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Investigation of changes in compressed moso bamboo (*Phyllostachys pubescens*) after hot-press molding

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Abstract

In this study, molding moso bamboo strips to a curved shape using hot-press molding operation was explored. Bamboo strips with different thickness and moisture content (MC) were subjected to press molding under 120–210 °C for different time. Changes in the chemical components of bamboo were analyzed by Fourier-transform infrared spectroscopy (FTIR). Effect of MC on thermal mechanical behavior of bamboo was investigated using dynamic mechanical analysis (DMA). Results showed that the influencing degree of four variables on compression and recovery ratios decreased as: temperature > time > thickness > MC. Compression ratio increased and recovery ratio decreased dramatically when pressing temperature exceeded 180 °C. FTIR analysis indicated that polysaccharide (especially hemicelluloses) underwent a progressive thermal degradation during compression at 180 and 210 °C for 40 min, whereas relative content of lignin increased. DMA results showed that bamboo samples with a higher MC had a lower storage modulus value, confirmed water had a plasticizing effect. The loss factor of bamboo with higher MC (12 and 16%) exhibited two major transitions centred around 100 °C (α_1) and 50 °C (α_2), respectively. The temperature of these α transitions kept almost unchanged as moisture level increased from 12 to 16%. These findings provide fundamental information for the future preparation of curved bamboo as profiled components in engineered products.

Keywords Bamboo · Hot-press molding · Compression recovery ratio · Thermal degradation · Transition

Introduction

Bamboo is an under-utilized material that could be used more efficiently in structural or engineered products analogous to glue-laminated wood, oriented strand board and plywood [1]. As a renewable and inexpensive material, bamboo has the potential to be used as a substitute for wood or expensive timber in bamboo producing countries [2]. Bamboo has a unique fiber-reinforced composite-like structure, consisting of vascular bundles, relatively large arrangements of dense sclerenchyma cells supporting vessels, in a matrix of low density parenchyma [3].

As an existing technique for improving strength properties, compression has been performed on solid wood and bamboo, during which the material is densified by properly heating and a compression force [4, 5]. However, it is difficult to obtain such modified materials with curved or other complicated shapes because this technique is often performed on a plate-pressing. Pressing the materials in molds is one method to make curved products in large numbers. Molding method has long been used for fabrication of wood and other biomass-based composites from powder, particle, or veneer forms. However, the fiber structure of bamboo, which is mechanically beneficial, is damaged in these forms. Bamboo has conventionally been processed by cutting and gluing, which limits the shape of the products and the productivity. It is necessary to investigate changing the shape of bamboo strips using the mold pressing operation, which provides the potential to manufacture profiled bamboo components for utilization in engineering products.

The feasibility of molding solid wood has been examined by Yamashita et al. [6]. They found that the pliability of wood improves as its MC increases, which is due to the softening of hemicellulose and lignin in the presence of water. It is known that wood undergoes elastic-visco-plastic deformation during compression. When the compressed sample is

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moistened and heated in the absence of any external force, it generally returns to its original shape, to a degree depending on the process parameters, i.e., temperature, time and MC during the compression [7]. This phenomenon is known as compression-set recovery. Hydrothermal treatment is well known as an effective method of wood plasticization. It reduces the softening temperature of the amorphous components, which is also an important process to fix the shape of compressed and bent wood [8]. During compression at elevated temperature, the presence of water serves to plasticize cell wall components, while the effect of heat is to bring the cell wall above its glass transition temperature, which has been reduced by plasticization effect [1].

The deformation and fixation of compressed wood have been studied and widely reported [9–12]. However, the information on basic properties of compressed bamboo for supporting higher utilization of such materials are still lacking, especially those related to softening behavior, deformations and setting of compressed conditions. We, therefore, studied the practice of molding bamboo strips to a curved shape with a small radius of curvature using this hot-press molding operation. In this paper, bamboo strips with different thicknesses and MC were subjected to press molding under 120–210 °C for different time. The influence of these variables (temperature, time, MC and thickness) on compression and recovery ratio, color changes, microstructure, mass loss, chemical changes and thermal–mechanical behavior were investigated to give fundamental information on bamboo press molding under hydrothermal conditions.

