Effects of Thermo-Hydro-Mechanical Treatments on Various Physical and Mechanical Properties of Poplar (*Populus*) Wood

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Poplar (*Populus*) wood was subjected in this work to thermo-hydro-mechanical treatment. The influence of the treatment parameters on the physical and mechanical properties were investigated. The wood samples were densified under three compression ratios (0%, 30%, and 50%), and thermally treated at three temperatures (180 °C, 200 °C, and 220 °C), at three thermal treatment durations (3 h, 4 h, and 5 h). The density, modulus of elasticity, modulus of rupture, radial hardness, and thickness swelling were measured. The results showed that the densities of the samples increased by 36.6% to 49.7%. As the compression rate increased, the temperature, duration, modulus of elasticity, modulus of rupture, and hardness increased. However, the dimensions of the densified samples were less stable. Compared to the densified samples, the maximum thickness swelling could be reduced by 74% (from 29.7% to 7.8%) when subjected to a thermal treatment at 220 °C for 3 h.

Keywords: Densification; Heat treatment; Physical properties; Mechanical properties

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INTRODUCTION

Wood has been used as a structural material for building and furniture construction for thousands of years, primarily due to its low cost, abundance, and renewability (Boonstra and Tjeerdsma 2006). However, some qualities of wood are unsatisfactory for many applications, *i.e.*, low density, poor mechanical properties, poor dimensional stability, and poor durability (Drafz *et al*. 2005; Kutnar and Kamke 2012). The physical and mechanical properties of wood are closely related to its density (Tu *et al*. 2014). Thus, considerable wood modification technologies have been proposed to increase the density of wood-based materials. Generally, the densification of wood materials can be accomplished *via* three different methods: mechanical compression, resin impregnation, or a combination of mechanical compression and impregnation method (Kollmann *et al*. 1975; Navi and Heger 2004; Kutnar *et al*. 2008). Among those densification methods, mechanical compression is the simplest and probably the most cost-efficient method (Dwianto *et al*. 2000).

During mechanical compression, the wood is compressed to the desired thickness between heated plates and the deformation is finalized in the compressed state. The void volumes in wood may be reduced by compressing the wood structure (usually in the radial direction), under the suitable moisture and temperature conditions, without the addition of

any chemicals. After mechanical densification, the physical and mechanical properties of the wood is improved, *e.g.*, the hardness, abrasion resistance, modulus of elasticity (MOE) and modulus of rupture (MOR), primarily due to the density of the wood increasing (Sandberg *et al*. 2013). Generally, most of the physical and mechanical properties of wood largely depend on the compression ratio (Belt *et al*. 2013). A study by Pelit *et al*. (2015) densified Scots pine and Eastern beech samples using a specially designed hydraulic press, with target compression ratios of 20% and 40%. Pelit *et al*. (2015) found that the density was increased by 42% for the Scots pine samples in proportion to the compression ratios, and 35% for the Eastern beech samples. In another study, Laine *et al*. (2016) studied the influence of the compression ratio (CR = 40%, 50%, and 60 %) on the hardness and set-recovery of densified wood. Based on the findings of Laine *et al*. (2016), they concluded that the hardness drastically increased (in some circumstances doubled) relative to non-densified samples with a CR of 50%. Song *et al*. (2018) processed wood to have a higher specific strength than most structural metals and alloys. Song *et al*. (2018) reported a two-step process involving the partial removal of lignins and hemicelluloses from the natural wood, following by hot-pressing, which led to the total collapse of the cell walls and the complete densification of the natural wood with highly aligned cellulose nanofibres.

Although mechanical densification has been developed, the set recovery of wood densification remains in question. Although the plasticized deformation of compressed wood does not change under dry conditions, wood completely recovers when the compressed wood is re-wet or boiled in water (Welzbacher *et al*. 2008). The structure of wood cells and the properties of cell wall polymers are responsible for the compression-recovery behavior of wood (Kutnar and Šernek 2007). Post-high-temperature heat-treatment also could assist in solving set recovery problem through the modification of chemical components in compression wood (Gong *et al.* 2010). Many researchers have attempted to fix the shape recovery of densified wood, through thermo-hydro-mechanical (THM) compression and thermo-mechanical (TH) compression, which promotes cross-linking of chemical components, or releases inner chemical stresses to fix the deformation of the compressed wood (Navi and Heger 2004; Kutnar and Kamke 2012; Gao *et al*. 2016).

