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Effects of pre-heating treatment parameters on dimensional stability and mechanical properties of densified Chinese fir

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ABSTRACT

Pre-heating treatment performed by heat plate is an efficient wood softening method that could improve the dimensional stability and mechanical properties of densified wood, having great potential to industrialize. To optimize the properties of densified Chinese fir softened by this method, an improved understanding of the pre-heating treatment parameters is required. An orthogonal experiment with three factors and levels was used in this study to investigate the effects of the pre-heating treatment parameters including heating temperature, duration time and moisture content on set recovery, static bending strength (MOR), modulus of elasticity (MOE) and compression strength parallel to grain (CSP). Chinese fast-growing fir with dimensions of 400 mm(L) 60 mm (T) 40 mm(R) was prepared as raw materials, and the wood densification process including softening, pressing and fixation was performed using a hot-press. The test results suggested that moisture content was the most significant factor influencing set recovery, MOR, MOE and CSP. Although the effect of temperature on MOE and CSP was not significant, it greatly affects set recovery and MOR. Moreover, the effect of duration time changes within the range of 10 min to 30 min was slight in all of the responses in this study. The mechanical properties of densified Chinese fir improved substantially with the increase in moisture content. The values of MOR and MOE raised to 95.17 MPa and 15.43 GPa respectively when moisture content raised from 30 % to 50 %, increased by 32.5 % and 21.8 %. The set recovery induced by moisture adsorption and water absorption decreased from 7.23 % to 3.09 % and from 20.9 % to 12.7 % respectively indicating that the dimensional stability of densified Chinese fir improved significantly with the increase of moisture content from 30 % to 50 %. The conditions of pre-heating treatment at a temperature of 120 °C, duration time of 20 min and moisture content of 50 % were found to be the optimum parameters for manufactured densified Chinese fir. The proposed pre-heating conditions could be a recommendation for industrial production.

1. Introduction

Wood has been widely used as a green and sustainable material for thousands of years because of the advantages in renewable, aesthetically comforting, cheap, structurally strong, non-toxic, and carbon neutral [1]. Building constructed with wood can reduce energy consumption and carbon footprint, meeting the demand for a more sustainable and eco-friendly economy. Although the potential of building with wood in relieving environmental problems has been recognized, wooden buildings still have a limitation on development and application in some regions due to the lack of wood resources. To meet this challenge, the development of structural timber products from plantation wood species is needed [2].

Chinese fir is one of the vital softwood species widely planted in South China and has the highest value in the rate of stock volume. The stock volume of plantation Chinese fir reaches 755 million m^3, up to a third of the stock volume of plantation Chinese arbor forest [3]. However, the main application of plantation Chinese fir is the production of low-value wood products, for instance, chipboard, fiberboard or paper [4], the structural application is hindered by instinctive defects such as

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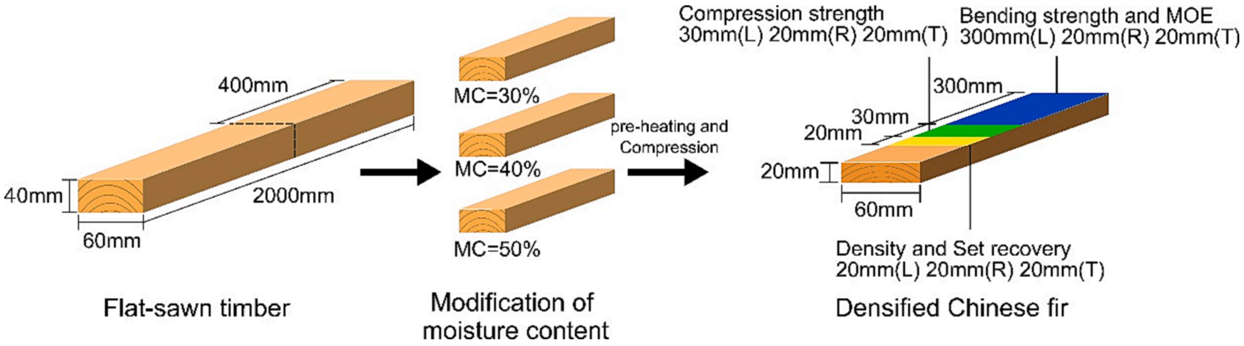

Fig 2.1. Schematic diagram for specimen preparation and densification process.

low density and strength, and poor dimensional stability. In order to utilize plantation fir instead of hardwood species in applications of higher value, improving the physical and mechanical properties of Chinese fir by wood modification technic is required.

Wood densification is one of the effective techniques for improving the density and mechanical properties of low-density wood, and the densified wood could be used to fabricate engineered wood products like laminated timber or plywood [5,6]. Mechanical properties of wood generally correlate positively with density [7], therefore, the properties such as strength and Yong's modulus, of the high-density wood are usually more desirable than the low-density one. In light of this, increasing the density of wood can improve its mechanical properties effectively and then widen the range of applications of low-density wood. The density of wood depended on the thickness of the cell walls and the size of the lumen due to the roughly same density of the cell walls regardless of the wood species or cell type [8]. With this in mind, the density can be greatly improved by reducing the pores and voids between the cell walls through mechanically compressing the wood cells, or by impregnating the void volume with fluids [9]. Among these two methods, the mechanical compression method is more adorable ascribed to the advantage of cost-effective, technically simple and easy recycling when compared to chemical impregnation [10]. Densified wood fabricated by mechanical compression was used as an alternative material of metal in military aircraft by America and Germany, as early as the 1930s [11]. Now it has been widely applied to improve the density and mechanical properties of softwood and various approaches have been reported, including thermo-mechanical (TM), thermo-hydro-mechanical (THM) and viscoelastic thermal compression (VTC). Densification can be carried out in open or closed system. Although a closed system provides the possibility of controlling all processing parameters, the industrial applications were hampered by the equipment costs. In an open system, the moisture content of the to-be-densified specimens was generally limited to 13 %. Under this condition, the dimensional stability of specimens was poorer compared to the wood densified in a closed system [12]. In light of this, this study provided a less studied method to manufacture densified wood in an open system using wood with high moisture content.

It is essential to soften the wood structure before mechanical pressing. The process of softening is known as a type of pretreatment and can be explained by the viscoelastic character of wood. Wood exhibits glass behavior at low moisture contents and temperatures, and exhibits rubbery behavior at high moisture contents and temperatures [13]. Through softening, densification can occur without breaking wood cell walls.

Wood can be softened by using chemical or mechanical treatment. Chemical treatment is costly and could raise an environmental issue [14], therefore, mechanical treatment may be an attractive method for widespread industrialization. The strategies of mechanical softening that have been used commonly include hot plate heating, microwave heating, subjecting the wood to steam, or combined application of steam, temperature and pressure. However, most of these processes have the disadvantages of high energy consumption, long processing time and high apparatus requirement, which hinder the utilization of densified wood as commercial materials [1]. Among different mechanical softening methods, heating the to-be-softened wood with a hot plate has the advantage of easy processing, money saving and eco-friendly. In light of this, hot plate heating was selected in this study as the softening strategy to cut costs, reduce energy consumption and moderate the apparatus demands.