Experimental section

Materials

Moso bamboo (*Phyllostachys pubescens*) aged 3–4 years was obtained from Fujian province. Tangentially oriented strips (22 mm tangential, 100 mm longitudinal) with different thickness (0.9, 2.3, 3.6 or 4.9 mm radical) were cut from the moso culm section (Fig. 1). The average density of the air-dried strips is 700 kg/m³.

Determination of compressive deformation and recovery ratios of bamboo

The bamboo specimens were conditioned to arrive at 4, 8, 12, or 16% MC in a humidity chamber, respectively. Then they were wrapped in aluminum foil and placed in a circular arc mold with radius of 210 mm (Fig. 2). Hot pressing was carried out in the radial direction at a pressure of 1.5–2.0 MPa at different temperatures (120, 150, 180 or 210 °C) for 10, 20, 30 or 40 min, respectively. When reaching the prescribed press time, the compressed samples were
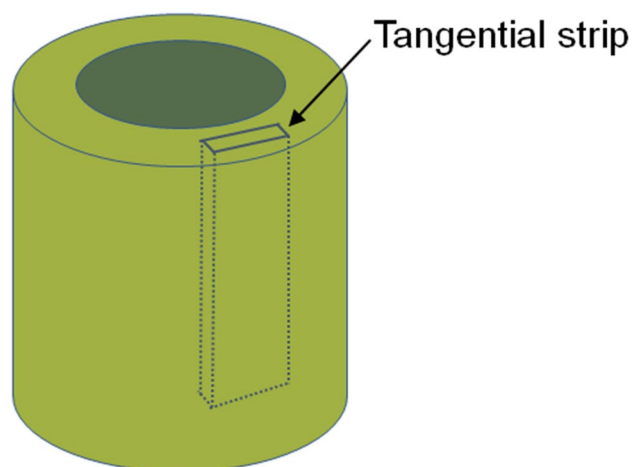

Fig. 1 Tangentially oriented strips cut from moso bamboo culm

taken out of the mold, and then conditioned at 20 °C and 65% relative humidity in a humidity chamber for 3–4 weeks.

The influence of different variables, namely specimen thickness, MC, hot-press temperature and time on deformation and recovery ratio of bamboo specimen was simultaneously evaluated using Taguchi's orthogonal array L_{16} (4^4, 4 factors and 4 work levels). Table 1 demonstrates the different variables evaluated and the conditions chosen for the different experimental groups. 12 replicates were used in each condition and the thickness at three points in each specimen was measured to calculate the average values. The compression ratio (R_c) and recovery ratio (R_r) were determined as follows:

$$R_c = \frac{T_0 - T_c}{T_c} \times 100\%, \tag{1}$$

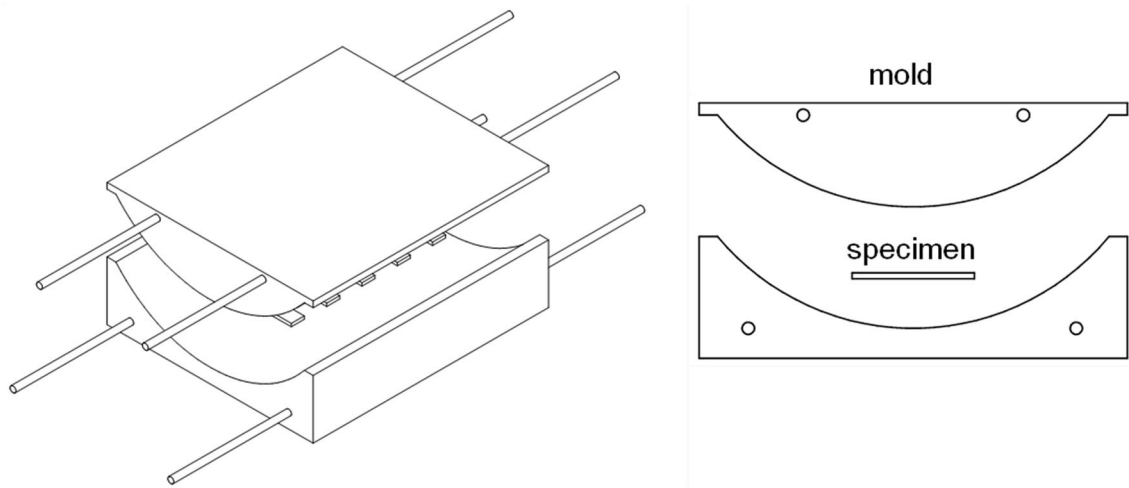$$R_r = \frac{T_f - T_c}{T_0 - T_c} \times 100\%, \tag{2}$$

where T_0 and T_c are the thickness before and after compression, respectively, and T_f is the thickness after equilibrium.

