situation reduces the dimensional stability of the wood as well as its durability against biological hazards (Li, 2002). The ash trees used in this study are included in the slightly durable group, according to the natural durability classification of wood, with the natural durability persisting less than five years (Findlay, 1985).

This study examined the effect of heat treatment applied at different temperatures (120, 160, 190 and 210°C) and times (3, 6, 9 h) on the WA and water repellent effectiveness (WRE) values and the dimensional stability of ash wood grown in natural forests. Thus, the aim was to gather data on the effect of heat treatment on the working characteristics of natural ash wood and to enhance the low-dimensional stability of the ash wood.

MATERIALS AND METHODS

Materials

In the study, raw wood samples were obtained from floodplain forestlands belonging to Süleymaniye sub-district Directorate of Forestry and Hendek Directorate of Forestry. In accordance with the study, a stand including the same properties of habitat was specified. The research area is located on a plain bottomland where the Dinsiz and Mudurnu Streams flow and join with the Sakarya River. With the increasing precipitation in the fall, the forest first becomes a swamp. Later, the level of water reaches up to 1 or 1.5 m in the period between January and May (especially in spring when the snow melts) and the forest can be reached via a boat. The vegetation period ranges between 230 and 240 days from April through November. During the vegetation period, the monthly temperature is >10°C and the monthly precipitation is 56 mm. This region lies in the climate transition zone between the Marmara marine climate and the Black Sea climate.

Selection of Sample Trees

This study used NLA wood from the Adapazarı Province, where this species is most extensively found. Considering habitat properties such as direction, bend, diameter, height and sequence, four sample trees with similar mean diameter at breast height (DBH) and having no cracks or abnormal canopy development were cut down and removed from the area. The general properties of the sample trees were determined according to TS 4176 (1984) principles (Table 1).

Table 1. General properties of the ash (*Fraxinus angustifolia* Vahl.) trees

Tree No	Age (years)	Diameter (cm)	Height (m)	Nodes (number)	Branchless Stem (m)
1	40	33	28	15	17
2	32	28	23	9	10
3	38	31	25	9	11
4	32	27	24	10	12

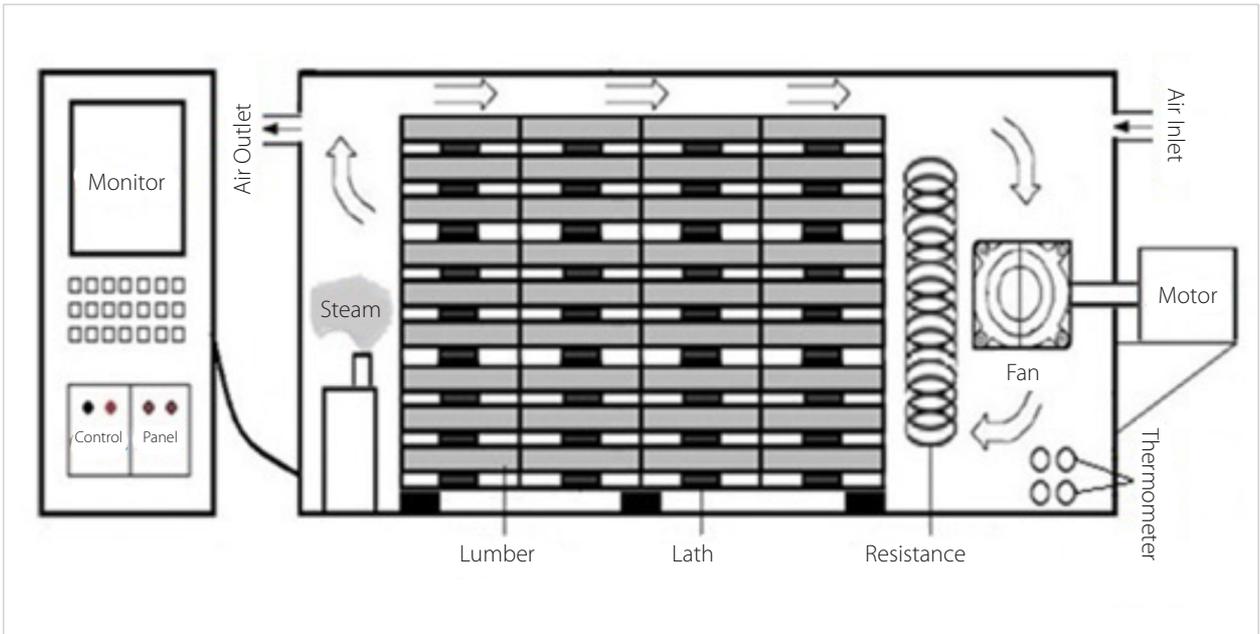


Figure 1. A schematic view of heat treatment kiln (Şahin 2014)

Preparing Test Samples

For the test samples used in the study, suitable healthy sections between 2 and 4 m above the ground root were utilized. The felled trees were cut into logs 1 to 1.5 m in length and stacked in the Adapazarı-Hendek Forest Sub-District Directorate lumber yard. Later, the logs were stripped of bark and 6-cm-wide boards were sawn, with the heartwood positioned in the center. The sawing procedure was carried out in accordance with the TS 2470 (1976) principles. The boards were tallied again and stacked in wood dunnage where they were stored in a sheltered area for air seasoning. To minimize the differences in the samples caused by the wood structure, the experimental and control samples were prepared having the same consecutive annual rings in the longitudinal axis of the boards. Experiment numbers for each trial were written on the experimental samples with an acetate pen.

