Long-Term Hygroscopic Thickness Swelling Rate of Hydrothermally Treated Beech Wood / Polypropylene Composites

ABSTRACT • Long-term hygroscopic thickness swelling rate of polypropylene (PP) composites filled with thermally treated wood flour was investigated. The beech wood chips were heat treated at 120 °C, 150 °C or 180 °C for 30 or 120 min using saturated steam in a digester. The composites based on PP, beech wood flour (BF), and coupling agents (PP-g-MA) were made by melt compounding and injection molding. The weight ratio of BF to PP was controlled at 50/47 for all blends. The amount of coupling agent was fixed at 3 wt.% for all formulations. Further study was conducted to model thickness swelling of the composites, a swelling rate parameter ($K_{SR}$). The thickness swelling of thermally-treated samples at 120 ºC for 30 min and at 150 ºC for 30 min were lower than that of control samples, followed by thermally-treated samples at 180 ºC for 120 min, at 180 ºC for 30 min, at 120 ºC for 120 min, and at 150 ºC for 120 min, respectively. Furthermore, the thickness swelling of the BF/PP composites decreased with increasing time and temperature of the thermal-treatment. In addition, at 120 ºC for 30 min, the composites showed a lower swelling rate than control samples. The $K_{SR}$ of the composites was influenced both by the time and temperature of thermal treatment.

KEYWORDS: thickness swelling rate; thermal-treatment; lignocellulusic filler; polypropylene
Thermal treatment has been used by many researchers to improve dimensional stability of wood and wood-based composites (Kazemi Najafi et al., 2007; Kaboorani et al., 2008). It decreases the water absorption of wood by the crystallization of cellulose and extraction of hemicelluloses from wood (Wallenberger and Weston, 2004; Yildiz and Gümüşkaya, 2007; Hosseinihashemi et al., 2016). The enhancement of the dimensional stability, reduction of the swelling, and alteration of the chemical composition of wood have been found in thermally modified wood (Tjeerdsma et al., 2000; Militz and Tjeerdsma, 2001; Yildiz et al., 2004; Temiz et al., 2006; Rezayati Charani et al., 2007; Koubaa et al., 2011; Hadi et al., 2016).

Under high-temperature conditions, a series of complex chemical reactions takes place in the wood cell wall, such as degradation and condensation reactions (Yin et al., 2010). This leads to changes in the amount of the components of wood and also in its physical and chemical properties. After the heat-treatment process, the dimensional stability and the durability of wood increase, which is strongly associated with the reduction in hygroscopicity (Bekhta and Niemz, 2003; Cao et al., 2012; Olarescu et al., 2014).

Amorphous region of cellulose by heating of wood at high temperature results in an increase in the degree of crystallinity of this polymer. A cross-linkage between the lignin and the polymers occurs because of the thermal degradation of wood, which is responsible for the decrease in the hygroscopicity of wood and the improvement of its dimensional stability (Jämsä and Viitanen, 2001; Waskett and Selmes, 2001; Bekhta and Niemz, 2003; Wikberg and Maunu, 2004; Metsä-Kortelainen et al., 2006; Calonеno et al., 2010).

The improvement in the hygroscopic and micro-mechanical properties of heat-treated wood occurred with an elevation in the steam temperature, which correlated well with this pattern of degradation in the constituents of the bio-composite matrix in the cell wall (Yin et al., 2010). Also, the improvement in the dimensional stability of thermally treated wood could be related to a reduction in the number of free hydroxyl groups with chemical reactions (Dale Ellis, 1994; Zhang et al., 2006; Deka and Saikia, 2000). This could also be related to the chemical modification in the cell wall of the fiber during the hydrothermal treatment (Yildiz et al., 2004; Hadi et al., 2016; Rowell and LeVan-Green, 2005). Hemicelluloses degraded by thermal treatment positively affect the dimensional stability of wood (Garrote et al., 1999; Tjeerdsma and Militz, 2005). In addition, the reduction of the thickness swelling could be related to the increase of the crystalline regions in the cellulose microfibrils (Wallenberger and Weston, 2004; Yildiz and Gümüşkaya, 2007). Previous studies reported that the swelling loss could occur as a result of esterification of the cellulose microfibrils (Tjeerdsma and Militz, 2005; Boonstra and Tjeerdsma, 2006). In general, the degradation of wood components and particularly of hemicelluloses negatively affects the mechanical properties of wood (Yildiz et al., 2006; Korkut et al., 2008). Although the effect of heat treatment of wood on thickness swelling of wood plastic composites were investigated by previous studies (Ayrilmis et al., 2011; Tufet et al., 2016), the long term thickness swelling has not been extensively investigated. In the present study, the effect of thermal-treatment of beech wood on the long-term thickness swelling of the thermoplastic composites was investigated.

