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Influence of hydrothermal treatment on the dimensional stability of beech wood

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ABSTRACT
Hydrothermal treatment of wood material products with many interesting properties such as enhanced dimensional stability, lower equilibrium moisture content and increased biological durability. The effects of hydrothermal treatment on dimensional stability, oven dry density and water absorption of beech wood (*Fagus Orientalis*) naturally grown, has been studied in present research that extremely used in Iran. Hydrothermal applied to the wood samples (2.5Cm×2.5Cm×2.5Cm) in three different temperatures (150 oC, 160 oC, 170 oC) and four different durations (1h, 3h, 5h and 7h) in reactor. Then, the samples and their controls were immersed in water at 25 oC, 65% relative humidity for various periods. Volumetric swelling percentage and rate of the water absorption values of the samples and their controls were measured at a period of 24 hour in 8 steps on basis of oven dried dimension. The obtained data were analyzed using Microsoft Office (EXCEL 2003). The data analysis indicated that best anti-swelling efficiency value was achieved at 170 oC (treating temperature) within 1 hour (treating time) and 8 steps of soaking - dried measurement by hydrothermal treatment. Consequences for anti-swelling efficiency, decreasing of samples specific gravity, water absorption and water repellency effectiveness values obtained 27.95%, 8.49%, 56.41% and 20.20% respectively. Therefore, hydrothermal treatment showed a little decreasing at 150 ^{o}C to 170^{o}C in specific gravity of treatment samples because maximum lessening of specific gravity obtained 8.02% in the highest condition of treatment after final soaking.

Keywords: Hydrothermal treatment, Dimensional stability, Water absorption, Specific gravity, Beech wood.

INTODUCTION
The use of wood in many applications is limited by decay, mould formation or by dimensional changes due to varying moisture content. These adverse effects are usually combated by treating wood thermally.

There are various thermal treatment processes that they have been developed to improve dimensional stability of wood (Tjeerdsma *et al.*, 1998; Militz and Tjeerdsma, 2001; Vernios *et al.*, 2000; Hietala *et al.*, 2002; Sivonen *et al.*, 2002; Brischke and Rapp, 2001; Rep *et al.*, 2004; Westin *et al.*, 2004). Synonyms for "Hydrothermal treatment" are "Hydrothermal modification", "Thermal modification", "Thermal treatment" and "Heat treatment". It is a physical method, which changes the chemical constitution of the martial without impregnating it with additional chemical (Rapp *et al.*, 2001; Brisch and Rapp, 2004).Hydrothermal modification is a technique to produce wood products with increased dimensional stability and lower equilibrium moisture (Yildiz, 2004a).

Plato treatment is a well – known heating process to manufacture wood products with improved qualities. It was reported that the plato-treated beech wood have a great decreasing ASE in radial and tangential 10%, 13% respectively (Militz and Tjeerdsma, 2001). It was reported a method of dimensional stabilization of wood that was based on the use of no chemicals such as use of heat a lone in vacuum that showed after the third cycle of soaking/drying test (Rep *et al.*, 2004). According to this report, Anti-Swelling Efficiency was observed between 31%-44% for 190 °C and 200 °C respectively as maximum treatment in radial direction. Also, ASE was not additionally improved at higher temperature.

In general, aims of hydrothermal modification of wood are improving the dimensional stability in an environment friendly way by toxifying, by altering the substrate. Parts of the cell wall polymers are altered in thermal wood modification; this may lead to cross-linking, reduction of OH-groups and /or (undesired) cleavage of the chains. The reduction of accessibility of OH-groups leads to a limited interaction with water compared to untreated. Examples of thermal treatments are the PLATO process, PERDURE process and the STELLAC (Homan *et al.*, 2000, Syrjänen, 2001). In addition, it was found that dimensional stability could be improved by either steaming or heating while the wood was in a compressed status. Dimensional stability was achieved by heat plasticization of the thermoplastic matrix (lignin and hemicellulose) which is reset in its compressed status and not by degradation of hemicelluloses. Results showed that minimizing the degradation of hemicelluloses also minimizing the decrease in mechanical properties of the compressed wood (Yildiz *et al.*, 2004a, Inoue *et al.*, 1993).

