

High-Pressure Treatment of Chinese Fir Wood: Effect on Density, Mechanical Properties, Humidity-Related Moisture Migration, and Dimensional Stability

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A new densification technique for industrial uses of plantation wood was studied. Chinese fir wood was treated by high pressure (HP) at 50 to 200 MPa for 5 min. The density and mechanical properties, moisture sorption isotherm (MSI), and dimensional changes of the pressure-treated wood under various relative humidity (RH) storage conditions were evaluated. The densities of HP-treated wood ranged from 0.79 ± 0.01 g/cm³ after treatment at 50 MPa to 0.92 ± 0.02 g/cm³ at 200 MPa, which was significantly higher ($p < 0.05$) than that of the control (0.35 ± 0.01 g/cm³). Hardness values in radial and tangential fiber alignment faces also significantly increased by 370% to 470% and 350% to 460%, respectively, as compared with the control. The modulus of elasticity and the modulus of rupture of pressure-treated wood increased by 48% to 88% and 89% to 170%, respectively. The equilibrium moisture content varied with RH, decreasing slightly at 33% and 52% while significantly increasing ($p < 0.001$) at 86% and 93% RH. Radial, tangential, and volumetric dimensions of densified wood were relatively stable at 33%, 52%, and 67% RH, while remarkable swelling occurred at 86% and 93% RH.

Keywords: High-pressure treatment; Chinese fir wood; Density; Mechanical properties; Moisture sorption isotherm; Dimensional changes

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INTRODUCTION

Chinese fir [*Cunninghamia lanceolata* (Lamb.) Hook] is an industrial plantation species with a short rotation cycle that has a high survival rate, durability, and high productivity (Yu 2000). However, the wood from Chinese fir plantations is very soft, light, has low strength, and is easily abraded and unstable, which considerably limits its commercial utilization (Song 2000). The key for successful commercial utilization of this fast-growing wood resource depends primarily on improving the density and mechanical properties.

Wood properties such as hardness and bending strength generally correlate positively with density; therefore, wood species with higher density are usually more desirable than others (Laine *et al.* 2016). Published research shows that the density of the cell wall is roughly the same (1.5 g/cm³) regardless of the wood species or cell type

(Kellog and Wangaard 1969). However, the density of the wood material is dependent on the cell wall thickness and size of lumen, and the density is generally increased by reducing the void space through various densification processes, which will also help to improve the mechanical properties.

There are several densification methods that are used to improve various physical and mechanical properties of wood (Kamke and Sizemore 2008; Rautkari *et al.* 2008, 2009; Rautkari and Hughes 2009; Gabrielli and Kamke 2010). These methods are generally composed of the same steps: compressing the wood between heated metal plates to a desired thickness and fixing the deformation in the compressed state under suitable moisture and temperature conditions. He *et al.* (2008) and Kutmar and Kamke (2012) have used this method to obtain compression wood after a softening treatment, and the modulus of rupture (MOR) and the modulus of elasticity (MOE) of compressed wood increased by 20% and 62% at the compression ratio of 50%. Gao *et al.* (2016) developed this method by adjusting the moisture and temperature distributions within the lumber. Using this developed method, the MOE and MOR values of compressed poplar wood could be increased by 73.2% and 88.9% at the compression ratio of 47%. In addition, two typical after-treatments to fix the compressed state – thermal modification (Hill 2006; Rautkari *et al.* 2011; Laine *et al.* 2016) and impregnation with low molecular weight plasticizers (Deka and Saikia 2000; Gabrielli 2010; Hosseinpourpia *et al.* 2016), introduced in subsequent research – can reduce set-recovery. However, the resins used for this purpose can affect both the environment and human health, and thermal modification requires a lot of energy. Another novel technology for wood densification is CaLignum (Blomberg *et al.* 2005), which is through semi-isostatic compression in a Quintus flexform press. This process could give compressed wood irregular shape but homogenous density. The pressure rises successively up to 140 MPa and is then immediately lowered to atmospheric pressure; the process will take about 3 min. No heating needed (Blomberg 2005).

