

Effects of Pressurized Superheated-steam Heat Treatment on Set Recovery and Mechanical Properties of Surface-compressed Wood

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The recovery behavior and selected mechanical properties were studied for *Populus tomentosa* wood subjected to surface compression followed by heat treatment. The surface compression of wood was carried out in an open hot-pressing system at 180 °C with compressed thickness of 2 to 18 mm. The surface-compressed wood was treated by atmospheric heat treatment or 0.30 MPa pressurized superheated-steam heat treatment at 180 °C for 2 h. The results showed that the set recovery of compressed wood decreased with increasing compressed thickness before post-treatment. Atmospheric and pressurized heat treatment reduced the average set recovery of compressed wood significantly from 12.9% to 4.1% and 1.5% respectively, after conditioning at 40 °C and 90% relative humidity. Moreover, mechanical properties including the modulus of elasticity (MOE), modulus of rupture (MOR), hardness, and surface hardness increased with elevating compressed thickness. Both atmospheric and pressurized heat treatment reduced the MOR, hardness, and surface hardness of compressed wood. Analysis of variance showed that the effects of heat treatment on mechanical properties was not significant, except pressurized heat treatment decreased hardness significantly. With a compressed thickness of 10 mm, MOE, MOR, hardness and surface hardness were increased by 52.6%, 36.4%, 122.0% and 129.6%, respectively, compared with untreated wood.

Keywords: Surface compression; Pressurized superheated-steam; Compressed thickness; Set recovery; Mechanical properties

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INTRODUCTION

Wood compression treatment is an eco-friendly wood modification technology that is based on the combined treatment of wood by elevated temperature, moisture, and application of mechanical forces (Navi and Pizzi 2015). In addition, this technology can significantly improve physical and mechanical properties of low-density wood (Fukuta *et al.* 2007; Laine *et al.* 2016; Zhan and Avramidis 2017; Li *et al.* 2018). Most researchers have studied bulk compression, a process in which wood is first softened by a hydrothermal treatment and then compressed throughout its entire thickness (Kamke and Sizemore 2008; Kutnar *et al.* 2009). For many applications, compression of the wood surface layers alone may be sufficient, such as in the case of flooring (Belt *et al.* 2013; Zhan *et al.* 2015). Surface compression has some distinct advantages over bulk compression: the compression process is fast and energy efficient, and the wood volume lost is significantly less (Laine *et al.*

2016; Zhan and Avramidis 2017). To obtain surface-compressed wood with high-density of the surface layer and low-density of the core layer, the distributions of moisture content (MC) and temperature of wood need to be controlled by soaking and heating treatments (Inoue *et al.* 1990; Gong *et al.* 2010; Tu *et al.* 2014; Gao *et al.* 2016). The wood was compressed in an open hot-pressing system to densify the cell lumen in the wood surface layer, and the mechanical properties are obviously improved (Laine *et al.* 2016; Zhan and Avramidis 2017).

Compressed wood produced in an open hot-pressing system without any deformation fixation treatment is sensitive to moisture, leading to deformation and set recovery when exposed to liquid water or humid environments (Morsing 2000; Navi and Girardet 2000; Kutnar and Kamke 2012). Atmospheric heat treatment is a commonly used method to fix the deformation of compressed wood. The set recovery of compressed wood decreases by more than 70% after 24 h water immersion after atmospheric heat treatment at 200 °C (Laine *et al.* 2013; Zhan *et al.* 2015). Furthermore, heat treatment in the steam environment also helps the fixation of compressive deformation (Gong *et al.* 2010; Tu *et al.* 2014; Laine *et al.* 2016). Inoue *et al.* (1993a) and Navi and Heger (2004) found that saturated steam treatment above 180 °C can achieve permanent fixation of compression deformation and the set recovery of compressed wood after boiling can be controlled to 2%. However, the saturated steam treatment at high temperature and high pressure (180 °C, 1.0 MPa) requires high pressure resistance equipment, and it is currently mainly used in laboratory research on the bulk compression of wood. Limited work has been done in terms of the fixation of surface-compressed wood using pressurized superheated-steam heat treatment.

