

## Effect of Hot Pressing Temperature on the Density Profile of Compressed Solid Wood

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To expand the applications of low-density wood, solid white poplar (*Populus tomentosa*) wood with a moisture content of 10.6% was compressed 5 mm radially at 90 °C, 120 °C, 150 °C, 180 °C, and 210 °C, and the density profile characteristics were analyzed. It was found that compressed wood with a non-uniform density profile contributed to the peak density and average density of the compressed layer, where the highest values were 0.87 g/cm<sup>3</sup> and 0.72 g/cm<sup>3</sup>, respectively. The density peak and compressed layer were formed at various distances from the wood surface according to the pressing temperatures tested. It was concluded that the pressing temperature had a great effect on the density profile of the compressed wood and the pressing temperature effectively controlled the shape of the density profile.

*Keywords:* Compressed solid wood; Pressing temperature; Density profile

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### INTRODUCTION

Wood density is known to correlate with the mechanical properties; when the density is higher, the mechanical properties values are higher. High-density wood is demanded to a greater degree. Many attempts have been made to develop a suitable process for preparing high-density wood, one of which is thermo-hydro-mechanical (THM) treatment (Navi and Girardet 2000; Blomberg and Persson 2004). The THM treatment is an effective process that increases the density of wood using heat, moisture, and mechanical compression perpendicular to the grain. In this way, the mechanical properties of low-density wood can be improved and the range of applications of low-density wood can be expanded without otherwise changing the wood characteristics.

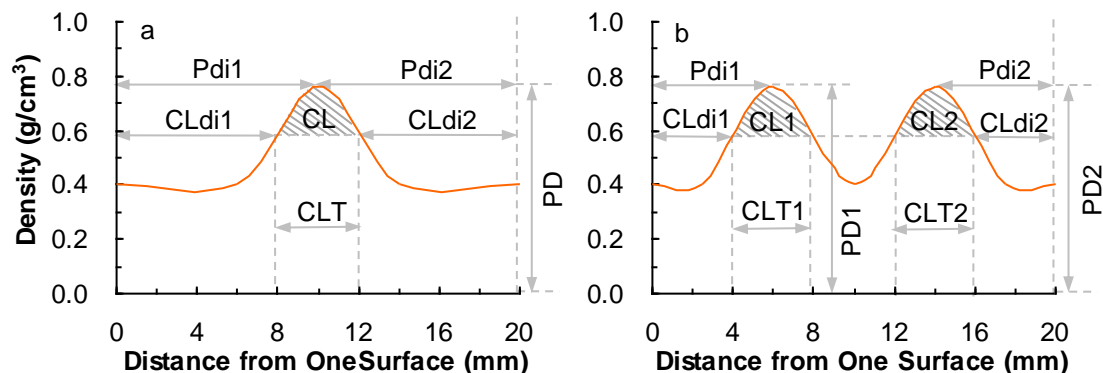
The hot pressing temperature is an important process parameter during THM because the temperature and moisture distributions through the lumber are affected by the hot pressing temperature. For wood with the same moisture content of 100%, the temperature at the surface increases more quickly than at the center, which forms a notable temperature gradient; thus, different temperature gradients are formed at different hot pressing temperatures (Beard *et al.* 1983, 1985). Also, the hot pressing temperature affects the moisture diffusion coefficient, and thus different pressing temperatures can lead to different moisture gradients (Fotsing and Tchagang 2005; Hřčka *et al.* 2008). The variations of moisture gradients under high temperatures were studied and discussed in a numerical study on the hydro-thermal behaviour of wood under surface densification (Fortino *et al.* 2013).

The softening behavior of wood is dependent upon the temperature and moisture. There are two different behavior states, which are called the glassy and rubbery states.

Wood softening is mainly thought to be dependent upon the main chemical components in wood and that the softening properties of each component are different from each other. For cellulose, the softening temperature is 200 °C to 250 °C and is not affected by the moisture content (Uhmeier *et al.* 1998). In contrast, the softening temperatures of hemicellulose and lignin are sensitive to the moisture content (Hillis and Rozsa 1978). For hemicellulose, the softening temperature is 200 °C at a 0% moisture content, 100 °C at a 20% moisture content, and 20 °C at a 60% moisture content. For lignin, the softening temperature is 150 °C at a 0% moisture content and 80 °C at a 20% moisture content, and then the softening temperature decreases only slightly when the moisture content is 60% (Furuta *et al.* 2010). Temperature and moisture contents also affect the properties of the cell wall layers and the consequent response of wood under compression (Fortino *et al.* 2015).