Compaction procedures have also been widely used for making densified materials for various purposes, such as biofuels, pulp, and fiber boards for furniture. Araújo *et al.* (2016) used wood particles of *Eucalyptus grandis* and *Eucalyptus* spp. to form briquettes using pressing at 120 °C for 7 min and 6 min cooling time, under pressures of 6.9, 10.3, and 13.8 MPa. The briquetting compacting pressure showed no significant influence on the briquette's properties. Seki *et al.* (2016) used 33 to 218 MPa extrusion force to form wood flow obtained using wood impregnated with low-molecular weight phenol formaldehyde (PF) resin. Rhén *et al.* (2005) used high pressure (46 to 114 MPa) combined with temperature (26 to 144 °C) and moisture content (6.3% to 14.7%) to form Norway spruce sawdust pellets. It was found that high compression strength was strongly correlated with the density of the pellets obtained.

In this study, a novel wood densification method, high-pressure (HP) treatment, was introduced. The HP technology is a non-thermal processing technique by which products, already sealed in their package, are introduced into a vessel and subjected to a high level of isostatic pressure, which is transmitted by water. This technology is widely used in the food industry (Norton and Sun 2007; Balasubramaniam *et al.* 2016). According to our preliminary experiments, there are five distinct advantages in using this technology for densification of wood: 1) A much higher range of 100 to 600 MPa isostatic hydrostatic pressure can be employed, more than the traditional compressed pressure; 2) There is no need for thermal treatment, saving a lot of energy; 3) The process

time is very short, as the complete cycle time is not more than 20 min; 4) Densification occurs uniformly from all sides, rather than the unilateral compaction used in most traditional cases, and the density is uniform; and 5) There is a low set-recovery rate for densified wood. A patent has been applied for in China for using HP technology for densification of wood (Yu *et al.* 2016).

As discussed above, there is a scarcity of information related to the use of HP technology for densification of wood. The aim of this study is to illustrate the effect of HP treatment on wood densification and to evaluate the resulting mechanical properties of Chinese fir. Moreover, the equilibrium moisture content (EMC) and dimensional stability under various relative humidity storage conditions at room temperature (25 °C) were also evaluated to determine the moisture stability of the product.

EXPERIMENTAL

Materials

Fast-growing Chinese fir logs with diameters of 75 ± 5 mm were harvested from a plantation forest located in Taizhou City, Zhejiang province, China. All logs were peeled first (removal of bark); then, 300-mm-long disks with no abnormalities (defects) were prepared. The softwood was then air-dried at room temperature (25 °C) for two months to reach about 12% moisture content (dry basis). A total of 25 specimens, with no visible drying defects, were cut to 300 mm in length and 75 ± 5 mm in diameter. These were then wrapped in a plastic bag and stored at room temperature until use.

Methods

High-pressure equipment

The HP treatments were executed in laboratory-scale high-pressure equipment. Figure 1 is a schematic illustration of the high-pressure treatment process. Figure 2 is a photograph of the high-pressure operation unit. The system consists of a HP unit (UHPF-750, 5L, Kefa, Baotou, China) equipped with a K-type thermocouple (Omega Engineering, Stamford, CT, USA) and a data logger (34970A, Agilent Technologies GMBH, Germany) for temperature measurement, as well as a thermostat jacket connected to a water bath (SC-25, Safe, China) for maintaining the processing temperature. The intensifier used for generating the pressure is a batch-type unit, which builds up the pressure in a stepwise ladder-like process. Water was used as the pressure transmitting medium in this study, and the pressure vessel was maintained at 25 °C before pressurization. Normally, the sample temperature is expected to increase by 3 °C for every 100 MPa pressure rise because of adiabatic compression. However, to minimize this adiabatic heating, a low rate of pressurization was maintained (~100 MPa/min) so that the sample temperature was easily equilibrated to the set point temperature of 25 °C. The pressure release time was kept at less than 5 s.