Methods

Heat Treatment

The experimental samples used in the study were prepared from flawless specimens in standard specified dimensions of 30 × 30 × 15 mm (T × R × L). Before heat treatment, the samples were kept in a climatic chamber at 20±2°C and 65±5% relative

humidity for 4 weeks to ensure a moisture balance of 11-14%. The samples were subjected to heat treatment in a total of 12 variations including four different temperatures and three different time durations. The heat treatment procedure was carried out in a heat treating furnace with the capacity of 1 m³, ±1°C equipped with a computerized precise temperature control system (Figure 1). The heat treatment parameters could be controlled automatically or manually on the control panel of the heat treating furnace.

The samples were heat-treated in a water vapor atmosphere at temperatures of 120, 160, 190 and 210°C over a period of 3, 6 and 9 h. During heat treatment, both the samples and air temperature were measured. In the first phase, the temperature of the furnace was raised to about 100°C. In the procedure known as high-temperature drying, vapor is released into the furnace to avoid surface checking or internal splitting caused by the shock of drying. At the same time, the wood material inside the furnace is prevented from burning. During this phase, the moisture of the wood was lowered to nearly zero. When the internal temperature was equivalent to the ambient temperature, the heat of the furnace was cautiously increased to the target heat treatment temperatures. The procedure continued at these temperatures throughout the specified time periods.

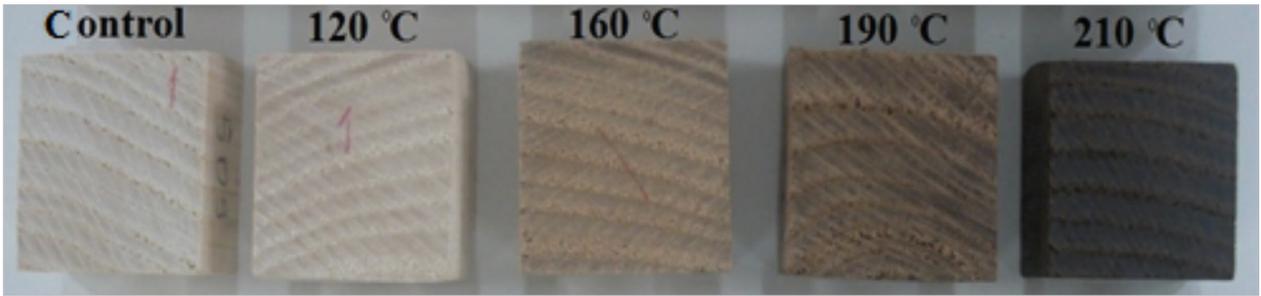


Figure 2. The test and control samples used in WA experiment (Şahin 2014)

The last phase was the cooling and conditioning. During this phase, the aim was to prevent the wood, which had reached a high temperature in the furnace, from cracking from the effects of the colder outside atmosphere. The internal temperature was lowered to 80 or 90°C with the help of a water-spray system and the samples were conditioned under the control of the water vapor atmosphere. At the end of this stage, the moisture content of the wood ranged between 4% and 7%, depending on the temperature of the heat treatment. The test samples remained inside the furnace for an average of 27 h in all combinations applied (including the heating, pre-dry and conditioning procedures), excluding the heat treatment period (Şahin, 2014). After heat treatment, the test samples were placed in the climatic chamber again in order to reach equilibrium moisture content (Figure 2). The experiments carried out on the heat-treated samples and the sample numbers used for each experiment were determined according to TSI CEN/TS 15679 (2011) standards.

Water Absorption and Water Repellent Effectiveness

The experimental samples used to identify WA and WRE values were prepared in dimensions of 30 × 30 × 15 mm (Figure 2). In the experiments, 20 test samples were used separately for control or experimental samples.