Microscopic observations

Cross-section samples were cut from the bamboo specimens before and after compression. The fractured surface was coated with gold and observed using a Hitachi S-3400N scanning electron microscope with an acceleration voltage of 5 kV.

Mass loss measurement

The mass loss of specimens after hot-press molding was estimated according to Eq. (3):

Fig. 2 Schematic of the circular arc mold used for compression

Table 1 The influence of different compression parameters determined by Taguchi's orthogonal array L_{16} (4^4) on compression and recovery ratios of bamboo

Coding	Thickness (mm)	MC (%)	Temperature (°C)	Time (min)	Compression ratio (%)	Recovery ratio (%)	Mass loss (%)
1	0.9	4	120	10	5.0	98.2	0.37
2	0.9	8	180	40	17.5	19.6	3.95
3	0.9	12	210	20	19.1	15.5	6.68
4	0.9	16	150	30	8.6	58.4	6.23
5	2.3	4	150	20	2.3	99.7	0.54
6	2.3	8	210	30	23.1	8.8	6.45
7	2.3	12	180	10	6.5	48.4	3.44
8	2.3	16	120	40	9.6	27.2	3.12
9	3.6	4	180	30	3.9	57.5	1.79
10	3.6	8	120	20	2.4	63.8	0.87
11	3.6	12	150	40	7.3	39.1	2.73
12	3.6	16	210	10	18.2	13.2	4.67
13	4.9	4	210	40	25.8	6.0	7.79
14	4.9	8	150	10	5.4	46.7	1.36
15	4.9	12	120	30	11.4	24.9	2.33
16	4.9	16	180	20	11.3	24.1	3.89

MC moisture contents

$$\text{Mass loss} (\%) = \frac{w_1 - w_0}{w_0} \times 100\%, \tag{3}$$

where w_0 and w_1 represent the mass of sample before and after compression on oven-dry basis, respectively.

Fourier transform infrared spectroscopy (FTIR) analysis

Chemical changes in the bamboo samples before and after compression were characterized by FTIR (NEXUS 870, Nicolet). The spectra were recorded with 4 cm^{-1} resolution at a wavenumber range of 4000 – 400 cm^{-1}.

Dynamic mechanical analysis (DMA)

Specimens (60 mm × 12 mm × 2.25 mm, L × T × R) were conditioned to reach 8, 12 and 16% MC, respectively, and then wrapped in aluminum foil. The oven-dried specimens with 0% MC were also tested as control. The temperature-dependent dynamic thermomechanical properties were tested using a dynamic mechanical analyzer (DMA 2980, TA) in a three-point bending mode. The experiments were

conducted from 30 to 150 °C at 3 °C/min with a frequency of 1 Hz. The displacement amplitude was set at 15 μm.
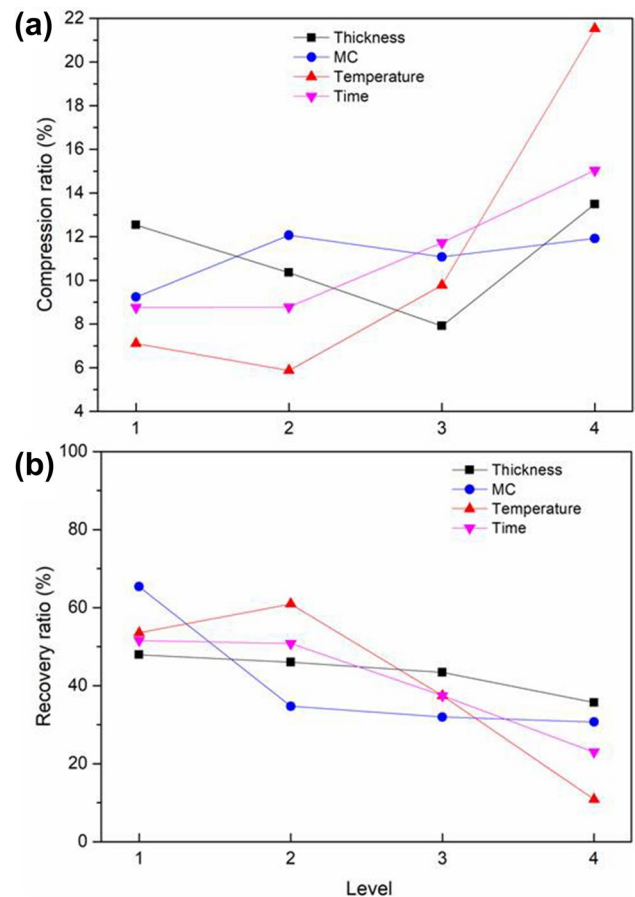
Statistical analysis