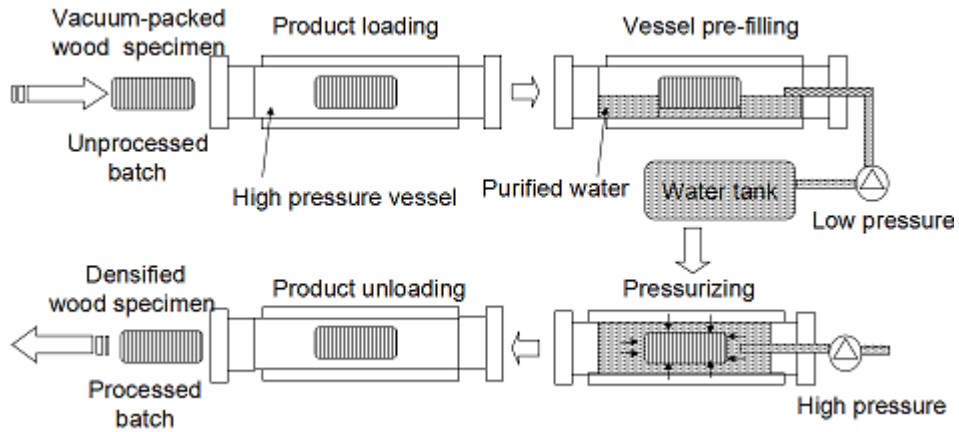


Fig. 1. Schematic illustration of the high-pressure densification process

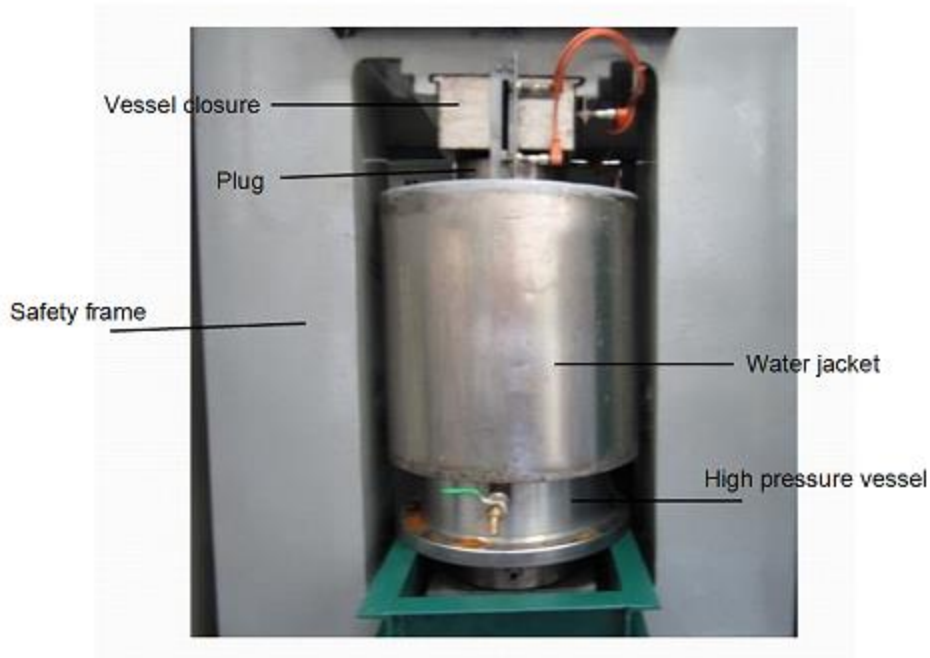


Fig. 2. Photograph of the high-pressure operation unit

High-pressure treatment

Wood specimens prepared as detailed earlier were separated into five groups as follows: one for control, and one each for four HP treatments. Each treatment was performed using one piece of wood at a time, and then repeated again with other samples to obtain five replicates. For HP treatment, the wood specimens were packed in a polythene bag, vacuum sealed, and then placed into the HP chamber. The treatment time was 5 min at four different pressure levels (50, 100, 150, and 200 MPa). Each pressure treatment was replicated five times. Wood specimens without HP treatment were used as control samples.

Measurement of Densities and Mechanical Properties

After HP treatment, the wood specimens were uniformly compressed. All specimens were conditioned in a controlled environment of 65% RH and 20 °C for 4 weeks before measuring the mechanical properties. Then one compressed wood specimen was cut into one specimen with the size of 300 mm (longitudinal) × 20 mm (tangential) × 20 mm (radial) for MOR and MOE measurement, and several specimens with size of 20 mm (longitudinal) × 20 mm (tangential) × 20 mm (radial) for density measurement and the experiment of equilibrium moisture content, and one specimen with size of 35 mm (longitudinal) × 25 mm (tangential) × 25 mm (radial) for hardness measurement.

The modulus of rupture (MOR) and modulus of elasticity (MOE) were determined by a three-point bending method according to ISO 3133 (1975).