2 MATERIALS AND METHODS

2.1 Materials

The polymer matrix comprised of V30S polypropylene (PP), with a melt flow index of 16 g/10 min and a density of 0.87 g/cm³, was supplied by Marun Petrochemical Co. (Mahshahr, Iran). The lignocellulosic material used as the reinforcing filler in the composites was beech (Fagus orientalis L.) wood flour, which was ground by a grinder. A maleic anhydride grafted polypropylene (MAPP) as a coupling agent, which was PPG101, was provided by Kimia Javid Sepahan Co. (Tehran, Iran), with a melt flow index of 64 g/10 min, and a density of 0.91 g/cm³. The amount of the MAPP in all the specimen groups was 3 wt.%.
2.2 Thermal-treatment of wood chips

Before the preparation of the composites, beech logs were chipped by a drum-type chipper. Prior to the heat treatment, the wood chips were dried at room temperature for 24 h. After cooling in a desiccator containing silica gel powder, the wood chips were heat treated for 30 or 120 min at different temperatures (120 °C, 150 °C, or 180 °C) using saturated steam in a digester. Then beech wood flour (BWF) was prepared from the treated chips using laboratory type grinder. The wood flour was dried until 0 to 1 % moisture content in an oven at (103±2) °C for 24 h. Polypropylene, beech wood flour, and the coupling agent were then weighed and bagged according to the formulations given in Table 1.

2.3 Preparation and testing of WPCs

The mixing of raw materials was carried out with a counter-rotating intermeshing twin-screw extruder (Model T20, 1990, Dr. Collin GmbH, Germany), with its barrel temperature ranging from 180 °C at six zones, from feeding zone to the die zone, at a screw speed of 60 rpm for 14 min. The pasty compound produced was cooled to room temperature and then grinded to produce suitable granules for further processing. Grinding was carried out in a laboratory mill (Wieser, WGLS 200/200 Model, Germany) and the granulated materials were dried at 105 °C for 4 h. Test specimens were prepared by injection molding machine (Model EM80, Aslanian Co., Iran) set at a temperature ranging from 160 to 180 °C. A complete set of specimens for different tests were produced for each molding operation. Finally, the specimens were conditioned at a temperature of 23 °C and relative humidity of 50 % for at least 40 h, according to ASTM D 618-99 prior to testing. The water absorption (WA) was determined according to ASTM D 570 standard. The water absorption of WPC specimens with nominal dimensions of 5 mm x 11 mm x 80 mm was determined after 2, 4, 6, 8, 10, 12, 24, 48, 72, 168, 336, 504, 720, and 1440 h immersion in distilled water at room temperature. Three specimens of each type of WPC were dried in an oven for 24 h at (103±2) °C. The dried specimens were weighed with a precision of 0.001 g and then they were placed in distilled water. At the end of immersion periods, the specimens were removed from the distilled water and the surface water was wiped off using blotting paper. Weight of the specimens was measured at different time intervals during the long-time immersion. The measurements were terminated after the equilibrium weights of the specimens were reached. The values of the water absorption and thickness swelling in percentage were calculated using Eq. 1 and 2:

\[ WA(t) = \frac{W(t) - W(o)}{W(o)} \times 100 \]  

(1)

Where, \( WA(t) \) is the water absorption at time \( t \), \( W(o) \) is the initial weight of specimens, and \( W(t) \) is the weight of specimens at time \( t \) (Equation 1).

\[ TS(t) = \frac{T(t) - T(o)}{T(o)} \times 100 \]  

(2)

Where, \( TS(t) \) is the thickness swelling at time \( t \), \( T(o) \) is the initial thickness of specimens, and \( T(t) \) is the thickness of specimens at time \( t \) (Eq. 2).

