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The Effect of Heat Modification and Densification on Physical Properties of Poplar Wood

Utjecaj toplinske modifikacije i ugušćivanja na fizikalna svojstva topolovine

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ABSTRACT • *In this study, density, volumetric swelling, mass loss, volume weight, fiber saturation point and water absorption of the poplar wood (Populus usbekistanica) were investigated with the effect of heat modification (HM) and heat-modified densification (HMD). Poplar samples were modified with steam at 120 °C, 160 °C and 200 °C for 1 and 3 h. After heat modification, the samples were compressed in hot press at a temperature of 120 °C, press pressure of 5 MPa and press time of 30 minutes for densification. Physical properties of the samples were determined according to Turkish standards. The results showed that heat modification affected densification and increased density. Densification had a positive effect on oven dry density (D_0), oven dry density after soaking (D_{0S}), volume weight (R) and fiber saturation point (FSP), except for volumetric swelling. Similarly, the densification process had an increasing effect on the water absorption, excluding 360 hours.*

Keywords: *poplar wood, densification, heat modification, physical properties*

SAŽETAK • *U ovoj je studiji istražen utjecaj toplinske modifikacije (HM) i ugušćivanja (HMD) na gustoću, volumno bubrenje, gubitak mase, volumnu težinu, točku zasićenosti vlakanaca i upijanje vode drva topole (Populus usbekistanica). Uzorci topolovine modificirani su parom pri 120, 160 i 200 °C tijekom jednoga i tri sata. Nakon toplinske modifikacije uzorci su radi ugušćivanja stlačeni u vrućoj preši, pri temperaturi 120 °C, tlaku prešanja 5 MPa i vremenu prešanja 30 minuta. Fizikalna svojstva uzoraka utvrđena su prema Turskim standardima. Rezultati su pokazali da je toplinska modifikacija utjecala na proces ugušćivanja i pridonijela povećanju gustoće. Ugušćivanje je imalo pozitivan učinak na gustoću topolovine u apsolutno suhom stanju (D_0), na njezinu gustoću u apsolutno suhom stanju nakon natapanja (D_{0S}), na volumnu težinu (R) i na točku zasićenosti vlakanaca (FSP), ali ne i na volumno bubrenje. Također, ugušćivanje je znatnije utjecalo na sposobnost upijanja veće količine vode, osim za vrijeme od 360 sati.*

Ključne riječi: *topolovina, ugušćivanje, toplinska modifikacija, fizikalna svojstva*

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1 INTRODUCTION
1. UVOD

There have been many studies on heat modification recently, due to its many advantages such as improving the dimensional stability and durability of wood, without using chemicals (Tjeerdsma *et al*.; 1998; Weiland *et al*.; 2003; Garcia *et al*., 2012). This technology was applied to wood at a temperature of about 200°C for several hours at low oxygen level, a non-inflammable gas like oil or nitrogen. The main aim of this technique is to convert the chemical composition of wood using heat, which results in a change of physical properties. The process of thermal modification is based on high temperatures, which cause the decomposition of basic wood component like hemicelluloses and celluloses (Tjeerdsma and Militz, 2005; Rowell *et al*., 2009). Schneider (1973) noted that treatment, at temperatures above 200 ºC and of longer duration, decreased swelling and shrinkage up to 50 %. Besides, he stated that sorption and desorption characteristics also changed in heat modified wood.

In 1980s, densified wood products were produced from low density wood species, especially for utilization of some fast growing trees (Wang *et al*., 2000). The utilization of hardwood species for flooring and furniture reduced their supply all over the world. Various efforts have been made to modify the surface of low-quality softwood and planted fast-growing species. The surface densification technology has been developed and several researches have studied this technology (Rautkari *et al*., 2008; Gong and Lamason, 2007; Diouf *et al*., 2011). The thermal compression process might affect the drying duration, dimensional stability, surface quality, hygroscopicity, durability, and mechanical properties (Welzbacher *et al*., 2008).