In the following, experimental investigation of dimensional stability of beech wood for hydrothermal modification presented.

METHODS

Raw material

Raw material was beech wood (Fagus Orientalis) obtained from north region of IRAN. beech wood was cut in parallel to grain direction and sawn into (specimens measuring) 25 mm ×25 mm ×25 mm long. Samples were conditioned for approximately two weeks. Mean wood absolute moisture content determined on a series of representative samples was about 12%, while the mean specific gravity determined at the above moisture content amounted to about 0.58 gr/cm^3.

Specimens were sorted into 12 treatment groups and for each group, 20 test and 20 control samples were used. Prior to test, the dimensions were measured to the nearest 0.01 mm and their weights were recorded to the nearest 0.001gr.

Hydrothermal Treatment

Hydrothermal treatment was then applied on the test samples in a 21-L batch cylindrical mini digester (stainless steel 321) which controlling to the temperature ± 5 oC sensitively, at different temperatures and duration under reactor pressure (Table 1). The mini digester includes an electrical heater, a motor actuator and required instruments for measurement and control of pressure and temperature. The solid/ distilled water ratio was fixed (1/20 d. w.).

Soaking/Oven Drying Test

After hydrothermal treatment and conditioned (12% moisture content) under atmosphere pressure and in present of air, Improvement of Dimensional Stability, Water Repellency Effectiveness (WRE)[1] and Specific Gravity (SG) of hydrothermal treated beech wood were evaluated using soaking /oven drying test for 8 cycles. All of treated and untreated (control) specimens (dimensions: l × r × t: 2.4 mm ×2.4 mm ×2.4 mm) for each maximum temperature were saturated with distilled water.

They were submersed for the distilled water at 20 oC in plastic basins for 24 hour in each of cycles. Separate basins were used for the different modified and untreated materials. Radial (r), tangential (t), longitudinal and volume (v) (by means of summation of values of main direction dimensions) dimensions of saturated specimens were determined. After that all specimens were air and oven dried to constant weights and dimensions.

[1]. Water Repellency Effectiveness, SG: Specific Gravity, ASE: anti-swelling efficiency.

Table 1. Conditional of hydrothermal treatment of beech wood.

Thermal treatment(°C)	150				160				170			
Thermal duration(h)	1	3	5	7	1	3	5	7	1	3	5	7

The WRE and oven dried weight were determined again. Whole cycle was repeated for 8 times.

The Anti-swelling efficiency and water repellency effectiveness values of the treatments and controls samples were determined on basis of oven dried measurement. After water – soaking/oven dried test, anti-swelling efficiency (ASE), water absorption, WRE and SG were calculated as:

$$a = \frac{l_s - l_o}{l_o} \times 100$$

a : Swelling
l_s : Length, swollen
l_o : Length, oven dried

$$ASE_{(r,t,v)}(\%) = \frac{\alpha_c(r,t,v) - \alpha_t(r,t,v)}{\alpha_c(r,t,v)} \times 100$$

$a_{c,}$: average swelling of untreated (control) samples each one of groups.
α_t : average swelling treated samples each one of groups in certain direction.

$$T = \frac{W_w - W_{O.D.}}{W_{O.D.}} \times 100$$

T: rate of the water absorption specimen.
W_w : weight of wet samples.
$W_{O.D.}$: weight of oven drying samples.

$$WRE\ (\%) = \frac{T_1 - T_2}{T_1} \times 100$$

T_1: rate of the water absorption of control specimen.
T_2 is the rate of the water absorption of the test specimen.

$$SG = \frac{M_0}{V_o} (g / cm^3)$$

M_0: the dry weight of the treatment and control.
V_0 : the volume of the samples when they are oven dried