The heat-treated experimental samples and the control samples having the same annual rings as the experimental samples were dried to constant weight at 103±2°C and their dimensions and dry condition weights were established as 0.01 mm and 0.01 g, respectively. The control and experimental samples were then soaked in water at 20±1°C. At the end of 2, 4, 8, 24, 48 h and 1 and 2 week periods, the amount of water absorbed by the control and test samples was measured. For this procedure, at the end of each soaking period, the samples were removed from the water and wiped with a napkin. They were weighed on a scale and the amount of water absorbed (A_{bs}) was recorded. Using the dry weight at the beginning, the WA value (%) of each experimental sample (P_{a0}) or control sample (A_0) was calculated separately for each period, according to the formula below (Yıldız, 2002).

$$WA = \frac{A_{bs} - P_{a0}(\text{or } A_0)}{P_{a0}(\text{or } A_0)} \times 100$$

The WRE value (%), defined as the decline that occurs in the WA of heat-treated wood samples compared to untreated wood, was calculated separately for each period and for each experimental and control sample according to the formula below.

$$WRE = \frac{(WA_c - WA_t)}{WA_c} \times 100$$

In the equation,

WRE: Water repellent effectiveness,

WA_c : WA (%) of the control sample at the end of a specified period,

WA_t : WA (%) of the experimental sample at the end of a specified period.

Swelling and Dimensional Stability

The samples used in the WA and WRE tests were utilized in the tests for the amount of swelling and dimensional stabilization. In the relevant tests, the dimensions of the test and control samples in the tangential direction under dry conditions were determined by calipers with 0.01 mm sensitivity. The samples were soaked in water until their dimensions remained constant and then were measured again from the same points. After the measurements were made by following the general principles in TS 4084 (1983), the percentage of tangential swelling (α_{tg}) was calculated according to the formula below.

$$\alpha_{tg} = \frac{(T_2 - T_1)}{T_1} \times 100$$

In the equation;

T1: The dimension of samples in the tangential direction under dry conditions (mm),

T2: The dimension of samples in the tangential direction after soaking in water for 2 weeks (mm).

The ASE values (%), which describe the decrease in the amount of swelling of the test samples compared to the control samples and accordingly, the dimensional stabilization gained through heat treatment, were calculated using the formula below (Yıldız, 2002).

$$ASE = \frac{(c\alpha_{tg} - t\alpha_{tg})}{c\alpha_{tg}} \times 100$$

Table 2. Mean WA of heat treated ash wood test and control samples at different temperatures and times (%)

	Temperature (°C)	Time (h)		Immersion Time				p		
		2 h	4 h	8 h	24 h	48 h	72 h		1 wk	2 wk
120	K	21.19 ^{bc*}	27.25 ^d	33.91 ^f	48.41 ^g	58.49 ^h	64.44 ^j	72.49 ^m	78.99 ^o	0.000
		(2.35)**	(2.63)	(2.73)	(4.22)	(4.87)	(4.9)	(4.95)	(4.79)	
	3	19.09 ^a	24.97 ^c	31.21 ^e	48.20 ^g	61.27 ⁱ	65.50 ^{jk}	70.69 ^l	76.60 ⁿ	
		(0.19)	(0.3)	(0.89)	(2.39)	(2.12)	(3.63)	(2.81)	(2.45)	
6	18.65 ^a	24.26 ^c	31.48 ^e	47.11 ^g	60.97 ⁱ	66.30 ^k	72.11 ^{lm}	78.34 ^{no}		
	(0.48)	(0.48)	(1.29)	(2.17)	(1.95)	(2.29)	(2.24)	(1.49)		
9	18.28 ^a	24.25 ^c	30.78 ^e	46.96 ^g	59.98 ^{hi}	64.93 ^k	70.64 ^l	77.20 ⁿ		
	(0.51)	(0.34)	(1.09)	(2.68)	(2.7)	(2.51)	(3.2)	(1.31)		
160	K	21.19 ^c	27.25 ^f	33.91 ⁱ	48.41 ^l	58.49 ^o	64.4 ^{pr}	72.49 ^u	78.99 ^y	0.000
		(2.35)	(2.63)	(2.73)	(4.22)	(4.87)	(4.9)	(4.95)	(4.79)	
	3	19.33 ^b	25.10 ^e	30.01 ^{gh}	41.79 ⁱ	50.73 ^m	57.69 ^o	67.06 ^s	73.63 ^u	
		(0.43)	(0.78)	(0.84)	(1.00)	(1.21)	(1.73)	(1.84)	(1.86)	
6	17.8 ^{ab}	23.6 ^{de}	30.94 ^h	46.50 ^k	58.34 ^o	63.40 ^p	69.92 ^t	77.16 ^v		
	(0.71)	(0.63)	(1.07)	(2.78)	(2.6)	(2.73)	(3.15)	(3.94)		
9	17.00 ^a	22.43 ^{cd}	28.6 ^g	42.11 ^j	52.72 ⁿ	58.42 ^o	65.97 ^{rs}	73.48 ^u		
	(0.9)	(1.02)	(1.37)	(1.35)	(1.8)	(3.14)	(2.35)	(3.05)		
190	K	21.2 ^{bc}	27.25 ^e	33.91 ^g	48.41 ^j	58.49 ^o	64.44 ^p	72.49 ^s	78.99 ^u	0.000
		(2.35)	(2.63)	(2.73)	(4.22)	(4.87)	(4.9)	(4.95)	(4.79)	
	3	16.33 ^a	22.58 ^c	29.31 ^f	42.33 ⁱ	51.72 ^l	57.32 ^{no}	66.57 ^r	75.18 ^s	
		(0.67)	(1.03)	(1.41)	(1.51)	(1.87)	(2.03)	(2.16)	(2.74)	
6	16.51 ^a	20.36 ^b	27.33 ^e	41.21 ⁱ	50.23 ^{kl}	55.94 ^{mn}	65.62 ^{pr}	74.69 ^t		
	(0.8)	(0.95)	(0.67)	(2.17)	(2.06)	(2.13)	(2.45)	(3.49)		
9	16.04 ^a	20.25 ^b	24.56 ^d	39.54 ^h	48.77 ^k	54.49 ^m	64.52 ^p	73.52 st		
	(0.66)	(1.16)	(0.88)	(2.04)	(2.81)	(2.84)	(2.98)	(1.53)		
210	K	21.19 ^c	27.25 ^f	33.91 ^g	48.41 ^l	58.49 ^o	64.4 ^{pr}	72.49 ^s	78.99 ^u	0.000
		(2.35)	(2.63)	(2.73)	(4.22)	(4.87)	(4.9)	(4.95)	(4.79)	
	3	16.35 ^a	20.37 ^{bc}	25.28 ^e	39.80 ⁱ	49.41 ^l	54.94 ^m	63.28 ^p	71.47 ^s	
		(0.84)	(0.88)	(0.77)	(0.95)	(1.15)	(1.6)	(1.45)	(2.41)	
6	15.73 ^a	19.54 ^b	23.46 ^d	38.17 ⁱ	49.36 ^l	56.47 ⁿ	65.78 ^r	74.35 ^t		
	(0.79)	(0.93)	(0.97)	(1.15)	(1.51)	(1.4)	(2.2)	(2.1)		
9	14.99 ^a	19.01 ^b	22.89 ^d	35.91 ^h	46.57 ^k	54.67 ^m	63.24 ^p	71.71 ^s		
	(0.93)	(1.15)	(1.3)	(1.29)	(1.69)	(1.17)	(1.43)	(1.97)		