Analysis of variance (ANOVA) and analysis of range (ANOR) were performed for compression and recovery ratios and mass loss data using SAS software (Version 8.0, SAS Institute, Cary, NC, USA). If the *P* value is less than 0.05, it indicates that values are significantly different at a significance level of 5%.

Results and discussion

Compression and recovery ratios

The compression and recovery ratios of compressed bamboo for different conditions are shown in Table 1. Both the specimen conditions (thickness and MC) and compression parameters (temperature and time) had significant effects on the compression and recovery ratio. ANOVA revealed that the influencing degree of four variables on compression ratio decreased as: temperature > time > thickness > MC, and recovery ratio exhibited a similar decreasing trend. Generally, compression ratio increased significantly with prolonging compression time. Furthermore, compression at higher temperature resulted in lower recovery ratio after equilibrium. The highest compression ratio and lowest recovery ratio were obtained in specimen with 4.9 mm thickness and 4% MC that was pressed at 210 °C for 40 min.

The significance of difference between four levels for each factor was determined by ANOR (Fig. 3). For hot-press temperature, 120 and 150 °C showed negligible difference on compression ratio, 180 °C led to a remarkable increase, and 210 °C resulted in a compression ratio over 21% (Fig. 3a). Very high recovery ratio was observed for samples compressed at low temperatures. The recovery ratio decreased dramatically when pressing temperature exceeded 180 °C (Fig. 3b). This indicated that higher temperature was beneficial for compression deformation fixation. Hot-press time exhibited a similar trend with temperature. The samples with 0.9 and 4.9 mm thickness showed higher compression ratios than medium thickness. This phenomenon is probably due to the unique microstructure of bamboo culm, which consists of many vascular bundles surrounded by bundle sheaths, reinforcing the bamboo culm. The vascular bundles are distributed densely in the outer side of the culm wall, and sparsely in the inner side [3, 13]. Therefore, the outer region of bamboo possesses a higher density and strength than the inner region. The thicker samples contain more inner parts of culm wall, thus giving larger deformation when subjected
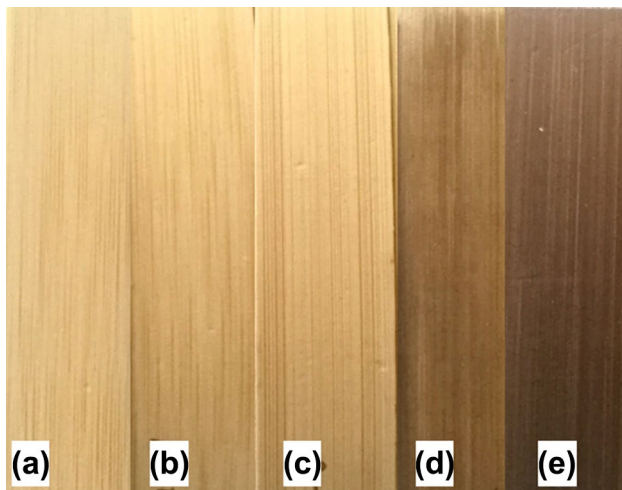

Fig. 3 Influence of variable levels on the compression and recovery ratios of bamboo. Note: level 1 (0.9 mm, 4%, 120 °C, 10 min), level 2 (2.3 mm, 8%, 150 °C, 20 min), level 3 (3.6 mm, 12%, 180 °C, 30 min), level 4 (4.9 mm, 16%, 210 °C, 40 min). *MC* moisture contents

to compression. The compression and recovery ratios of compressed bamboo with various MC did not show significant difference from each other except for 4% MC, at which the compression ratio was lowest and recovery ratio was highest. This indicated that keeping MC above 8% would facilitate compression and subsequent fixation.