High-temperature heat treatment, especially superheated-steam treatment, improves the dimensional stability of wood by degrading the wood cell wall components cellulose, lignin, and especially the hydrophilic hemicellulose (Ding *et al.* 2011; Rautkari *et al.* 2014). The latent heat of superheated steam vaporization is larger, and heat treatment under low pressure and high temperature conditions can be realized. However, it also decreases the mechanical properties of wood. The MOR and hardness of wood decrease by about 10% and 6%, respectively, after atmospheric heat treatment at 180 to 210 °C (Gong *et al.* 2010; Laine *et al.* 2016). Moreover, Ding *et al.* (2011) investigated the effects of atmospheric steam and pressurized steam on the mechanical properties of wood, which shows that heat treatment, no matter whether in atmospheric steam or in pressurized steam, make fracture toughness and MOR drop. However, when comparing the samples treated in atmospheric steam to those treated in pressurized steam, it can be found that increased pressure pulls down the values of fracture toughness and MOR, but the differences are not statistically significant ($P = 0.05$). The accumulation of acidic degradation products and the increase in active hydration ions directly accelerates wood degradation in the pressurized steam environment (Borrega and Kärenlampi 2008a; Ding *et al.* 2012). At present, there are few reports on the effects of pressurized superheated-steam treatment on the deformation fixation and physical and mechanical properties of compressed wood.

This paper examined the influence of 0.30 MPa pressurized superheated-steam heat treatment at 180 °C on the set recovery of surface-compressed white poplar wood after conditioning at 40 °C and 90% relative humidity (RH) or immersion in water. The effects of the heat treatment and compressed thickness on the MOE, MOR, hardness, and surface hardness of compressed wood were also investigated. This study can potentially promote the commercial production of surface-compressed wood.

EXPERIMENTAL

Materials

Preparation of wood specimens

Twenty-five-year-old Chinese white poplar (*Populus tomentosa*) trees, with diameters from 25 to 35 cm at breast height and air-dried density of 0.44 g·cm⁻³, were harvested from a plantation forest. After the timber was dried to 12% MC, the specimens with the size of 400 mm (longitudinal) × 120 mm (tangential) and six thicknesses of 20, 22, 25, 30, 33 and 38 mm (radial direction) were prepared. Six replicates were performed for each thickness.

Surface compression

The surface compression parameters used in this study were chosen based on a previous study (Gao *et al.* 2016). After cross section and the radial section of the specimens were sealed with paraffin, they were immersed in the distilled water for time periods between 0.5 and 5.5 h (Table 1). The average MC of specimens was 17% after water immersion.

The compression of specimens was implemented with a hot-press machine (JICA). The specimens were placed onto the bottom plate of the hot-press machine and then the top plate quickly contacted the specimens surface. The temperatures of the top and bottom plates of the hot-press were controlled at 180 °C. Afterwards, surface compression in the radial direction was carried out with a pressure of 6.0 MPa. Due to different compressed thickness, the total closing time lasted from 20 to 200 s in order to achieve the target thickness of 20 mm. Compressed specimens were under pressure for 30 min (Table 1). Finally, the specimen was taken out until the temperature was reduced to 60 °C.

Table 1. Process Parameters of Wood Surface Compression

Original thickness (mm)	Compressed thickness (mm)	Soaking time (h)	Total closing time (s)	Holding time (min)
22	2	0.5	20	30
25	5	1.0	50	30
30	10	2.5	120	30
33	13	4.0	160	30
38	18	5.5	200	30

The fixation of compressive deformation by heat treatment

The compressed wood specimens were divided into three groups before post-treatment: compressed wood without post-treatment (Compressed), compressed wood with atmospheric heat treatment at 180 °C for 2 h (Compressed + AHT), and compressed wood with pressurized (0.30 MPa) heat treatment at 180 °C for 2 h (Compressed + PHT).

Heat treatment was carried out in three stages with a sealed heat treatment tank (Xinandrying 0938). In the first stage, high-temperature drying (maximum temperature: 130 °C) was performed prior to the heat treatment to approximately 0% MC. Then, heat treatment at the proposed temperature 180 °C was applied to the specimens for 2 h in atmospheric steam or 0.30 MPa pressurized steam, respectively. In pressurized-steam treatment, the pressure increased along with the temperature and reached 0.30 MPa at 180 °C. After the treatments, the lumbers were kept in the dryer until the medium temperature fell close to the room temperature.

