Effect of High-pressure Densification on Moisture Sorption Properties of *Paulownia* Wood

Yong Yu,[a,b] Xiuping Jiang,[a,b] Hosahalli S. Ramaswamy,[c] Songming Zhu,[a,b] and Huanhuan Li [a,b]*

The effect of high-pressure (HP) densification (30, 90, and 150 MPa for 3, 30, and 300 s) on the moisture sorption properties of *Paulownia* wood was investigated. After the densification, samples were conditioned at three temperatures (20, 30, and 40 °C) and five equilibrium moisture contents (from 11.20 to 95.62%) during the study, after which the equilibrium moisture contents of the control and treated samples were measured. The HP-treated groups had higher equilibrium moisture contents than the controls at higher relative humidity levels. The hysteresis phenomenon and the scanning electron microscopy observations were explained by the transformation of the structural elements by the HP treatment. Finally, two moisture sorption isotherm (MSI) models (a linear polynomial model for adsorption and a quadratic polynomial model for desorption) were established with good performance to describe the relationship between HP treatment parameters, environmental conditions, and equilibrium moisture contents.

Keywords: High-pressure treatment; Paulownia wood; Equilibrium moisture content; Moisture sorption

*Contact information: a: College of Biosystems Engineering and Food Science, Zhejiang University, 866 Yuhangtang Road, Hangzhou 310058, China; b: Key Laboratory of Equipment and Information in Environment Controlled Agriculture, Ministry of Agriculture, 866 Yuhangtang Road, Hangzhou, 310058, China; c: Department of Food Science and Agricultural Chemistry, McGill University, St-Anne-de-Bellevue, Quebec, Canada; *Corresponding author: huanhuanlee@zju.edu.cn*

INTRODUCTION

Paulownia elongata, commonly known as *Paulownia* (or empress tree) and belonging to the family Paulowniaceae, is a popular hardwood tree species native to China and widely distributed throughout North America, Australia, Europe, and Japan (Ayrilmis and Kaymakci 2013). *Paulownia* is highly adaptable to different growing conditions and has a very high growth rate under favorable conditions. Compared with other traditional hardwoods, it is the fastest growing species and grows 15 feet or more in the first year (Kaygin *et al.* 2009). It can be harvested for producing usable wood products within 4 to 7 years; other hardwoods take decades to mature (Ayrilmis and Kaymakci 2013).

Other advantages that make the *Paulownia* tree more popular in the world market include its short rotation cycle, high survival rate, high disease and rot resistance, good dimensional stability, very low thermal conductivity, very high ignition point, and knot-free appearance with a satiny luster (Ates *et al.* 2008). However, compared with other traditional woods, *Paulownia* is much softer, lighter, and easily abraded, and has a low density and low dimensional stability that considerably limits its commercial utilization.

To overcome these problems, several studies have focused on different wood processing technologies to improve the wood quality, especially density, and commercial value of *Paulownia* (Saipnami *et al.* 2007; Rautkari *et al.* 2008; Kutnar and Kamke 2012; Gao *et al.* 2016; Hosseinpourpia *et al.* 2016; Laine *et al.* 2016; Sun *et al.* 2016). Among these, high pressure (HP) processing, a non-thermal processing technology, is one of the more effective and environmental friendly technologies that are widely used in the food industry (Yu *et al.* 2016; Sun *et al.* 2016; Zhu *et al.* 2016).

High-pressure (HP) processing for the densification results in a significant improvement in wood microstructure, density, mechanical strength, and other wood quality properties (Li *et al.* 2016; Sun *et al.* 2016). Regardless of the size, shape, and composition, the pressure applied in HP treatments is instantaneous and uniformly transmitted throughout the wood. HP is an energy-efficient process because it requires no additional energy once the desired pressure is reached (Balasubramaniam *et al.* 2015). It also extends the shelf life of the wood by killing several pathogenic microorganisms present, especially discoloration-causing fungi, thereby increasing the cost efficiency of using this technology.

Changes in the morphological properties of wood generally result from initial changes to its anatomical structure, which can be easily affected by the moisture content and the form in which it exists (free water, hygroscopic water, and bound water) in the wood. Siau *et al.* (1984) reported that moisture content had a significant effect on the physical and chemical properties of wood. Once the absorbed water is elevated beyond the bound water stage, wood undergoes swelling, which causes dimensional instability, decreases the density and mechanical strengths, and increases the thermal and electrical conductivities (Passarini *et al.* 2014). Being somewhat hygroscopic, the compressed or densified wood exchanges water molecules with the surrounding environment in a dynamic state, reaching an equilibrium moisture content (EMC) at any given relative humidity (RH) condition (Zhou *et al.* 2013). EMC is affected by temperature, relative humidity, species, and wood properties, and the moisture/RH relationships at a given temperature can be described using a moisture sorption isotherm (MSI).