Approximately 15 to 20 small wood specimens were determined according to the ISO 3131 (1975) standard. The wood density was calculated using the following equation (Eq. 1),

$$\rho_w = \frac{m_w}{v_w} \quad (1)$$

where ρ_w is the density of the wood specimen; m_w is the mass of the wood specimen; and v_w is the volume of wood specimen, all at moisture content w .

The hardness measurements were performed according to ISO 3350 (1975). This was principally calculated using the area of the surface that a metal ball of (5.64 ± 0.01) mm radius created on the wood and the applied force, from the following equation (Eq. 2),

$$H_{12} = KP \quad (2)$$

where H_{12} is the hardness of the wood specimen at 12% moisture content; P is the applied force (N); and K is the coefficient of radius for the metal ball indenter at a depth of 5.54 mm, which is 1.

Equilibrium Moisture Content and Dimensional Stability

The moisture sorption isotherm (MSI) experiments were conducted with groups of 25 matched cubic samples of 20 mm (longitudinal) × 20 mm (tangential) × 20 mm (radial) of densified and untreated wood each. The sorption behavior was investigated using static and dynamic methods at a constant temperature of 25 °C (Hosseinpourpia *et al.* 2016). Saturated inorganic salt solutions can be used to maintain stable relative humidity (RH) conditions. In this study, five constant RHs (33%, 52%, 69%, 86%, and 93%) were provided by five saturated inorganic salt solutions (MgCl₂, Mg(NO₃)₂, KI, KCl, and KNO₃ solutions). During the sorption experiments, five wood specimens were placed in a basket within a hermetic jar. Each pressure treatment group involved five of these jars. These small sorption chambers were placed in temperature-controlled incubators kept at 25 ± 1 °C, allowing a precise RH control. A similar procedure has previously been described by Passarini and Hernández (2016). Wood specimens were weighed periodically until the change in mass was negligible (at least < 0.01% per day), and under this condition, it was assumed that the EMC was reached. Once EMC had been reached, the samples were individually weighed using an analytical balance to the nearest 0.0001 g to determine the EMC of each sample precisely. Immediately, the radial and

tangential dimensions were measured with a micrometer to the nearest 0.01 mm. Dimensional measurements were used to calculate, for each EMC, the radial, tangential, and volumetric swellings.

Equilibrium relative humidity (ERH) was then taken as the RH of the jar (specific for each of the salts used in the experiment), and the MSI diagram was prepared by plotting EMC vs. ERH.

Statistical Analysis

All measured values are represented as mean \pm standard error. Results were compared using one-way analysis of variance (ANOVA). A Duncan's multiple range test was conducted to assess the significant differences among experimental mean values ($p < 0.05$). All statistical computations and analyses were conducted using SPSS version 20.0 (IBM, America).

RESULTS AND DISCUSSION

Densification

The typical cross section of densified wood is shown in Fig. 3. Compared with the control sample (with an oval cross section), the cross section of densified wood represented an irregular shape (very small deformation in longitudinal direction). The diameter of densified wood was notably reduced compared with the control. Moreover, the heartwood and sapwood were found to be identically compressed after densification.



Fig. 3. Cross sections of Chinese fir wood: control (left) and densified wood at 50 MPa for 5 min (right). Heartwood and sapwood of Chinese fir had identical compression. The cross section represented an irregular shape (very small deformation in longitudinal direction) after densification.

The densities of Chinese fir after HP treatment under various pressure conditions for 5 min are presented in Table 1. The densities of wood samples were significantly enhanced by HP treatment at different pressure levels, with an increase of 126% (at 50

MPa), 163% (at 100 MPa), 154% (at 150 MPa), and 143% (at 200 MPa) compared with the control density. The highest density was achieved with HP treatment at 100 MPa ($0.92 \pm 0.02 \text{ g/cm}^3$), while treatments from 50 to 100 MPa resulted in an approximately 25% increase in density. Pressure values beyond 100 MPa decreased the density of treated wood. This could be related to the oil/moisture from the wood, which oozed out when the pressure values were higher than 100 MPa. Such events had to decrease wood properties due to mass loss.