Further study was conducted to model long-term thickness swelling behavior of the composites. The swelling rate parameters in the model were obtained by fitting the model predictions with the experimental

| WPC code | Treatment type | Beech wood flour, wt.% | Polypropylene (PP), wt.% | MAPP*, wt.% |
|----------|----------------|------------------------|--------------------------|-------------|
| Oznaka WPC-a | Vrsta tretmana | Drveno brašno od bukovine, wt.% | Polipropilen (PP), wt.% |         |
| A        | WPC-30 min-120 °C | 50                     | 47                       | 3           |
| B        | WPC-30 min-150 °C | 50                     | 47                       | 3           |
| C        | WPC-30 min-180 °C | 50                     | 47                       | 3           |
| D        | WPC-120 min-120 °C | 50                     | 47                       | 3           |
| E        | WPC-120 min-150 °C | 50                     | 47                       | 3           |
| F        | WPC-120 min-180 °C | 50                     | 47                       | 3           |
| G        | WPC-control       | 50                     | 47                       | 3           |

*MAPP – maleic anhydride grafted polypropylene / polipropilen grafiiran anhidridom maleinske kiseline
data. Shi and Gardner (2006) studied to quantify the thickness swelling rate of WPCs for more convenient comparisons. They developed a swelling model describing the hygroscopic swelling process of wood based composites. In this model, a swelling rate parameter \((K_{SR})\), as determined using the test data, can be used to quantify the swelling rate. The swelling model is described by the following Eq. 3:

\[
T(t) = \frac{T_e}{1 + \left(\frac{T_e}{T_i} - 1\right)e^{-K_{SR}t}}
\]

Where, \(T(t)\) is the thickness swelling at time \(t\). \(T_e\) and \(T_i\) are the initial and equilibrium board thickness, respectively. \(K_{SR}\) is a constant referred to as the initial (or intrinsic) relative swelling rate.

The values of \(K_{SR}\) in Eq. 3 depend on how fast the composites swell and also on their equilibrium thickness swelling. Non-linear curve fitting was used to find the swelling rate parameter \((K_{SR})\) that provided the best fit between the equation and the experimental data. This algorithm seeks the parameter values that minimize the sum of the squared differences between the observed and predicted values of the dependent variable as seen in Eq. 4,

\[
SS = \sum_{i=1}^{n} (y_i - \overline{y})^2
\]

Where, \(SS\) is the sum of squared difference and \(y_i\) and \(\overline{y}\) are the observed and predicted values of the dependent variable, respectively.

### 2.6 FT-IR analysis

**2.6. FT-IR analiza**

FT-IR measurements were carried out in an Equinox instrument (Bruket Co., Germany) by direct transmittance using KBr pellet technique. Each spectrum was recorded at a rate of 10 scans, in the range from 3500 to 800 cm\(^{-1}\) with a resolution of 4 cm\(^{-1}\).

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**Figure 1** Effect of thermal-treatment severity on long-term water absorption of WPCs

**Figure 1.** Utjecaj jačine toplinskog tretmana na dugotrajno upijanje vode WPC-a
**Figure 2** Effect of thermal-treatment severity on long-term thickness swelling of WPCs
Slika 2. Utjecaj jačine toplinskog tretmana na dugotrajno debljinsko bubrenje WPC-a

**Figure 3** Independent effect of thermal-treatment times on long-term thickness swelling of WPCs
Slika 3. Neovisni učinak trajanja toplinskog tretmana na dugotrajno debljinsko bubrenje WPC-a

**Figure 4** Independent effect of thermal-treatment temperatures on long-term thickness swelling of WPCs
Slika 4. Neovisni učinak temperature toplinskog tretmana na dugotrajno debljinsko bubrenje WPC-a

\[ y = -2E-0.6x^2 + 0.006x + 0.40 \quad R^2 = 0.99 \]

\[ y = -2E-0.6x^2 + 0.005x + 0.36 \quad R^2 = 0.99 \]

\[ y = -2E-0.6x^2 + 0.0056x + 0.36 \quad R^2 = 0.998 \]

\[ y = -1E-06x^2 + 0.0046x + 0.46 \quad R^2 = 0.997 \]

\[ y = -2E-06x^2 + 0.0058x + 0.33 \quad R^2 = 0.998 \]
Hosseinihashemi, Arwinfar, Najafi, Özdemir, Ayrilmis, Tamjidi: Long-Term Hygroscopic Thickness Swelling Rate...

maximum value of $K_{SR}$ with beech wood flour treated at 120 °C for 30 min. The thickness swelling is given in Table 3.

The correlation between predicted and experimental value determined in the wood flour filled composites. This can be explained by very high $K_{SR}$ness for thermally treated beech wood flour/PP composites (Shi and Gardner, 2006; Kord, 2013).

swelling but also on the equilibrium thickness swelling equilibrated. It is dependent not only on the initial rate of swelling but also on the equilibrium thickness swelling of the composites (Shi and Gardner, 2006; Kord, 2013).