Thermo-hydro-mechanical (THM) processes as a physical modification were developed in late 20th century, which includes mechanical and hydrostatic compression *via* the addition of steam and/or heat (160 °C to 250 °C) (Inoue *et al.* 1993; Kutnar *et al*. 2009; Kutnar *et al*. 2015). The THM processes, at thermal treatment temperatures ranging from 160 °C to 250 °C, were found to improve the decay resistance, lower hygroscopicity, and enhance dimensional stability. Popescu *et al*. (2014) studied the influence of thermal treatments on wood samples. The set recovery tests showed that a higher temperature and longer duration improved the dimensional stability of the wood sample. Yin *et al*. (2016) used Raman microscopy (CRM) and X-ray diffraction to investigate the changes in the chemical composition and cellulose crystalline structure of spruce wood cell walls *via* THM treatment. The CRM results revealed that the crystallinity index and crystallite thickness of the samples increased due to the crystallization of the semicrystalline region and β-aryl-ether links. The crystallization process was associated with the guaiacyl units of the lignins, which were depolymerized after the re-condensation reactions. The crystallization of cellulose and lignin β-aryl-ether links are considered to be the reasons causing an increase in dimensional stability. However, the thermal treatment caused a loss of mass and consequently, a decrease in the density of the wood. Tomislav *et al*. (2019) studied changes in the Brinell hardness rating of beech wood and hornbeam wood, and found that thermal modification reduced the Brinell hardness rating of beech wood and

hornbeam wood by 3% and 6% for the cross sections, by 15% and 18% for radial sections, and by 25% and 13% for tangential sections, respectively. This result indicated that the thermal treatment negatively influenced the Brinell hardness value of wood.

Thermo-hydro-mechanical treated wood has all the advantages of compressed wood and thermal treatment wood, with higher density and mechanical properties, as well as better dimensional stability. Therefore, the aim in this study was to examine the influence of the compression ratio, thermal treatment temperature, and duration on the density profile, MOE, MOR, hardness, and set recovery of THM treatment wood. In addition, this study examined how density influences the hardness and mechanical properties of THM treated wood.

EXPERIMENTAL

Materials

Preparation of the specimens

Beijing Poplar (*Populus* x *beijingensis* W. Y. Hsu) specimens without any defects were obtained from Inner Mongolia as logs with diameters between 28 and 38 cm. Sample dimensions were as follows: 400 mm (longitudinal) x 150 mm (tangential) x 30 mm (radial). Because the growth cycle is short, Beijing poplar wood with low density, low strength, poor material, and no decay or other defects was obtained. The oven-dried density of the wood was 395 kg/m^3. The initial moisture content of all the samples was 25% to 30%, and the initial moisture content of the samples was measured with a hygrometer (KT-50B, Klonteser Co., Rome, Italy).

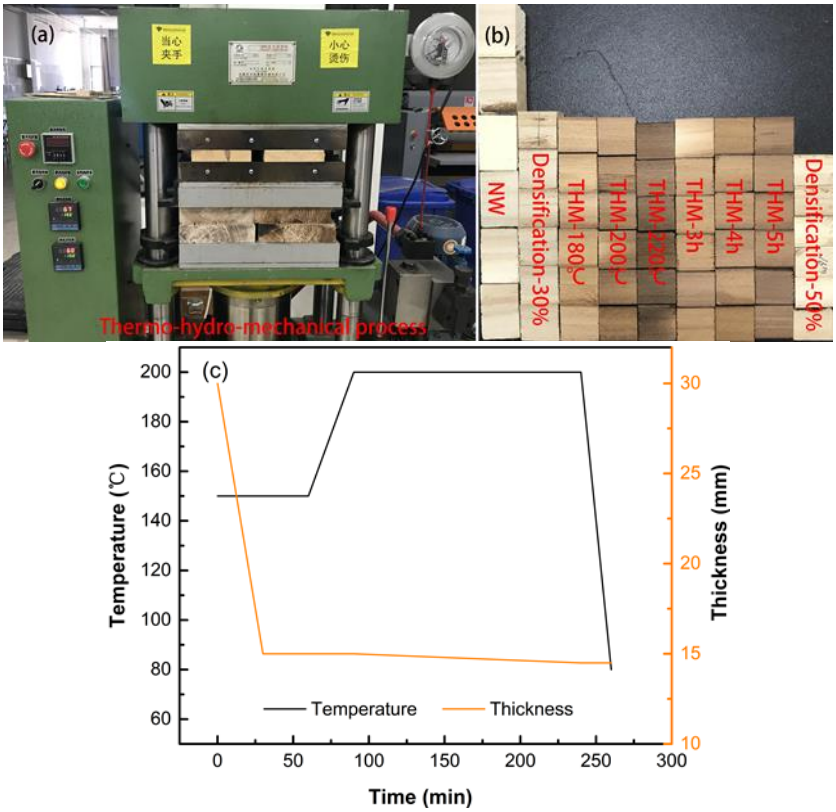

Fig. 1. The processing steps of the thermo-hydro-mechanical process

Thermo-hydro-mechanical process

The thermo-hydro-mechanical process was composed of three processing steps (Fig. 1). First, the specimens were pressed in the radial direction at 150 °C under a pressure of approximately 4 MPa for approximately 30 min to obtain the compressed wood. The thickness of the specimens was controlled with metal stops at three different target compression ratios: 0%, 30%, and 50%. Then, the samples were dried under a constant temperature (150 °C) and pressure (4 MPa) for approximately 30 min. Heat treatment can reduce the set-recovery of compressed wood. Therefore, after drying, the samples were directly heat-treated with a hot press at the designated temperatures and durations (Table 1). The three important processes of THM processing are all completed in the open press system (as shown in Fig. 1a).The specimens were stored in a chamber at 20 °C and 65% relative humidity (RH) until their equilibrium moisture content was reached.