Table 1. Densities and Mechanical Properties of Densified and Untreated Wood

Pressure (MPa)	Density (g/cm^3)	Hardness values (kN)		MOE (GPa)	MOR (MPa)
		Radial face	Tangential face		
0.1 (control)	0.35±0.01 a	1.16±0.09 a	1.27±0.06 a	9.81±1.49 a	65.9±7.73 a
50	0.79±0.01 b	5.42±0.31 b	6.32±0.75 b	14.52±2.16 b	124.62±15.32 b
100	0.92±0.02 d	8.59±0.49 d	9.17±0.25 d	18.47±1.86 d	179.53±28.51 d
150	0.89±0.02 c	8.03±0.35 c	8.75±0.17 c	17.68±1.69 c	162.98±21.34 c
200	0.85±0.03 c	7.85±0.26 c	8.42±0.23 c	16.73±2.03 c	160.25±19.86 c

Measured values are represented as mean \pm standard error of five replicates. Letters represent difference levels.

As noted earlier and can be visualized in Fig. 3, the compression of heartwood and sapwood were uniform and the directional effect dominated, indicating that the void spaces in Chinese fir were uniformly compressed, with no density gradient generated. This result was similar with those of Blomberg and Persson (2004), who used a novel method, CaLignum, for wood densification. This method could give compressed wood homogenous density, and avoid unfavorable density gradient effect. In contrast, density gradient effect is typical for common methods, which higher density is generated near the surface and lower toward the center of board (Gao *et al.* 2016; Laine *et al.* 2016). These may be related to the much higher pressure levels used in HP treatment. Gao *et al.* (2016) employed a sandwich compression of wood with 6 MPa pressure, which is far less than the lowest pressure level (50 MPa) used in this study. Another distinct advantage of using HP compaction as used in this study is the relatively short processing time (5 min) as compared with long compaction processing times (0.5 to 5 h) in traditional methods (Morsing 2000; Kamke 2006; Kutnar and Kamke 2012; Gao *et al.* 2016; Laine *et al.* 2016). Furthermore, in most traditional methods, the process requires long treatment times at elevated temperatures (mostly above 150 °C) which indicates that the HP compression of wood for densification not only saves time but also saves energy (no need for heating during processing).

Mechanical Properties

As expected, the hardness values of the tangential and radial faces were significantly increased by densification (Table 1). The average hardnesses of the radial and tangential faces for untreated wood specimens were 1.16 ± 0.09 and 1.27 ± 0.06 kN, respectively, while the hardnesses of the radial and tangential faces for samples treated at 50, 100, 150, and 200 MPa were significantly increased, by 367%, 468%, 419%, and 404%, respectively, for the radial face and 350%, 464%, 431%, and 405%, respectively, for the tangential face. The hardness values of the radial and tangential faces of wood

densified at 100 MPa were significantly higher than those for the other pressure levels, showing a similar trend with density. For both untreated and treated wood, the hardness of the tangential face was higher than that of the radial face. This hardness increase in densified wood could open up new opportunities in, for example, the flooring industry. Kutnar *et al.* (2008) used a surface densification method, which was to bulk compress the thin boards and combine these with untreated wood using an adhesive, to increase the hardness of densified wood, quite different from the HP densification.

The MOE and MOR values of densified wood at various pressure levels are also shown in Table 1. The HP treatment could significantly increase the MOR and MOE of Chinese fir wood. At 100 MPa, the MOE increased by 88.3% and MOR increased by 172%, compared with that of untreated Chinese fir. At pressures greater than 100 MPa (150 MPa and 200 MPa), the MOE and MOR decreased with increasing pressure. MOR and MOE values have been shown to significantly increase with density (Liu and Zhao 2004; Gao *et al.* 2016). In this study, the MOE and MOR of wood densified at 50, 100, 150, and 200 MPa increased by 48.0%, 88.3%, 80.2%, and 70.5%, respectively, and 89.1%, 172%, 147%, and 144%, respectively.

These MOE and MOR increments after densification by HPP were higher than that of traditional compression wood, in which wood MOR and MOE increased by 20% and 62% at a compression ratio of 50% (He *et al.* 2008; Kutnar and Kamke 2012), or the MOE and MOR of compressed poplar wood could increase by 73.2% and 88.9% at a compression ratio of 47.0% by adjusting the moisture and temperature distributions within the lumber (Gao *et al.* 2016). The densification by HPP significantly improved the mechanical properties of Chinese fir.

Equilibrium Moisture Content

The equilibrium moisture contents (EMCs) of treated and untreated Chinese fir wood samples at various relative humidities (RH) are presented as bar graphs in Fig. 4.