HP treatment has been successfully used in wood processing for densification purposes. However, little is known about the wood/water relationship in HP-treated *Paulownia* or the resulting morphological changes. Characterization of the EMC of *Paulownia* at different relative humidities, *i.e.*, evaluation of the MSI, would help to provide insight into the wood/water relationship and how processing technology affects wood quality. The purpose of this study was therefore to evaluate and model the MSI behavior of HP-treated *Paulownia* wood and to establish the relationship between HP treatment variables and MSI.

EXPERIMENTAL

Sample Preparation

P. elongata logs with diameters of 50 ± 5 mm were harvested from a plantation forest located in Xingyang, Zhengzhou, Henan Province, China. Wood logs were cut into boards after peeling, and then equilibrated in an incubator at 20 ± 1 °C and 60 ± 2% relative humidity to reach an equilibrium moisture content (EMC) of 10% (Kartal *et al.* 2007). After establishing the EMC, the boards were cut into smaller boards (8 mm x 20 mm) from which thirty sapwood boards were selected. Twenty-seven of the boards were set aside as test specimens to be subjected to HP-treatments at different pressure levels (30, 90, and 150 MPa) levels and holding times (3, 30, and 300 s). Levels of pressure and holding time were selected based on results from the preliminary experiments (data not shown). The treatment conditions were 30 MPa and 3 s, 30 MPa and 30 s, 30 MPa and 300 s, 90 MPa and 3 s, 90 MPa and 30 s, 90 MPa and 300 s, 150 MPa and 3 s, 150 MPa and 30 s, and 150 MPa and 300 s, and three boards were used for each treatment. Before being subjected to the HP treatments, the nine test groups were vacuum-packaged with polythene bags and clamped to both tangential surfaces by iron plates with rubber bands. A similar packaging method was also described in a previous study (Yu *et al.* 2017). The remaining three boards were left untreated as a control (0.1 MPa and 0 s).

The HP equipment used for giving the treatment was a high-pressure processor (UHPF-750, Kefa, Baotou, China) with a maximum chamber capacity of 5 L. The HP unit was connected to a data logger for recoding temperature and pressure during HP treatment. Purified water was used as the pressure-transmitting medium. The pressure vessel was maintained at room temperature (~20 °C) (Yu *et al.* 2016).

Using a freshly sharpened knife, all treated boards and the control were cut into cubes measuring 8 mm x 8 mm x 8 mm. Each group had five replicates. All samples were carefully selected to be free of knots or visible defects. Prior to the MSI experiment, all specimens were numbered for easy identification. The average initial moisture content was determined by oven-drying at 103 °C, and weights were recorded periodically until a constant value was reached, in accordance with ISO 3130 (1975).

Equilibrium Moisture Content

The sorption experiments were carried out at three different temperatures and in five different relative humidity environments. Five salts were selected to provide relative humidities ranging from 11% to 94% (saturated solutions of LiCl, $MgCl_2$, NaBr, NaCl, and KNO_3). The corresponding ERHs at different temperatures are shown in Table 1. Five hermetic jars (500 mL) with rubber plugs were used in the experiment for achieving ERH/EMC conditions. Each jar was filled 1/3 with a saturated inorganic salt solution with excess salt. A welded-wire mesh basket was suspended from the middle of the rubber plug above the saturated salt solution. A schematic diagram of the apparatus is shown in Fig. 1. EMC values were determined at the different constant ERH conditions and temperatures (20, 30, and 40 °C). Within this setup, the samples with high moisture content would lose moisture, and those with low moisture content would gain moisture.

During the adsorption and desorption experiments, thirty wood samples representing the different treatment conditions (30 MPa and 3 s, 30 MPa and 30 s, 30 MPa and 300 s, 90 MPa and 3 s, 90 MPa and 30 s, 90 MPa and 300 s, 150 MPa and 3 s, 150 MPa and 30 s, and 150 MPa and 300 s) and the control (0.1 MPa and 0 s) and three replicates were placed each in a hermetic bottle and distributed into the three temperature controlled incubators maintained at 20, 30 or 40 °C. These ovens provided a temperature control within ±1 °C during the extended periods employed for moisture equilibration, thus promising precise MSI data.

The adsorption test was started from the lowest RH (11%) and proceeded to the highest (94%). The samples were weighed periodically until a constant mass was attained. After the samples in the lowest RH had equilibrated, the saturated salt solutions in each bottle were changed to a higher RH. The adsorption experiments were accomplished when the RH in each bottle was changed from the lowest to highest (with the sequence of salts LiCl, $MgCl_2$, NaBr, NaCl, and KNO_3).