Because of the temperature and moisture gradients formed during hot pressing and their relationship to wood softening where the compression stress is applied, a thickness density profile is created in the compressed wood, which varies with the pressing temperature (Rautkari *et al.* 2011). Density profile analysis is a general method applied to wood-based panels, such as particleboard and medium density fiberboard, and has not been widely applied to compressed solid wood. Research has mainly been performed with the aim of compressing the whole thickness or to increase the density of only the first few cell layers. The shape of the density profile affects the properties of the end-product, such as the hardness and bending strength (Wong *et al.* 1998, 1999, 2000); thus, it is important to control the density profile formation to optimize the properties.

The purpose of this experiment was to study the effects of the pressing temperature on the formation of density profiles in compressed solid wood. The density profile characteristics were analyzed. The density profile characteristics included in the analysis were the average density, peak density, peak distance, compressed layer distance, and compressed layer thickness (Fig. 1).



**Fig. 1.** Examples of the density profile that show the density profile characteristics: (a) one compressed layer and (b) two compressed layers; PD – peak density; Pdi – peak distance; CL – compressed layer; CLdi – compressed layer distance; and CLT – compressed layer thickness

## EXPERIMENTAL

### Materials

Twenty-five-year-old white poplar (*Populus tomentosa*) trees with a diameter of 25 cm to 35 cm at breast height were harvested from a plantation forest in Guan county,

Shandong province, China. Lumber with the dimensions 500 mm (longitudinal) × 150 mm (tangential) × 50 mm (radial) was cut from these poplar logs. After the lumber samples were kiln-dried, they were further machined into sapwood specimens (MC = 10.6%) with the dimensions 400 mm (longitudinal) × 110 mm (tangential) × 25 mm (radial) for hot pressing in the radial direction.

## Methods

### *Hot pressing process*

The sapwood specimens were preheated at 90 °C, 120 °C, 150 °C, 180 °C, and 210 °C for 22 min. After the preheating process, intermittent compression was applied to the preheated specimens at a pressure of 6.0 MPa. Each intermittent compression cycle consisted of a 3-s compression time and a 30-s unloading time. The specimens were compressed from the original thickness of 25 mm (radial) to a target thickness of 20 mm with 10 compression cycles. The final compression thickness of 20 mm was maintained at the pressing temperature for 30 min. Then, the heated platens were cooled with a water-cooling system to attain a temperature below 100 °C before releasing the load. Three replicates were analyzed for each pressing temperature.

### *Density profile*

Before preparing the test samples, the compressed wood was conditioned at 20 °C and a 65% relative humidity (RH) to obtain a constant moisture content. All of the samples with a dimension of 50 mm (longitudinal) × 50 mm (tangential) × 20 mm (radial) were cut from the middle of the width of the compressed wood for the density profile tests. The density profiles of the thermally compressed and untreated control specimens were determined by using an X-ray density profiler (DENSE-LAB X, Electronic Wood Systems GmbH, Hameln, Germany) with a step of 20 μm.

### *Morphological structure*

The poplar wood control, wood compressed at 180 °C, and compressed layers of the wood compressed at 90 °C, 120 °C, 150 °C, and 210 °C were analyzed with a scanning electron microscope (SEM) (S-3400N, Hitachi, Tokyo, Japan) to investigate the morphological structure changes. Specimens were cut from the transverse section.

### *Analysis of the compressed layer*

The average and maximum densities (65.0% RH and 20 °C) of the poplar wood control were 0.44 g/cm<sup>3</sup> and 0.49 g/cm<sup>3</sup>, respectively. The areas with a density 20.0% higher than the maximum density of the control were defined as compressed layers.