The swelling rate parameter ($K_{SR}$) and maximum values of thickness swelling of the composites are given in Table 2.

The composites produced by a 120 min / 150 °C treatment had the highest thickness swelling. The minimum $K_{SR}$ was calculated for the composites produced with beech wood flour treated at 120 °C for 30 min. The maximum value of $K_{SR}$ was found in the composites produced with wood flour at 150 °C for 30 min. It is important to note that in the swelling model $K_{SR}$ was obtained considering the whole thickness process until it was equilibrated. It is dependent not only on the initial rate of swelling but also on the equilibrium thickness swelling of the composites (Shi and Gardner, 2006; Kord, 2013). Less time was required to reach the equilibrium thickness for thermally treated beech wood flour/PP composites (Figure 2). This can be explained by very high $K_{SR}$ value determined in the wood flour filled composites. The correlation between predicted and experimental thickness swelling is given in Table 3.

Table 2 Swelling rate parameters for studied composites

| WPC code / Oznaka WPC-a | Maximum thickness swelling, % | Maximum value of $K_{SR}$, $10^{-3}$, h$^{-1}$ | Sum of squared |
|-------------------------|-------------------------------|---------------------------------------------|---------------|
| A                       | 3.35                          | 0.0023                                     | 2.51          |
| B                       | 3.99                          | 0.0018                                     | 0.27          |
| C                       | 4.70                          | 0.0021                                     | 1.74          |
| D                       | 4.88                          | 0.0019                                     | 0.99          |
| E                       | 5.23                          | 0.0021                                     | 2.08          |
| F                       | 4.27                          | 0.0021                                     | 0.44          |
| G                       | 4.15                          | 0.0020                                     | 0.73          |

* R – squared / $R – kvadrat

Table 4 Relationship between long-term water absorption ($WA$) and thickness swelling ($TS$) in studied composites

| WPC code / Oznaka WPC-a | Equation | $R^2$ |
|-------------------------|----------|-------|
| A                       | $TS = 0.30 WA + 0.02$ | 0.92  |
| B                       | $TS = 0.42 WA - 0.63$ | 0.92  |
| C                       | $TS = 0.47 WA - 0.32$ | 0.93  |
| D                       | $TS = 0.53 WA - 0.56$ | 0.90  |
| E                       | $TS = 0.57 WA - 0.34$ | 0.95  |
| F                       | $TS = 1.05 WA - 0.56$ | 0.96  |
| G                       | $TS = 0.46 WA - 0.25$ | 0.98  |

* R – squared / $R – kvadrat

Figures 5 and 6 indicate fitting predicted thickness swelling with the experimental data obtained from thermally treated beech wood flour/PP composites for calculating the swelling rate.

The relationship between long-term water absorption and thickness swelling in the composites is given in Table 4. Thickness swelling is a response to absorbed water in the composites. The $R$-squared values of all the composites were found to be 0.90 (Table 4).

3.2 FT-IR analysis

FT-IR spectroscopy is a simple technique applied to determine the effect of various applications used in order to obtain information about the structure of wood components causing changes in the chemical structure of wood. It is preferred because it needs only a small sample size and short analysis time for test application, as well as because it does not disrupt wood structure. Due to their complex nature, spectra are considered as two regions for examination. The first region is expressed as 2700-4000 cm$^{-1}$ band where the OH and C-H stretching vibrations are included, while the second region is defined as the “Fingerprint” region between at 1100-1800 cm$^{-1}$ where different vibration extension regions of wood components are identified.

Band assignment of wood material in the 4000-800 cm$^{-1}$ region is presented in Table 5 (Li et al., 2015). FT-IR spectroscopy of the test specimens treated at 120

Table 3 Correlation between predicted and experimental thickness swelling

| WPC code / Oznaka WPC-a | $R^2$ |
|-------------------------|-------|
| A                       | 0.98  |
| B                       | 0.99  |
| C                       | 0.99  |
| D                       | 0.99  |
| E                       | 0.99  |
| F                       | 1.00  |
| G                       | 0.99  |