The aim of this research is to determine the interactive effect of heat modification and post-heat densification on the poplar wood. The specific objective of the study was to analyze volumetric swelling and other chosen properties of heat threated and densified samples compared with samples densified without heat modification. These properties are: density, mass loss, volume weight, fiber saturation point and water uptake.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

Populus trees were obtained from Kahramanmaraş province in Turkey. Wood samples used in the study were prepared in dimensions 20×20×30 mm. For each group of modification, 27 samples were prepared from logs. Before testing, each sample was conditioned at 20 °C temperature and at 65 % relative humidity until reaching a moisture content of 12 %.

The samples were first heat modified by using laboratory drying oven at a temperature of 120 °C, 160 °C and 200 °C and modification time of either 60 or 180 min (Table 1). During heat modification, 100 ml water vapor was heated up to 100 °C degrees in the oven, and then the vapor was removed and kept still until the desired temperatures reached 120 °C, 160 °C and 200 °C. After the heat modification, samples were hot pressed by using a laboratory hot press at 120 °C temperature and 5 MPa press pressure for 30 minutes (Table 1).

The obtained results were statistically analyzed by using one-way ANOVA and Duncan's mean separa-

Table 1 Parameters used in tests of samples - undensified and densified
Tablica 1. Parametri obrade toplinski modificiranih uzoraka, neugušćenih i ugušćenih

Treatment *Obrada*	**Heat temperature** *Temperatura zagrijavanja* °C	**Heat time** *Vrijeme zagrijavanja* min	**Pressure** *Tlak* MPa	**Pressure temperature** *Temperatura prešanja* °C	**Pressure time** *Vrijeme prešanja* min
Undensified / *Neugušćeni uzorci*					
Control / *kontrolni uzorak*	-	-	-	-	-
Heat modified (HM) *Toplinski modificirani uzorci*					
HM11	120	60	-	-	-
HM13	120	180	-	-	-
HM21	160	60	-	-	-
HM23	160	180	-	-	-
HM31	200	60	-	-	-
HM33	200	180	-	-	-
Densified / *Ugušćeni uzorci*					
Control / *kontrolni uzorak*	-	-	5	120	30
Heat modified (HM) *Toplinski modificirani uzorci*					
HM11	120	60	5	120	30
HM13	120	180	5	120	30
HM21	160	60	5	120	30
HM23	160	180	5	120	30
HM31	200	60	5	120	30
HM33	200	180	5	120	30

tion test to populate homogeneity groups that showed significant differences at the 95 % confidence level.

Oven dry density (D_0): This term expresses the amount of substance in the full dry unit volume and it is calculated according to TS 2472.

Volume weight value (*R*): This value describes the amount of substance in the full wet unit volume and it is determined according to TS 2472.

Fiber saturation point (*FSP*) was calculated according to Turkish standards TS 2371 and Equation 1 given below:

$$FSP = \frac{V_S}{D_0} \cdot 100 \quad (1)$$

where V_S is volumetric swelling (%).

Oven dry density after soaking (D_{0S}): After soaking for 15 days in water, samples were dried to 0 % moisture content and oven dry densities were determined according to TS 2472. Mass loss (*ML*), volumetric swelling (%) and water absorption (W_A) were calculated by equations (2), (3) and (4), respectively,

$$ML = \left(\frac{M_0 - M}{M_0}\right) \cdot 100 \quad (2)$$

ML – mass loss (%), M_o – mass of oven-dry sample before heat modification (gr), *M* – mass of the sample after heat modification (gr).

$$V_S(\%) = \left(\frac{L - L_0}{L_0}\right) \cdot 100 \quad (3)$$

V_S – volumetric swelling (radial, tangential and longitudinal), *L* – wet dimension after immersion in water (mm), L_o – oven-dry dimension (mm),

$$W_A(\%) = \left(\frac{W - W_0}{W_0}\right) \cdot 100 \quad (4)$$

W_A – water absorption, *W* – weight after immersion in water (gr), W_o – oven-dry weight before immersion in water (gr).

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

Average oven density values of densified and undensified poplar wood samples with and without heat modification are shown in Table 2.