### *Proportion of the compressed layer thickness*

The proportion ( $P$ ) was calculated using Eq. 1,

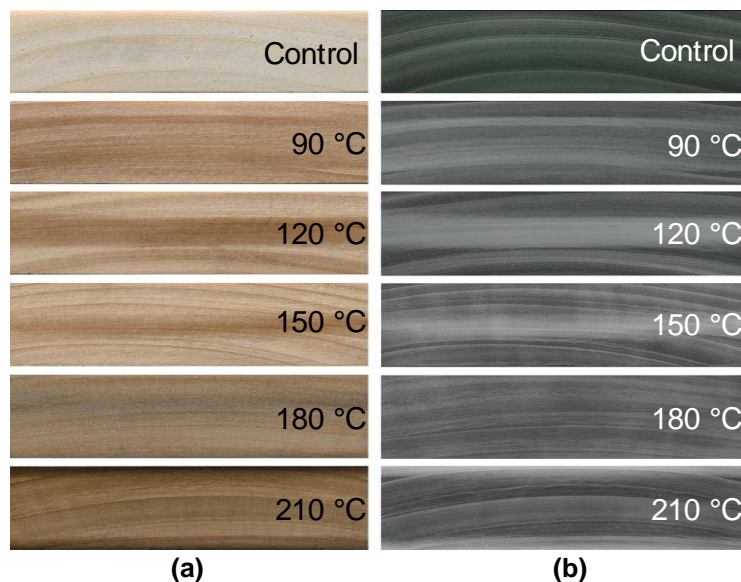
$$P (\%) = (CLT / CWT) \times 100\% \quad (1)$$

where  $CLT$  is the thickness of the compressed layer (mm),  $CLT$  is the sum of the thicknesses when there were two compressed layers (mm), and  $CWT$  is the thickness of the compressed wood (mm).

## RESULTS AND DISCUSSION

### Formation of the Compressed Wood

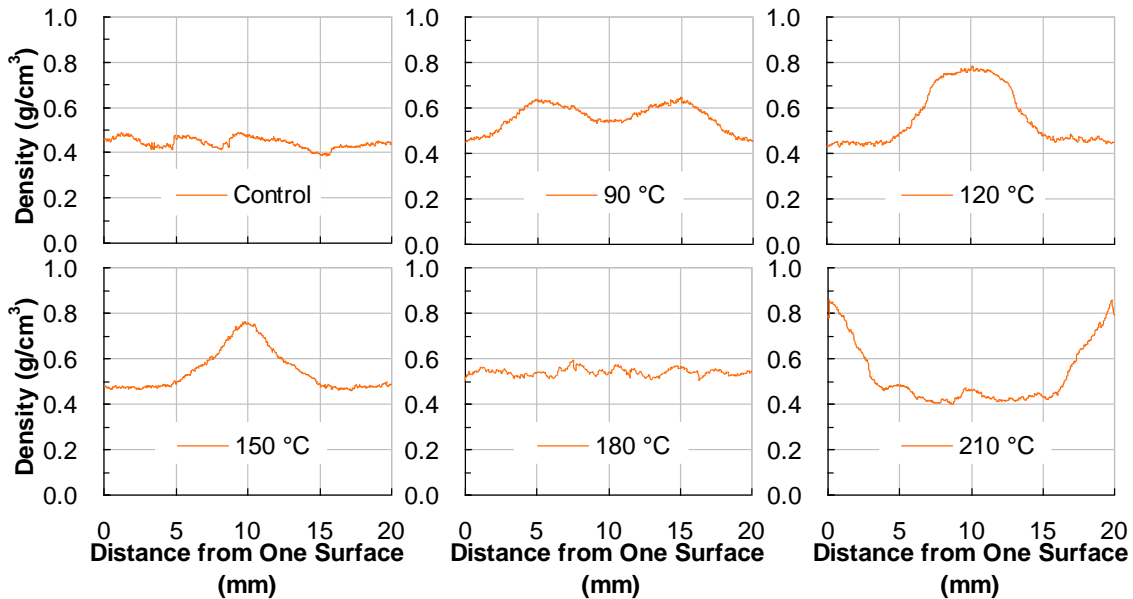
Figure 2 shows photographs and associated soft X-ray images of the individual transverse sections after compression at different pressing temperatures. In the photographs, there were some dark layers in the transverse sections, which was because those sections had higher densities than the others. The X-ray images confirmed this result. When the image was brighter, the material density was higher. Additionally, the distance between the compressed layer and wood surface changed as the pressing temperature increased. At 90 °C to 150 °C, the compressed layer was formed inside the compressed wood and the distance of the compressed layer from the wood surface became larger as the pressing temperature increased. At 180 °C, the compressed layer disappeared and the compressed wood had a uniformly high density. When the pressing temperature was increased from 180 °C to 210 °C, compressed layers were formed at the surface. Moreover, the pressing temperature also affected the number of compressed layers. At 90 °C, two compressed layers were observed. When the pressing temperature was 120 °C and 150 °C, there was one compressed layer. The compressed layer at 150 °C was narrower than the one at 120 °C. There were no compressed layers when the pressing temperature was 180 °C, and two compressed layers appeared again at 210 °C.



