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## The Effect of Industrial Heat Treatment on The Wettability and Dimensional Stability of Ash (*Fraxinus excelsior*) Wood

### Şemsettin KILINÇARSLAN<sup>\*1</sup>, Yasemin ŞİMŞEK TÜRKER<sup>1</sup>

<sup>1</sup>Suleyman Demirel Univesity, Faculty of Engineering, Department of Civil Engineering, 32600, Isparta, Turkey

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<b>Keywords</b> Heat treatment, Ash, Wettability, Dimensional stability	<b>Abstract:</b> In this study is to examine the effect of industrial heat treatment application on the dimensional stability and dynamic wettability properties of ash ( <i>Fraxinus excelsior</i> ) wood. Ash samples that were heat treated for 120 minutes at 210 degrees according to the ThermoWood process were used. In this study, a total of 30 samples, including 15 heat treated samples with dimensions of 20x20x20, mm, and 15, wheat treated complex were subtract the dynamic							
	wettability and dimensional stability properties were examined.							
	The results obtained in the study showed that the wettability property of the wood material decreased with the application of heat treatment. It has been determined							
	that the dimensional stability is higher especially in tangential section compared to							
	dynamic wettability property and dimensional stability.							

# Endüstriyel Isıl İşlemin Dişbudak (*Fraxinus excelsior*) Odununun Boyutsal Stabilite ve Islanabilirliği Üzerine Etkisi

#### Anahtar Kelimeler Isıl işlem, Dişbudak, Islanabilirlik, Boyutsal kararlılık

**Özet:** Çalışmada, endüstriyel ısıl işlem uygulamasının dişbudak (*Fraxinus excelsior*) odununun boyutsal stabilite ve dinamik ıslatma özelliği üzerine etkisini incelemektir. ThermoWood prosesine göre 210 derece 120 dakika boyunca ısıl işlem görmüş Dişbudak numuneleri kullanılmıştır. Çalışmada 20x20x30 mm boyutlarına sahip 15 adet ısıl işlem görmüş numune 15 adet ısıl işlem görmemiş numune olmak üzere toplam 30 numune biçilmiş ve dinamik ıslatma özelliği ile boyutsal stabilite özellikleri incelenmiştir.

Çalışmada elde edilen bulgular, ısıl işlem uygulaması ile birlikte ağaç malzemenin ıslanabilme özelliğinin azaldığını göstermiştir. Özellikle radyal kesite nazaran teğet kesitte boyutsal kararlılığın daha yüksek olduğu tespit edilmiştir. Ayrıca dinamik ıslatma özelliği ile boyutsal kararlılık arasında önemli bir korelasyon olduğu belirlenmiştir.

### 1. Introduction

Demand for sustainable construction materials is increasing in order to reduce energy consumption and CO<sub>2</sub> emissions throughout the World [1-4]. Wood is one of the oldest building materials. Among the materials used in construction, only the source of the wood material can be renewed [5]. Wood is a material that is frequently used in building industry and it can be easily found [2, 6]. For many years wood materials have been used in building sector but reinforced concrete structures have gained importance in time and the application areas of wood material has decreased. In recent years, the use of wood and wood composites has become popular

again with the importance of cost [7-9]. There is a wide range of applications such as furniture, building, road works and water supply works [2]. Due to the easy processing and shaping of wood materials, it has expanded its usage area continuously. With the opportunities provided by today's technology, the usage area has diversified in time [10]. However, as it is an anisotropic material, it has some negative results due to its limited resistance and water supply. Due to the changes in the size of the water contact or in areas where high humidity is used for a long time is possible after some operations [11]. Recently, the most common method is to impregnate toxic chemicals on wood. However, the treatment of wood with toxic chemicals has a negative effect on both human health and the environment. Therefore, various heat treatment methods have been developed in order to process wood material without treatment with chemicals [12, 13].

Heat treatment methods applied for experimental and commercial purposes; Fin (ThermoWood) method, Plato method, Rectification method, Oil heat treatment methods [14]. The elasticity modulus, bending strength and compressive strength are important for the load-bearing structural elements. Dimensional stabilization is an important factor for wood materials used in damp environments such as sauna, bathroom and garden furniture [15]. Knowing the dimensional stability property of wood material is important in determining the place of use of this material.