*Same letters appearing in a column indicate no statistical differences between the means (p<0.05), ** Standard deviation values are given in parentheses, C: Control sample, wk: week, h: hour, p: Significance level

In the equation;

ASE: Anti-swell effectiveness,

catg: The percentage (%) of swelling of control samples in the tangential direction,

tatg: The percentage (%) of swelling of experimental samples in the tangential direction.

ATR-FTIR Analysis

Ash wood powder obtained from three stands and passed through an 80-mesh sieve was used for the Fourier transform infrared spectroscopy (FTIR) analysis. According to the attenuated total reflection (ATR) method, absorption peaks were directly obtained without applying any pre-treatment. The FTIR analyses

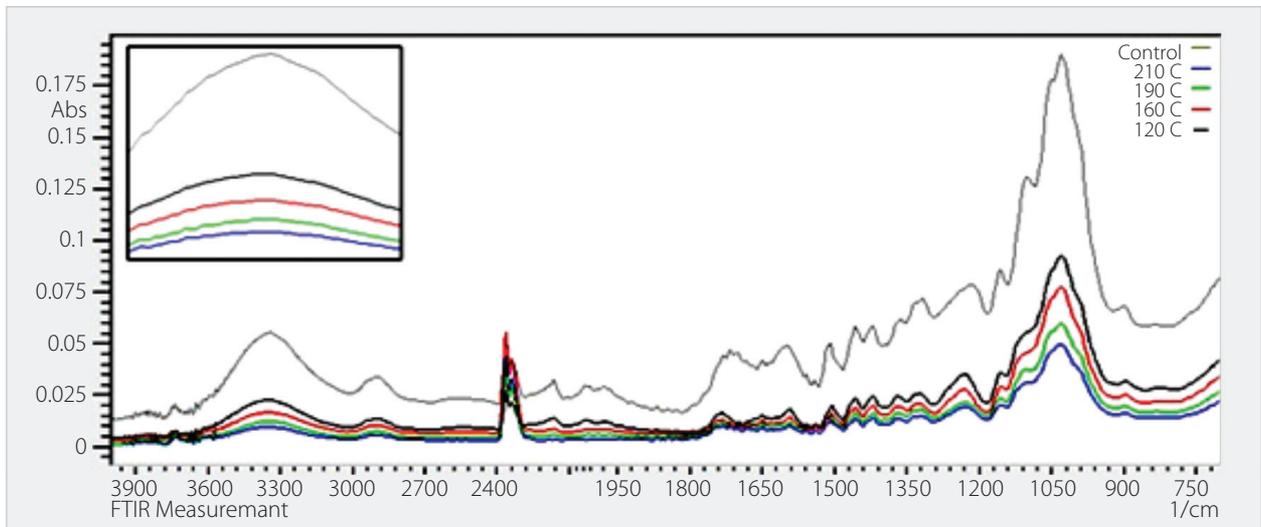


Figure 3. ATR-FTIR spectra of heat-treated ash wood for 3 h at different temperatures and control samples