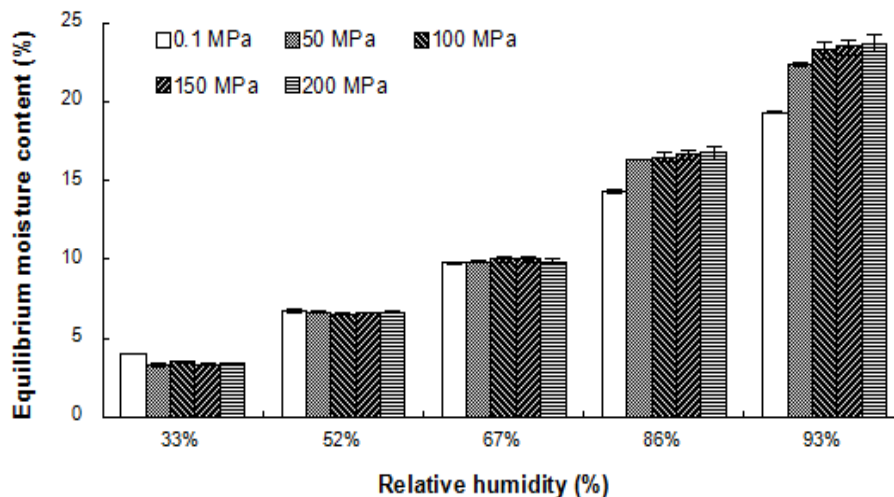


Fig. 4. Moisture Sorption Isotherm: equilibrium moisture contents (EMC) of HP densified and control (0.1 MPa) samples of Chinese fir wood at various relative humidities at 25 °C (error bars represent standard deviation)

The HP treatment at different pressure levels reduced the EMCs at 33% and 52% RH, which were at a range of 3.28% to 3.48% and 6.53% to 6.65%, respectively (almost doubled at 52% RH compared with that at 33% RH), with no significant differences from the control. However, the EMC increased dramatically ($p < 0.05$) at 86% and 93% RH, to 16.3% to 16.7% and 22.4% to 23.7%, respectively, also showing significant increases compared with the control. At 67% RH, the HP treatment at different pressure levels slightly increased the EMC. The EMCs of Chinese fir densified at various pressure levels and stored at various RH were not significantly ($p > 0.05$) different, except at 86% and 93% RH. The EMC of Chinese fir compressed at 50 MPa was significantly lower ($p < 0.05$) than that of other pressure levels.

These results indicate that pressure could significantly increase the permeability of water in wood, allowing a reduction of their moisture content at low RH and increasing it at higher RH, which is somewhat similar to results reported by Iida (1992) and Iida *et al.* (2002). Their results showed that, with a conventional compression method, the compressive deformation process resulted in separating and destroying aspirated pits in the wood, leading to increased liquid uptake during the recovery process of the deformation because of volumetric pressure. These results also indicate that the pre-compression treatment method increased the penetration level. In our pre-experiment, a water absorption test showed that the water absorption of wood samples densified by HP treatment was 2 or 3 times higher than that of untreated wood (data not shown). Combining with an observation about overflowing of gases from the wood after densification by HP treatment, a reasonable speculation can be derived, that the HP treatment destroyed the structural integrity of the wood by collapsing the cells and expelling the entrapped air from the void spaces. Following the HP treatment, the hygroscopic sugar polymers (hemicellulosic constituents and cellulose) (Fengel and Wegener 1989) in densified wood may absorb moisture from environment, resulting in increased degrees of moisture absorption. This process is commonly recognized as hysteresis, which contributes to the differences in the moisture sorption isotherms between desorption (drying) and adsorption (rehydration) behaviors commonly seen in drying applications.

Dimensional Changes

Dimensional changes in the control (0.1 MPa) and HP densified Chinese fir wood samples at various RH levels after EMCs of wood was reached are shown in Fig. 5. The volumetric swelling of densified wood is obvious at 86% and 93% RH, while the control kept its original shape at various RH levels. In addition, the edges of densified wood specimens showed only slight deformations at 33%, 53%, and 67% RH, compared with the control, indicating a lower sensitivity of densified wood to moisture.