Visual observation of surface appearance

The hot-press molding process had a similar effect on the bamboo color with heat treatment. Bamboo samples darkened with increased temperature (Fig. 4). When the temperature exceeded 150 °C, the changes in color were quite apparent. At 210 °C, the color of bamboo turned from light yellow to black. The significant changes in color at high temperatures are probably due to lignin polymerization reactions, which produces intermediate lignin degradation compounds such as quinone and quinone [14]. Besides, transfer

Fig. 4 Photographs of bamboo samples after compression: **a** untreated, **b** Group 8 (120 °C), **c** Group 11 (150 °C), **d** Group 2 (180 °C), **e** Group 13 (210 °C). *Note* The group number refers to Table 1
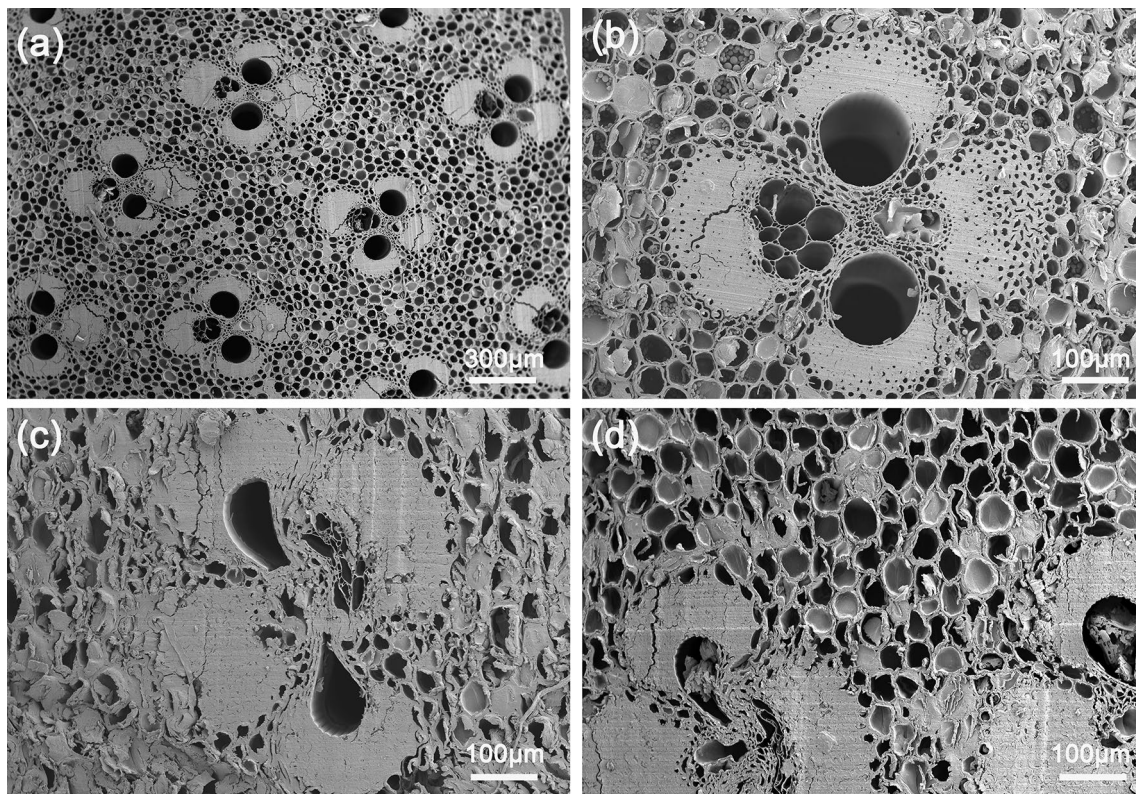
of carbohydrates and other extracts from the interior of sample to the exterior accompanied with the evaporation of moisture also attributed to the darkened color [15].
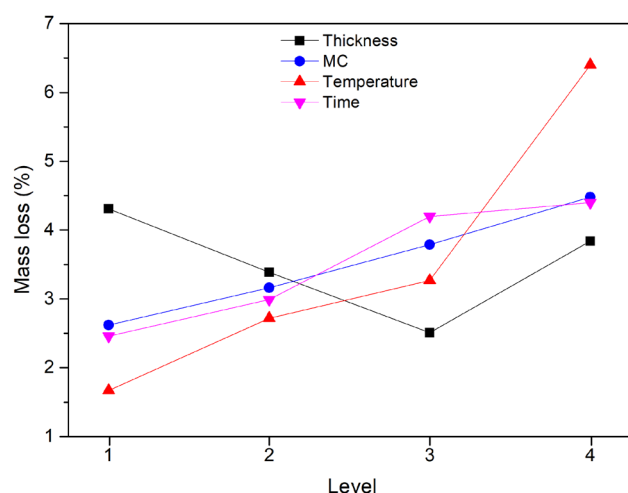
Microstructure of compressed bamboo