RESULTS AND DISCUSSION

Anti-swelling efficiency

The main effect of hydrothermal treatment was a reduction of wood hygroscopicity (Feist and Sell 1987). For this purpose, it was calculated water absorption and ASE which is the most known method of evolution of the dimensional stabilization processes of wood (Santo, 2000). Fig. 1 shows the variation of ASE after 1 and 8 periods of soaking the specimens for all conditions of treatment time and temperature. According to these figs., average ASE percentage is improved by use of senior conditions and when specimens were treated at 150 °C, the ASE value was found superior for 7 hour than for 1 hour for all of period of soaking. Also, At 170 °C for 1 hour was nearly similar to the ASE value from variation at 170 °C for 7 hour for 1 period soaking. Consequently, the upp-ermost ASE (47.43%) was obtained at 170°C with 1 period of soaking/drying test. This result showed an analogue with the study of Yildiz. Yildiz *et al.,* (2004b) recorded that heating at 130 °C and 150 °C for 2, 6 and 10 h; ASE of wood exhibited an increase with increasing exposure temperature and time.

ASE of main directions of the specimens which soaked in water for the time of 24 hour for 8 cycle were observed as a result of the different temperatures and durations (Table 2). According to Table 2, ASE values were be dwindling by use of more than one cycle of soaking after treatment. For the reason that by use of many cycle of soaking, the specimens swelling efficiency (%) was being reduced (Yildiz, 2004a). However maximum swelling of all treated specimens with 8 cycles of soaking / oven drying was decreased. An ASE approximately 35.93% was found for specimens treated at 170 °C.

Water absorption

Commonly, water absorption decreased at all heat modified for instance hydrothermal treatment wood. But, when cracks was been increased, they were caused to increase the water absorption. Hence with increasing treatment condition for instance temperature or soaking cycle, their strength of the some specimens decrease for this reason they are caused to increasing the water absorption (Tjeerdsma, 1998; Vernois, 2000; Welzbacher and Rapp, 2004).

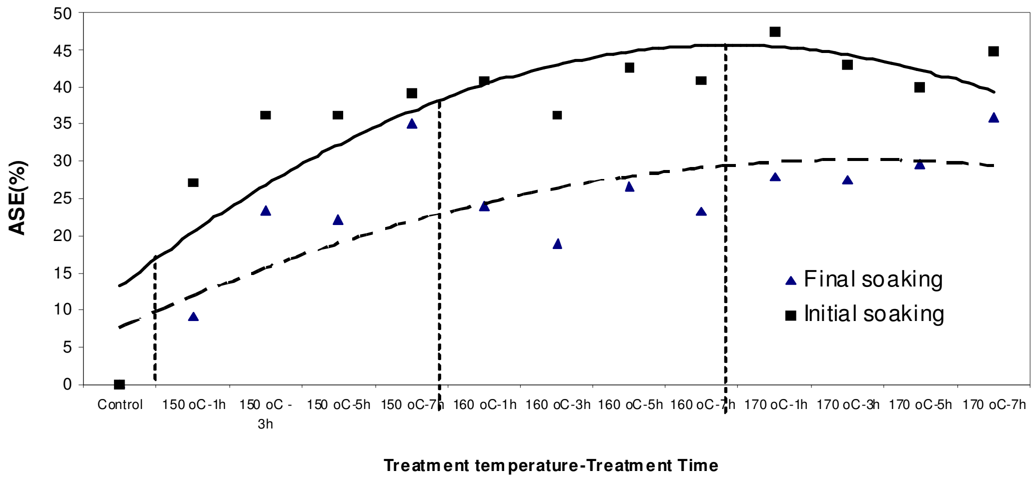


Fig 1. Variation of volumetric ASE percentage after initial (1 step) and final (8 step) soaking /oven drying hydrothermal treatment specimens.

Table 2. ASE values in radial, tangential direction and volumetric observed for hydrothermal treated beech at different temperatures and times after 8 soaking/ oven drying cycle.