were conducted via 4000 and 700 cm^{-1} wavelengths and 4 cm^{-1} solubility using the Shimadzu IR Prestige-21 spectrophotometer. The analyses were carried out by completing 20 scans for each sample.

Statistical Analysis

The data were analyzed using the statistical package program SPSS 19 software. In all comparisons, the level of significance was taken as 0.05 (95%). Simple analysis of variance (ANOVA) was used to determine any statistically significant differences between the experimental groups, with four different temperatures and three different times applied during heat treatment, and the control groups. The means obtained via the ANOVA were compared using Duncan's new multiple range test separately for each variation.

RESULTS AND DISCUSSION

Effect of Heat Treatment on Water Absorption (WA)

The means of the WA and statistical results for the different water immersion times of the NLA test samples subjected to heat treatment at different temperatures (120, 160, 190 and 210°C) for different times (3, 6 and 9h) and of the control samples are presented in Table 2. According to the results obtained, in each variation applied, the increases in WA values of the test and control samples were dependent on the increase of the immersion time. The results of the ANOVA indicated that the differences between the mean WA values of each variation depending on the immersion time were statistically significant ($p < 0.05$, Table 2).

In the test and control samples of NLA, the mean values of WA increased depending on the immersion time. At the end of the 2, 4 and 8h immersion times, the WA values of all test samples to which heat treatment was applied were found to be different from the control samples ($p < 0.05$). Depending on the increase of heat treatment temperature and time period, the WA values

decreased. For example, the WA value of the control sample was 21.19% at the 2h immersion time while the WA value was decreased to 14.9% for test samples (210°C and 9h) at the same immersion time. The WA values of the test and control samples especially regarding the heat treatment at 190 and 210°C were found to be statistically different from one another for each immersion time ($p < 0.05$).

In some studies in the literature, it has been reported that, depending on commercial heat treatment, the WA values of eucalyptus (Cademartori, 2014), acacia (Van Chu, 2013), juniper (Kasemsiri, 2012), black pine (Dündar et al., 2012) and fraxinus ash (Korkut et al., 2012) woods decreased. In another research, at the end of thermal treatment applied at 180°C over 2, 4, 6, 8, and 12h, the water adsorption of *Castanea sativa* wood samples decreased with the rise in the duration of heat treatment (Gündüz & Aydemir, 2008).

In their research, Kocaefe et al. (2008) determined the physical, mechanical and fungal properties of heat-treated pine and populus wood. They stated that an extraordinary decrease occurred in the WA values of wood samples soaked in water for 24 h. The decrease in the pine WA was 38%, while this value in the populus wood was 14%.

In a study which examined the effect of heat treatment at different temperatures on the WA properties of heartwood and sapwood of pine and spruce, it was reported that the heartwood of both wood types absorbed less water than the sapwood. The heat treatment visibly decreased the WA values of pine and spruce heartwood. The WA values of the pine sapwood decreased at 230°C (Metsä-Kortelainen et al., 2006).

In general, for all immersion times, the WA values of the test samples treated at different temperatures decreased to some extent when compared to the WA values of the control samples (Table 2). The modification in the chemical structure of

Table 3. Mean WA of heat treated ash wood test and control samples at different temperatures and times (%)