The processing parameters of heating treatment including heating temperature, duration time and moisture content of specimens have been reported by previous literature that have a great effect on dimensional stability, physical and mechanical properties of densified wood. Wood softening behavior depends on temperature and moisture content ascribed to the glass transition temperature is depressed as the moisture content rises [15], so wood softening could occur at lower temperatures when moisture content increases. The mechanical and physical properties of densified wood highly depend on the density profile [16], which is affected by the degree of wood softening[17], therefore, temperature and moisture content are two main parameters that have a significant influence on the physical and mechanical properties of densified wood. Additionally, increasing the heating temperature properly could improve the dimensional stability of densified wood due to the decomposition of hemicellulose[18]. Kariz et al. [19] reported that set recovery of surface densified spruce [*Picea abies* (L.) Karst.] decreased from 81.8 % to 73.4 % as the heating temperature increased from 170 °C to 230 °C. However, excessive increases in temperature and duration time cause mass loss, which is the result of the decrease in the holocellulose ratio, resulting in a decrease in physical and mechanical properties [20]. Although the effect of duration time is not significant compared to temperature and moisture content, it is an important factor for improving the efficiency of densification production. Accordingly, to fabricate dimensionally stable densified wood with high physical and mechanical properties, an improved understanding of pre-heating treatment parameters is required.

This study aimed to improve the dimensional stability, physical and mechanical properties of densified plantation Chinese fir by optimizing the pre-heating treatment parameters. Chinese fast-growing fir with high moisture content was softened and compressed by a hot-press using pre-heating treatment and thermo-mechanical compression respectively. The softening and densification process reported by previous studies was not continuous, wood specimens after being heated were conditioned in a cabinet before densification [21], which is detrimental to production efficiency. To address this drawback, a continuo process was implemented in the presented study, which is less studied to the authors' knowledge. To determine the optimal pre-heating treatment parameters of densified Chinese fir wood, the effects of heating temperature, duration time and moisture content on the set recovery, physical and mechanical properties of densified Chinese fir were discussed in this study. Furthermore, the optimal pre-heating treatment

Table 1
Factors and levels.

Factors	Level		
	1	2	3
Heating temperature, *T*, (℃)	80	100	120
Duration time, *t*, (min)	10	20	30
Moisture content, *MC*, (%)	30	40	50

parameters were determined in this work.

2. Materials and methods

2.1. Raw materials

Fig. 2.1 shows the process of specimen preparation. 15 Chinese plantation fir [*Cunninghamia lanceolata* (Lamb.) Hook.] harvested in Jiangxi province, China, were used as the raw materials in this study. These stems were flat-sawn into timbers with dimensions of 2000 mm (L) 60 mm (T) 40 mm (R) . The flat-sawn timbers were then equilibrated in a conditioning room at a temperature of 20 ℃ and relative humidity of 65 %. After conditioning, each flat-sawn timber was processed to 4 timber boards with dimensions of 400 mm (L) 60 mm (T) 40 mm (R) for densification and mechanical property tests, and processed to 2 specimens with dimensions of 20 mm (L) 20 mm (T) 20 mm (R) for determination of density and moisture content according to GB/T 1933-2009 [22] and GB/T 1931-2009 [23] respectively. The average density and moisture content of natural Chinese fir were 0.399 ± 0.032 g/cm^3 and 16.52 ± 0.012 %.

2.2. Experimental design

Table 1 gives detailed information on experimental design. An orthogonal experiment with three factors and levels was used in this study. Three factors that have the greatest impact on wood softening behavior were investigated in this study: heating temperature, duration time and moisture content. The responses considered were set recovery, static bending strength (MOR), modulus of elasticity (MOE) and compression strength parallel to grain (CSP). 6 timber boards were contained by each test group and two replicates were prepared (a total of 12 specimens). The values were an average of 12 specimens.

2.3. Modification of moisture content

The moisture content of the specimens was conditioned by soaking the to-be-densified specimens in distilled water at room temperature until the mass obtained through weighting operation and the mass calculated according to equation (1) did not differ by more than 0.5 g. The specimens with target moisture content were then wrapped in plastic film and stored under light-proof conditions to ensure that the moisture content remained unchanged before testing.

$$m_1 = (1 + W_1)\, m_0 / (1 + W_0) \quad (1)$$

Where

m_1 is the mass of specimens at the target level of moisture content, in g;
W_1 is the target moisture content, in %;
m_0 is the mass of specimens prior to soaked, in g;
W_0 is the moisture content of the specimens prior to soak, in %.

2.4. Densification process

Densification containing three phases, heating, pressing and pressure holding procedure was performed by a hot-press (XLB-IMND, YADONG GROUP, China). Fig. 2.2 shows the schematic image of densification.

The pressing parameters were determined by preliminary test to ensure the pre-heating treatment parameters were the only variables affecting the properties of densified Chinese fir [24]. The pressing parameters determined in this study were found to be compression ratio = 50 %, pressing temperature = 140 ℃, compression pressure = 6 MPa, and pressure holding time = 3 h.

The specimens were compressed in the radial direction. Each specimen with target moisture content (30 %, 40 % and 50 %) was placed in contact with the hot platens which were already heated to target temperature (80 ℃, 100 ℃ and 120 ℃), then the specimens were softened using the heated plates in a load less than 0.5 MPa for different duration time (10 min, 20 min and 30 min). Then increased the temperature of the plates to 140 ℃ with a mean heating speed of 1.9 ℃/min. The segmented pressurization process was used in this study with the intention of avoiding cracks in densified wood. A 1-minute interval was set when the pressure applied to the specimens reached 2 MPa. The thickness of densified Chinese fir was monitored with mechanical stops,

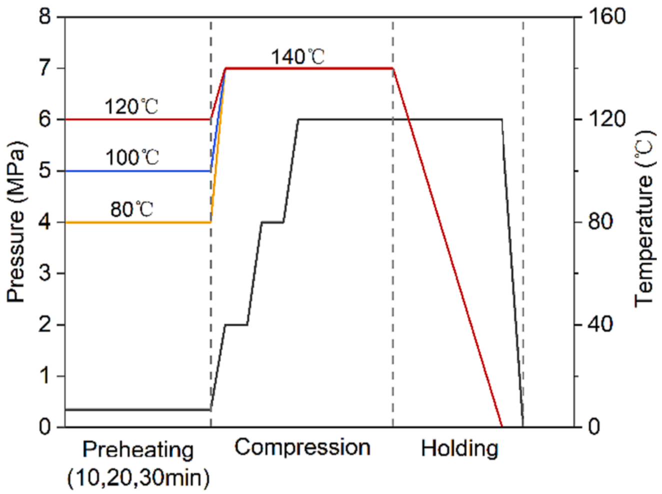

Fig 2.3. Schematic image of segmented pressurization process.