The samples were finally suspended in a hermetic bottle filled with distilled water (RH ~100%) to achieve the maximum EMC. During the desorption experiments, the samples were also placed in hermetic bottles (Fig. 1) with the same saturated inorganic salt solutions, but the sequence moved from the highest RH to the lowest this time. Finally, the sorption isotherm curves, including the adsorption and desorption of wood samples with different HP-treatments at different temperatures, were obtained.

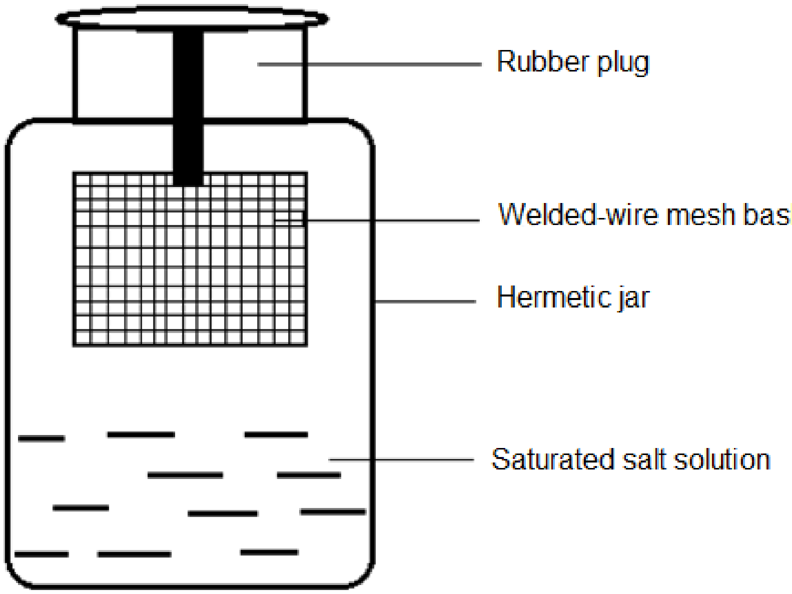

Fig. 1. Schematic diagram of the apparatus for the EMC experiment: a rubber plug, a welded-wire mesh basket, a hermetic jar, and saturate salt solution

Table 1. Corresponding Equilibrium RHs Provided over the Saturated Inorganic Salt Solutions at Three Different Temperatures

Saturated Salt Solution	20 °C	30 °C	40 °C
LiCl	11.31	11.28	11.20
$MgCl_2$	33.07	32.44	31.60
NaBr	59.14	56.03	53.17
NaCl	75.47	75.09	74.70
KNO_3	94.62	92.31	89.03

Scanning Electron Microscopy

Scanning electron microscopy (SEM) was employed to study the microstructure of HP-treated *Paulownia* wood samples. Samples from the control and test groups were observed before and after the adsorption experiment to study the effect of HP treatment on the microstructural properties of the wood (Li *et al.* 2016). Before the observations were made, each wood cube was cut into 1 mm-thick slices in the transverse direction (Ling *et al.* 2015). The wood slices were then attached to a specimen holder using a silver plate and coated with a thin film of gold (10 nm) in a vacuum evaporator. The obtained specimens were viewed in a scanning electron microscope (SEM; XL30-ESEM Philips. Holland) at the accelerating voltage of 10 kV.

Statistical Analysis

Linear and quadratic polynomial equations were established to describe the relationship between the EMC, RH, temperature, and HP treatment parameters (holding times and applied pressures) as follows,

$$Xe = \mathrm{a}x + \mathrm{b}y + \mathrm{c}z + \mathrm{d}t + \mathrm{p}$$

$$Xe = \mathrm{a}x + \mathrm{b}y + \mathrm{c}z + \mathrm{d}t + \mathrm{e}x2 + \mathrm{f}y2 + \mathrm{g}z2 + \mathrm{h}t2 + \mathrm{i}xy + \mathrm{j}xz + kxt + lyz + myt + nzt + q$$

where Xe was EMC obtained from different conditions; x, y, z, t represented HP treatment duration, pressure level, temperature, and relative humidity, respectively; and a, b, c, d, e, f, g, h, I, j, k, l, m, n, and q were estimated constants. To evaluate the correlation of each model to fit the data, the mean relative percent error (*MRE*), the standard error (*SE*), and the coefficient of determination (r^2) between the experimental and predicted data were calculated using the following equations,

$$MRE\ (\%) = \frac{100}{N}\sum_{i=1}^{N}\left|\frac{Mi,exp - Mi,pre}{Mi,exp}\right| \tag{1}$$