Since hemicelluloses have the lowest molecular weight, they begin to degrade at higher temperatures in heat treatment. The degradation of hemicellulose leads to a decrease in the assembly of the OH bond and the O-acetyl group [16, 17]. Pavlo and Niemz (2003)[18] found that heat treatment applied at high temperatures to the material obtained from spruce wood species caused darkening of color, increased dimensional stability but impaired mechanical properties [19]. Kocaefe et al. (2008) [16] examined the effect of heat treatment on the wettability of ash and maple samples. In their study, they determined the axial contact angles of maple and ash samples subjected to heat treatment at different temperatures. They found that the contact angle values were higher in the heat treated samples.

Petrissans et al. (2003) [20] treated with chemical and heat treatment spruce poplar, beech and pine wood in their study on the wettability of the material, heat treated materials have determined that a higher rate of hydrophobicity. Hakkou et al. (2005) [21] state that the change in wettability is a result of the plasticization of free water or lignin. Hill (2006) [12] stated that there is a decrease in hydrophilicity with an increase in the contact angle, so the rate of swelling and shrinkage of the material decreases with an increase in the contact angle values.

It is important both industrially and academically to know the changes in the technological properties of the trees of industrial importance after heat treatment application. Dimensional stability properties of wood material are one of the most important issues to be considered especially in materials used in outdoor weather conditions. Therefore, there are many studies on dimensional stability and wettability of wood material. In this study, heat treated (treatment) and unheat-treated (control) Ash (Fraxinus excelsior) woods were used for wettability, dimensional stability tests. The primary aim of this study is to examine the wettability and dimensional stability properties of heat treated materials obtained from different types of trees in tangential and radial sections. In addition, heat treated samples are compared with unheat treated samples to examine surface wettability, swelling-shrinking behaviour.

#### 2. Material and Methods

### 2.1. Preparation of Test Samples

In this study, Ash (Fraxinus exelsior L.) woods, which are widely used in wood working and furniture industry, were used. The wood materials used were obtained from the Naswood plant operating in Dosemealti, Antalya (Nasreddin Forest Products). It has been ensured that this timber provided is perfect and that the fibers are smooth, without backing, without reaction wood, and has not suffered from fungal and insect damages. The lumber supplied was divided into two parts from the core, and one part was heat treated, while the remaining part remained as a control sample. The ash wood used in the study was subjected to heat treatment in Nasreddin Forest Products Factory according to "ThermoWood" process for 120 minutes at 210°C (This time refers to the temperature and time applied to the heat treatment in the second stage).

#### 2.2. Measurement of Contact Angle

Firstly, timber (control and treatment) was cut with a motorized saw blade in dimensions of 20x20x30 mm (thickness x width x length) to make the surface wettability test. A total of 30 samples, 15 from control samples and 15 from treatment samples, were studied.

After the samples were taken, the surfaces of the samples were sanded to obtain surface smoothness. The samples were stored at  $20\pm5$  °C at  $60\pm5$  % relative humidity until they reached equilibrium humidity. In this study, contact angle values were determined to determine the surface wettability of ash samples. A schematic view of the system established to indicate the surface contact angles of the samples is given in Figure 1.



**Figure 1.** Schematic view of contact angle measuring device a: Light and control center, b:Micro syringe, c: Camera, d: Sample placement table

The distilled water was drilled down to 5  $\mu$ l with the help of a syringe. With a syringe, a drop of water was dropped on the surface of the material and an image was taken every 5 seconds. Therefore, 7 images were taken for 30 seconds for one sample [16, 22]. The contact angle values were determined on the images taken with the image analysis program "Image J". It is shown in Figure 2 that the drops drawn for 30 seconds every 5 seconds are seen on the control and treatment samples of the ash wood.

The contact angle values measured by the image analysis program are measured according to results of Štrbová et al. (2013) [23] study and Cengiz, 2010 [24](Figure 3).



Figure 3. Determination of contact angle in image analysis program

Comparison tables with the contact angle values obtained using the image analysis program (Image J) were created.