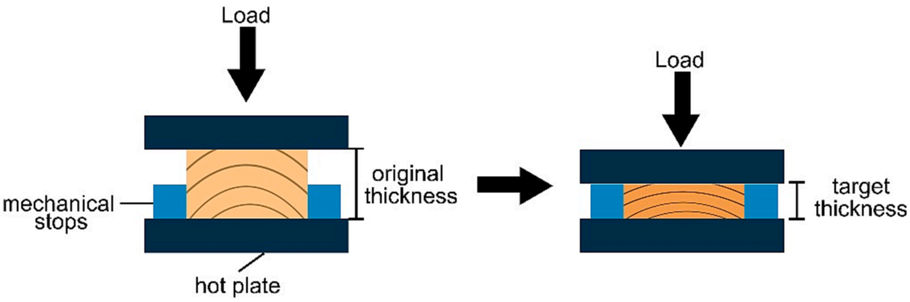

Fig 2.2. Schematic image of densification.

Table 2
Dimensions of test specimens and relevant standards for physical and mechanical tests.

Properties	Dimensions of specimens	Standards
Density	20 mm (L) 20 mm (T) 20 mm (R)	GB/T1933-2009 [22]
Moisture content	20 mm (L) 20 mm (T) 20 mm (R)	GB/T 1931-2009 [23]
Set recovery	20 mm (L) 20 mm (T) 20 mm (R)	GB/T 1934.2-2009 [25]
MOR	300 mm (L) 20 mm (T) 20 mm (R)	GB/T1936.1-2009 [26]
MOE	300 mm (L) 20 mm (T) 20 mm (R)	GB/T 1936.2-2009 [27]
CSP	30 mm (L) 20 mm (T) 20 mm (R)	GB/T 1935-2009 [28]

Table 3
Air-dry density, oven-dry density and moisture content of natural specimens.

	Minimum value	Maximum value	Mean value	Number	Standard deviation
Air-dry density (g/cm^3)	0.374	0.486	0.399	30	0.032
Oven-dry density (g/cm^3)	0.303	0.463	0.361	30	0.034
Moisture content (%)	15.36	18.52	16.52	30	0.012

the target thickness of 20 mm was achieved when the pressure was up to 6 MPa. Once the designated pressure and thickness of densified wood were reached, the specimens were kept under pressure for 3 h to fix the deformation. The hot platens were cooling at room temperature during the load-holding process. The whole densification process is depicted in Fig. 2.3.

After compression, the specimens were conditioned at a temperature of 20 ℃ and relative humidity of 65 %, waiting for the test of determination of physical and mechanical properties selected in this study.

2.5. Set recovery

The compressive deformation recovery of specimens resulting from moisture adsorption and water absorption were investigated separately when the moisture content of specimens was lower than the fiber saturation points and higher. The determination of set recovery induced by moisture adsorption was as follows: The specimens with a size of 20 mm (L) 20 mm(T) 20 mm(R) which were cut from the densified Chinese fir were dried to absolute dry in the oven and measured the thickness in a radial direction in accordance with GB/T 1934.2-2009 [25]. The samples were then conditioned using the humidity chamber which was set up at a temperature of 25 ℃ and relative humidity of 65 % for 90 days. The dimensions of the specimens after various intervals in the humidity chamber were recorded. Subsequently, the specimens were oven-dried again using the same method and the measuring operations. The determination of set recovery due to water absorption was conducted using specimens prepared and dried by the same method. After the first drying, the samples were soaked in water for 10 days. The measuring operations for the change in radial dimensions of the soaked specimens were performed every 2 days. The specimens were then dried to absolute dry again and measured the radial dimensions. The set recovery was calculated according to equation (2):

$$R = \left(\frac{Tr - Tc}{To - Tc}\right) \times 100\% \tag{2}$$

Where the R is the set recovery due to moisture adsorption or water absorption, in %.

T_r is the thickness of specimens after condition or soak, in mm;
T_c is the thickness of densified specimens, in mm;
T_o is the thickness of raw specimens, in mm.

2.6. Physical and mechanical properties

Density, moisture content, MOR, MOE and CSP of densified Chinese fir were determined according to the relevant standards given in Table 2. The mechanical property tests were performed by a universal testing machine (AG-IG, SHIMADZU, Japan).

2.7. Statistical analysis

The statistical program SPSS 24.0 was used for data analysis. The experiment results of orthogonal tests are presented as means (M) ± standard deviation (SD). Firstly, the normality of experiment results distribution was checked with the Shapiro-Wilk test. The results of the Shapiro-Wilk test show that all data follow normal distribution ($P > 0.05$). The experiment results of orthogonal tests were then subjected to range analysis and analysis of variance to determine the influence degree and optimum level of different factors.

Range analysis is a statistical method to investigate the sensitivity of factors to the experiment results. Range is defined as the distance between the extreme values of the data. The greater the range is, the more sensitive the factor is. The Table of the range analysis contained three variables of Ki, ki and R. Ki is the sum value of the experimental results which contain a factor at the i level. ki is the average value of the experimental results which contain a factor at the i level. The optimum level was considered the i level at which the ki value reaches the maximum. R is the difference between the maximum and minimum values of ki, which stands for the influence degree of the factor. When the R value is high, the influence degree of the factor is great as a result.

Analysis of variance allows a statistical analysis of three factors with different levels in order to determine the effect of the three factors on individual response. The significance level was set at 0.05. The results of the analysis of variance include the degrees of freedom (D*f*), sums of squares (SS), mean square (MS), F-value (*F*) and P-value (*P*).

3. Results and discussion

3.1. Density and moisture content

Table 3 shows the results obtained from the density and moisture content determination of the natural specimens. After densification, the mean values of air-dry density, oven-dry density and moisture content were 0.744 g/cm^3, 0.752 g/cm^3 and 14.48 %, respectively. The oven-dry density of densified wood corresponds to a 108.3 % increase in relation to untreated wood. Fig. 3.1 depicts the comparison between the density of untreated specimens and densified specimens. The result indicates that the influence of pre-heating treatment on density was not significant, which conforms to the study reported by Sözbir et al.[21].

3.2. Set recovery

Dimensional instability due to set recovery could be detrimental to some densified wood applications such as in laminas for the fabrication of glued laminated timber. Set recovery is a phenomenon that densified wood partially or fully returns to its original dimensions when exposed to moisture. This moisture-dependent swelling of densified wood is attributed to reversible and irreversible swelling [13]. Reversible swelling can be ascribed to the natural hygroscopicity of wood, and irreversible swelling is thought to be a result of the release of the elastic-strain energy stored by densification.

Table 4 shows the results of compression deformation recovery measurement of densified Chinese fir. The mean values and standard deviations are presented in the Table. The experiment results of orthogonal tests were then subjected to range analysis to determine the influence degree and optimum level of different factors, as shown in Table A.1.