Table 1. Groups of Specimens According to Treatment

Sample Groups		Compression Ratios (%)	Thermal Treatment Temperature (℃)	Thermal Treatment Duration (h)
NW	Natural Wood	-	-	-
Densification	Densification-30%	30	-	-
	Densification-50%	50	-	-
THM-CR	THM-0%	0	180	3
	THM-30%	30	180	3
	THM-50%	50	180	3
THM-T	THM-180 °C	30	180	3
	THM-200 °C	30	200	3
	THM-220 °C	30	220	3
THM-D	THM-3h	30	200	3
	THM-4h	30	200	4
	THM-5h	30	200	5

Methods

Density and testing the density profile

The densities of the wood samples were measured with a standard procedure described in the GB/T 1933 (2009). The samples were kept in the oven chamber at a temperature of 103 °C, until a constant weight was reached. The mass and dimensions (longitudinal, tangential, and radial) of each sample were measured with an analytical balance (± 0.001 g) and a vernier caliper (± 0.001 mm), which were used to determine the weight (m_0) and volumes (V_0), respectively. The oven-dried density (ρ_0) (g/cm^3) was calculated using Eq. 1:

$$\rho_0 = \frac{m_0}{V_0} \quad (1)$$

Determining the density profile of the wood samples was performed with a GreCon DAX 5000 tester. Specimens with dimensions of 50 mm (longitudinal) x 50 mm (tangential) x compressed size (radial) were prepared from the oven-dried compressed specimens, and were used to measure the density profile *via* a combination of X-ray sensors (where measurements can be made up to 1 mm per second). In this experiment, the narrow X-ray beam was projected to the specimens tangentially and then scanned in the thickness direction to generate a density profile.

Testing the modulus of elasticity (MOE) and modulus of rupture (MOR)

The bending deformation was measured using a Tinius Olsen H5KT tester with a standard procedure described in the GB/T 1963.1-2009 and GB/T 1963.2-2009. The sample dimensions were approximately 300 mm (longitudinal) by 20 mm (tangential) by the compressed size (radial). Three-point bending tests were conducted on each sample, with the span between the two bottom rollers equaling 240 mm and the top roller pressing down at the centre at a speed of 2 mm min^{-1} .

Testing the radial hardness

The radial hardness of the wood samples was determined with a Tinius Olsen H5KT tester with a standard procedure described in the GB/T 1941-2009. The dimensions for hardness samples were approximately 50 mm (longitudinal) x 50 mm (tangential) x compressed size (radial). The pressing ball had a diameter 5.64 mm with a penetration depth of 5.64 mm. The test was performed with a rate of penetration of 3 mm min^{-1}. The static radial hardness was calculated as K x P, where K is the coefficient of the penetration depth (5.64) and P is the applied maximum force.

Testing the thickness swelling

Determination of thickness swelling of the wood samples was based on the standard procedure described in GB/T 1934.2-2009. Treated specimens were oven-dried for more than 12 h to determine their dry dimensions and masses. After that, all specimens with dimensions of approximately 20 mm (longitudinal) x 20 mm (tangential) x compressed size (radial) were placed in a chamber under conditions of 25 °C and 60% RH for 1 week to determine their stable dimensions and masses. Next, all specimens were soaked in water at 25 °C, and the dimensions and masses after soaking in water were recorded. The thickness swelling was calculated using Eq. 2,

$$R_{max} = \frac{L_{max} - L_0}{L_0} \times 100 \quad (2)$$

where R_{max} (mm) is the compression set recovery along the thickness direction after swelling, L_0 (mm) is the oven-dried thickness before setting recovery occurred, and L_{max} (mm) is the thickness of the wood sample after swelling.