The effect of heat modification on density is given in Table 2. According to this Table, it can be seen that oven dry density of densified samples differs from that of undensified samples. Heat modification has not shown significant effect on undensified poplar and has caused 1 % decrease in density. The highest decrease in density among undensified poplar was observed at 200 ºC heat modification (HT31 and HT33). Means of samples densified with heat modification (897 kg/m^3) were compared to density of densified control samples (668 kg/m^3), and the results clearly showed that heat modification increased density by 34 %. The density of samples densified with and without heat modification was increased by 102 % and 174 % on average, respectively. Gong *et al*. (2010) studied the effect of heat treatment on density between undensified and densified poplar (*Populus tremuloides*) and concluded that wood density in densified poplar decreased to 11 % during the heat treatment, while density decreased to 2 % in undensified poplar wood. In another words, they concluded that the heat treatment of densified poplar increased density. Wang and Cooper (2005) found that the density of densified wood was affected by compress temperature, press duration time, press closing time and humidity of samples before compressing

The average volumetric swelling is given in Figure 1 for undensified and densified poplar samples with and without heat modification. The undensified volumetric swelling values at 120 and 160 ºC are higher than those of control samples but at 200 ºC they are lower. These differences are insignificant (p<0.062). Besides, the duration of heat modification did not indicate significant differences in all parameters, except for control samples. On the other hand, in densified samples, all temperatures and durations had a significant effect (p<0.001) on respective values. Moreover, as the duration of heat modification rose, volumetric swelling decreased. The mean volumetric swelling of undensified and modified samples (10.1 %) decreased by 3.7 % in comparison with the undensified control group (10.49 %). The value of modified and densified samples (180.35 %) increased by 85.68 % compared to their control samples (97.13 %).

Table 2 Average oven density (kg/m^3) values of undensified and densified poplar wood; control and heat modified
Tablica 2. Srednja vrijednost gustoće neugušćenih i ugušćenih uzoraka topolovine u apsolutno suhom stanju; vrijednosti kontrolnih i toplinski modificiranih uzoraka

Treatment *Obrada*	**Densified** / *Ugušćeni*	**Undensified** / *Neugušćeni*	**Increase** / *Povećanje* **%**
Control / *kontrolni uzorak*	668	331	102
Heat modified / *Toplinski modificirani uzorci*			
HM11	950	334	184
HM13	859	335	156
HM21	944	331	185
HM23	884	330	168
HM31	878	310	183
HM33	869	321	171
Average / *prosječna vrijednost*	897	327	174

HM: Heat modified / *toplinski modificirani uzorci*

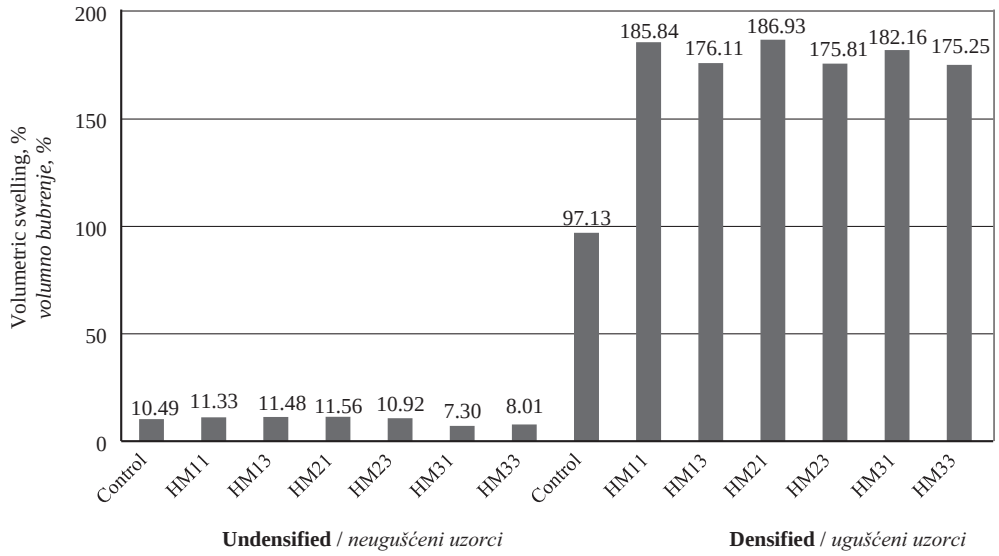

Figure 1 Average volumetric swelling of undensified and densified poplar wood with and without heat-modification
Slika 1. Prosječno volumno bubrenje neugušćenih i ugušćenih te nemodificiranih i toplinski modificiranih uzoraka topolovine