Fig. 3.2 and Fig. 3.3 were plotted based on the results of the range

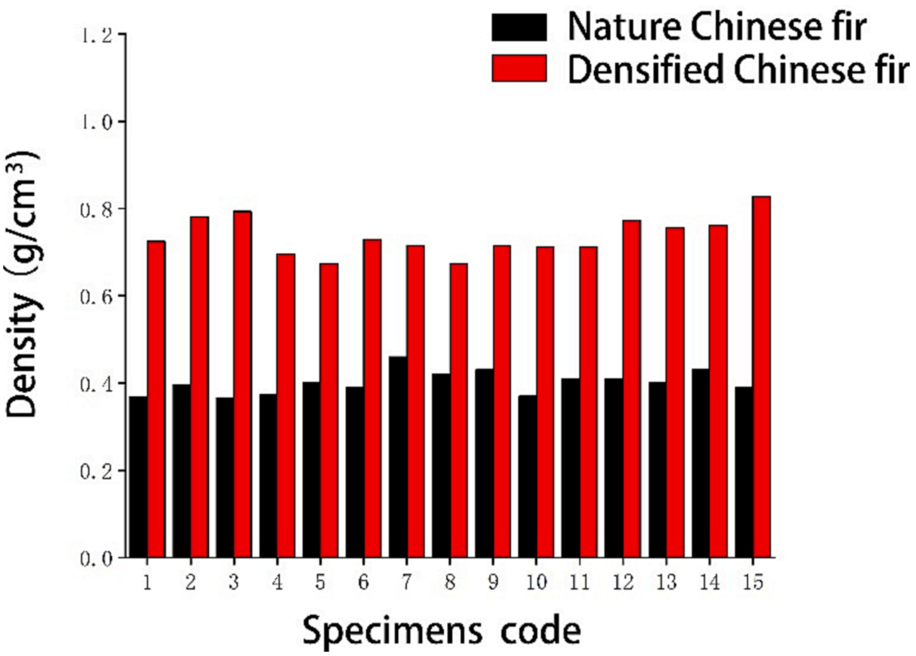

Fig. 3.1. Density comparison before and after densification.

Table 4
Orthogonal test result of recovery rate of densified Chinese fir.

Code	Factor			Moisture adsorption recovery (M ± SD, %)	Water absorption recovery (M ± SD, %)
	T (℃)	*t* (min)	*MC* (%)		
1	80	10	30	9.73 ± 1.36	27.86 ± 2.41
2	100	10	40	4.73 ± 0.47	22.73 ± 2.18
3	120	10	50	2.42 ± 0.31	10.61 ± 1.64
4	80	20	40	5.72 ± 0.83	17.69 ± 1.93
5	100	20	50	2.71 ± 0.42	13.53 ± 1.31
6	120	20	30	5.65 ± 0.67	15.49 ± 2.15
7	80	30	50	4.15 ± 0.84	13.96 ± 2.41
8	100	30	30	6.30 ± 1.18	19.53 ± 2.27
9	120	30	40	3.83 ± 0.69	11.15 ± 1.85

analysis. Moisture content was the most significant factor influencing set recovery resulting from moisture adsorption and water absorption, followed by heating temperature and duration time. The influence degree of factors affecting set recovery due to water absorption was consistent with that of moisture adsorption. The optimum processing parameters to reduce moisture adsorption set recovery were found to be heating temperature = 120 ℃, duration time = 20 min, and moisture content = 50 %, and to reduce water absorption set recovery were found to be heating temperature = 120 ℃, duration time = 30 min, moisture content = 50 %. A significant reduction in set recovery with the increase of moisture content can be observed from the figure. Moisture content increased from 30 % to 50 % resulting in the absolute value of moisture adsorption recovery reaching 3.09 %, which had decreased by 57.3 %. The absolute value of water absorption recovery reached 12.70 % when moisture content increased from 30 % to 50 %, which decreased by 39.4 %. The results show that the dimensional stability of densified Chinese fir was improved significantly by increasing moisture content. The elastic-strain energy stored in the semi-crystalline microfibrils and lignin of wood is thought to be the main cause of set recovery [10], therefore, the improvement of dimensional stability could be attributed to the reduction of this energy. Three points may result in this reduction, one is the hydrolysis of hemicellulose when wood with high moisture content was softened by pre-heating treatment. Hydrolysis of hemicellulose leads to a weakening of the connection between the microfibrils and the lignin, then allows the internal stresses of wood to relax [29]. Another is the improvement of wood softening behavior, which is the result of the decrease in glass transition temperature, increasing the compressibility of wood. Additionally, water acts as a plasticizer during the softening procedure improving the plasticity of wood also contributing to the reduction of elasticity-strain energy stored by compression.

The heating temperature was found to be another important factor

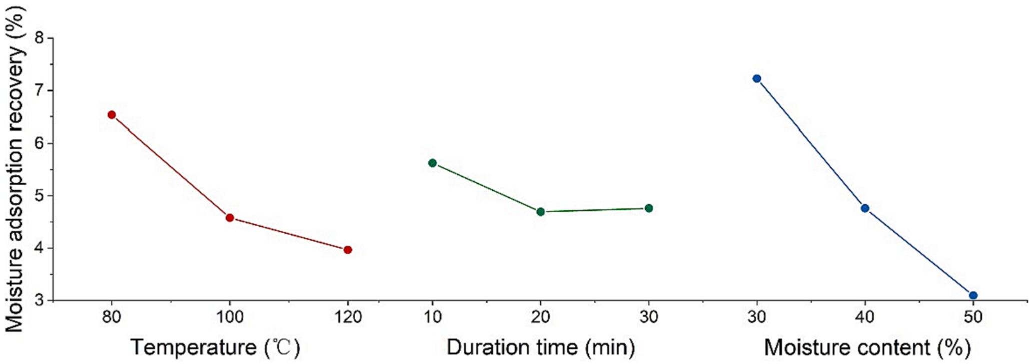

Fig 3.2. Values of moisture adsorption recovery of densified Chinese fir.

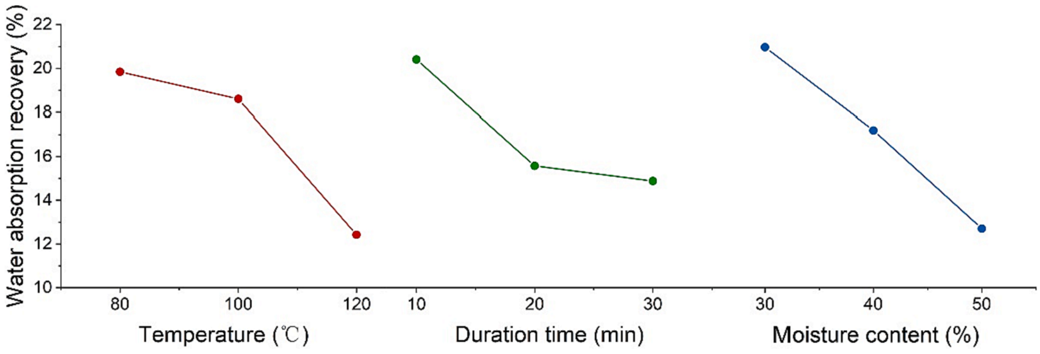

Fig 3.3. Values of water absorption recovery of densified Chinese fir.