RESULTS AND DISCUSSION

Density Profile Analysis

The density profile is the density distribution along the thickness that affects the mechanical and physical properties of wood (Rautkari *et al*. 2013). The density profiles of the untreated and treated wood samples (CR 30% and CR 50%) were presented in Fig. 2a and 2b, respectively. Because densification occurs in two surface layers, due to softening under the high temperature of the press plate, a symmetrical density profile was expected. The surface layers have higher densities than the core area; however, the density at the core layer was still greater than the density of untreated wood. An increase in the exerted pressure can increase the maximum density as well as the minimum density. For the samples with a CR of 50% the maximum and minimum densities were 941.8 and 634.8 kg/m^3, respectively, for samples with a CR of 30% the maximum and minimum densities were 759.5 and 406.9 kg/m^3, respectively, while for samples with a CR of 0% the

maximum and minimum densities were 479.7 and 376.1 kg/m^3, respectively. The maximum and minimum densities of the untreated wood samples were 473.3 and 388.5 kg/m^3, respectively, which corresponded to the densities of latewood and earlywood. Different studies have shown that density increases as the compression ratio increases (Blomberg *et al*. 2005; Arruda and Menezzi 2013).

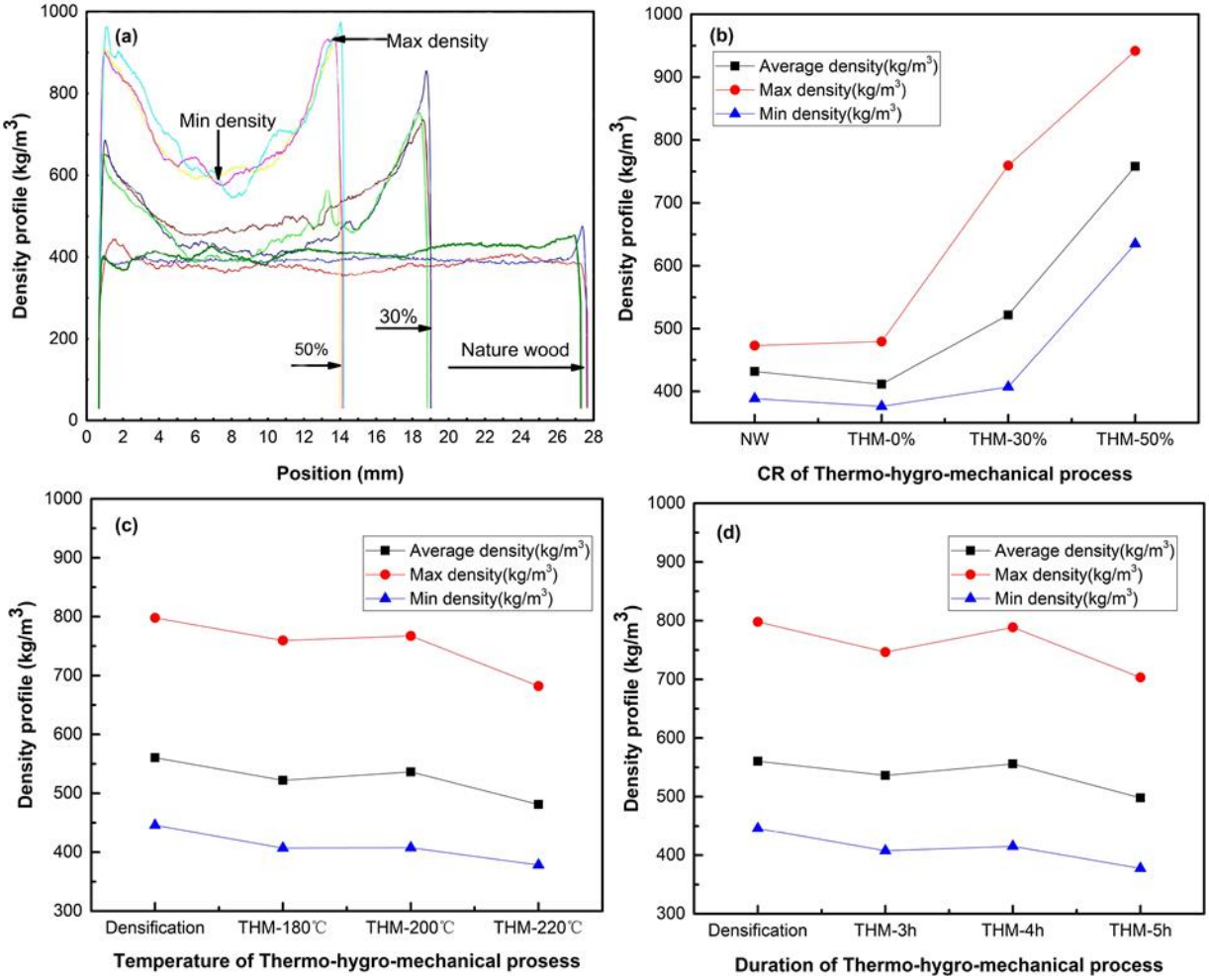

Fig. 2. (a): The density profile of the specimens with different degree of compression ratios; (b): Influence of the compression ratio of the thermo-hydro-mechanical process on the average (*n* = 18) density profile of poplar (*Populus*) wood; (c): Influence of the thermal treatment temperatures of the thermo-hydro-mechanical process on the average (*n* = 18) density profile of poplar (*Populus*) wood; and (d): Influence of the thermal treatment duration of the thermo-hydro-mechanical process on the average (*n* = 18) density profile of poplar (*Populus*) wood