$$S.E. = \sqrt{\frac{1}{N}\sum_{i=1}^{N}(Mi,exp - Mi,pre)^2} \tag{2}$$

$$r^2 = 1 - \frac{\sum_{i=1}^{N}(Mi,exp - Mi,pre)^2}{\sum_{i=1}^{N}(Mi,exp - \bar{M}exp)^2} \tag{3}$$

where Mi,exp and Mi,pre were the i^{th} experimental and predicted equilibrium moisture contents (%), $\bar{M}exp$ was the average experimental equilibrium moisture content (%), and N was the total number of data points included in the regression. Generally, the best-fit models were determined to have high r^2 values and low *MRE* and *SE* values (Bahloul *et al.* 2008; Yu *et al.* 2016).

RESULTS AND DISCUSSION

Effect of High-pressure Treatment

The experimental EMC *vs*. ERH data for HP-treated and control wood samples at 20 °C are shown in Fig. 2, and similar trends were observed at 30 °C and 40 °C. The EMCs increased with increasing ERH. At each same ERH, the EMC values for the test group were different. For example, at an ERH of 11.3% and 94.6%, the HP-treated samples had higher EMC values than the control, while from 33.1% to 75.5% the control sample had comparatively higher EMC values than did the HP-treated groups. This may have been due to the existence of internal stress in the samples caused by the HP treatment. Similar results have been reported in some densified woods by other researchers. Wood is a material of porous, anisotropic, and inhomogeneous structure, and whether before or after HP treatment (Zauer *et al.* 2016), wood samples exposed to a high-humidity external environment would experience a high driving force of water vapor from the internal pressure deficit, resulting in the infusion of moisture and consequent swelling of the wood, depending on the level and nature of the compaction. Li *et al.* (2016) reported that the residual stress generated during the thermo-compression was released after the moisture absorption and equilibration. When the samples were placed in a relatively low-RH environment, the original stable state was generally maintained because the water vapor pressure deficit was small and insufficient to drive the moisture in. This result signified the impact of relative humidity on the moisture absorption of wood samples. In terms of pressure treatment, HP-treated sample had a comparatively lower EMC in most experimental cases. However, the differences among the treated samples were not significant, which turned out to be a useful result for energy-efficient development patterns for the future large-scale manufacturing of HP compacted wood products. In addition, at RH below 75%, the EMCs of all HP treated boards were all below 12%, which signified a stable condition, especially for use as construction materials for indoor use applications.