Comparable results were recorded in other studies. For instance, Gong et al. (2010) studied the effect of heat modification on densification and stated that heat modified wood is more resistant to swelling than densified wood. Also, Bal and Bektaş (2012) noted that heat treatment without steam decreased thickness swelling specially above 180 °C. In some studies, it was noted that the relationship between volumetric swelling and oven dried density was potently positive (Table 3) (Kord *et al*., 2010; Kurt, 2010). Candan *et al*. (2013) noted that all thermally compressed boards increased thickness swelling values. This result could be explained by springback behavior of wood due to densification (Abraham *et al*., 2010).

The results of physical properties of undensified and densified poplar samples without and with heat modification are given in Table 3.

Densification process with and without heat modification increased density values according to Table 3. The highest density for undensified poplar was observed for heat modification temperature of 120 °C compared to control group. Table 3 shows that the densities measured at 200 °C in undensified samples seem to be lower, but this decrease is not significant. As seen from statistical results in Table 3, while heat temperature duration had no effect on density in undensified samples, a significant increase in density was observed when the duration of heat modification decreased from

Table 3 Comparison of physical properties of undensified and densified poplar wood
Tablica 3. Usporedba fizikalnih svojstava neugušćenih i ugušćenih uzoraka topolovine

Treatment / *Obrada*	D_o, g/cm³	D_{os}, g/cm³	*ML*, %	*R*, g/cm³	*FSP*, %
Undensified / *Neugušćeni uzorci*					
Control / *kontrolni uzorak*	0.331a(*)	0.328ab	__	0.299a	31.74a
Heat modified / *Toplinski modificirani uzorci*					
HM11	0.334a	0.333	0.30a	0.299a	33.92a
HM13	0.335a	0.324b	0.44a	0.299a	34.33a
HM21	0.331a	0.329ab	1.05ab	0.296a	34.89a
HM23	0.330a	0.328ab	1.82bc	0.297a	33.11a
HM31	0.310a	0.303a	8.40e	0.289a	23.41a
HM33	0.321a	0.316ab	6.61d	0.296a	24.99a
Densified / *Ugušćeni uzorci*					
Control / *kontrolni uzorak*	0.668b	0.376cd	__	0.320b	146.02b
Heat modified / *Toplinski modificirani uzorci*					
HM11	0.950d	0.425e	0.42a	0.335b	195.58c
HM13	0.859c	0.394d	0.66ab	0.323b	205.06c
HM21	0.944d	0.386cd	0.97ab	0.337b	197.98c
HM23	0.884c	0.366c	1.49abc	0.301a	223.36d
HM31	0.878c	0.396d	6.36d	0.322b	205.99c
HM33	0.869c	0.393cd	2.60c	0.327b	202.56c
ANOVA	p<0.001				

D_0 - Oven dry density / *gustoća u apsolutno suhom stanju*; D_{OS} - Oven dry density after soaking / *gustoća u apsolutno suhom stanju nakon natapanja*; *ML* - Mass loss / *gubitak mase*; *R* - Volume weight / *volumna težina*; FSP - Fiber saturation point / *točka zasićenosti vlakanaca*. *Means with the same small letter are not significantly different in Duncan's mean separation test. / *Srednje vrijednosti označene istim malim slovom nisu signifikantno različite prema Duncanovu testu*.