Densification is an effective way to convert low-density wood into a denser, harder material (Dwianto *et al.* 1998; Kariz *et al*. 2017; Kúdela *et al*. 2018). Thermal modification is the simplest and probably the most cost-efficient method of doing so (Wehsener *et al*. 2018; Tomislav *et al*. 2019). It was found that thermal modification at temperatures greater than 160 °C, in an oxygen free environment, causes permanent changes to the mass, density, and mechanical properties of wood (Li *et al*. 2017, 2018). This study also examined the effects of the temperature and duration on the density profile of densified wood (Fig. 2c and 2d). The density profile of the densified samples that underwent thermal posting

treatment did not differ drastically from the density profile of densified wood that did not undergo thermal posting treatment. However, in samples with thermal modification temperatures of 180 °C and 200 °C, a decrease in density was observed, while the samples with thermal modification temperature of 220 °C differed from the densification-30% samples (Fig. 2c). The effects of the thermal modification duration on the density profile of the sample led to a similar conclusion, in which the samples with thermal modification duration of 5 h differed from the densification-30% samples (Fig. 2d). During treatment, the loss of wood mass and the decrease in equilibrium moisture content lead to a decrease in total wood density (Pelit *et al*. 2018). Thermal treatment causes wood degradation, especially from the decomposition of hemicelluloses. Hemicelluloses generate acetic acid during heat treatment, which further catalyzes carbohydrate cleavage, resulting in a decrease of the degree of carbohydrate polymerization (Popescu *et al.* 2011). Thermal modification causes mass loss, and mass loss directly affects the density profile of wood (Esteves *et al*. 2008; Rautkari *et al*. 2013; Pelit *et al*. 2019).

MOE and MOR Analysis

The mechanical properties of the densification samples were drastically higher than untreated wood. The MOE and MOR of the treated samples, under different THM treatment conditions, are presented in Fig. 3a and 3b. These results showed a major increase in the MOE and MOR due to the increase in the density resulting from the increase in the compression ratio (ranging from 0% to 50%). The densified wood with a compression rate of 50% demonstrated the greatest MOE and MOR values (13,900 MPa and 131 MPa, respectively), which were 3.84 and 2.61 times greater than natural wood.

The thermal modification temperature and duration affects the mechanical strength of densified wood. To characterize mechanical strength changes due to variation in the temperature and duration and the effect of thermal modification on the MOE and MOR, the treated samples at various temperatures (180 °C, 200 °C, and 220 °C) and durations (3 h, 4 h, and 5 h) were measured. The MOE value of compressed wood with a CR of 30% was 11,800 MPa under thermal modification conditions of 3 h at 200 °C, which was noticeable higher than the MOE values at thermal modification temperatures of 180 °C and 220 °C (Fig. 3a). However, thermal modification did noticeably influence the MOR values. Thermal modification temperatures of 180 °C and 200 °C reduced the MOR value to 84.3 MPa and 87.6 MPa, respectively (Fig. 3b). When the thermal modification temperature reached 220 °C, the MOR value sharply dropped. The MOE and MOR were also measured for samples with different treatment durations. A longer duration did not affect MOE values. A thermal modification duration of 4 h and 5 h reduced the MOE to 69.7 MPa and 67.2 MPa. The MOR values were reduced after wood samples were subjected to higher treatment temperatures (220 °C) and longer treatment times (5 h).

The mechanical properties noticeably increased due to densification, as well as increasing with an increase in compression ratios (Zhan and Avramidis 2017). In terms of natural wood samples, the highest air-dried density was found in densification samples with a CR of 50%, and the value increased by approximately 84.2%. The mechanical strengths (MOE and MOR) and density exhibited a positive linear correlation with the correlation coefficients, which were 0.86 and 0.8206, respectively (Fig. 3c and 3d).