The radial (Fig. 6(a)), tangential (Fig. 6(b)), and volumetric (Fig. 6(c)) dimensions of Chinese fir wood specimens densified by HP treatments followed by storage at various RH levels, are presented in Fig. 6. Radial, tangential, and volumetric dimensions of both untreated (control) and treated wood samples significantly increased as RH increased ($p < 0.05$). The change in trends observed at various RH levels for radial, tangential, and volumetric swelling of treated Chinese fir wood samples were similar to those described earlier for EMC, but mostly occurred at 86% and 93% RH ($p < 0.01$) compared with the control, while no significant differences ($p > 0.05$) occurred in treated and untreated

wood stored at 33%, 52%, and 67% RH conditions. At 33% RH, the HP treatment slightly reduced the radial, tangential, and volumetric dimensions of Chinese fir wood.

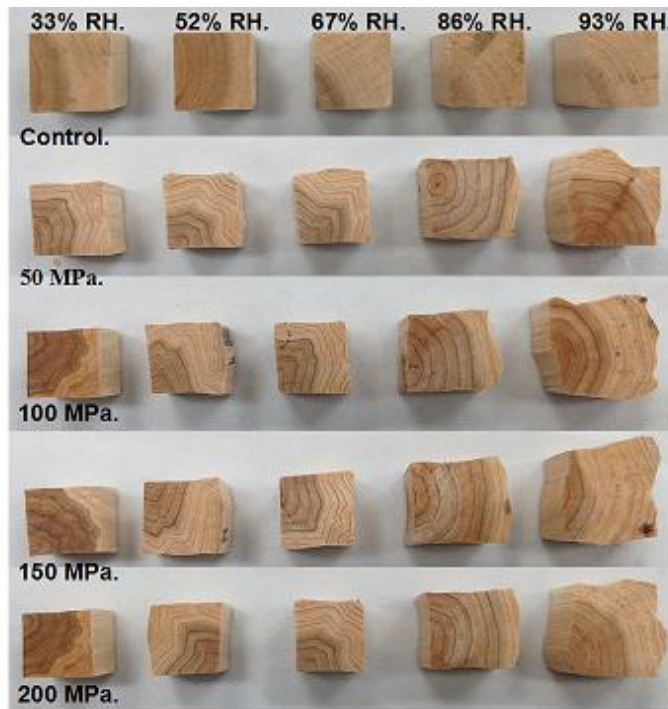


Fig. 5. Dimensional changes in the control (0.1 MPa) and HP densified Chinese fir wood samples at various relative humidities at 25 °C after a month when EMCs of wood was reached. The dimensions of all densified and control wood specimens were 20 mm (longitudinal) × 20 mm (tangential) × 20 mm (radial) mm³ before the moisture sorption experiments.

The effect of different pressure levels on radial, tangential, and volumetric dimensional changes were similar at different RH conditions, except at 93%. Again, the radial, tangential, and volumetric dimensional swelling at 50 MPa at 93% RH was significantly lower than that at other pressure levels. Another finding was that the tangential swelling of densified wood was larger than radial swelling.

A major finding from these results was that the recovery rates in radial, tangential and volumetric dimensions were small at low or moderate RH conditions (Figs. 5 and 6) after nearly a month of balance time, which was quite different from traditional thermo-compression methods (Mao *et al.* 2009; Gao *et al.* 2016; Laine *et al.* 2016). The high recovery rates of wood densified by HP treatment had a close relationship with high RH in the environment. These results indicate that the dimensional stability should be improved by preventing the passage of moisture into the wood specimen. In our preliminary experiment, the volume of Chinese fir densified by 100 MPa treatment for 5 min was reduced by 2.78% when preserved in an air-conditioned room for three months (data not shown). It has been previously found that higher compression ratio leads to greater immediate spring back because higher inner stresses are created during traditional compression (Gong *et al.* 2006). These observations necessitate further studies, perhaps involving some surface modification pretreatments for the wood prior to HP densification or impregnating hydrophobic substances in the wood during the HP treatment.