**Fig. 2.** Photographs (a) and soft X-ray images (b) of the transverse sections of the control and compressed wood at different pressing temperatures

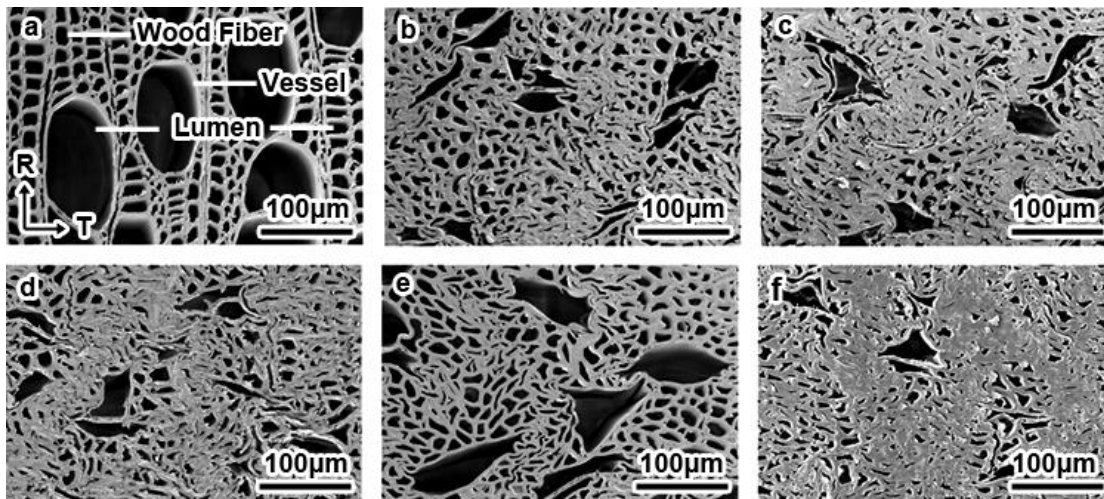
The density profiles of the control, uncompressed specimens, and compressed wood specimens are presented in Fig. 3. The control exhibited an almost uniform density throughout its thickness, and the variations in the density throughout the thickness were because of the density difference between the earlywood and latewood. Hot pressing caused an increase in the density, and different pressing temperatures resulted in different density profiles. At 90 °C, two compressed layers formed inside the compressed wood. As the pressing temperature increased, the two compressed layers shifted towards the core layer and integrated into one compressed layer at 120 °C. With a further increase in the pressing temperature, the thickness of the compressed layer formed at the core layer

narrowed at 150 °C and disappeared at 180 °C. When the pressing temperature was 210 °C, the two surface layers were compressed. It was concluded that the pressing temperature effectively controlled the shape of the density profiles.



**Fig. 3.** Density profiles of the control and compressed woods at different pressing temperatures

A microscopic examination of the transverse section was done to examine the extent and characteristics of deformation in the wood (Fig. 4). It was observed that the cells collapsed into different shapes after compression at different pressing temperatures and large distortion of the cell walls occurred in the vessels and wood fiber. The wood deformation under compression is strictly related to the temperature and moisture effects along with the influence of the microfibril angles of the wood cell layers (Fortino *et al.* 2015). The vessels were fully collapsed, but the cells were not deformed entirely and the lumen stayed open.