3 to 1h for densified poplar wood. As for densified samples, density decreased depending on the increase of the process duration (from 1 to 3h), except for the temperature of 200 °C. No significant difference was observed. This is in accordance with literature data. For example, Metsä-Kortelainen and Viitanen (2011) determined that 120 °C heat modification increased density compared to control samples, while 200 °C heat modification decreased density due to degradation of hemicellulose and cellulose. In another study, Cao and Huang (2012) noted that steam-heat-modification increased wood thermal conductivity and density.

Comparing the mean oven dry density after soaking (D_{0S}) with oven dry density (D_0), the density decreased 1 % in undensified samples and decreased 55 % in densified samples as seen in Table 3. This reduction occurred due to the permanent springback effect of the compressed wood. Densified and undensified poplar oven-dry densities after soaking (D_{os}) decreased compared to D_0. However, D_{0S} of densified poplar wood was still higher after soaking compared to undensified samples.

Values given in Table 3 demonstrate that densification process did not cause mass loss but heat modification increased mass loss with increased temperature. ML values of poplar at 200 °C were greater than at 120 and 160 °C. The average mass loss at 200, 160 and 120 °C was 6 %, 1.33 % and 0.45 %, respectively for all samples. A notably increased dimensional stability following thermal modification above 180 °C was noted in many studies (Welzbacher *et al*., 2008). This could be explained by the decreased hygroscopicity of components on wood cell walls (Fang *et al*., 2012). Decomposition of hemicelluloses and lignin at elevated temperatures and stress relaxation in samples caused a reduction in the hygroscopicity of wood (Cai *et al*., 2013). The value of volume weight of densified poplar (*R*) increased because density of densified poplar was higher than that of undensified poplar. The volume weight of densified wood increased by 9.31 %. The value of volume weight of undensified poplar (*R*) decreased at 200 °C with heat modification compared to other temperatures. Furthermore, *FSP* is directly related to volumetric swelling and oven dry density according to Equation (1) mentioned in the Method section. As is known, there is a linear relationship between *VS* and *FSP*. The *FSP* value of the control group (undensified and control) was 31.74 %. After the samples were heat modified at 200 °C (the mean of HT31 and HT33), the *FSP* values decreased to 23.75 % in poplar wood. Furthermore, the *FSP* values at 120 °C and 160 °C increased by 7.3 %. The *FSP* value of the control group (densified and control) was 146.02 %. After the densification, the *FSP* values increased to 40.47 %. Bal and Bektas (2012) noted that heat treatment at higher temperatures (180 °C) decreased values of *FSP*. Additionally, the volumetric swelling is very high due to the back-spring effect of wood. Abraham *et al*. (2010) also noted that the densified wood exhibited this behavior. For this reason, the high springback effect has caused the *FPS* value to increase up to 223 %.

Table 4 shows that the effect of heat modification on water absorption (%) differed between undensified and densified samples. It can be seen from Table 4 that, in the densified samples, the heat modification time had no statistically significant effect on water absorption at 120 ºC (HM11, HM13), whereas at 200 ºC (HM31, HM33) the respective effect was significant (p<0.000). As shown in Table 4, the modification temperature and duration have no statistically significant effect in the first 48 hours only in heat modified (not densified) wood. The water absorption values of heat modified samples decreased after 48 hours compared to control group. However, in 72 h and 96 h there was no statistically sig-

Table 4 Water absorption (%) of undensified and densified poplar wood without and with heat-modification
Tablica 4. Upijanje vode (%) ugušćenih i neugušćenih te nemodificiranih i toplinski modificiranih uzoraka topolovine