Micrographs of cross sections of bamboo before and after compression are shown in Fig. 5. The microstructure of bamboo culm consists of many vascular bundles embedded in parenchyma tissue. Densification of the parenchyma and closure of the vessels are evident for compressed bamboo from the micrographs. Most cells appeared to be deformed after compression. The parenchyma cells have undergone large plastic deformations without noticeable fracture or failure of the cell walls.

Mass loss of compressed bamboo

Mass loss of samples after hot-press moulding is presented in Table 1. Mass loss was less than 3.5% when compressed at 120 °C, which is mainly attributed to water evaporation in bamboo. The effect of heating condition on the mass loss of compressed bamboo shows that the mass loss increased with increasing temperature and heating time, and reached the maximum reduction of 7.8% when samples were compressed at 210 °C for 40 min (Table 1). ANOVA revealed that the influencing degree of four variables on mass loss decreased as: temperature > time > MC > thickness (Fig. 6).

Fig. 5 Scanning electron micrographs of cross section of **a, b** untreated and **c, d** compressed bamboo culm (Group 13). Note: the magnifications of the images are **a** × 50, **b** × 150, **c** × 150, **d** × 150, respectively
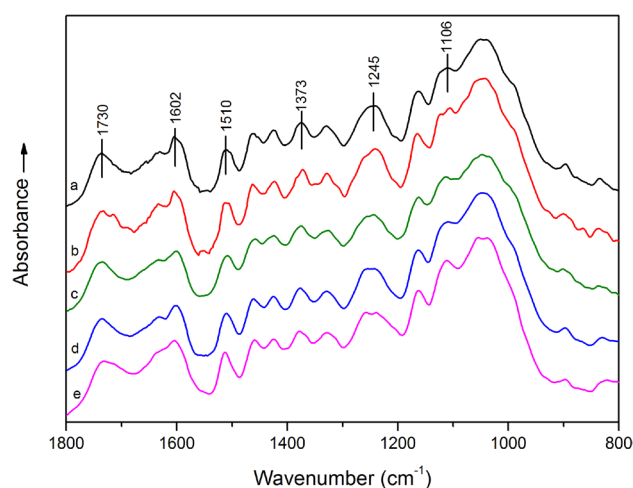
Fig. 6 Influence of variable levels on the mass loss of compressed bamboo. *Note* The level number refers to Fig. 3. *MC* moisture contents

Fig. 7 FTIR spectra of compressed and control bamboo samples: **a** control, **b** Group 8 (120 °C), **c** Group 11 (150 °C), **d** Group 2 (180 °C), and **e** Group 13 (210 °C)

The pressing temperature had a much greater effect on mass loss than did the other three factors. Starke et al. [16] also found that difference in the thermal treatment temperature induced a greater mass loss than an extension of the holding time. Mass loss varied greatly at four different temperature levels from 120 to 210 °C. Zhang et al. [17] studied the effect of thermal treatment at temperature 100–220 °C for 1–4 h on bamboo. They found the sample remained stationary when heat treated below 160 °C, and mass loss reached maximum of 29% when heat treated above 160 °C for 4 h. Previous study by Zhong et al. [15] showed that 175 °C was a key turning point, at which thermal decomposition occurred in bamboo and the mass loss began to increase rapidly. Therefore, it is speculated that the mass loss of samples compressed at 180 and 210 °C is mainly due to the chemical components degradation.