#### 2.3. Measurement of Swelling and Shrinkage

In this study, dimensional change was examined in terms of shrinkage and swelling. In this study, TS 4083 [25] was used to determine the shrinkage value and TS 4084 [26] standard was used to determine the swelling value. The weight of the test pieces was immersed in distilled water at 20  $\pm$  5 ° C in a container until the weight became unchanged and

then measurements were made and recorded. The test pieces were dried to about 12-15% humidity in normal room conditions. Humidity values of the samples were measured and checked with Gann brand moisture meter. The test pieces were dried in the drying cabinet until they reached a constant weight of  $103\pm2^{\circ}$ C. Measurements were taken when the samples became fully dry.

The following Equation was used in the calculation of the shrinkage quantities [25];

$$\beta = \frac{L_2 - L_1}{L_2} x100 \ (\%) \tag{1}$$

 $L_2$ , dimensions of the test piece which has a moisture level on the fiber saturation point in radial (R), tangential (T) and longitudinal (L) directions (mm);  $L_1$ , the dimensions of the test piece in R, T and L directions after drying (mm).

The T, R and L direction measurements were used to determine the percentage of the shrinkage in the tangent ( $\beta_t$ ), radial ( $\beta_r$ ), and the longitudinal direction ( $\beta_l$ ). The volumetric shrinkage ( $\beta_v$ ) was calculated by the following Equation [26].

$$\beta_{\nu=}\beta_{t+}\beta_{r+}\beta_{l} \tag{2}$$

The test pieces were dried in the drying cabinet until they reached constant dimensions at  $103 \pm 2$  °C. Then, the test pieces were immersed in distilled water in a container until the dimensions of the test pieces remained unchanged. After the samples were removed from the water, the measurements were taken and the following Equation was used to calculate the amount of swelling ( $\alpha$ ).

$$\alpha = \frac{L_{2} - L_{1}}{L_{2}} x100$$
 (%) (3)

Where; L<sub>2</sub>, dimensions of the test piece which has a moisture level on the fiber saturation point in R, T and L directions (mm); L<sub>1</sub>, the dimensions of the test piece in R, T and L directions after drying (mm).



Figure 2. Water drop on ash wood (control and treatment) samples (0-30 seconds)

The T, R and L direction measurements were used to determine the percentage of the swelling in the tangent ( $\alpha_t$ ), radial ( $\alpha_r$ ), and the longitudinal direction ( $\alpha_l$ ). The volumetric swelling ( $\alpha_v$ ) was calculated by the following Equation [26].

$$\alpha_{\nu=}\alpha_{t+}\alpha_{r+}\alpha_{l} \tag{4}$$

After determining the contact angle, swelling and shrinkage percentages, graphs were generated and values were met for both contact angle and swellingshrinkage values. The data were entered into the SPSS analysis program and analyzed.

#### 3. Results

The tangential and radial direction contact angle values of the Ash (Control-Treatment) test samples are given in Figure 4.



Figure 4. Contact angle values of Ash samples (0-5-10-15-20-25-30 sc.)

Table 1 shows ash wood contact angle measurement results.

When the measurements taken for thirty seconds were evaluated, it was determined that the contact angle value decrease rate of the control samples was 39.53 in the contact angle values are approximately 2-6 degrees lower than tangential surface contact angle values. According to the T-test results, the contact angle values of the heat treated samples have increased significantly compared to the control sample (p < 0.05).

Hill (2006) [12] stated that there is a decrease in hydrophilicity with an increase in the contact angle, and thus the rate of expansion and contraction of the material with an increase in contact angle values. Since the surfaces of the heat treated wood are less polar, they are waterproof and thus have less wettability than wood without heat treatment. After determination of dynamic wettability properties, radial, tangential and volumetric, swelling and shrinkage amounts of wood species were determined. The results of the swelling and shrinkage of the ash samples are given in Table 2, Figure 5 and Figure 6.