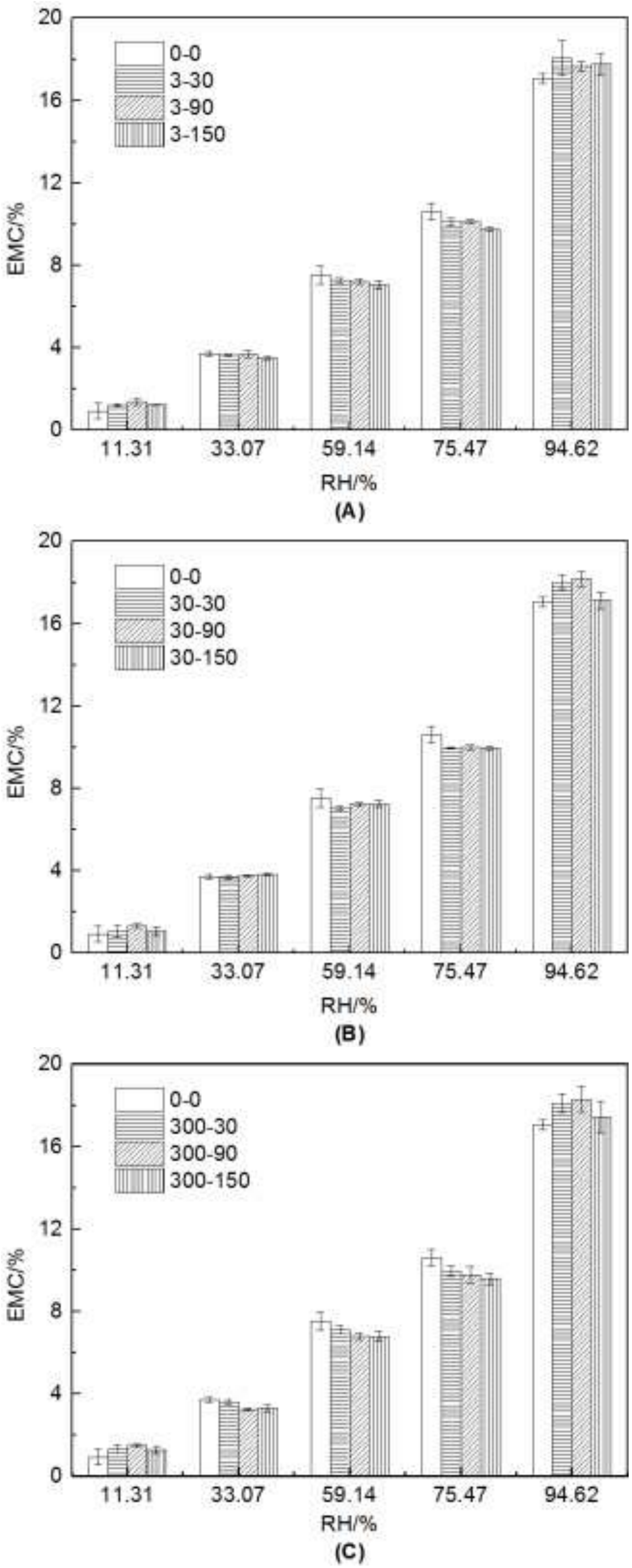

Fig. 2. EMC *vs*. ERH data for HP-treated and control wood samples at 20°C

Effect of Temperature

The sorption isotherms are essential for selecting the optimal processing and storage conditions in the wood industry. The experimental moisture adsorption isotherms of control and HP-treated wood samples (30 s and 30 MPa, 30 s and 90 MPa, and 30 s and 150 MPa) at 20, 30, and 40 °C are presented in Fig. 3 (similar results not shown). In general, from the curve trend in the figures, both the temperature and HP treatment had a qualitatively similar effect on the equilibrium moisture content.

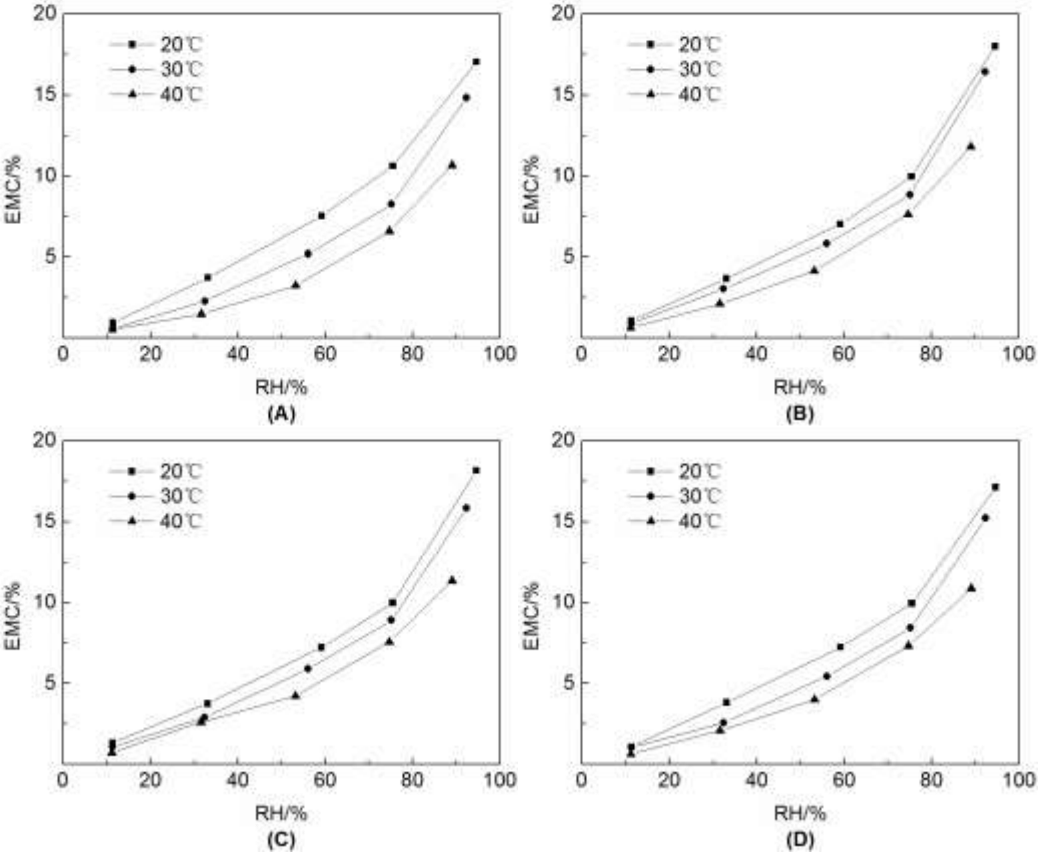

Fig. 3. The moisture adsorption isotherms of control and HP-treated wood samples at 20 °C, 30 °C, and 40 °C; (A) 0 s and 0.1 MPa, (B) 30 s and 30 MPa, (C) 30 s and 90 MPa, and (D) 30 s and 150 MPa