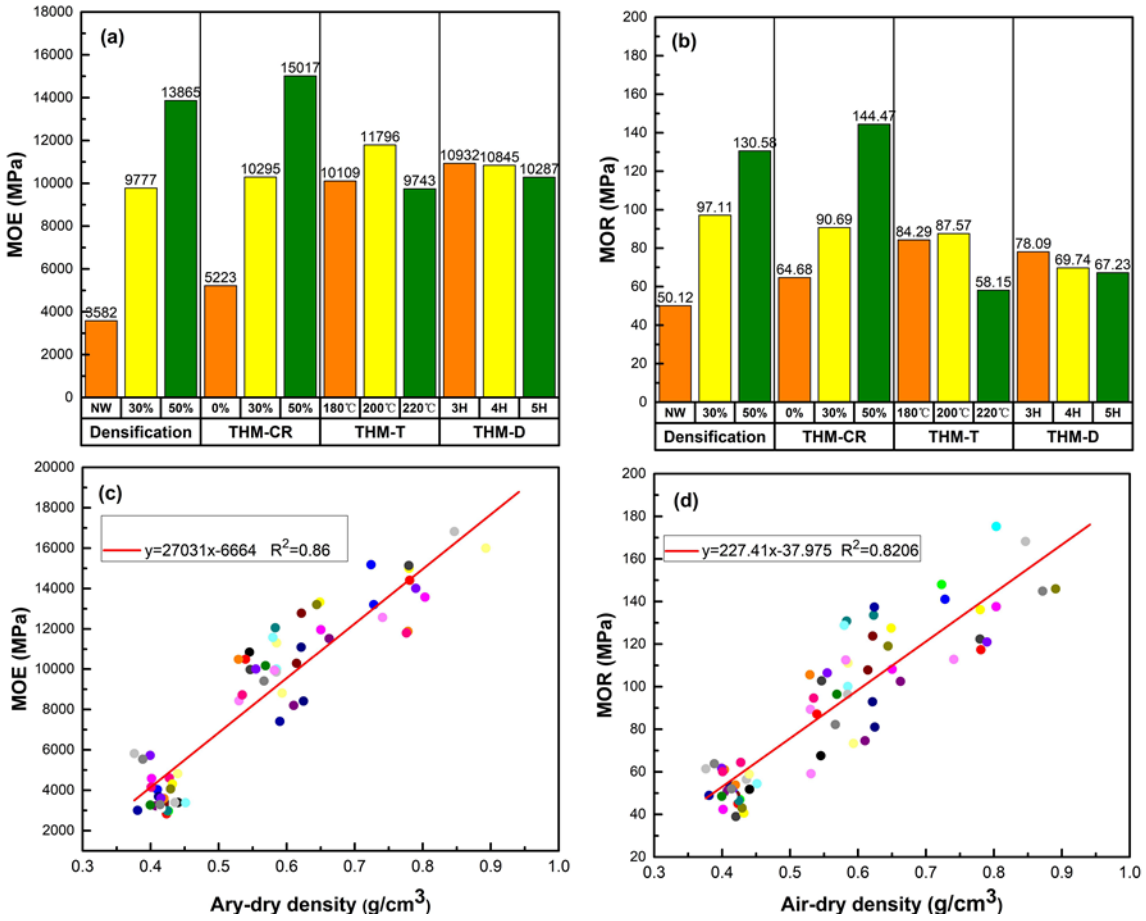

Fig. 3. (a): Influence of the different parameters of the thermo-hydro-mechanical process on the average (n = 10) MOE values of poplar (*Populus*) wood; (b): Influence of the different parameters of the thermo-hydro-mechanical process on the average (*n* = 10) MOR values of poplar (*Populus*) wood; (c): Correlation between Air-dry density and MOE of nature wood and thermo-hydro-mechanical modified poplar (*Populus*) wood; and (d): Correlation between Air-dry density and MOR of nature wood and thermo-hydro-mechanical modified poplar (*Populus*) wood

In general, deformation had a greater change of occurrence in the earlywood, which has thinner and weaker cell walls in comparison to latewood. However, a higher heat treatment temperature and longer heat treatment duration will thermally decompose chemical components, which results in quality loss, and a reduction of the mechanical properties of the treated sample (Esteves and Pereira 2009).

Radial Hardness Analysis

In this study, the radial hardness of the treated samples and natural wood control were measured. The radial hardness values when the compression ratio, heat treatment temperature, and duration were altered, as presented in Fig. 4. The radial hardness increased with an increase in the compression ratio. According to results shown in Fig. 4a, the highest radial hardness (3.09 KN) was found in the samples with a compression ratio of 50%, whereas the lowest hardness value (1.47 KN) was found in the undensified samples. The mean difference in the hardness values was approximately 110%, while the density values were higher in samples with a compression ratio of 50% (768 kg/m^3) than in the undensified samples (395 kg/m^3). In other studies, it has been reported that the

hardness of woody materials is closely related to the compression ratio, and that the hardness values increase as the compression ratio is increased (Ünsal *et al*. 2011; Gašparík *et al*. 2016). The correlation between the radial hardness and the air-dried density of the samples that underwent thermal treatment and the control are shown in Fig. 4d. The changes in the density of the wood sample affected the radial hardness of the samples that underwent thermal treatment, relative to the control.