Figure 5. Radial, tangential, longitudinal and volumetric swelling graph



Figure 6. Radial, tangential, longitudinal and volumetric shrinkage graph

In Table 2, it is seen that the volumetric swelling percentage is 3.94%, the volumetric shrinkage percentage is 3.97% in the control samples, the volumetric swelling percentage is 3.77% and the volumetric shrinkage percentage is 3.77% in treatment samples.

When the contraction and swelling amounts are examined, it is seen that the shrinkage and swelling values of the heat treated samples are significantly lower (p < 0.05).

In particular, when the percentages of swelling and shrinkage in the R and T directions are examined, the surface swelling and shrinkage values of the control samples are respectively 1.91% - 1.91% in the radial direction and 1.98% - 2.00% in the tangential direction, these values of the process samples are respectively 1.83% - 1.81% in the radial direction and 1.90% - 1.91% in the tangential direction. It is seen from these data that the percentage of swelling and shrinkage of radial surface is lower than tangential surface (p<0,05).

Mean (sc.)																
	N	0		5		10		15		20		25		30		
		84,30		75,91		66,62		62,58		56,35		50,98		45,70		
UT (R)	15	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	
		78,15	94,24	69,75	85,75	54,40	74,70	51,94	67,16	40,50	62,66	38,16	58,69	35,40	52,20	
		86	,71	80,15		71,73		65,67		60,32		53,67		48,68		
UT (T)	15	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	
		81,75	96,48	73,67	89,37	60,73	77,37	53,65	69,79	50,97	65,85	42,98	59,37	38,14	55,40	
		96	<b>5</b> ,84	91	1,05	87,62		80,61		74,01		67,51		62,46		
HT(R)	15	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	
		90,48	102,19	81,37	97,38	80,27	92,94	70,26	89,36	67,48	82,20	61,39	75,44	53,79	69,75	
		99,13		95,60		91,43		83,80		77,84		73,01		66,15		
НТ (Т)	15	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	
		92,15	105,18	90,95	102,37	88,37	95,18	74,86	90,64	61,38	84,46	59,63	79,37	59,25	74,79	
						St	andard	Deviatio	n							
UT (R)	15	5,	,14	4,92		5,50		4,34		7,26		7,07		5,72		
UT (T)	15	5,18		4,51		5,83		4,40		5,46		5,30		5,77		
HT(R)	15	3,81		3,77		3,46		5,98		4,47		4,23		3,92		
HT (T)	15	3,84		3,17		2,37		5,33		7,	43	6,38		4,57		
							Standar	d Error								
UT (R)	15	1,32		1,27		1,42		1,12		1,87		1,82		1,47		
UT (T)	15	1,33		1,16		1,50		1,13		1,40		1,36		1,49		
HT(R)	15	0,98		0,97		0,89		1,54		1,15		1,09		1,01		
HT (T)	15	0,	,99	0,81 0,6			61	1,	37	1,91		1,64		1,17		
							Su	m								
UT (R)	15	126	64,45	1138,61 999,33			9,33	938	3,65	845,27		764,63		685,45		
UT (T)	15	1300,65		1202,27		1075,95		985,02		904,75		805,05		730,14		
HT(R)	15	1452,55		1365,80		1314,34		1209,11		1110,12		1012,57		936,93		
HT (T)	15	1486,88		1433,93		1371,43		1256,98		1167,54		1095,08		992,26		
Coefficient of Variation																
UT (R)	15	0,	,06	0,06		0,08		0,07		0,13		0,14		0,13		
UT (T)	15	0,	,06	0	,06	0,08		0,07		0,09		0,10		0,12		
HT(R)	15	0,	,04	0	0,04 0,04		04	0,	0,07		0,06		0,06		0,06	
HT (T)	15	0,04		0,03		0,03		0,06		0,10		0,09		0,07		

#### Table 1. Ash wood contact angle measurement results

N: Number of Sample, UT: Unheat-treated, HT: Heat-treated, R: Radial direction, T: Tangential direction, Min: Minimum, Max: Maximum