Treatment Condition	Radial	Tangential	Volumetric
150 °C-1h	0.80	17.71	9.15
150 °C -3h	23.37	23.37	23.37
150 °C-5h	13.01	25.47	22.02
150 °C-7h	18.36	23.48	31.00
160 °C -1h	15.61	24.51	24.02
160 °C -3h	12.81	22.73	18.99
160 °C -5h	19.42	28.06	26.49
160 °C -7h	18.15	27.62	23.34
170 °C -1h	20.58	31.72	27.95
170 °C -3h	17.77	31.37	27.55
170 °C -5h	25.52	32.42	29.63
170 °C -7h	26.01	41.27	35.93

Figure 2 shows variations of water absorption at all of treatment condition after final soaking / oven drying cycle. It can be seen that when specimens is treating at 1 hour, for 170°C, water absorption is found greater than for 150°C and 160°C while at one hour treatment water absorption of specimens for 150°C is seems lower than control samples. Nevertheless it is increasing by use of great temperatures such 160°C and 170°C. In other section of this figure, water absorption is found greater than for 150°C when specimens are treating at 3 and 5 hour for 160°C and 170°C. It is at 170°C for 3 and 5 hour nearly the same of the water absorption at 150°C. Also, the highest water absorption percentage obtained at 5 hour for 160°C (60.86%).

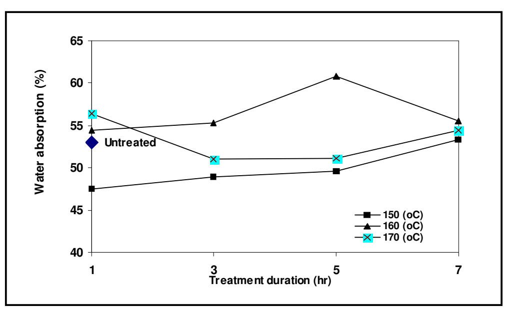


Fig 2. Water a bs or ption in hydrothermally modiried beech wood.

Consequently, it seems the water absorption is not increasing absolutely parallel to increasing treatment duration and temperature and the low percentage of water absorption at 150 °C is related to primary effect of treatment on chemical structure of samples that caused to reduce the interesting to water absorption by wood, while by increasing of temperature, temperature caused to some damaging in wood structure that sourced to increasing the water absorption. However the behavior of wood material in water absorption need to be investigating at another paper that it will be report soon.

Water repellency effectiveness

Water repellency effectiveness (WRE) of the samples which soaked in water during 24 hour at each one of cycles was observed as a result of the different temperatures and duration.

The result of WRE is shown for 24 hour soaking in figure 3. It was observed that WRE values commonly exhibited an increase with increasing exposure temperature for 1 hour duration of treatment. In spite of this fact, when specimens were treated at 3 and 5 hour, the WRE value was found greater for 150°C than for 160°C and 170°C. The WRE value was nearly at 7 hour for 170°C the same of the WRE value at 150°C and 160°C. Furthermore the greater WRE (22.20%) was obtained from the variation at 170°C for one hour.

Obtained results can be explained by the sorption behavior of the chemical wood components such as cellulose, lignin and their proportions in the chemical composition of the wood as well as their different thermal constancy (Kollman and Schneider, 1963).

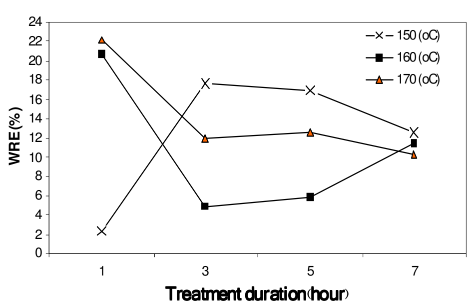


Fig 3. Variations of water absorption at all of treatment condition after 24 hour soaking/oven drying period.