FTIR analysis

Infrared spectra of control and compressed bamboo were compared to investigate the structural changes that occur during hot-press molding. Since the pressing temperature had a much greater effect on mass loss than did the other three factors, specimens compressed at 120, 150, 180, and 210 °C for 40 min (Group 2, 8, 11 and 13) were selected for FTIR analysis. It has been reported that C-H aliphatic stretching (around 2900 cm^{-1}) is more stable than C–OH, C–O–C, and R–COO–R bands and can be expected to stay intact during heat treatment [18]. Therefore, all the spectra were normalized to the absorption band around 2900 cm^{-1} which is chosen as a reference. The FTIR spectra of samples compressed at different temperatures in the studied region present similar patterns (Fig. 7).
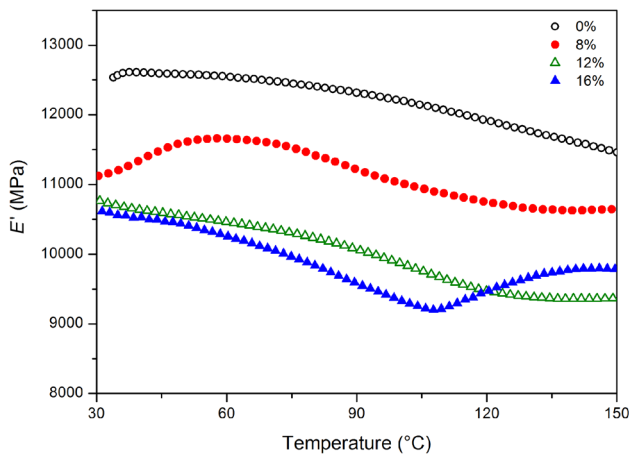
To further quantitatively evaluate the changes of chemical components at various pressing temperatures, the average relative intensities of peaks at 1730, 1602, 1510, 1373, 1245, and 1106 cm^{-1} against peak at 2900 cm^{-1} were calculated using peak heights, and the results are presented in Table 2. There was no significant difference of the relative intensities of these characteristic bands between control and sample compressed at 120 °C. However, the difference gradually became enlarged with increasing temperature, especially at 180 and 210 °C. Relative intensity of peak at 1730 cm^{-1}, which is assigned to non-conjugated C=O stretching in xylan of hemicellulose, decreased after compression at elevated temperature. This is probably caused by the preferential degradation of hemicelluloses over other components. The intensity of peaks at 1602 and 1510 cm^{-1} (aromatic skeletal vibrations of lignin) increased after treatment, indicating increase in relative contents of lignin. In addition, the relative intensity of peak at 1106 cm^{-1} that is attributed to C–H stretching of aromatic ring in lignin also increased after compression. Similar trend has been previously observed by Meng et al. [18] who found that heat treatment led to an increase in relative contents of lignin due to reductions in polysaccharide contents. Relative intensities of the peak at 1373 cm^{-1}, representing C–H deforming and stretching of cellulose and hemicelluloses, decreased at 180 and 210 °C. Another feature that needs to be taken into consideration is the decrease in the peak at 1245 cm^{-1} (guaiacyl ring breathing with CO-stretching of lignin and hemicellulose, esters) after treatment. This indicates the cleavage of acetyl groups in hemicelluloses, which form carbon acids, especially acetic acid. These results suggested that polysaccharide (especially hemicelluloses), whose branched structure and amorphous tissues are more susceptible to thermal degradation than

Table 2 Ratio of the normalized heights of the absorption peaks of control and treated bamboo samples

Samples	I_{1730}/I_{2900}	I_{1602}/I_{2900}	I_{1510}/I_{2900}	I_{1373}/I_{2900}	I_{1245}/I_{2900}	I_{1106}/I_{2900}
Control	1.03	1.45	0.92	0.55	1.14	0.40
Group 8 (120 °C)	1.04	1.44	0.93	0.53	1.15	0.38
Group 11 (150 °C)	1.05	1.46	0.96	0.48	1.07	0.43
Group 2 (180 °C)	1.00	1.47	1.00	0.46	1.05	0.50
Group 13 (210 °C)	0.98	1.49	1.17	0.37	1.03	0.76

The heat treatment time for all these samples is 40 min

I_{1730}/I_{2900}, I_{1602}/I_{2900}, I_{1510}/I_{2900}, I_{1373}/I_{2900}, I_{1245}/I_{2900}, and I_{1106}/I_{2900} refer to the average relative intensities of peaks at 1730, 1602, 1510, 1373, 1245, and 1106 cm^{-1} against peak at 2900 cm^{-1}, respectively

