Influences of the hydro-thermal treatment on physical properties of beech wood (*Fagus orientalis*)

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Paper prepared for 36th Annual Meeting
Bangalore, India
24 – 28 April 2005
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Abstract

Influences of the hydro-thermal treatment on physical properties of beech wood were studied. Wood specimens (2×2×2cm) were treated in two steps. At first step, samples were treated at 160, 180 & 200°C for 4, 5 & 6 hours. At second step, treated samples were cured based on their first step treatment temperatures (160, 180 & 200°C) for 16 hours. The treated samples were soaked in water and oven dried for 24 hours. The soaking-drying cycles were repeated for seven times. Oven dried density (initial and final), swelling (initial and final), water absorption (initial and final) and density loss were analyzed. Results revealed that swelling was decreased due to the hydro-thermal treatment; while the water absorption was increased in wood. And oven dried density was slightly lost due to the treatment. The density loss and increase of water absorption are related to initial pyrolysis of wood which consequences with increase of wood porosity due to the treatment.

**Keywords:** thermal wood modification, hydro-thermal treatment, oven dried density, swelling, water absorption, Anti-Swelling Efficiency (ASE)

Introduction

Wood and lignocellulosic materials are composed of three major polymers; cellulose, hemicellulose and lignin. Their all physical, chemical and mechanical properties are influenced of them. Wood is an engineering material. In spite of its good technological properties, it has some limiting properties that can affect its applications; e.g. dimensional instability, flammability, degradation due to UV and biodeterioration.

Different techniques have been used to improve those properties; such as, wood preservation by using preservatives and pesticides, UV-absorbent paints, etc. However, many of them have environmental impacts. For this reason, their applications are being restricted in different countries due to laws and media pressures (Mohebby, 2003a). Since the last decade, scientists are looking for techniques to modify wood structure and lignocellulosic materials chemically to enhance higher properties.

Wood modification has been found as a better solution. Different techniques have been studied or used for wood modification. Heat (thermal) treatment is known as one of the oldest, easiest and cheapest methods, which improves water repellency, dimensional stability and bio-resistance in wood.

Since the last decade, different techniques have been invented for the thermal wood modification; e.g. PLATO (Tjeerdema et al., 1998, 2000; Boonstra et al., 1998).
Le Bois de Perdure (Vernois, 2001; Gohar & Guyonnet, 1998), Retification (Militz, 2002), VTT (Syrjänen & Oy, 2001; Jäsmä & Viitaniemmi, 2001) and OHT (Rapp, 2001; Rapp & Sailer, 2002; Thévenon, 2002). Some techniques have been industrialized in different countries; such as France, Germany, The Netherlands and Sweden. Annually 165000 m$^3$ of thermally treated woods are produced (Militz, 2002). According to the reports, the thermal treatment of wood improves its different properties; such as water repellency, dimensional stability, anti-swelling efficiency (ASE), UV resistance (Militz, 2002). After using different chemicals and improving dimensional stability of wood, Stamm et al. (1946) reported that achieving dimensional stability is neither due to applying heat during the treatment nor using the chemicals.

It was reported that the thermal treatment increases bio-resistance of wood against rot fungi (Kamdem et al., 2002; Welzbacher & Rapp, 2002; Mazela et al., 2003; Momohra et al., 2003).

Kamdem et al. (2002) suggested that the thermal treatment of wood decreased modulus of rupture (MOR) about 10-50%. Reports indicate that any raised treatment temperature and duration increase modulus of elasticity in beech wood (Yildiz et al., 2002; Repellin & Guyonnet, 2003).

Since, there is no report about the influences of the hydrothermal treatment on Persian beech wood; the current research has been set up to study its effects on density, swelling, water absorption of beech wood.

**Material and methods**

Wood blocks (5×5×20cm) were prepared from oriental beech wood (*Fagus orientalis* Lipsky) and cut into sizes 2×2×2cm. Samples were dried in an oven for 24h at 103±2ºC to determine dry weights before treatment. Thereafter, the treatment was carried out in two steps, hydrothermal and curing respectively.

Samples (20 replicates) for each treatment were placed in a stainless steel chamber and filled with water ($\frac{\text{Liquid}}{\text{Dry Wood}}=10$ g/g) and heated at 160, 180 and 200ºC for 4, 5 and 6 hours. After the hydrothermal treatment, they were cured in an oven for 16 hours based on their initial treatment temperatures (160, 180 and 200ºC). Oven dry weights of the treated samples and their initial dimensions were measured to determine the density loss due to the hydrothermal treatment and samples' initial dimensions before any soaking-drying cycles. Afterwards, they were dipped in water for 24h to determine anti-swelling effect (ASE) of the hydrothermal treatment. Oven weights and dimensions were measured to determine the water absorption and swelling after soaking. At next step, samples were oven dried and their dry weights and dimensions were also determined. The soaking-drying cycles were carried out for 7 times. At each step, the mentioned measurements were done to determine the ASE and density loss. ASE and swelling were calculated based on the following equations:

\[
\text{Eq. 1} \quad \text{ASE} \, (\%) = \frac{(S_2- S_1)}{S_1} \times 100
\]

Where:

- ASE (\%) = Anti-Swelling - Efficiency
- $S_1$ (\%) = Swelling Coefficient after each soaking-drying cycle
- $S_2$ (\%) = Swelling Coefficient before each soaking-drying cycle
Eq. 2\[ S (\%) = \frac{V_2 - V_1}{V_1} \times 100 \]

Where:
- $S (\%)$ = Volumetric Swelling
- $V_1 (cm^3)$ = Volume after soaking in water
- $V_2 (cm^3)$ = Volume in dry condition (before soaking)

Oven dried densities before and after the hydrothermal treatment, and also during the soaking-drying cycles, water absorption during the soaking-drying cycles, swelling and also anti-swelling efficiency during the soaking-drying cycles were measured to determine any changes which were occurred due to the repeated cycles.