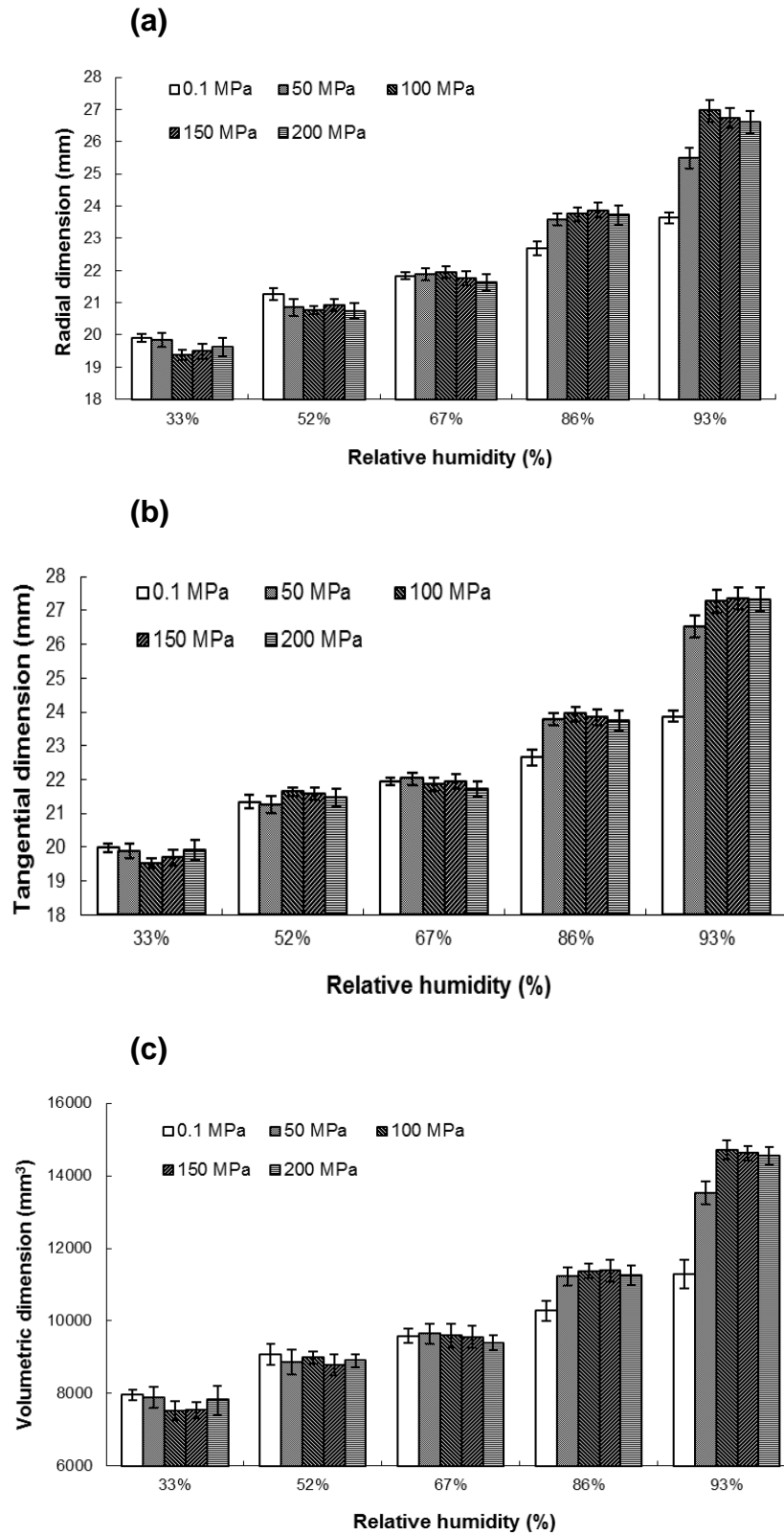


Fig. 6. The (a) radial, (b) tangential, and (c) volumetric dimensions of HP densified and control (0.1 MPa) Chinese fir wood samples at various relative humidities at 25 °C (error bars represent standard deviation)

CONCLUSIONS

1. HP treatment has a great potential for plantation wood densification applications. The density and mechanical properties of wood samples could be significantly increased by 2 or 3 times within a short treatment time using the HP densification process.
2. The optimum compression pressure was 100 MPa. Higher pressure treatment decreased density and mechanical properties of wood.
3. Pressure treatment significantly increased the permeability in wood, making it more susceptible to dimensional and water exchange activities when stored at various RH environments. These results indicate significantly low recovery rates at low or moderate RH levels, but extreme and dimensionally unstable conditions at high RH values.

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