Methods

Determination of density profiles

Sections of 50 mm (longitudinal) × 50 mm (tangential) × 20 mm (radial) were cut from the center of the specimens with or without heat treatment, respectively. They were conditioned in a controlled environment of 65 % RH and 20 °C for 4 weeks. Their densities were then measured using a cross-sectional X-ray densitometer (D-31785 Hameln) with a step of 20 μm. Sections were scanned from the top surface to the bottom surface.

Determination of set recovery

Set recovery was measured after moisture absorption at high humidity environment or immersion in water. Specimens for set recovery determination were cut from the surface-compressed wood with or without heat treatment, respectively, to obtain two pieces of specimens with the size of 10 mm (longitudinal) × 80 mm (tangential) × 20 mm (radial). The two specimens were used for moisture absorption and water absorption respectively.

For the set recovery due to moisture absorption, the specimens were treated at 40 °C under the relative humidity of 90% until the thickness of the specimen along the compression direction was constant. The thickness along the compression direction of the specimen after drying at 103 °C was recorded.

With respect to the set recovery due to water absorption, the specimens were immersed in water under vacuum for 1 h, then further soaked in water for 6 h to saturation. The specimens were dried at room temperature for 7 days and 60 °C for 1 day, then finally dried at 103 °C to constant weights. The thickness of the specimens after oven dried at 103 °C were recorded. Set recovery was calculated by Eq. 1,

$$\text{Set recovery (\%)} = [(t_r - t_c) / (t_i - t_c)] \times 100\% \quad (1)$$

where t_i , t_c , t_r , are the thicknesses (mm) of oven dried wood before compression, after compression and after moisture absorption or water absorption respectively.

Test Standards of Mechanical Properties

Specimens for mechanical tests were sawn from the compressed and control wood (Fig. 1). The MOE, MOR, hardness, and surface hardness were measured with a universal mechanical testing machine (Instron 5580) according to standards GB/T 1936.1 (2009), GB/T 1936.2 (2009), GB/T 1941 (2009), and JIS Z2101 (1994), respectively. All specimens were conditioned in a controlled environment of 65 % RH and 20 °C for 4 weeks before testing.

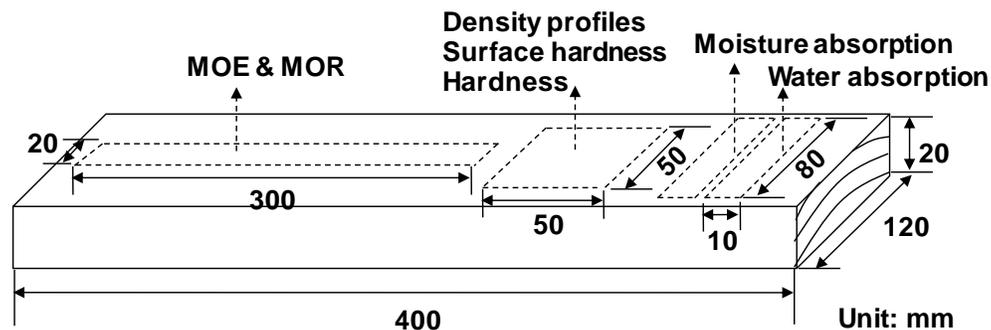


Fig. 1. Schematic diagram of specimens sawing

RESULTS AND DISCUSSION

Density Profiles

In past studies it was found that the main issue associated with the process of compression in an open system, the fixation of compressive deformation, can be overcome by hydrothermal post-treatment (Cai *et al.* 2013; Laine *et al.* 2013; Zhan and Avramidis 2017). However, a post-treatment causes wood degradation and hemicelluloses hydrolysis, which directly affects the density of wood (Rautkari *et al.* 2013). The influence of hydrothermal post-treatment on the density profile of surface-compressed wood of different compressed thickness was determined and compared with the density profile of surface-compressed specimens that were not post-treated. Results are shown in Fig. 2. The surface density peak increased from 0.49 to 1.20 g/cm³ as the compressed thickness rose to 13 mm. Also, some decrease in density can be observed in surface layers of the specimens with compressed thickness of 5 to 18 mm after atmospheric or pressurized heat treatment (Rautkari *et al.* 2013). But no changes were found after heat treatment for the control group and the mildly compressed wood with compressed thickness of 2 mm.