The effect of temperature on the MSI of both control and HP-treated wood samples was significant. EMC increased with the decreasing temperature at each constant relative humidity. EMC reduced with the increasing temperature, during both the adsorption and desorption processes. A similar tendency has been observed in the literature (Demertzis *et al.* 1989). This can be explained by the kinetic energy of the associated water molecules (Zauer *et al.* 2016). At a high temperature, water molecules have a higher excitation state and get activated at a higher energy level, which makes them become less stable and leads to a reduction in the attractive forces among the molecules. The EMC values of the HP-treated wood samples at the three temperatures were closer to each other than to the control. This result indicated that HP-treated wood samples were more stable in the face of temperature variation than were the untreated samples.

Hysteresis

The MSI curves (adsorption and desorption) of the control and HP-treated wood board samples at 20 °C are shown in Fig. 4. Differences were observed between the MSI curves for the adsorption and desorption processes which is generally recognized as the hysteresis phenomenon. Generally, it is understood that at a given constant temperature and RH, samples' EMC for desorption is comparatively higher than for adsorption, indicating the moisture content associated with desorption rather than adsorption especially at the middle regions generating a hysteresis loop (as shown in the Fig.4). Such a hysteresis phenomenon has been reported frequently in the literature (Bahloul *et al.* 2008). Taking as an example the sorption isotherm of (f) in Fig. 4, during the adsorption and desorption processes, the slope of the isotherm curve increased with the increase in relative humidity, becoming markedly steep in the range from 75% to 95% RH. Finally, the values of adsorption and desorption got much closer at 95% RH, which showed the trend that the hysteresis cycle was coming to an end. Similar results have been observed in other studies (Li *et al.* 2011; Yu *et al.* 2016). According to the capillary theory, as pointed out by Raji and Ojediran (2011), when the EMC increases during the adsorption process, the capillary swells, which mean the capillary pore diameter increases. Moreover, before desorption started, samples were placed in a relatively high RH environment (100%, as stated in the methodology part), which made the capillary pore size larger at the beginning of desorption than at the end of the adsorption experiment. These differences become even more pronounced in the normal drying and rehydration situation as compared to the hydration and dehydration adapted in the present study. Moreira *et al.* (2005) also reported that this effect could be explained by the pre-drying step, which modifies and deactivates by physical and chemical processes (collapse the tissue structure and terminates the biochemical processes) in the case of adsorption. There are many different possible explanations. For example, during the dehydration, the moisture present is mostly entrapped within the shrinking cells, while during rehydration it can enter the intercellular regions and provide more free water with higher water activity (ERH). However, the differences between the control and HP-treated samples were not obvious.