Table 5
Analysis of variance of moisture adsorption recovery and water absorption recovery.

	Source	Df	SS	MS	F	P
Moisture adsorption recovery	*T*	2	10.779	5.390	15.195	0.062
	t	2	1.627	0.813	2.293	0.304
	MC	2	25.947	12.973	36.576	0.027
	Error	2	0.709	0.355		
Water absorption recovery	*T*	2	94.786	47.393	20.688	0.046
	t	2	54.275	27.138	11.846	0.078
	MC	2	102.601	51.300	22.393	0.043
	Error	2	4.582	2.291		

Table 6
Orthogonal test result of MOR and MOE of densified Chinese fir.

Code	Factor			MOR (M ± SD, MPa)	MOE (M ± SD, GPa)
	T (℃)	*t* (min)	*MC* (%)		
1	80	10	30	57.6 ± 6.43	12.4 ± 1.27
2	100	10	40	72.7 ± 7.84	13.9 ± 1.66
3	120	10	50	89.9 ± 9.15	16.7 ± 1.56
4	80	20	40	69.5 ± 6.91	14.0 ± 1.44
5	100	20	50	106.5 ± 8.37	15.7 ± 1.62
6	120	20	30	81.2 ± 6.28	14.2 ± 1.57
7	80	30	50	89.1 ± 7.52	13.9 ± 1.36
8	100	30	30	76.7 ± 6.79	11.4 ± 1.08
9	120	30	40	87.3 ± 8.16	12.5 ± 1.29
Normal				36.6 ± 4.50	7.5 ± 1.12

affecting set recovery. The values of moisture adsorption recovery and water absorption recovery reached 3.97 % and 12.42 % respectively, decreased by 39.2 % and 37.4 % when heating temperature increased from 80 ℃ to 120 ℃. This improvement could be ascribed to the reduction of the hygroscopicity of wood. Hygroscopicity is highly dependent on the number of accessible hydroxyl groups, and a decrease in accessible hydroxyl groups could be observed accompanying an increase in temperature during heat treatment [30]. It can be explained by a cross-linking formed among the aromatic units in the lignin [31]. The value of water absorption recovery reduced to 14.88 %, which decreased by 27.1 %, when the duration time was raised from 10 min to 30 min. However, it was found that the effect of duration time on moisture adsorption recovery was slight. The moisture adsorption recovery reduced from 5.63 % to 4.69 % when the duration time was raised from 10 min to 20 min, which decreased by 16.7 %.

Results obtained from the measurement of moisture adsorption and water absorption recovery of densified Chinese fir were subjected to analysis of variance, and the result was shown in Table 5. The Table suggested that the effect of moisture content and heating temperature on set recovery was significant, although the difference in heating temperature did not reach statistical significance in the case of moisture adsorption recovery ($0.05 < P < 0.1$). Water absorption recovery varied between different levels of duration time. However, the difference was not statistically significant. Lastly, moisture adsorption recovery was not found to be dependent on duration time ($P > 0.1$). The analysis of variance gives good agreement with the range analysis.

3.3. Static bending strength and modulus of elasticity in static bending

Bending strength and stiffness have significant effects on some utilization, for example, for the production of engineered wood products. In fact, lots of wood species with low strength and stiffness are considered to be unsuitable for use as laminae. A previous study reported that cross-laminated timber fabricated by poplar failed to comply with the minimum allowable strength set by ANSI/APA PRG 320-2012 [32]. The low stiffness of poplar is responsible for this result. Therefore, one way to account for the widening of wood application is to improve the strength and stiffness.

Table 6 shows the MOR and MOE values of Chinese fir before and after densification. The maximum and minimum MOR value of densified Chinese fir was 106.5 MPa and 57.6 MPa respectively, which was higher by 190.98 % and 57.38 % compared to natural specimens. The

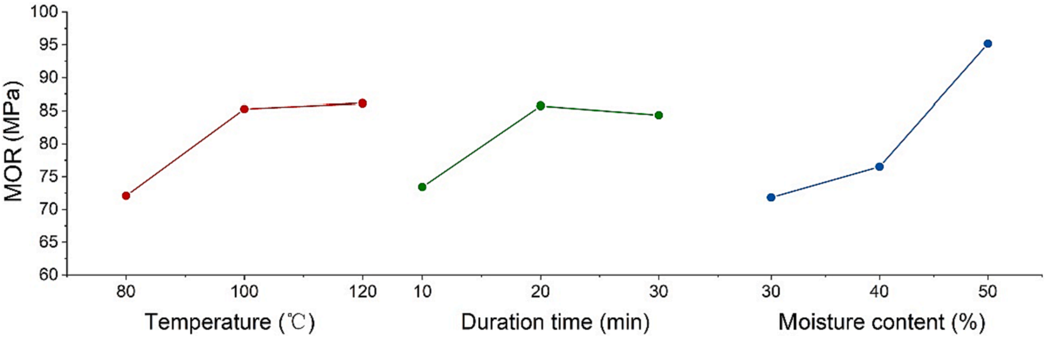

Fig 3.4. MOR values of densified Chinese fir.

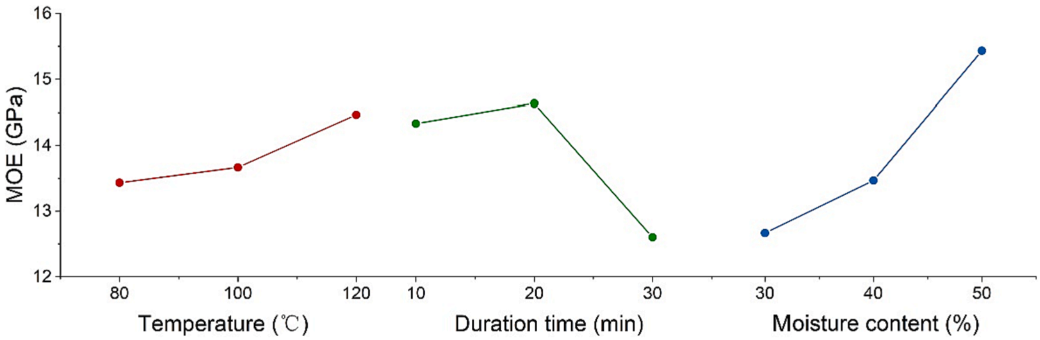

Fig 3.5. MOE values of densified Chinese fir.

Table 7
Analysis of Variance of MOR and MOE of densified Chinese fir.