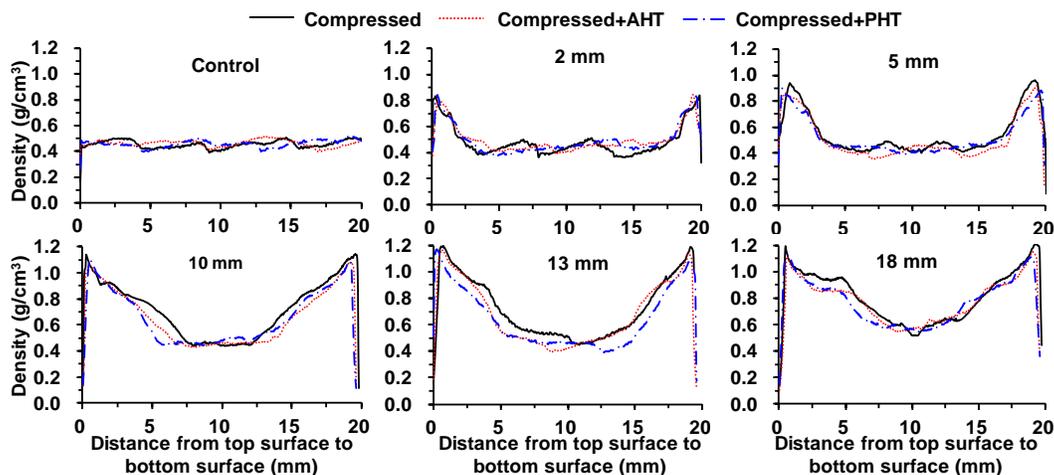


Fig. 2. Density profiles of specimens before and after heat treatment

Effect of Pressurized Superheated-steam Heat Treatment on Set Recovery

The changes of set recovery of compressed wood due to moisture absorption and water absorption are shown in Fig. 3. Without post-treatment, the set recovery of compressed wood was reduced with the increase in compressed thickness. The degree of buckling deformation of the softened cell wall increased with greater compressed thickness. Although serious delamination and rupture are not observed in the cross section of the compressed wood specimen under optical microscopy or scanning electron microscope, some microcrackings within the microfibrils occur in wood cell walls (Guo *et al.* 2015; Navi and Pizzi 2015; Chen *et al.* 2018; Li *et al.* 2018). These microcrackings may affect the release of the compressive stress, which decreases the set recovery of compressed wood due to moisture absorption or water absorption (Inoue *et al.* 1993b; Pelit *et al.* 2016). Moreover, the specimens with higher compression ratio were subjected to longer closure time, which may contribute to the decline of set recovery (Cai *et al.* 2013). Specimens with higher compression ratios also have a higher initial rebound rate when the compression is completed (Laine *et al.* 2016); that is, some internal stress release had occurred before the post-deformation fixation.

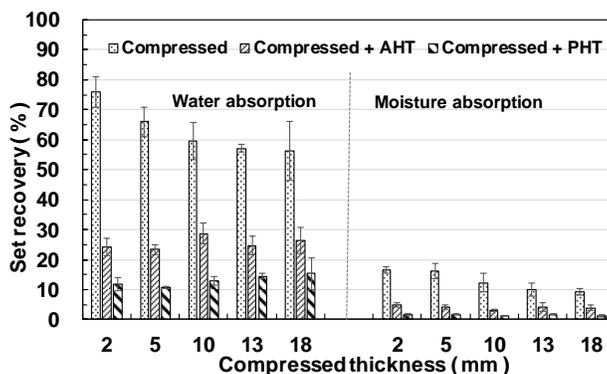


Fig. 3. Effects of compressed thickness and heat treatment on set recovery of compressed wood. The error bars represent standard deviations.