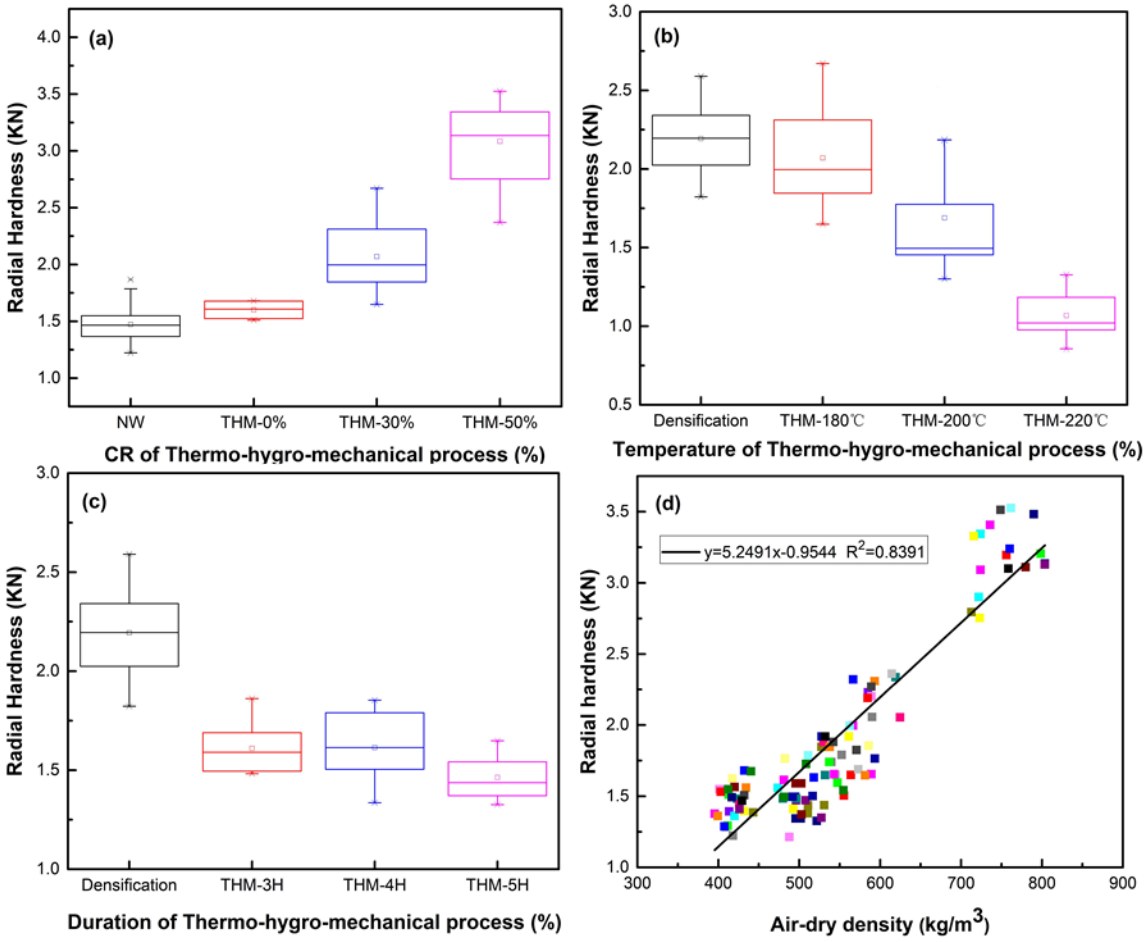

Fig. 4. (a): Influence of the CR of the thermo-hydro-mechanical process on the average (n = 15) radial hardness of poplar (*Populus*) wood; (b): Influence of the temperature of the thermo-hydro-mechanical process on the average (n = 15) radial hardness of poplar (*Populus*) wood; (c): Influence of the duration of the thermo-hydro-mechanical process on the average (n = 15) radial hardness of poplar (*Populus*) wood; (d): Correlation between the air-dried density and radial hardness of natural wood and thermo-hydro-mechanical modified poplar (*Populus*) wood samples

The radial hardness decreased as the treatment temperature and duration increased (Figs. 4b and 4c). The densified wood subjected to a treatment temperature of 180 °C had a lower radial hardness (6.4%) and densified wood subjected to a treatment temperature of 200 °C and 220 °C had a decrease in radial hardness of 22.8% and 51.6%, respectively. When a thermal treatment duration of 3 h, 4 h, or 5 h was applied with a treatment temperature of 200 °C, the mean radial hardness was reduced to 1.69 KN, 1.61 KN, and 1.46 KN, respectively. The radial hardness values gradually decreased as the thermal treatment duration increased. However, the hardness value gradually decreased as the thermal treatment temperature and duration increased (Perçin 2012; Fang *et al*. 2012b;

Agnieszka 2017). The temperature and duration of the thermal treatment accounted for 30% of the wood hardness variability, whereas the interaction between temperature and duration accounted for 23% of the wood hardness variability (Agnieszka 2017).