 Table 2. Results of the swelling and shrinkage test of the Ash samples

					Swell	ing								
Direct.	Treatment	Ν	Mean (%)	SD	CV	Min	Max	SE	Mean (%)	SD	CV	Min	Max	SE
R	UT	15	1,91	0,06	0,03	1,80	1,99	0,01	1,91	0,06	0,03	1,8 2	2,00	0,01
	НТ	15	1,83	0,07	0,04	1,71	1,96	0,01	1,81	0,06	0,03	1,70	1,91	0,01
т	UT	15	1,98	0,06	0,03	1,90	2,07	0,01	2,00	0,06	0,03	1,91	2,16	0,01
	НТ	15	1,90	0,06	0,03	1,83	1,99	0,01	1,91	0,06	0,03	1,83	2,00	0,01
L	UT	15	0,06	0,02	0,32	0,037	0,087	0,004	0,06	0,02	0,27	0,036	0,088	0,004
	НТ	15	0,04	0,01	0,30	0,021	0,071	0,003	0,05	0,02	0,34	0,021	0,724	0,004
v	UT	15	3,94	0,10	0,02	3,807	4,107	0,025	3,97	0,11	0,03	3,817	4,170	0,028
	НТ	15	3,77	0,11	0,03	3,568	3,988	0,028	3,77	0,11	0,03	3,596	3,964	0,027

SD: Standard Deviation, SE: Standard Error, CV: Coefficient of Variation, N: Number of Samples, R: Radial, T: Tangential, L: Longitudinal, V: Volumetric, Min: Minimum, Max: Maximum



Figure 7. Contact angle, swelling and shrinkage values in radial and tangential directions (R: Radial, T:Tangential)

Dimensional stability properties of wood material are one of the most important issues to be considered especially in materials used in outdoor weather conditions. Therefore, there are many studies on dimensional stability and wettability of wood material. The findings obtained from these studies show that the dimensional stability of wood material varies depending on many factors such as material type, heat treatment application conditions, heat treatment temperature and duration. In most studies on this subject, it has been stated that the dimensional stability of heat treated wood has increased and thus the swelling and shrinkage percentages of the wood material have decreased [27-39, 22].

Heat treatment of wood significantly reduces its water adsorption (p<0,05). Decrease in accessibility of free hydroxyl groups as a result of heat treatment application is undoubtedly obvious [12, 40]. In this study, especially in radial and tangential directions, when the contact angle, swelling and shrinkage values were compared, the contact angle values of the heat treated (HT) samples were higher than the unheat treated (UT) samples and the contraction and expansion values were lower (Figure 7).

In this study, when the contact angle, swelling and shrinkage values were compared, it was determined that there was a negative correlation between the contact angle values and swelling and shrinkage values, but there was a positive correlation between swelling and shrinkage values. In other words, with the increase in contact angle values, it was concluded that the amount of swelling and shrinkage decreased according to the statistical data results.

#### 4. Discussion and Conclusion

When the contact angle values of the heat treated and non-heat treated samples were examined, it was determined that the contact angle values increased with the heat treatment application.

With the increase in the contact angle, there is a decrease in hydrophilicity and thus the dimensional

stability of the material increases with the increase in contact angle values.

Therefore, according to the results of the dynamic wetting tests, it is seen that the heat treatment application reduces the wettability of the material. It has been found that the contact angle measurement values made in the tangential and radial directions give close results, but the contact angle values in the radial direction are lower than the contact angle values in the tangential direction. This result shows that the wettability in the tangential direction is lower than the radial direction.

When the percentages of swelling and shrinkage were examined, it was determined that with the application of heat treatment, the percentage of swelling and shrinkage of wood material decreased and therefore dimensional stability property increased.

In conclusion, the contact angle values of the heat treated materials and the percentages of swelling and shrinkage gave similar results. As a result of both experiments, it is seen that heat treatment reduces the wettability of wood material. The wettability of wood materials can be shown as an indicator of dimensional stability. For this reason, it is possible to get a preliminary idea about this feature with the contact angle method instead of long-term and laborious studies to obtain information about the dimensional stability of wooden materials.

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#### **Declaration of Ethical Code**

In this study, we undertake that all the rules required to be followed within the scope of the "Higher Education Institutions Scientific Research and Publication Ethics Directive" are complied with, and that none of the actions stated under the heading "Actions Against Scientific Research and Publication Ethics" are not carried out.

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