	Temperature (°C)	Time (h)			Immersion Time				p	
		2 h	4 h	8 h	24 h	48 h	72 h	1 wk		2 wk
120	3	9.94 ^{bcd*}	8.36 ^{de}	7.96 ^{de}	0.43 ^{ghi}	-4.75 ^k	-1.65 ^{ij}	2.48 ^{fg}	3.02 ^f	0.000
		(0.89) ^{**}	(1.11)	(2.62)	(4.93)	(3.63)	(5.64)	(3.87)	(3.1)	
		6	11.99 ^{ab}	10.97 ^{bc}	7.17 ^e	2.67 ^{fg}	-4.24 ^k	-2.89 ^k	0.52 ^{ghi}	
(2.27)	(1.78)		(3.81)	(4.48)	(3.34)	(3.56)	(3.09)	(1.89)		
9	13.72 ^a		11.01 ^{bc}	9.24 ^{cde}	2.99 ^f	-2.55 ^k	-0.77 ^{hij}	2.55 ^{fg}	2.27 ^{fg}	
	(2.39)	(1.23)	(3.22)	(5.53)	(4.61)	(3.9)	(4.41)	(1.66)		
	160	3	8.78 ^{gh}	7.88 ^{gh}	11.52 ^{de}	13.67 ^{cd}	13.26 ^d	10.47 ^{ef}	7.48 ^{gh}	6.78 ^h
(2.01)			(2.85)	(2.48)	(2.06)	(2.07)	(2.68)	(2.54)	(2.36)	
6			15.84 ^{bc}	13.50 ^{cd}	8.76 ^{gh}	3.94 ⁱ	0.26 ^j	1.62 ^{ij}	3.54 ⁱ	2.31 ^{ij}
	(3.36)	(2.31)	(3.14)	(5.74)	(4.45)	(4.24)	(4.35)	(4.99)		
	9	19.78 ^a	17.70 ^{ab}	15.72 ^{bc}	13.01 ^d	9.86 ^{efg}	9.35 ^{efgh}	9.00 ^{fgh}	6.97 ^h	
(4.26)		(3.75)	(4.03)	(2.78)	(3.08)	(4.87)	(3.24)	(3.87)		
190		3	22.95 ^{cd}	17.13 ^{efg}	13.57 ^{hij}	12.56 ^{ijk}	11.58 ^{kl}	11.06 ^{kl}	8.17 ^{mn}	4.82 ^o
	(3.18)		(3.77)	(4.16)	(3.12)	(3.19)	(3.16)	(2.98)	(3.47)	
	6		22.09 ^d	25.26 ^{abc}	19.41 ^e	14.88 ^{ghi}	14.13 ^{hi}	13.19 ^{hijk}	9.48 ^{lm}	5.44 ^o
(3.75)		(3.47)	(1.98)	(4.48)	(83.53)	(3.3)	(3.38)	(4.42)		
9		24.33 ^{bcd}	25.67 ^{ab}	27.57 ^a	18.33 ^{ef}	16.62 ^{fg}	15.44 ^{gh}	10.99 ^{kl}	6.92 ^{no}	
	(3.13)	(4.25)	(2.58)	(4.21)	(4.81)	(4.4)	(4.11)	(1.93)		
	210	3	22.86 ^e	25.25 ^d	25.44 ^d	17.78 ^g	15.53 ^h	14.74 ^h	12.71 ⁱ	9.52 ^j
(3.98)			(3.22)	(2.26)	(1.96)	(1.97)	(2.49)	(2)	(3.06)	
6			25.75 ^d	28.31 ^c	30.81 ^{ab}	21.15 ^{ef}	15.62 ^h	12.37 ⁱ	9.26 ^j	5.87 ^k
	(3.73)	(3.41)	(2.87)	(2.38)	(2.59)	(2.17)	(3.04)	(2.65)		
	9	29.25 ^{bc}	30.22 ^{bc}	32.50 ^a	25.82 ^d	20.38 ^f	15.17 ^h	12.75 ⁱ	9.21 ^j	
(4.37)		(4.22)	(3.84)	(2.68)	(2.9)	(1.81)	(1.97)	(2.5)		

*Same letters appearing in a column indicate no statistical differences between the means (p<0.05), ** Standard deviation values are given in parentheses, wk: week, h: hour, p: Significance level.

the wood by heat treatment applied at above 200°C even in a shielding gas environment influenced all properties of the wood. When wood absorbs moisture in the environment, water molecules try to enter through the wood polymers (hemicellulose and amorphous cellulose). The OH groups are loosened and crosslinks of wood fibers are formed as a result of heat treatment, which significantly lessens the capacity of water to enter the wood (Homan et al., 2000; Tuong and Li, 2010). This fact indicates the main reason that the test samples exhibited less WA than the control samples.

The ash wood samples heat-treated at different temperatures are characterized via ATR and FTIR spectral analysis peaks in Figure 3. The FTIR analyses were applied to all temperature variations, and the 3-hour differences for each temperature are marked in the figure below.

The ash wood samples showed similar spectra results after the heat treatment. The fact that most of the hemicelluloses were removed in the heat-treated test samples was understood from the decrease in the volume of the absorption peaks in the FTIR spectra at 1740 and 1590 cm⁻¹ emerging from the C=O and C-O groups which contain hemicellulose (xylan) (Figure 3). In addition, the fact that the free hydroxyl groups (-OH) which cause significant decreases in the WA of ash wood had actively loosened can be explained by the decrease in the absorbance values of the peaks observed at the 3350 cm⁻¹ frequency, with the OH stretching vibration depending on rising temperature. Similar results were found in research carried out by Miklečić et al. (2011).

Effect of Heat Treatment on Water Repellent Effectiveness (WRE)

The WRE values indicated the decrease in the WA of the heat-treated test samples compared to the control samples.