After heat treatment at 180 °C, the set recovery of the heat-treated wood due to moisture absorption or water absorption decreased remarkably. The set recovery after heat treatment was similar for specimens with different compressed thicknesses (Fig. 3). After atmospheric steam heat treatment at 180 °C, the average set recovery of compressed wood after moisture absorption and water absorption were 4.1% and 25.5%, respectively, which decreased by 68.3% and 58.9% relative to that of compressed wood without post-treatment. Zhan *et al.* (2015) and Gong *et al.* (2010) reported that the set recovery after 24 h water immersion was 16.0 to 35.0% after 180/190 °C atmospheric steam heat treatment, which is in agreement with the results in this study. Except for atmospheric heat treatment, the pressurized superheated-steam heat treatment was also carried out to fix the surface-compressed deformation in this paper, which led to mean set recovery decreasing to 1.5% and 13.1% after moisture absorption and water absorption, respectively. Comparing the samples treated in pressurized steam to those treated in atmospheric steam, the set recovery decreased by about 63.8% and 48.9% after moisture absorption and water absorption respectively. Therefore, pressurized-steam heat treatment was more effective than atmospheric heat treatment in the fixation of compressed deformation.

In a pressurized steam environment at 180 °C, the acidic degradation products accelerated the degradation of hemicellulose and changes in the cell wall structure of compressed wood (Ding *et al.* 2012). These changes led to the reduction in accessible hydroxyl groups as well as hygroscopicity of wood (Hillis 1984; Yin *et al.* 2011) and thus limited the deformation recovery of compressed wood. Structural changes including the increase of cellulose crystallinity, the increase of crystallization region width, and the crosslinking of lignin in the cell wall after pressurized superheated-steam treatment might contribute to the release of stress in compressed wood (Ito *et al.* 1998; Chen *et al.* 2018).

Effect of Pressurized Superheated-steam Heat Treatment on Mechanical Properties

The MOE and MOR of compressed wood before and after heat treatment are shown in Fig. 4. Compared with the control, the MOE and MOR before heat treatment both improved with increasing compressed thickness. After heat treatment at 180 °C, the MOE of compressed wood remained the same, but the MOR was slightly reduced. The average loss ratio of MOR was 3.24% and 5.85% after atmospheric and pressurized heat treatment, respectively, in this study. The MOR loss may be attributed to surface density slightly declining after heat treatment. Pelit *et al.* (2017) also found that the MOE and MOR of

compressed black poplar wood decreased by 0.82% and 10.36% after heat treatment at 185 °C. Moreover, the MOR of *Pinus sylvestris* dropped by more than 30.0% after saturated steam treatment at 180 °C (Rautkari *et al.* 2014). Despite the elevated temperature and increased steam pressure having a negative impact on wood mechanical properties (Gong *et al.* 2010; Ding *et al.* 2011), the post-treatment significantly reduced the set recovery of compressed wood. The results confirmed that pressurized-steam heat treatment can effectively control the set recovery; furthermore, as shown in Table 2, there were no significant effects of pressurized heat treatment or atmospheric heat treatment on the MOE and MOR of compressed wood ($P > 0.05$). The reasons for the no significant effects on MOE and MOR after heat treatment are attributed to the increased cellulose crystallinity. Another important reason is that the lower equilibrium moisture content of heat-treated wood; mechanical properties increase with decreasing moisture content (Borrega and Kärenlampi 2008b; Guo *et al.* 2014).

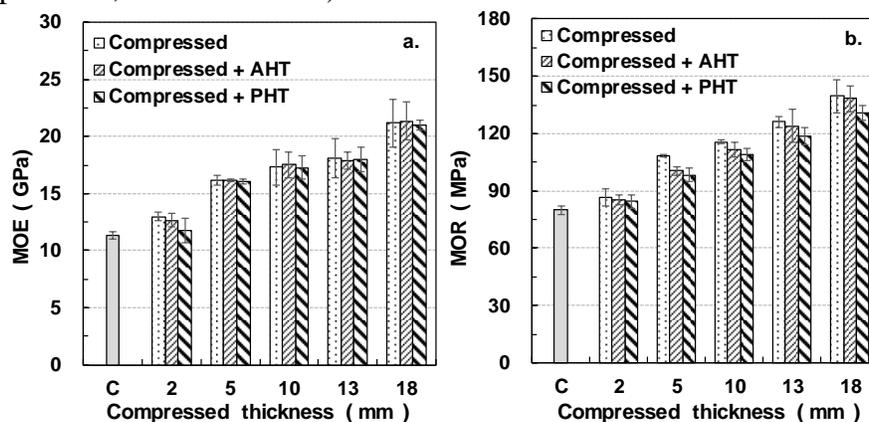


Fig. 4. MOE (a) and MOR (b) changes of compressed wood after atmospheric and pressurized heat treatment. C means control group. The error bars represent the standard deviations.