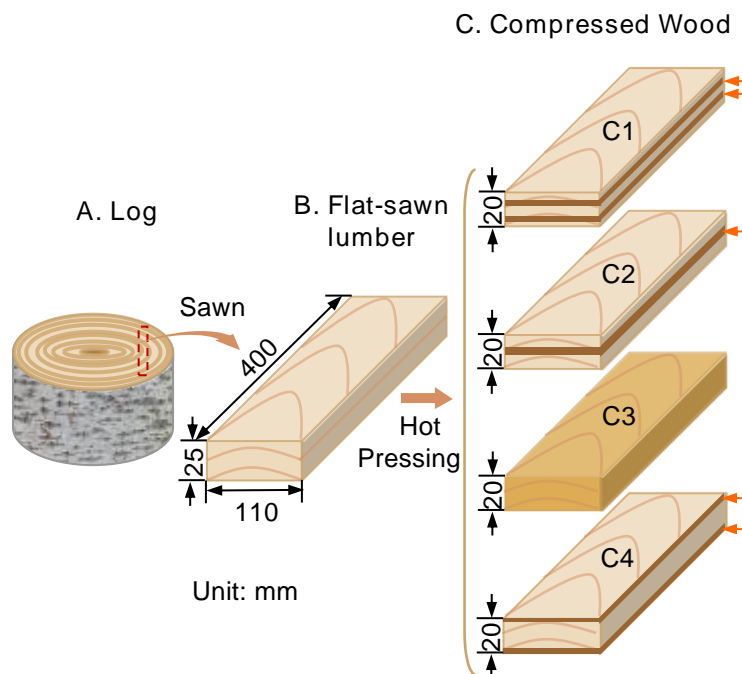


**Fig. 4.** SEM images of the control and compressed woods at different pressing temperatures: (a) control, (b) 90 °C, (c) 120 °C, (d) 150 °C, (e) 180 °C, and (f) 210 °C



While large cell wall deformation was observed in the wood fiber, some fiber cells were almost completely closed. Also, the extent of deformation in the wood compressed at different temperatures differed. The most visible difference was that the lumen of the wood fiber cells at 210 °C were closed more than at the other temperatures. Even though deformation occurred in the wood cells after compression, there were no compression failures in the wood subject to compression at different pressing temperatures. The microscopic examination revealed that the lumen volumes were greatly reduced and the deformations were the consequence of cell wall buckling in the compression direction, without marked cell wall failures compared with the control.

It was concluded that the pressing temperature had a noticeable effect on the density profile of the compressed wood (Fig. 5). Depending on the pressing temperature, the compressed wood showed four distinct types of behavior: 1) two compressed layers formed between the surface and core layers (C1), 2) one compressed layer formed at the core layer (C2), 3) the compressed wood had a uniform density profile (C3), and 4) two compressed layers formed at the surface layers (C4).



**Fig. 5.** Schematic indicating the formation of the compressed wood: C1 – compressed at 90 °C, C2 – compressed at 120 °C and 150 °C, C3 – compressed at 180 °C, and C4 – compressed at 210 °C; the dark layers indicated by arrows are the compressed layers

The literature shows that during compression, different pressing temperatures resulted in different temperature distributions throughout the thickness of the wood (Beard *et al.* 1983; Beard *et al.* 1985; Tang *et al.* 1994). Meanwhile, the pressing temperature as a driving force for the diffusion of moisture had an impact on the moisture diffusion coefficient (Fotsing and Tchagang 2005; Hrčka *et al.* 2008). When the temperature is above 100 °C, water in wood is transformed into vapor and the vapor pressure also becomes a driving force for the diffusion of moisture (Hunter 1993). Moreover, the vapor pressure is affected by the temperature; when the temperature is higher, the vapor pressure is higher (Udaka and Furuno 2005). Thus, the different pressing temperatures caused different

moisture distributions in the thickness direction. Wood softening depends on the moisture and temperature, and the softening temperature decreases with an increase in the moisture content (Furuta *et al.* 2010). When the moisture content is 60%, the softening temperatures of hemicellulose and lignin are only 20 °C and 70 °C, respectively. Also, increasing the temperature and moisture content reduces the yield stress (Inoue *et al.* 1990; Yu *et al.* 2011; Ozyhar *et al.* 2012; Jiang *et al.* 2017). Therefore, regions with a high moisture content and high temperature in the thickness direction were compressed more easily and the compression at various temperatures contributed to the four different types of compressed wood (Fig. 5).

This study was based on our previous research (Li *et al.* 2018). In the previous research, the lumber samples were kiln-dried, then they were further machined into sapwood specimens. Before compression, these samples were first coated with paraffin on the transverse sections, then soaked in water for 2 h to wet the surface and stored in sealed plastic bags for 18 h (MC=18.3%), then these samples were preheated and hot compressed at 60 °C, 90 °C, 120 °C, 150 °C, 180 °C and 210 °C. After compression, three kinds of compressed wood were obtained through this method. It provides a promising technology for full valorization of low-density plantation wood. But the process that samples were first coated with paraffin on the transverse section, then soaked in water for 2 h to wet the surface and stored in sealed plastic bags for 18 h is a disadvantage for industrial applications.