Table 4. Mean ASE values of heat treated ash wood at different temperatures and times (%)

Temperature (°C)	Time (hour)	ASE (%)	HG	S	Min	Max	p
120	3	3.62	a*	1.00	1.56	5.58	0.000
	6	5.28	b	0.76	3.56	6.25	
	9	7.66	c	0.85	6.31	9.27	
160	3	15.66	d	1.03	13.62	17.86	
	6	17.14	e	0.96	15.38	18.88	
	9	21.06	f	1.11	19.54	23.82	
190	3	40.17	g	1.17	37.62	42.39	
	6	46.50	h	1.51	43.20	48.52	
	9	49.44	i	2.08	46.22	54.43	
210	3	51.08	j	1.29	48.35	53.69	
	6	54.29	k	2.19	48.27	58.61	
	9	58.36	l	1.68	55.08	62.06	

*Same letters appearing in a column indicate no statistical differences between the means (p<0.05), ASE: anti-swell effectiveness, HG: Homogeneous group, S: Standard deviation, Min: Minimum value, Max: Maximum value, p: Significance level.

The arithmetic means, standard deviation, statistical homogeneity groups and statistical data related to WRE values of the test samples immersed in distilled water for various periods after heat-treatment are presented in Table 3.

In general, the highest WRE values were detected in the 9-hour heat treatment at all temperatures and periods of soaking in water procedures. According to Table 3, compared to the control samples, the WRE values in the ash wood samples heat-treated at 120°C were related to short-term (2, 4 and 8 h) immersion time increases. At the end of the 48-hour immersion time, the WRE values began to decline rapidly and decreased below those of the control samples (e.g. -2.55%). The WRE values at 48-hour and 72-hour immersion times became negative (in which case the heat-treated test samples absorbed more water than the control samples) for all heat treatment periods. After treatment via some water-repellent enabling processes, the test wood samples absorbed less water than the control samples. However, when the water immersion time was increased, the amount of water that the test wood absorbed approached that of the control samples (Yıldız, 2002).

The WRE values obtained from the heat treatment at 190°C for 3 and 6 h decreased depending on the increase in the periods of soaking in water. The WRE values increased for the 9-hour heat treatment up to the completion of the 8-hour immersion time, after which they decreased (Table 3). According to the results, the highest WRE value among heat-treatment periods was obtained at the 8-hour periods of soaking in water with the 9-hour treatment (27.6%) and this value was found to be statistically different from all other variations (p<0.05).

The effect of the heat treatment applied at various temperatures and times on the dimensional stability of acacia wood was

studied by Vah Chu (2013), who stated that improvements of 15-46% in dimensional stability and 8-18% in water-repellent effectiveness were observed at the end of heat treatment carried out in air.

For each temperature application, the highest WRE values were obtained at the lowest periods of soaking in water (2, 4 and 8 h) procedures. A decrease in WRE values was observed, especially in the period of 24 h and after, depending on the increase of the immersion time. Dündar et al. (2012) applied heat treatment at 180 and 210°C for 3 h on black pine timber which included compression and opposite wood by using the commercial Thermowood method. Consequently, while the maximum WRE values were obtained in the short-term immersion time periods for all processes, the heat-treated wood lost that positive effect with the increase of the application period. Once again, the highest WRE values were attained from the 9-hour heat treatment at all immersion time periods. In addition, when the temperature of heat treatment was increased, an overall increase in the WRE values was observed. The lowest WRE values were seen at 120°C, while the highest values were determined at 210°C with the 9-hour treatment. In one study, after an 8-hour periods of soaking in water, the WRE values of beech heat-treated at 200°C over 6 and 10 h were respectively 36% and 42.5% (Yıldız, 2002). Heat-treatment was applied on beech and spruce wood with various vegetable oils by Tomak et al. (2011) and in all applications, the WA% decreased compared to control samples, and increases in WRE values were determined.

Effect of Heat Treatment on Anti-Swelling Effectiveness (ASE)

The decrease in the swelling amount of the ash wood test samples taken from growth forests and heat-treated at various temperatures and times was compared to that of the control sam-

ples. The ASE values describing the dimensional stability gained as a result of heat treatment along with other statistical data are shown in Table 4. According to the ANOVA results, it was concluded that the difference between the mean ASE values of each variation was statistically significant ($p < 0.05$). For this reason, in order to reveal which of the variations was the source of the significant difference, Duncan's new MRT was conducted. According to the results, significant differences were found in the ASE values at all heat treatment temperatures and times (Table 4).

Increases in the ASE values were determined depending on increasing of the temperature and time period. Especially in the application at 190°C for 3 h, the ASE values rose rapidly to over 40%. The highest ASE values were obtained when the heat treatment was applied at the most severe variation. There have been a number of studies examining the fact that heat treatment increases ASE values in a directly proportional way to the increasing temperature and time (Esteves et al., 2007; Tuong and Li, 2010; Cao et al., 2012). In another study, the dimensional stability values of oriental beech wood heat-treated under atmospheric pressure between 130 and 200°C for 2, 6 and 10 h increased in direct proportion to the rise in the temperature and time; this value reached 50% at 200°C (Yıldız, 2002).