	Source	*Df*	SS	MS	*F*	*P*
MOR	*T*	2	373.687	186.843	9.547	0.095
	t	2	274.247	137.123	7.007	0.125
	MC	2	914.667	457.333	23.369	0.041
	Error	2	39.140	19.570		
MOE	*T*	2	1.762	0.881	3.758	0.210
	t	2	7.229	3.614	15.417	0.061
	MC	2	12.162	6.081	25.938	0.037
	Error	2	0.469	0.234		

Table 8
Orthogonal test result of CSP of densified Chinese fir.

Code	Factor			Compression strength parallel to grain (M ± SD, MPa)
	T (℃)	*t* (min)	*MC* (%)	
1	80	10	30	41.7 ± 3.96
2	100	10	40	58.4 ± 4.07
3	120	10	50	46.9 ± 2.78
4	80	20	40	58.6 ± 5.42
5	100	20	50	46.6 ± 2.49
6	120	20	30	42.5 ± 3.25
7	80	30	50	46.0 ± 3.52
8	100	30	30	42.0 ± 2.36
9	120	30	40	51.2 ± 4.24
Normal				23.7 ± 2.58

maximum MOE value was 16.7 GPa and the minimum value was 11.4 GPa, increased by 122.67 % and 52 % respectively. The influence degree and optimum level of different factors were then determined using range analysis, and the analysis result is shown in Table A.2.

Fig. 3.4 and Fig. 3.5 were plotted according to the result of range analysis. Fig. 3.4 shows that moisture content was the main factor affecting bending strength, followed by heating temperature and duration time. The optimum processing parameters to improve bending strength and MOE were found to be heating temperature = 120 ℃, duration time = 20 min and moisture content = 50 %. Fig. 3.5. shows that the most significant factor affecting MOE of densified Chinese fir was moisture content, followed by duration time and heating temperature. The optimum processing parameters to improve MOE were the same as bending strength.

MOR was increased by 6.5 % and 24.4 % at a moisture content of 40 % and 50 % respectively. The enhancement of MOR at higher moisture content was partly due to the improvement of wood compressibility. The hydroxyl groups of semi-crystalline cellulose and hemicellulose and water molecules formed hydrogen bonds during pre-heating treatment under the combined action of high temperature and hydro. The formation of hydrogen bonds expanded the spacing between molecular chains, enhancing the compressibility of wood [33]. In addition, the loss of strength is thought to affected by the microcracks of wood cell walls caused by densification [34]. Microcracking occurs between cells during densification could be avoided by the improvement of wood plasticity. When wood softening treatment was conducted at high moisture content conditions, the plasticization degree achieved a better level due to the decrease of the glass transition temperature of hemicellulose and lignin. Therefore, the bending strength was enhanced due to the improvement of wood plasticity. An increase of 18.4 % and 16.8 % in MOR was detected at a temperature of 100 ℃ and a duration time of 20 min

Table 9
Analysis of Variance of CSP of densified Chinese fir

	Source	*Df*	SS	MS	*F*	*P*
CSP	*T*	2	8.487	4.243	0.650	0.606
	t	2	14.687	7.343	1.126	0.470
	MC	2	307.520	153.760	23.571	0.041
	Error	2	13.047	6.523		

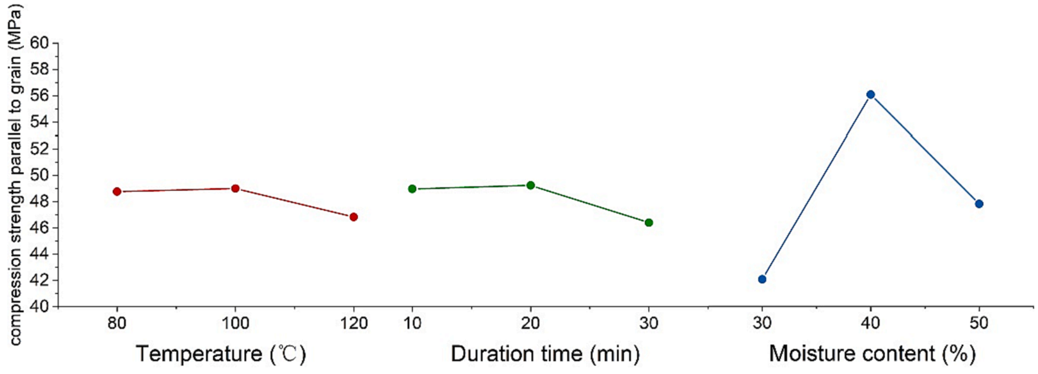

Fig 3.6. Compression strength parallel to grain of densified Chinese fir.

Table A.1
Range analysis of moisture adsorption recovery and water absorption recovery.

	Factor	K_1	K_2	K_3	k_1	k_2	k_3	R	Optimum level
Moisture adsorption recovery	*T*	19.60	13.74	11.90	6.53	4.58	3.97	2.57	3
	T	16.88	14.08	14.28	5.63	4.69	4.76	0.93	2
	MC	21.68	14.28	9.28	7.23	4.76	3.09	4.13	3
Water absorption recovery	*T*	59.51	55.79	37.25	19.84	18.60	12.42	7.42	3
	t	61.2	46.71	44.64	20.40	15.57	14.88	5.52	3
	MC	62.88	51.57	38.10	20.96	17.19	12.70	8.26	3

Table A.2
Range analysis of MOR and MOE of densified Chinese fir.

	Factor	K_1	K_2	K_3	k_1	k_2	k_3	R	Optimum level
MOR	*T*	216.20	255.90	258.40	72.07	85.30	86.13	14.07	3
	t	220.20	257.20	253.10	73.40	85.73	84.37	12.33	2
	MC	215.50	229.50	285.50	71.83	76.50	95.17	23.33	3
MOE	*T*	40.30	41.00	43.40	13.43	13.67	14.47	1.03	3
	t	43.00	43.90	37.80	14.33	14.63	12.60	2.03	2
	MC	38.00	40.40	46.30	12.67	13.47	15.43	2.77	3

Table A.3
Range analysis of CSP of densified Chinese fir.

	Factor	K_1	K_2	K_3	k_1	k_2	k_3	R	Optimum level
CSP	*T*	146.30	147.00	140.50	48.77	49.00	46.83	2.17	2
	t	146.90	147.70	139.20	48.97	49.23	46.40	2.83	2
	MC	126.20	168.20	139.40	42.07	56.07	46.47	14.00	2

Table A.4
Raw data of the experimental test results of moisture adsorption recovery.