In this study, after lumber samples were kiln-dried and further machined into sapwood specimens (MC=10.6%), sapwood specimens were compressed immediately. The results showed that four kinds of compressed wood could be obtained by the technology used in this study while there were three kinds of compressed wood in the previous research (Li *et al.* 2018). Additionally, the process in this study that after lumber samples were kiln-dried and further machined into sapwood specimens, sapwood specimens could be compressed immediately is simpler and more efficient than that in the previous research (Li *et al.* 2018). That is an advantage for industrial applications.

### Density Profile Characteristics

Table 1 shows that the pressing temperature influenced the average density of the control and compressed wood. From 90 °C to 150 °C, it was observed that the density was higher when the layer was deeper. The most likely explanation for this was that the samples began drying at the heated surface, and at the same time, the moisture migrated toward the center and the temperature inside the wood increased. This behavior was also found in spruce pine in a numerical study (Fortino *et al.* 2013), where it was shown that the peaks in moisture profiles under high densification temperature appear in locations close to those of the peaks of density. This thereby softened the deeper layers. At 180 °C, the compressed wood had a uniform density, which meant that the compressed wood had uniform plasticization throughout the thickness during compression. In contrast, the density was lower when the compressed layer was deeper at 210 °C, which suggested that the temperature had a stronger effect on wood softening than the moisture content when compression was performed at a high temperature. During this time, the surface reached the highest temperature. This led to better plasticization, and so the highest density layer was at the surface. Also, the pressing temperature had a negative influence on the average density of the entire thickness. The average density appeared to decrease slightly, which

was attributed to the degradation of wood with an increase in the pressing temperature; however, the differences among the average densities were not significant (F test,  $p > 0.05$ ).

**Table 1.** Average Density of the Control and Compressed Wood at Different Pressing Temperatures

Pressing Temperature (°C)	Different Surface Layer Thickness (g/cm <sup>3</sup> )			Entire Thickness (g/cm <sup>3</sup> )
	0.32 mm	2.82 mm	5.64 mm	
Control	0.44 (0.01)	0.44 (0.01)	0.44 (0.01)	0.44 (0.01)
90	0.46 (0.01)	0.48 (0.02)	0.54 (0.01)	0.56 (0.01)
120	0.43 (0.01)	0.44 (0.02)	0.45 (0.01)	0.56 (0.01)
150	0.47 (0.02)	0.47 (0.01)	0.48 (0.01)	0.54 (0.01)
180	0.53 (0.01)	0.53 (0.01)	0.53 (0.01)	0.53 (0.01)
210	0.83 (0.02)	0.72 (0.01)	0.60 (0.01)	0.52 (0.01)

Values in parentheses are the standard deviation of three runs

The values displayed in Table 2 are the peak density (PD) and peak distance (Pdi). For the PD, compression in a specific part of the wood instead of the whole thickness contributed to the PD. When the pressing temperature was 210 °C, the PD reached its highest value of 0.87 g/cm<sup>3</sup>, which was 47.5% higher than that of the sample compressed at 180 °C with a uniform density profile throughout its thickness. The Pdi increased from 90 °C to 120 °C, and when the density peak reached the center of the wood at 120 °C and 150 °C, the Pdi reached a maximum value. At 180 °C, the density peak disappeared, but when the pressing temperature increased from 180 °C to 210 °C, the density peak appeared at the surface.

**Table 2.** Peak Density

Pressing Temperature (°C)	PD (g/cm <sup>3</sup> )			Pdi (mm)		
	PD1	PD2	Average	Pdi1	Pdi2	Average
Control	—	—	0.49 (0.01)	—	—	—
90	0.63 (0.02)	0.64 (0.02)	0.64 (0.02)	5.07 (0.06)	4.99 (0.04)	5.03 (0.04)
120	—	—	0.76 (0.02)	10.03 (0.03)	9.96 (0.07)	10.00 (0.01)
150	—	—	0.75 (0.02)	10.13 (0.10)	9.86 (0.10)	10.00 (0.01)
180	—	—	0.59 (0.03)	—	—	—
210	0.87 (0.03)	0.86 (0.01)	0.87 (0.02)	0.14 (0.02)	0.15 (0.01)	0.15 (0.01)

Values in parentheses are the standard deviation of three runs; PD – peak density; Pdi – peak distance



The values displayed in Table 3 are the average densities of the compressed layer and compressed layer distance (CLdi). The average density of the compressed layer shared the same trend as that of the PD (Table 2). At 210 °C, the average density of the compressed layer reached the highest value of 0.72 g/cm<sup>3</sup> and was 63.6% higher than that of the control.