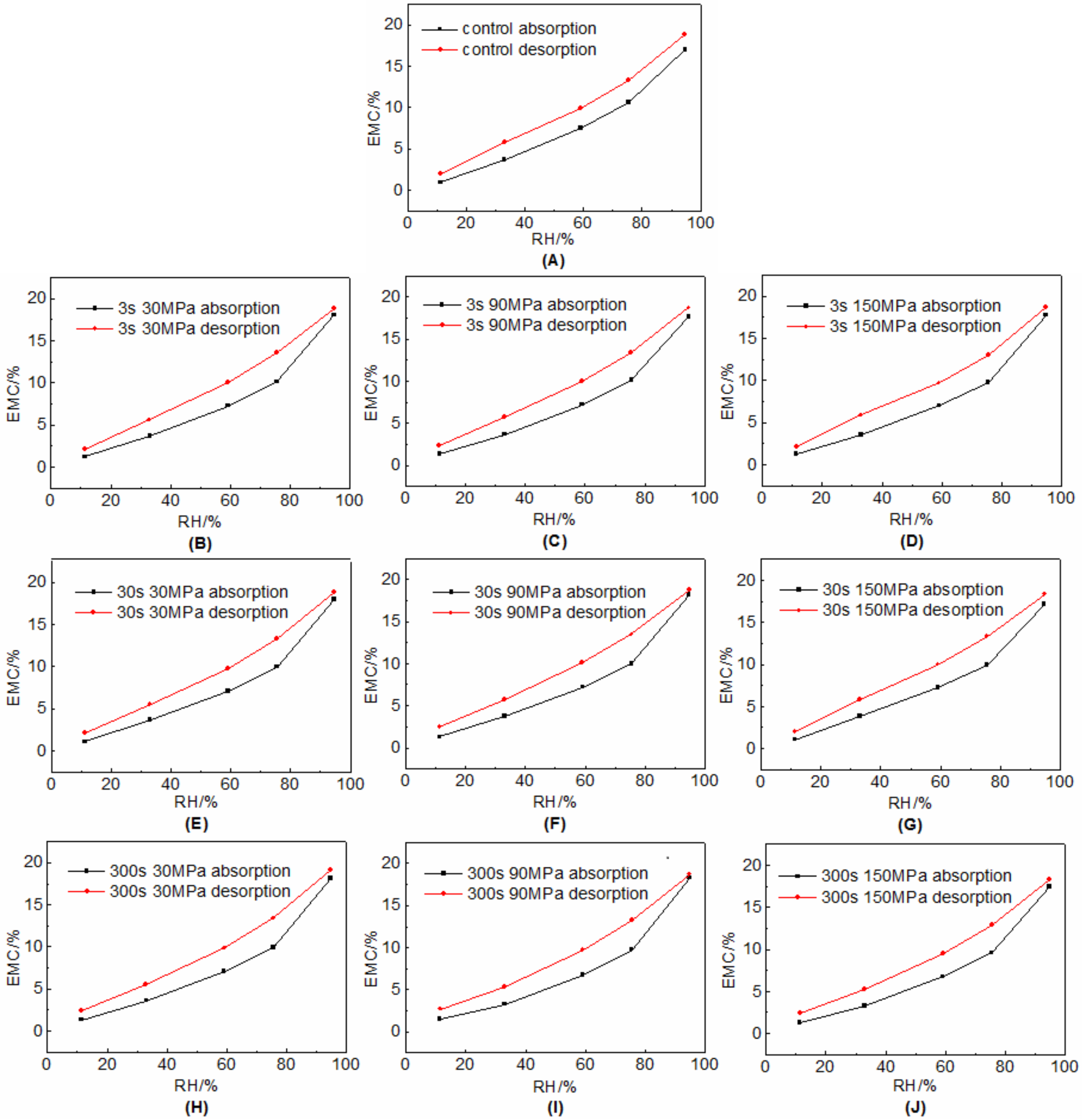

Fig. 4. Adsorption and desorption equilibrium moisture content of the control and HP-treated wood samples at 20 °C

Data Modeling

A nonlinear regression technique was used for fitting the adsorption and desorption equations to the experimental data, and the resulting models are presented in Table 2. The mean relative percent error (*MRE*), the standard error *(SE*), and the coefficient of determination (r^2) values of the experimental and predicted data were compared to select the best fitting model. The optimal fitted equilibrium moisture content model for adsorption and desorption of high-pressure treated *Paulownia* wood were as follows:

$$Xe,ads = 2.3763 \times 10^{-6}x + 1.5882\times10^{-6}y + 1.2880\times10^{-3}z + 1.6175\times10^{-3}t + 1.8241 \times 10^{-2}x^2 + 2.9199\times10^{-5}z^2 + 9.4885 \times 10^{-6}t^2 + 1.6683 \times 10^{-5}zt + 7.4727 \times 10^{-8}$$

$$Xe,des = -2.2177 \times 10^{-7}x - 2.2012 \times 10^{-6}y - 1.7821 \times 10^{-3}z + 1.6899 \times 10^{-3}t + 4.4461 \times 10^{-2}$$

where Xe,ads and Xe,des were EMCs obtained from adsorption and desorption, and x, y, z, and t represent HP treatment duration, pressure level, temperature, and relative humidity, respectively.

Table 2. Fitting Criteria of the Models Applied to Experimental Adsorption and Desorption Data of Wood

	Liner Polynomial Model		Quadratic Polynomial Model	
	Adsorption	Desorption	Adsorption	Desorption
MRE	1.3488	0.9254	1.3497	0.9254
S.E.	0.0162	0.1147	0.0162	0.0465
r^2	0.8993	0.9532	0.8994	0.2783

As shown in the Table 2, the equilibrium moisture content model of adsorption is established by the quadratic polynomial model, and the results indicated the goodness of fit evaluated by r^2. Moreover, the linear polynomial model gives the best fit to the experimental desorption data. A comparison of the experimental and predicted equilibrium moisture contents of adsorption and desorption, based on the quadratic polynomial model and the linear polynomial model for *Paulownia* wood samples, is shown in Fig. 5.