* R – squared / $R – kvadrat

Band assignment of wood material in the 4000-800 cm$^{-1}$ region is presented in Table 5 (Li et al., 2015). FT-IR spectroscopy of the test specimens treated at 120
and 180 °C with control samples was recorded. As shown in Figure 7, the peak intensity of the OH stretches at about 3400 cm$^{-1}$ in test specimens exposed to temperatures of 180 °C and 120 °C reduced as compared to the control sample. The C-H deformations in lignin and carbohydrates were observed around 1462 cm$^{-1}$. When the exposure time of the test specimens at 180 °C increased, aromatic and aliphatic C-H (methylene groups) stretches were determined between 2800 and 2920 cm$^{-1}$. Similarly, the C=O carboxyl groups were observed between 1731 and 1737 cm$^{-1}$. In 1596 cm$^{-1}$ C=C aromatic skeletal vibration (lignin) peak value, peak intensity showed an increase due to the temperature increase. The intensity of the C-O stretching peak of lignin at 1250 cm$^{-1}$ decreased with the effect of temperature. The C-O stretching vibration in cellulose and hemicelluloses formed at 1054 cm$^{-1}$ was slightly degraded. The cellulose and hemicellulose C-H deformations and C-O-C stretches formed at 1377 cm$^{-1}$ and 1169 cm$^{-1}$, respectively.

As a result, the IR peaks were not affected by the heat treatment temperature. However, the intensities of the functional groups, in particular for –OH stretches, decreased at 180 °C as compared to the control samples at lower temperature (150 °C and 180 °C). Thus, it can be said that some functional groups such as –OH or...
carboxylic acid C=O groups reacted with each other at 180 °C and formed new ester or etheric groups in addition to –OH or C=O stretches.

### 3.3 Morphological analysis

3.3. Morfološka analiza

The morphological analysis of the composites is presented in Figure 8. The SEM images revealed that there was some distortion and modification of the cell walls of wood due to the hydro-thermal treatment. The cracks in the cell walls of wood increased with increasing the treatment temperature and time. The melted PP polymer filled the cracks in the cell walls of wood based on the SEM images, thus decreasing the thickness swelling.

#### Table 5

| Wavenumber, cm⁻¹ | Band assignment |
|------------------|----------------|
| 3399             | O-H stretching in hydroxyl groups |
| 2921             | C-H asymmetric stretching in methylene groups |
| 1736             | C=O stretching vibration of carboxyl, carboxyl and acetyl groups |
| 1659             | Conjugated C-O in quinines coupled with C=O stretching of various groups |
| 1594             | C=C stretching of aromatic skeletal in lignin |
| 1508             | C=C stretching of aromatic skeletal in lignin |
| 1463             | C-H deformation in lignin and carbohydrates |
| 1423             | C-H deformation in lignin and carbohydrates |
| 1374             | C-H deformation in cellulose and hemicellulose |
| 1328             | C-H vibration in cellulose and C-O vibration in syringyl derivatives-condensed structures in lignin |
| 1266             | C-O stretching in lignin |
| 1234             | C-O stretching vibration of Ph-O-C coupled with aromatic ring vibration in lignin and C-O stretching vibration in xyloglucan |
| 1157             | C-O-C stretching vibration in cellulose and hemicelluloses |
| 1055             | C-O stretching vibrations in cellulose and hemicelluloses |
| 1034             | C-O ester stretching vibrations in methoxyl and β-O-4 linkages in lignin |
| 897              | Character of cellulose P-chains, C-H stretching out of plane of aromatic ring |

#### Figure 7

FT-IR spectrum of heat treated and untreated test samples (A, B, C, D, E, F and G according to Table 2)

**Table 5** Band assignment of wood samples in 4000-800 cm⁻¹ region (Li et al., 2015)

**Tablica 5.** Vrpce pripisane uzorcima drva u području 4000 – 800 cm⁻¹ (Li et al., 2015.)

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### 4 CONCLUSIONS

4. ZAKLJUČAK

The composites produced with thermally treated beech wood flour swelled and gained weight very slowly. The thickness swelling of the composites decreased with increasing time and temperature of the thermal-treatment. In addition, at 120 ºC for 30 min, the composites showed a lower swelling rate than control samples. The $K_{SW}$ of the composites was influenced by both the time of thermal treatment and temperature. A strong correlation was found between the long-term water absorption and thickness swelling in the composites. Based on the findings obtained from the present study, it can be said that thermal treatment of the
wood chips at 150 °C for 30 min result in optimal parameters for the wood flour reinforced polypropylene composites, as they have higher water resistance than other treatment groups.

Acknowledgements – Zahvala

The authors are grateful for the support of the Department of Wood Science and Paper Technology, Karaj Branch, Islamic Azad University.

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