Specific gravity

Table 3 shows that the effect of hydrothermal in different temperature and duration on specific gravity (SG) for beech wood as a comparatively between some conditions. As can be seen, the variation of SG loss is shown in Fig. 2. In present investigate the highest loss of specific gravity values were obtained in condition of 170°C for 7 h for 8 cycles of soaking/ oven drying (Figure 2 and Table 3). The rate of the SG loss in this condition was found as 11.02%. This loss can be related to this fact that it was recorded the thermal degradation and formation of volatile products proceed rapidly when the temperature is elevated above 200°C (Kotilainen, 2000).

As can be seen from table 3 and fig. 3; it was observed that specific gravity values generally exhibited a decreasing with increasing exposure temperature and duration compared to the control group. This result showed a parallelism with the study of Sanaee (2004). He recorded that during the heating of beech wood for 4 hour, afterward 7 soaking/oven drying cycle a loss weight began at 160 °C with 8.99% and increased to 32.54% at 180 °C for duration about 6 hour.

Commonly, the rate of the weight loss was not significantly decreasing by hydrothermal treatment at medium temperature treatment (160 °C to170 °C). According to the earlier researchers; the effects of heat treatment on different wood species were depending on the type and amount of hemicellulose (Caparros *et al.,* 2006). A hard wood like as beech, for instance showed more intense reaction than Pine (Yildiz *et al.,* 2003).

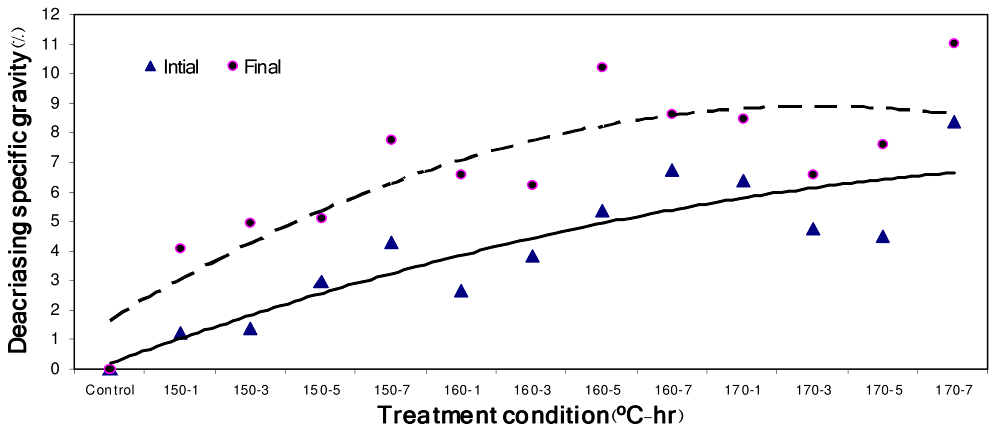
CONCLUSION

In this study the development of modified wood according to hydrothermal treatment was investigated. The study recommend that 170 °C for 1 hour was the critical point in terms of physical properties of treated specimens. Beside the changes in the physical properties of treated wood which are commonly attributed to the hydrothermal degradation of wood substance also they are accelerated by use of some soaking cycle.

It was found that the temperature of process is the most influential parameter. The ASE value of beech wood was increased by increment the exposure durations and temperatures and some soaking cycles.

Table 3. Average variation of specific gravity of specimens before and after treatment for moreover after soaking/ oven drying cycle.

specific gravity				
After final soaking/ oven drying	After initial soaking/ oven drying	After treatment	Before treatment	
0.55	0.55	0.55	0.58	150
0.53	0.54	0.55	0.58	160
0.53	0.54	0.54	0.58	170



In the other hand, the SG of treated specimens was decreased by use of great durations and temperatures. However, loss of SG was not considerable. For all studied, hydrothermal treated beech wood with acceptably high physical properties can be obtain at intermediate temperature (170°C) for short time(1 hour). In this condition, the result are: 27.95% (ASE), 20.20% (WRE), 8.49% (lose of SG), 56.41% (water absorption) compared to control specimens.

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