Changes in the hardness and surface hardness of compressed wood before and after post-treatment are shown in Fig. 5. As the compressed thickness increased, the hardness and surface hardness improved progressively, which can be attributed to the elevated surface density that was above 1.10 g/cm^3 as compressed thickness was 10 mm. After atmospheric heat treatment, the hardness and surface hardness of compressed wood was reduced, which was consistent with the variance of the density profile as the compressed thickness was above 5 mm (Fig. 5). The maximum hardness and surface hardness were 54.62 MPa and 17.67 MPa after pressurized heat treatment. Analysis of variance (ANOVA) revealed that the effect of atmospheric steam heat treatment at 180 °C on the mechanical properties of surface-compressed wood were not significant ($P > 0.05$), as shown in Table 2. The results in this paper were consistent with the findings of Morsing (2000) and Laine *et al.* (2016). The hardness and surface hardness of compressed wood were further reduced after pressurized superheated-steam heat treatment. The ANOVA showed that the pressurized heat treatment had no significant effect on the surface hardness of compressed wood ($P > 0.05$), but it had a significant effect on wood hardness ($P < 0.05$) while the compressed thickness was above 10 mm (Table 2). The heat transfers were much faster in higher density materials than lower ones. At the same treating condition, the high-density specimens might experience a longer time at the target high temperature (Gong *et al.* 2010). This effect might be responsible for the reduced hardness after superheated-steam heat treatment for specimens with compressed thicknesses above 10 mm.

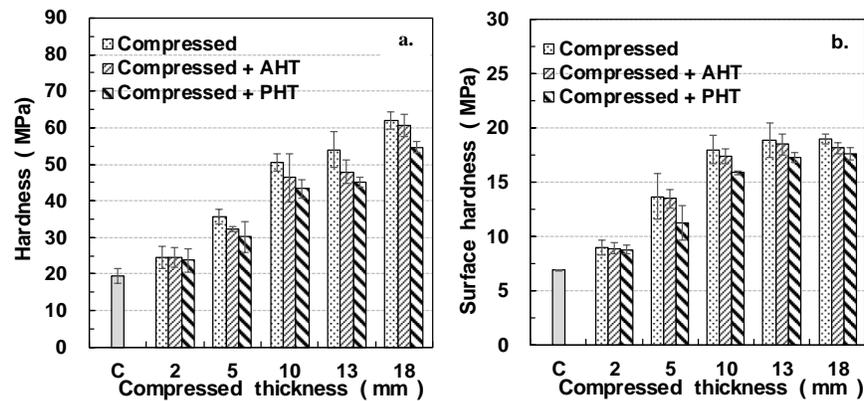


Fig. 5. Hardness (a) and surface hardness (b) changes of compressed wood after atmospheric and pressurized heat treatment. C means control group. The error bars represent the standard deviations.

Table 2. The Variance of Analysis of Mechanical Properties of Compressed Wood Before and After Heat Treatment

Compressed thickness (mm)	Compressed and Compressed + AHT				Compressed and Compressed + PHT			
	MOE	MOR	Hardness	Surface hardness	MOE	MOR	Hardness	Surface hardness
2	ns	ns	ns	ns	ns	ns	ns	ns
5	ns	ns	ns	ns	ns	ns	ns	ns
10	ns	ns	ns	ns	ns	ns	*	ns
13	ns	ns	ns	ns	ns	ns	*	ns
18	ns	ns	ns	ns	ns	ns	*	ns

Note: ns: $P \geq 0.05$; *: $P < 0.05$.