Dimensional stability values occurring with numerous heat treatments of between 100 and 240°C lasting 1 to 48 h on fir, spruce, beech, eucalyptus, oak, acacia and rubber tree (*Ficus elastic*) woods were studied. In general, it was reported that dimensional stability of between 40% and 90% was acquired with the increasing temperature and time of the heat treatments and also depending on the method (Edvardsen and Sandland, 1999; Yıldız, 2002; Srinivas, 2012; Cao et al., 2012; Van Chu, 2013; Cademartori, 2014).

While the ASE value was 49.4% at 190°C with the 9-hour application of heat treatment, this value reached 58.4% at the application of 210°C for 9 h. The lowest ASE value was achieved when the heat treatment temperature and period were at the lowest (3.62%).

Some physical and mechanical properties of hornbeam wood heat-treated at three different temperatures (130, 160 and 190°C) and times (3, 6 and 9 h) were investigated, and as a result of the heat treatment, the decrease in the ASE values were higher in the tangential direction of the wood than in the other (radial and longitudinal) directions. The highest ASE ratio (40.58%) was obtained at 190°C and a 9-hour application in the tangential direction (Ghalehno & Nazerian, 2011). In a study carried out with eucalyptus wood by Calonego et al. (2012), it was found that the thermal modification caused a decrease of 53.3% in the ratio of volumetric swelling.

Another study evaluated the effect of heat treatment on the physical, mechanical and color properties of eucalyptus wood. According to the results, there were significant decreases in the amount of tangential and radial swelling of the eucalyptus

wood depending on the increases in heat treatment temperature and period, with the highest ratio of decrease (21.5%) being observed with the most severe heat treatment (180°C for 10 h) in the tangential direction (Ünsal et al., 2003).

In the literature, several reasons are stated for the fact that dimensional stability is enhanced by heat treatment. Tjeerdsma et al. (1998) reported that the loss of methyl radicals belonging to some syringyl and guaiacyl units of lignin may be a possible cause for the enhancement of dimensional stability. In this case, there is an increase in the ratio of phenolic groups and units in free ortho positions. As a result of these chemical modifications, lignin generates some cross-links and passes more reactive positions, thus increasing dimensional stability. In addition, the elasticity of cellulose molecules is reduced by the increase in crosslinking. When water is absorbed, less swelling occurs in these molecules. Therefore, the amount of equilibrium moisture of the wood material is reduced and its dimensional stability increased.

Treatment at high temperature modifies the structure of wood and reduces the proportion of hemicellulose (Kantay et al., 1995). In addition, as the amount of hydroxyl groups decreases, the cell wall of the wood absorbs less water. For this reason, when heat treatment is applied to wood material, its dimensional stability is enhanced. Because hemicelluloses are highly hygroscopic, the decrease in the content of hemicellulose during heat treatment increases the dimensional stability of treated wood compared to control samples (Tjeerdsma et al., 1998; Yıldız et al., 2006; Tuong and Li, 2010; Priadi and Hiziroğlu, 2013; Akkılıç et al., 2014).

CONCLUSION

In this research, the effect of heat treatment in a water vapor atmosphere on the dimensional stability of ash (*Fraxinus angustifolia* Vahl.) The wood was examined and statistically significant differences were determined in the WA, WRE and ASE values between the test and control samples. The WA of the ash wood control and test samples increased depending on increasing immersion times. Generally, the WA of the test samples in different temperature groups decreased slightly for all immersion time when compared to the control samples (Table 2). However, longer immersion times reduced the effect of heat treatment on the WA. In this study, increases of WRE values in certain amounts took place in proportion to the increase in the temperature and period of heat treatment (Table 3). As a result, in order to increase the WRE values of ash wood, the temperature and time of heat treatment need to be at the highest level.

The temperature and time of the heat treatment applied affected the ASE values (dimensional stability) linearly. That is to say, as the temperature and time of the heat treatment increased, the dimensional stability also gained increases. The best results were obtained with the long heat treatment (9 h) at 210 °C (58.36%). According to these evaluations, in order to enhance the working property of ash wood, in a way similar to increasing WRE values, applying heat treatment at the highest tempera-

ture and duration will yield the best result. Therefore, heat-treated ash wood could be recommended for areas of use where dimensional stability is important (e.g., doors, windows, floors, bathrooms, saunas, decks and siding).

Like ash wood, other rapidly-growing tree species, especially those with low dimensional stability, could also be given added value via heat treatment applications and as a result could become competitive with the tropical tree species commonly used for many purposes nowadays.

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