Test group.	Moisture adsorption recovery of each test sample (%)											
	1	2	3	4	5	6	7	8	9	10	11	12
1	9.52	9.65	10.63	8.45	12.14	8.70	7.53	10.99	10.59	8.08	10.76	9.74
2	5.04	4.20	4.38	4.73	4.81	4.62	4.13	4.48	5.33	4.78	5.76	4.55
3	2.15	2.39	2.64	2.79	2.24	2.55	2.48	2.89	2.37	1.85	2.06	2.64
4	7.06	4.67	6.16	6.03	6.17	5.07	5.45	4.47	5.03	5.59	5.97	6.98
5	2.13	3.22	2.30	2.95	2.47	2.94	2.78	3.12	2.85	2.16	3.30	2.35
6	6.12	4.85	6.80	6.22	5.17	5.21	5.55	5.02	5.89	6.49	5.75	4.72
7	5.28	5.45	3.96	4.48	4.25	4.81	3.69	3.84	3.49	3.04	4.71	2.79
8	6.48	6.84	4.55	5.38	4.57	5.18	6.96	7.87	7.49	5.75	7.74	6.81
9	3.82	3.33	3.11	3.93	4.36	4.41	3.42	3.83	4.92	2.56	3.53	4.71

Table A.5
Raw data of the experimental test results of water absorption recovery.

Test group.	Water absorption recovery of each test sample (%)											
	1	2	3	4	5	6	7	8	9	10	11	12
1	25.83	29.63	27.87	23.57	25.23	32.24	27.55	26.16	28.42	27.9	30.29	29.62
2	24.31	25.89	22.43	20.22	21.67	22.98	23.66	20.08	24.53	25.60	19.20	22.19
3	13.40	9.22	8.42	10.37	12.65	11.62	9.82	10.63	10.61	12.28	8.14	10.16
4	18.09	17.04	16.18	16.79	19.97	21.59	17.84	19.05	14.54	18.67	16.41	16.11
5	15.36	12.56	13.05	11.49	11.78	14.76	14.43	14.26	13.59	12.55	15.35	13.22
6	14.22	16.53	16.87	15.78	17.97	18.47	15.09	11.35	14.17	17.82	14.42	13.16
7	12.02	13.48	17.55	15.83	12.55	14.36	14.01	18.19	13.35	11.01	10.30	14.90
8	20.32	20.32	18.37	23.78	18.79	21.87	20.82	17.35	21.26	16.11	16.61	18.77
9	8.69	9.19	9.94	12.72	11.27	10.62	13.88	12.61	12.57	11.24	12.90	8.20

respectively, however, the difference is barely noticeable when temperature and duration time were raised to 120 ℃ and 30 min. This phenomenon could be explained by a previous study, which states that the intermolecular and intramolecular chemical bonds begin to break with a rate when heated at a temperature above 100 ℃[35]. The break of the chemical bonds was a result of wood thermal softening and was responsible for the tight combination of cellulosic fibers and the amorphous matrix including lignin.

Fig. 3.5 shows that MOE increased by 6.3 % and 21.8 % when moisture content increased from 30 % to 40 % and 40 % to 50 % respectively. This could be ascribed to the improvement of wood plasticity. The plasticity of wood is mainly thought to be dependent upon the softening properties of the main chemical components, namely, cellulose, hemicellulose, and lignin. Although the softening temperature of

Table A.6
Raw data of the experimental test results of MOR.

Test group.	MOR of each test sample (MPa)											
	1	2	3	4	5	6	7	8	9	10	11	12
1	48.49	50.84	63.29	64.78	58.88	59.22	67.56	54.84	48.87	62.07	52.43	60.44
2	74.52	83.01	72.63	59.07	79.08	75.02	71.12	84.22	77.59	65.71	62.25	68.49
3	96.99	75.96	80.20	92.90	94.09	93.08	81.95	99.68	86.28	78.55	104.74	94.93
4	60.96	61.76	65.31	74.94	69.89	67.65	79.00	73.52	74.41	72.73	76.41	57.11
5	92.08	109.19	120.45	114.04	95.95	106.70	96.96	107.00	105.58	111.70	114.68	103.98
6	76.29	84.69	68.26	83.93	80.65	84.74	91.19	74.62	82.95	88.57	79.79	78.40
7	92.75	104.39	89.32	99.77	86.44	84.01	78.45	86.52	89.58	83.07	93.34	81.75
8	76.48	67.11	77.41	84.07	88.81	72.05	67.50	77.44	79.52	71.53	74.17	84.81
9	81.50	94.18	86.81	73.63	75.53	99.38	85.51	82.99	89.12	86.92	97.67	94.11
N	33.43	36.31	35.36	37.45	44.20	29.73	31.11	36.45	38.27	41.83	42.30	33.11

Table A.7
Raw data of the experimental test results of MOE.

Test group.	MOE of each test sample (GPa)											
	1	2	3	4	5	6	7	8	9	10	11	12
1	11.96	13.11	11.24	13.49	12.35	11.19	12.53	11.07	12.96	14.76	10.55	13.84
2	15.32	11.31	13.79	14.30	12.31	14.67	16.48	14.13	13.29	15.86	14.23	11.22
3	14.94	16.95	17.20	16.62	18.59	15.63	18.83	15.98	17.26	13.85	18.54	15.41
4	14.07	14.53	13.49	13.98	12.88	11.17	11.83	15.36	15.56	14.32	15.92	14.54
5	13.79	14.60	13.13	16.48	18.73	15.44	15.76	15.05	16.31	17.63	14.58	17.04
6	12.08	14.29	14.53	17.27	14.62	15.50	16.15	14.81	12.46	13.16	12.79	13.24
7	13.11	16.10	13.72	14.23	14.09	15.49	12.25	13.77	12.86	14.94	11.29	14.63
8	13.09	10.72	10.27	11.38	12.68	12.36	11.06	11.53	11.15	12.48	9.31	11.26
9	12.89	12.26	11.76	12.11	13.99	14.93	12.74	13.45	10.12	13.21	11.67	11.32
N	6.86	6.59	7.03	9.04	9.57	6.89	8.52	7.34	6.69	7.57	8.48	5.89

Table A.8
Raw data of the experimental test results of CSP.

Test group.	CSP of each test sample (MPa)											
	1	2	3	4	5	6	7	8	9	10	11	12
1	44.17	36.95	38.54	41.54	42.27	40.72	36.56	39.83	46.91	42.58	50.25	40.35
2	58.35	61.93	56.69	57.79	57.12	64.4	60.05	62	58.98	59.94	48.67	54.42
3	46.86	49.37	45.74	46.47	46.01	51.08	48.05	49.41	47.33	47.97	40.38	44.13
4	53.82	55.43	59.55	63.24	65.16	55.76	51.32	63.43	64.65	49.43	62.96	57.99
5	51.04	42.97	48.13	47.62	48.02	45.31	46.14	42.73	44.58	46.32	47.13	49.51
6	49.68	44.52	39.56	41.04	39.84	43.11	44.78	37.24	42.81	43.27	39.79	43.83
7	41.16	42.51	46.32	49.25	43.42	41.67	47.35	52.89	47.12	49.28	46.18	44.25
8	42.97	46.86	44.17	41.42	42.63	42.26	43.18	38.72	40.79	37.86	41.53	42.14
9	51.18	47.11	56.42	52.31	48.96	43.62	53.48	56.81	46.27	53.87	55.52	49.34
N	23.18	19.73	20.29	25.16	25.94	22.98	25.18	28.21	24.47	26.01	21.19	21.88