**Table 3.** Compressed Layer

Pressing Temperature (°C)	Average Density (g/cm <sup>3</sup> )			CLdi (mm)		
	CL1	CL2	Average	CLdi1	CLdi2	Average
Control	—	—	—	—	—	—
90	0.61 (0.01)	0.61 (0.01)	0.61 (0.01)	3.66 (0.13)	3.63 (0.04)	3.65 (0.06)
120	—	—	0.71 (0.01)	6.50 (0.25)	6.43 (0.20)	6.47 (0.04)
150	—	—	0.67 (0.02)	7.29 (0.08)	7.32 (0.16)	7.30 (0.06)
180	—	—	—	—	—	—
210	0.75 (0.02)	0.70 (0.01)	0.72 (0.01)	0.00 (0.00)	0.00 (0.00)	0.00 (0.00)

Values in parentheses are the standard deviation of three runs; CL – compressed layer; CLdi – compressed layer distance

For the CLdi, its change trend was similar to that of the Pdi (Table 2), but when the temperature was increased from 120 °C to 150 °C, the Pdi almost did not change, while the CLdi continued to increase. This was because of the decrease in the compressed layer thickness (CLT). From 90 °C to 150 °C, the CLdi increased with an increase in the temperature, which was attributed to the progressive increase in the temperature inside of the wood and the migration of moisture toward the center, which shifted the zone of maximum wood plasticization deeper into the sample. At 180 °C, there were no compressed layers and the compressed wood had a uniform density profile throughout the thickness. When the temperature was 210 °C, the compressed layers formed at the wood surfaces. It is known that compressed layers forming at the wood surface is good for bending properties and hardness, and compressed layers forming at the center of the wood is good for the nail withdrawal resistance. Therefore, controlling the density profile of compressed wood is important for the desired end-use properties. Also, less energy is used in the process, while the desired property improvements are achieved.

Figure 6 shows the proportion of the CLT. The proportion decreased when the temperature increased from 90 °C to 180 °C. This was because drying reduced the moisture content, and the zone of maximum wood plasticization became smaller. At 210 °C, the compressed wood may be oven dried, and the deformation was caused by the high temperature. Additionally, the proportions of the CLTs at 120 °C, 150 °C, 180 °C, and 210 °C were lower than that of the CLT at 90 °C. This suggested that the thickness of the compressed layer was affected by the interactive effect of the moisture content and temperature, and wood with higher moisture content contributes more to compressive deformation.

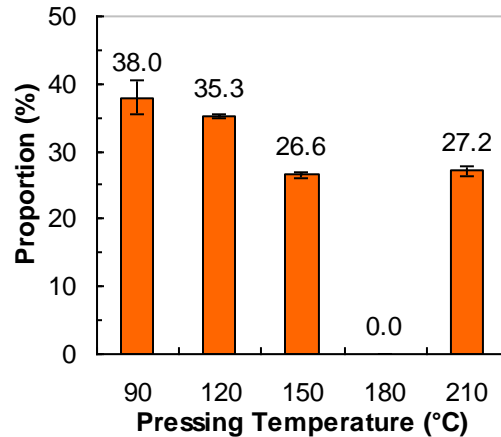


Fig. 6. Proportion of the compressed layer thickness at the different pressing temperatures

## CONCLUSIONS

1. The pressing temperature and the related moisture gradients effectively controlled the shape of the density profile. Depending on the pressing temperature, the compressed wood showed four distinct types of behavior: 1) two compressed layers formed between the surface and core layers, 2) one compressed layer formed at the core layer, 3) the compressed wood had a uniform density profile, and 4) two compressed layers formed at the surface layers.
2. The pressing temperature affected the average density for different thicknesses in the surface layer. From 90 °C to 150 °C, the density was higher when the layer was deeper, and the compressed wood had a uniform density at 180 °C. In contrast, at 210 °C, the density was lower when the layer was deeper.
3. Compression in a specific part of the wood instead of the whole thickness contributed to the peak density and average density of the compressed layer, which attained the highest values of 0.87 g/cm<sup>3</sup> and 0.72 g/cm<sup>3</sup>, respectively, when the compressed thickness was 5 mm.
4. The peak density and compressed layer could be formed at various distances from the wood surface and the thickness of the compressed layer was affected by the interactive effect of the moisture content and temperature.

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