Effect of Compressed Thickness on Mechanical Properties of Compressed Wood

The effects of compressed thickness on the MOE and MOR of compressed wood after pressurized superheated-steam heat treatment are shown in Fig. 6a. The MOE and MOR of uncompressed wood were 11.3 GPa and 79.8 MPa, respectively. With a compressed thickness of 2 mm, the MOE and MOR increased slightly. As the compressed thickness increased to 10 mm (compression ratio = 33%), the MOE and MOR increased by 52.6% and 36.4%, respectively. And the maximum MOE and MOR were 21.0 GPa and 130.6 MPa respectively as the compressed thickness increased to 18 mm. Bulk compression treatment improves the wood mechanical properties (Navi and Pizzi 2015). Kitamori *et al.* (2010) found that, at the same compression ratio (33%), the MOE and MOR of the bulk compressed wood without heat treatment increased by 35.0% and 30.0%, respectively. At the same compression level, surface-compressed wood showed superior MOE and MOR compared to bulk compressed wood, mainly due to the layered density distribution of surface-compressed wood. The surface-compressed wood forms a sandwich structure with high-density layers at the top and bottom and a low-density central layer. Commonly, the surface layers are subjected to the maximum tensile or compressive stress in the bending test, and the closer to the neutral layer, the smaller the stress (Anshari *et al.* 2012; Wang *et al.* 2018). This suggested that surface compression was an efficient method to improve the mechanical properties of low-density wood.

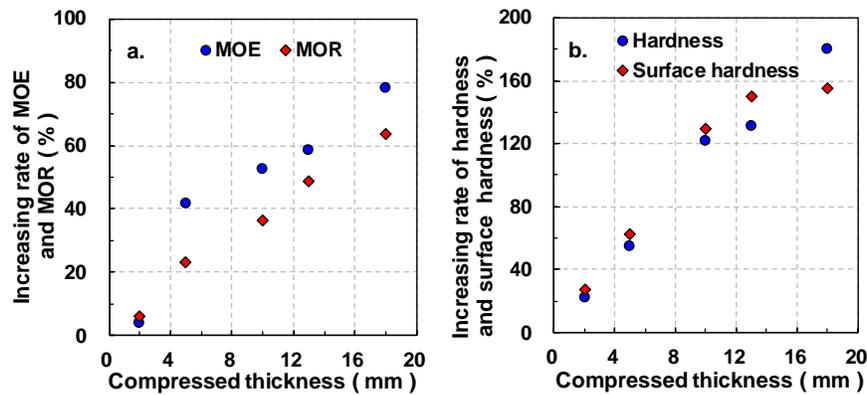


Fig. 6. Increasing rate of wood mechanical properties after compression and pressurized heat treatment, comparing to control group. a. MOE and MOR, b. hardness and surface hardness

Figure 6b. shows the effects of compressed thickness on wood hardness and surface hardness. As expected, the hardness and surface hardness of wood increased significantly with increasing compression ratio. With a compressed thickness of 5 mm (compression ratio = 20%), the hardness of compressed wood improved by 54.5% compared to uncompressed wood. This result was consistent with the results of Cai *et al.* (2013). Although pressurized heat treatment slightly reduced hardness, compared to that of control group, the hardness and surface hardness increased by 122.0% and 129.6%, respectively, with a compressed thickness of 10 mm (compression ratio = 33%). In comparison, the hardness of bulk compressed wood increased by 40.0% and 85.0% relative to uncompressed wood at compression of 20% and 33% respectively (Morsing 2000). The surface compression of wood had high-density of the surface layers, enabling surface compressed wood to resist greater pressure during the hardness test.

CONCLUSIONS

1. Surface-compressed wood was manufactured by radially compressing surface-soaked and preheated poplar wood at 180 °C. The set recovery of compressed wood due to moisture absorption was 1.5% after pressurized-steam heat treatment, which represents a decrease by about 63.2% compared to that of compressed wood after atmospheric heat treatment.
2. The MOE, MOR, hardness, and surface hardness of compressed wood gradually improved with increasing compressed thickness. The ANOVA showed that the atmospheric and 0.30 MPa pressurized heat treatment at 180 °C had no significant effect on the MOE, MOR, and surface hardness of compressed wood. With a compressed thickness of 10 mm (compression ratio = 33%), MOE, MOR, hardness and surface hardness increased by 52.6%, 36.4%, 122.0%, and 129.6%, respectively, compared to the control group.
3. Compared with the control, the mechanical properties of the white poplar wood as well as the dimensional stability were greatly improved after surface compression and 0.30 MPa pressurized steam heat treatment.

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