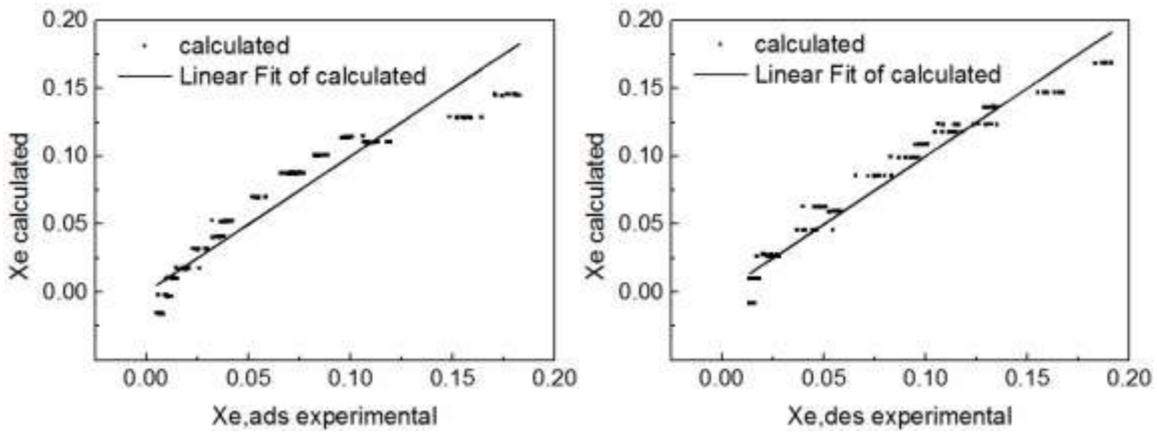

Fig. 5. Comparison of experimental and predicted adsorption and desorption equilibrium moisture contents of wood samples

Scanning Electron Microscopy

The scanning electron microscopy observations provided information on the characteristics of the morphological structure of the wood samples. The observations showed noticeable differences in the vessel elements between the control and HP-treated samples in the cross section, as shown in Fig. 6. Indeed, the vessel element pits observed on the transverse surfaces of each sample showed different sizes and forms as affected by the HP treatments. At the same magnification, the vessels were conspicuously collapsed along with the compressed parenchyma cells and internal cracks, which could account for the impact of applying intensely high pressure. A similar result was also found in a previous study on another species of HP-treated wood (Yu *et al.* 2017). The microstructure became smooth (as can be seen from the picture), and the sample decreased in size and became densified, in addition to undergoing improvement in mechanical properties.

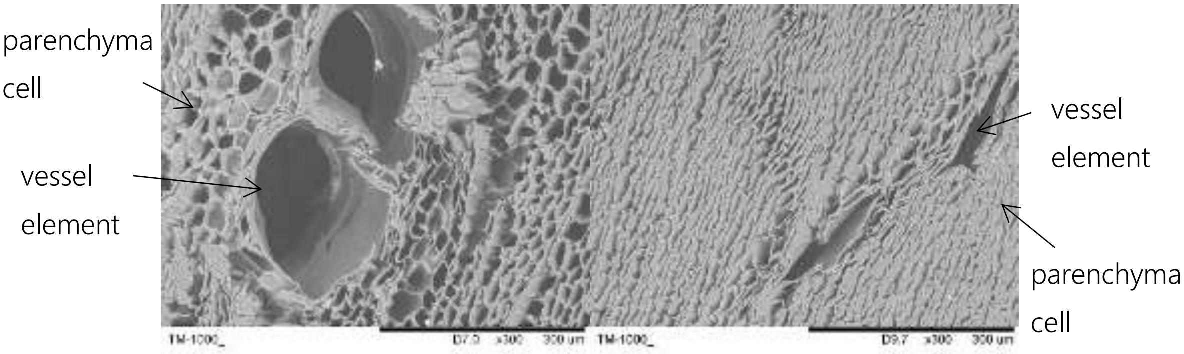

Fig. 6. Effect of HP treatment on the cross-section of wood samples (left: control; right: 3 s and 90 MPa)

CONCLUSIONS

1. *Paulownia* wood was treated under three pressure levels (30, 90, 150 MPa) and three holding times (3, 30, 300 s) for the purpose of densification. Moisture sorption isotherms were developed for each group at 20, 30, and 40 °C and in the RH range of 11 to 95%. The results showed that the HP treatment increased the equilibrium moisture content at high-RH conditions, while the treatment had no significant influence on moisture content at low-RH conditions. The effect of treatment time was negligible.
2. A linear polynomial model for desorption and a quadratic polynomial model for adsorption were established to describe the relationship between the EMC, RH, temperature, and high-pressure parameters. Both gave a high coefficient of correlation (0.95 for desorption; 0.90 for adsorption).
3. High-pressure treatment altered the morphological structure of the wood boards. The vessel element was transformed by the intense compression and therefore influenced the moisture sorption performance.

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