cellulose is not affected by the moisture content, for hemicellulose and lignin, the softening temperature decreased dramatically accompanying with the increase in moisture content [36]. Therefore, a better densification degree was achieved due to the improvement of wood plasticity when wood was treated at high moisture content then led to the enhancement of wood deformation-resistance capacity. As a result, MOE was significantly enhanced. MOE was increased weakly by 2 % when the duration time was raised from 10 to 20 min, while decreased dramatically by 13.9 % when the duration time was raised to 30 min. MOE is sensitive to variations in the material porosity, and the increased number of pores in the wood partly depends on the pyrolysis of hemicellulose [34]. Although the pyrolysis temperature of hemicellulose is above 180 ℃, a certain degree of pyrolysis will also occur when the heating temperature is below 180 ℃, and the degree will intensify as the heating time increases [37]. Therefore, the decrease of MOE when the duration time extended to 30 min could be ascribed to the pyrolysis of hemicelluloses that generated pores. Additionally, the weakening of wood compressibility as a result of moisture excessive evaporation due to the increases in duration time may be also responsible for this decrease. Finally, the increase of 1.7 % and 5.9 % in MOE at 100 and 120 ℃ indicates that MOE is not sensitive to the variance of temperature.

The test results of MOE and MOR of densified Chinese fir were subjected to analysis of variance. Table 7 shows the results of the analysis. The Table indicates moisture content had a strong effect on MOR and MOE, the difference was found to be statistically significant ($P < 0.05$). Heating temperature was found to have a medium-sized effect on MOR ($0.05 < P < 0.1$), while in the case of MOE, the effect was hard to observe($0.1 < P$). The effect of duration time on MOR was slight, while it influenced MOE greatly, although the difference did not reach statistical significance. In light of this, to avoid high loss of stiffness, the length of pre-heating treatment should be controlled strictly.

3.4. Compression strength parallel to grain

Table 8 shows the experiment results of CSP before and after densification. The experiment results were then subjected to range analysis as shown in Table A.3. Table 8 shows that the maximum and minimum CSP values of densified Chinese fir were 58.6 MPa and 41.7 MPa respectively, increased by 147.3 % and 75.9 % compared to natural wood. This significant improvement could be attributed to the increase in the number of wood fibers subjected to compression load per unit area.

Fig. 3.6 was plotted according to the results of range analysis. As presented in the Fig, the main factor affecting CSP was moisture content, followed by duration time and heating temperature. The optimum processing parameters to improve CSP were found to be temperature = 100 ℃, duration time = 20 min, and moisture content = 40 %.

The specimens that were pre-heated with a moisture content of 40 % exhibited a higher CSP than others but decreased with the increase in moisture content. CSP was increased by 33.3 % at the moisture content of 40 % and decreased by 17.1 % at 50 %. The enhancement could be ascribed to the increase in densification degree, which was the result of the improvement of wood plasticity upon the increase in moisture content. The decrease could be explained by the structure of wood fibers. The walls of wood fibers are a composite consisting of hemicellulose and lignin reinforced by cellulosic microfibrils. The excessive decrease in the glass transition temperature of hemicellulose and lignin due to the increase in moisture content, resulted in the wall of the fiber collapsing and folding easily during densification. Therefore, the stability of wood fiber was easier to lose when undergoing uniaxial compressive load. The difference between the variation trend of MOR and CSP accompanied by the increase in moisture content can be explained by the failure mode. The failure mechanism for the wood subjected to axial compressive load was the buckling of wood cell walls, while in the case of the three-point bending test, the failure mechanism was the breakage of wood fibers. There was a slight decrease of 5.2 % and 4 % in CSP when the duration time increased from 10 min to 30 min and the heating temperature from 80 ℃ to 120 ℃ respectively. Two relatively flat lines depicted in the figure revealed that the effect of duration time and heating temperature on CSP was very weak.

Table 9 gives the result of the analysis of variance of CSP. It reveals that moisture content had a statistically significant effect on CSP ($P < 0.05$), while the effect of heating temperature and duration time on CSP was negligible ($P > 0.1$). The result greatly agrees with the result obtained from the range analysis.

4. Conclusions

This study was aimed at improving the dimensional stability, physical and mechanical properties of densified Chinese fir by optimizing the pre-heating treatment parameters, the effect of heating temperature, duration time and moisture content on the set recovery, static bending strength, modulus of elasticity in static bending, compression strength parallel to grain of densified Chinese fir were evaluated in this study. This study did not take the influence of other factors, to exemplify, the age of the tree, the growing conditions or the species of wood, into consideration, which is a limitation and will be addressed in future work. The main findings could be summarized as follows:

(1) The experiment results indicated that the increase in moisture content improved dimensional stability, MOR and MOE of densified Chinese fir significantly. Additionally, the compression strength parallel to the grain could be improved by increasing moisture content within the range of 30 % to 40 %. These enhancements could be ascribed to the wood physical and chemical alterations, including the hydrolysis of hemicellulose, the formation of hydrogen bonds, and the decrease in glass transition temperature, which is a result of variation in moisture content during pre-heating. Accordingly, the moisture content of to-be-densified wood should increase properly.

(2) Raising the pre-heating temperature could improve dimensional stability and MOR of densified Chinese fir, while the effect on MOE and compression strength parallel to grain was negligible. The variation in pre-heating temperature affects the wood hygroscopicity and degree of wood substance degradation and further affects the wood quality. Therefore, a higher pre-heating temperature is not recommended by this study to avoid excessive wood degradation.

(3) Duration time was not the significant factor affecting the physical and mechanical properties of densified Chinese fir, while it affects the wood softening behavior to certain degree. To avoid excessive softening that induces the loss of wood strength, duration time needs strictly controlled. Meanwhile, the efficiency of densified wood production can be improved by the reduction of duration time in industrial production.

(4) The optimum pre-heating treatment parameters were found to be temperature = 120 ℃, duration time = 20 min, and moisture content = 50 %, when densification processing parameters were fixed at compression ratio = 50 %, pressing temperature = 140 ℃, compression pressure = 6 MPa, and pressure holding time = 3 h. Most of the properties of densified Chinese fir achieved optimal levels at this condition. Although the lowest water absorption recovery was achieved at the duration time of 30 min, and the highest compression strength was obtained at the temperature of 100 ℃, the effect of the difference between the two parameters was very weak. Considering the compression strength declined significantly when pre-heated at the moisture content of 50 %, densified Chinese fir manufactured at the conditions recommended by this study might be used as the beams instead of columns.

CRediT authorship contribution statement

Panpan Ma: Writing – review & editing, Writing – original draft, Visualization. **Xin An:** Writing – original draft, Data curation. **Feibin Wang:** Methodology. **Hui Huang:** Investigation. **Zhiyuan Chen:** Software. **Shuo Wang:** Validation. **Meng Gong:** Supervision. **Zeli Que:** Project administration, Conceptualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

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Appendix A

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