The decrease in hardness can be attributed to the degradation of polysaccharides and the loss of mass (Pelit *et al*. 2017). The total cellulose and hemicellulose content decreased with an increase in temperature and duration of treatment. The change in hemicellulose content was dependent on the temperature. Most of the hemicelluloses were degraded when the sample was exposed to thermal treatment temperatures and durations of 220 °C for 3 h or 200 °C for 5 h (Li *et al*. 2016).

Thickness Swelling Analysis

The results of analyzing the water-soaked thickness swelling values of the treated samples are illustrated in Fig. 5. Before the thermal treatments, when compared to the nondensified samples, the thickness swelling values were 29.7% and 41.6% at compression ratios of 30% and 50%, respectively. Compared to the untreated samples, the thickness swelling values were less in the treated samples. Wood has a shape memory effect, *i.e.*, when densified wood is soaked in water or exposed to a high relative humidity, it will bound back (Ugolev 2014). The internal stress generated by the compression is relieved, and the cell wall absorbs water and expands, returning to its original shape (Pelit *et al*. 2016).

The thickness swelling values decreased with an increase in the temperature and duration of treatment (Fig. 5).

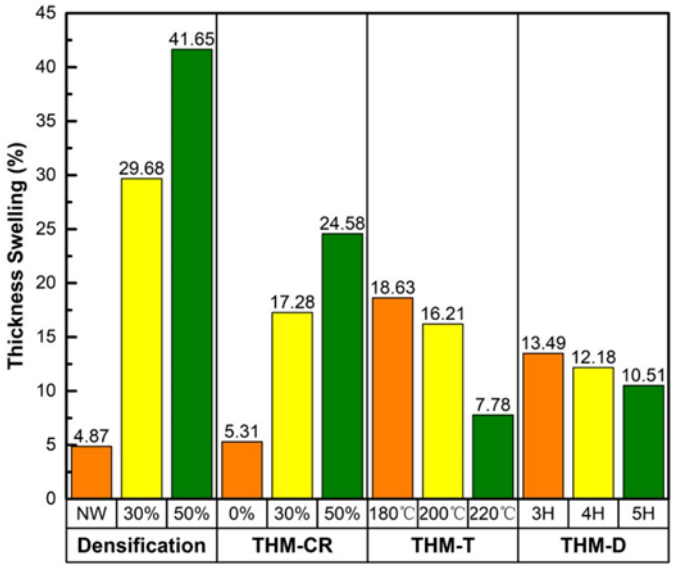

Fig. 5. Influence of different parameters of the thermo-hydro-mechanical process on the average (n = 20) thickness swelling of poplar (*Populus*) wood

The maximum thickness swelling was reduced by 74% (from 29.7% to 7.8%) in samples that underwent thermal treatment conditions of 220 °C for 3 h. The thermal temperature had more influence on the thickness swelling values of densified wood than the thermal treatment duration. The dimensional stability was greatly improved with thermal treatment temperatures greater than 180 °C (Fukuta *et al*. 2008; Fang *et al*. 2012a). Several factors contributed to the increase in the dimensional stability of the thermal treated

wood: less hygroscopic hemicelluloses, the bridging of cellulose chains, and the cross-linking of the aromatic rings found in the lignins (Kocaefe *et al*. 2015).

CONCLUSIONS

1. After densification due to heat treatment and compression, an increase in density was observed in poplar samples, in proportion to the compression ratio (CR). Thermal treatment causes wood degradation, especially from the decomposition of hemicelluloses. Thermal modification causes mass loss, and mass loss directly affects the density of wood. The density decreased 4.6% in samples with thermal treatment conditions of 200 °C for 5 h.
2. The modulus of elasticity (MOE) and modulus of rupture (MOR) values of the treated poplar samples increased as the compression ratio increased. The mechanical strengths and density have a positive linear correlation with the correlation coefficients. However, a higher heat treatment temperature and longer heat treatment duration will thermally decompose chemical components, which results in quality loss, and a reduction of the mechanical properties of the treated sample.
3. The hardness of poplar is closely related to the compression ratio, and the hardness value increases with the increase of compression ratio. At a compression ratio of 50%, the hardness increased from 1.47 to 3.09 KN. The radial hardness gradually decreased as the treatment temperature and duration increased. The decrease in hardness can be attributed to the degradation of polysaccharides and the loss of mass.
4. The dimensional stability was improved by the thermal treatment. Compared to the densified sample, the maximum thickness swelling value was reduced by 74% (from 29.7% to 7.8%) through thermal treatment conditions of 220 °C for 3 h duration. The reduction of hygroscopic hemicellulose, the bridging of cellulose chains, and the crosslinking of aromatic rings in lignin are the reasons for the increase of dimensional stability of the thermal treated wood.

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