**Results and discussions**

Changes of the initial and final densities (after first and seventh step of soaking-drying cycles) are shown in Fig. 1. It was revealed that density is decreased due to raised treatment temperatures and durations. Comparison between initial and final densities showed that final oven dried density was slightly lost after soaking-drying cycles in comparison with the initial density. Also Fig. 2 indicates that percentage of density loss increased due to the raised treatment conditions (temperature and time).

![Fig. 1- Initial and final swelling in hydrothermally treated beech wood](image)

As the thermal treatments were occurred at higher temperatures, it could be suggested that the increased oven dried density losses probably related to slight pyrolysis and/or deterioration of the cell wall polymers; especially hemicelluloses, which was sequenced by a mass loss in wood. Allen *et al.* (2002) studied thermo-chemical behavior of spruce (*Picea abies*) and indicated that the thermal treatment degrades hemicelluloses; while pyrolysis of cellulose and lignin is negligible.

Lower density of the treated samples after final step of soaking-drying cycle could be related to leached extractives that were formed during the hydrothermal treatment. Mayes and Oksanen (2002) reported that thermally treated Scots pine wood losses its weight after soaking-drying cycles. They suggested that leaching of the formed extractives during the thermal treatment was the main reason.
Figs. 4 & 5 show volumetric, axial, radial and tangential swelling of the treated beech after the initial and final steps of soaking-drying cycles. Results revealed that initial volumetric swelling of the samples was decreased at raised temperature. The highest initial volumetric swelling was measured in untreated samples as 17.31% and the lowest percentage of swelling was determined in treated samples (180°C for 4h) as 7.86% (Fig. 3) with an ASE of 45.4%. Also, it was revealed that the highest final volumetric swelling was determined in untreated samples as 13.29% and the lowest in treated samples at 180°C for 6h as 8.09% with an ASE of 60.87% (Fig. 4).

Fig. 5 indicates ASE in the hydrothermally treated beech wood. The results revealed that ASE was increased at raised temperature.

Shrinkage and swelling are the phenomenon, which are happened under fibre saturation point (FSP) due to moisture absorption. It sequences with dimensional
changes in wood. Above the FSP, no dimensional changes and swelling occur due to the water absorption.

Chemical structure of wood becomes altered due to the thermal treatment, which is followed by cross-linking in cell wall polymers and slight pyrolysis. It could be suggested that OH groups of the cell wall polymers are removed or cross-linked during the hydrothermal treatment and the curing step. Therefore, wood absorbs less moisture and becomes much water repellent.

![Graph showing final swelling of treated beech wood after the last step of soaking-drying cycle](image1)

**Fig. 4-** Final swelling of treated beech wood after the last step of soaking-drying cycle

![Graph showing Anti-Swelling-Effect of the hydrothermal treatment on beech wood](image2)

**Fig. 5-** Anti-Swelling-Effect of the hydrothermal treatment on beech wood

Leaching of the extractives during the soaking-drying cycles could be related to pyrolysis of the hemicelluloses that was occurred during the hydrothermal treatment and curing step. Not only removal of the hydrophilic hemicelluloses influences slightly wood mass, but also its hygroscopy. Therefore, increased ASE could also be
related to removal of the hemicelluloses and cross-linked cell wall polymers. Yildiz et al. (2002) reported an ASE of about 47.64% in the thermally treated beech wood at 180°C for 2, 4 and 10h of soaking-drying cycles. Viitanen et al. (1994) reported also an ASE between 50-80% based on applied thermal treatment.

![Fig. 6- Water absorption after initial and final steps of soaking-drying cycles](image)

![Fig. 7- Checks in hydrothermally treated beech wood; a: untreated, b: treated at 160°C, c: treated at 170°C](image)
The influence of hydrothermal treatment on moisture absorption is shown in Fig. 6. Results revealed that the moisture absorption was increased due to raised temperature. In spite of the lower ASE at higher temperatures, the water absorption was increased due to the treatment. The water absorption was also increased after soaking-drying cycles (Fig. 6). The highest absorption was determined in treated beech wood at 180°C for 4 and 6 hours (76.75% and 74.97% respectively). The lowest moisture absorption was measured in the treated samples at 160°C for 4h (31.86%). Comparison between the absorbed moisture after initial and final soaking-drying cycles indicated that untreated beech wood absorbed less moisture than the treated samples after soaking-drying cycles.

Visual analysis also showed small checks in the treated woods (Fig.7). The checks were mostly appeared in wide and multisierate rays due to their lower strengths.

The increased moisture absorption in the treated beech wood can be related to appeared checks due to the therm treatment. Wood became porous due to broken wide rays and removal of the hemicelluloses. Decreased moisture absorption in untreated beech wood is related to hysteresis that was occurred in wood after soaking-drying cycles.

It was observed that the treated samples at 200°C were deteriorated during curing step because of highly applied temperature. Therefore, they were removed from this experiment.

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