Treatment *Obrada*	1 h	2 h	3 h	24 h	48 h	72 h	96 h	360 h
Undensified *Neugušćeni uzorci*								
Control / *kontrolni uzorci*	7.64a(*)	13.77a	21.24a	51.49a	81.94bcd	92.05b	99.19b	129.62bcd
HM11	8.16a	13.48a	21.74a	48.26a	77.78bc	88.92b	95.13b	129.19bc
HM13	8.95a	15.75a	21.16a	51.67a	80.55bcd	91.00b	99.83b	130.42bcde
HM21	8.95a	15.04a	21.02a	49.43a	79.05bcd	90.78b	99.53b	131.44bcde
HM23	8.61a	14.42a	18.79a	45.03a	74.90bc	87.81b	94.32b	122.71bc
HM31	10.00a	17.57a	21.70a	50.57a	77.74bc	100.74b	112.24b	150.44def
HM33	6.25a	11.50a	17.39a	43.28a	68.51ab	87.52b	95.32b	131.78bcde
Densified / *Ugušćeni uzorci*								
Control / *kontrolni uzorci*	30.98cd	54.44b	56.96b	84.12b	96.54d	105.50b	108.78b	119.02b
HM11	31.21cd	49.44b	58.55b	112.48cd	126.23ef	132.82c	135.64cd	151.00ef
HM13	23.32bc	50.15b	62.84b	107.05c	122.02e	127.69c	130.33c	149.29cdef
HM21	36.20cde	59.74b	80.25c	126.09d	141.08f	156.15d	150.80d	167.31f
HM23	48.24e	62.70b	69.70bc	78.80b	90.08cd	94.59b	98.23b	111.81b
HM31	11.54ab	21.08a	25.60a	48.87a	58.09a	67.76a	68.01a	89.56a
HM33	41.33de	50.73b	58.65b	81.33b	91.55cd	98.68b	100.85b	118.49b
ANOVA	p<0.001							

HM – Heat-modified / *toplinski modificirani uzorci*. *Means with the same small letter are not significantly different in Duncan's mean separation test. / *Srednje vrijednosti označene istim malim slovom nisu signifikantno različite prema Duncanovu testu.*

nificant difference in water absorption compared to the control sample. As water absorption (%) changed with time, the lowest water absorption in undensified group was obtained in HT23 (122.71 %) after 360 hours. The lowest water absorption in the densified wood in the interval from 1 hour to 360 hours was determined in HT31 compared to the densified control. The water absorption in the first 24 hours in the densified wood continued to be water intake without heat treatment temperature and duration effective, and after 24 hours HT23, HT31 and HT33 showed lower water absortion than the densified control sample. When compared to the undensified control sample (129.62 %), the lowest water absorption percentage was determined in HM23 (122.71 %). Likewise, in densified samples, the lowest decrease in HM31 (89.56 %) was calculated according to control samples (119.02 %).Also, Cai *et al*. (2013) noted that the water absorption of densified wood with and without heat treatment was affected by compression temperature, compression ratio and pressure holding time. Similar findings were obtained by Unsal *et al*. (2009), and they indicated that the compressed pine decreased water absorption for 5MPa at 120 °C compared to uncompressed pine, while higher compression temperature and compression ratio increased water absorption.

4 CONCLUSIONS
4. ZAKLJUČAK

The main outputs of this study are summarized below:

1. Density of undensified poplar wood was minimally (1 %) affected by heat modification. However, density was influenced by heat modification before densification and it increased density by 34 %.
2. Volumetric swelling of undensified poplar wood decreased significantly only at 200 °C heat modification. Moreover, the densification increased the volumetric swelling due to the springback effect.
3. In comparison to oven dry density (D_0), the oven dry density after soaking (D_{0S}) decreased in undensified and densified samples at the rate of 1 % and 55 %, respectively. In addition, the mass loss increased with the increase of heat modification temperature in all samples, notwithstanding the densification process. Densification process increased the volume weight and fiber saturation point compared to heat modification.
4. No significant correlation was found between water absorption time and heat modification process for undensified samples. On the other hand, in densified samples, water absorption retention improved as the heat modification temperature and duration increased. Besides, the effect of the application time increased depending on temperature increase.
5. Poplar trees are fast growing and have low durability. However, poplar wood has become a material with high density and lower water absorption by heat modification and densification process. Thus, species with low durability can be converted to wood material of high density and extended service life by heat modification and densification process.
6. One of the remarkable characteristics of this study is that no chemicals have been used during the process and there have been no side effects that could adversely affect human and environmental health during and after the process.
7. In order to make the above physical properties meaningful and useful at the desired level, mechanical properties should also be researched in future as a part of this study.

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