Fig. 8 Storage modulus (E') of bamboo samples with different moisture contents

other components [19], underwent a progressive degradation during compression at 180 and 210 °C. This finding is in agreement with the mass loss data.
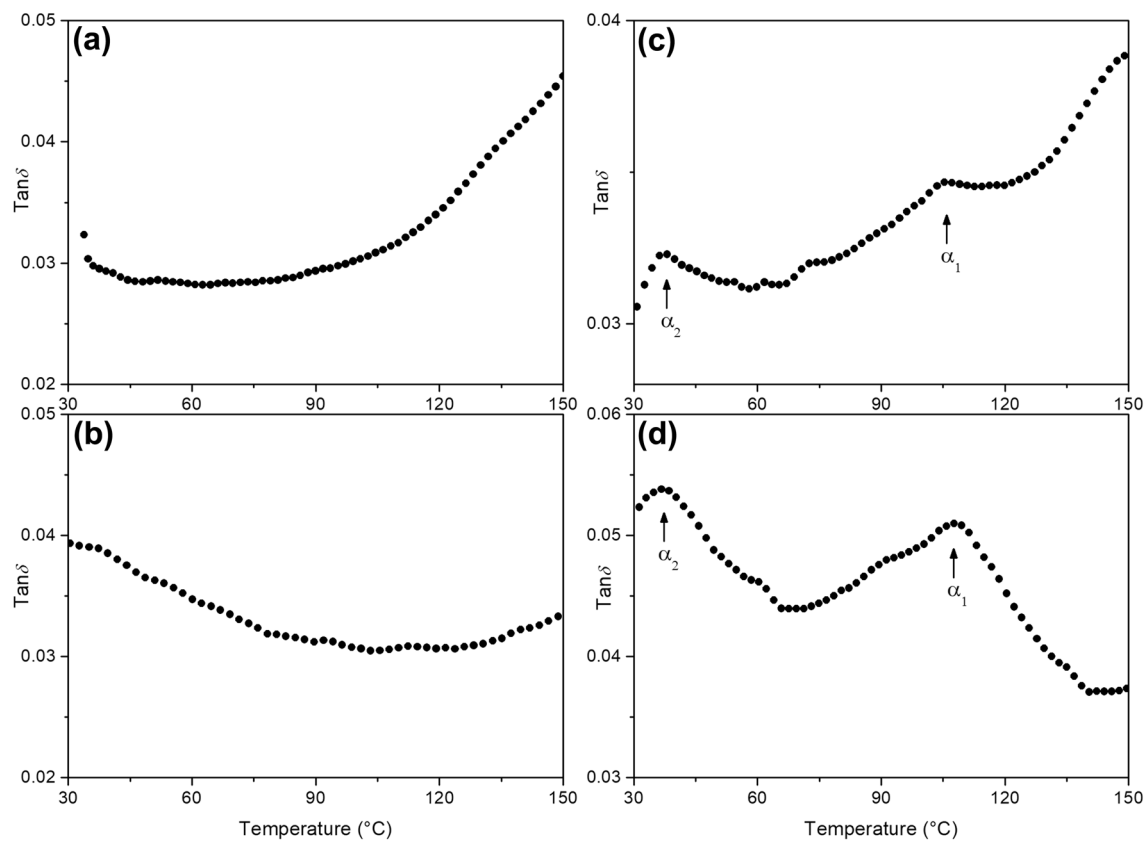
DMA

To clarify the role of MC during the mold pressing, bamboo specimens with different MC were subjected to DMA measurement. Storage modulus (E') values of samples, which are related to the ability of storing mechanical energy, are plotted as functions of temperature (Fig. 8). The general feature of E' variation with temperature for bamboo samples is similar to those observed in previous studies of wood [20, 21]. The decrease of the E' value of all samples with increasing temperature was attributed to the higher chain mobility of cell wall components at elevated temperature. At low temperature region, the kinetic energy of material molecules is very low, with motion occurring only in some small units such as side and branched chains. High temperature could result in crystallization or ordering of the molecules, causing the substantial decrease of E' [22]. It should be noted that, for sample with 16% MC, the E' gradually increased from

around 100 °C. One probable reason is the MC decreased with temperature increase. Because the DMA equipment used in this research lacks relative humidity control, and water started to evaporate from the samples when they were heated up to 100 °C. Over the entire temperature range, the bamboo with a higher MC generally had a lower E' value, indicative of a lower relevant stiffness. Liu et al. [22] also found the storage modulus decreased with water content increase. This phenomenon confirmed the expected role of water as having a plasticizing effect on the bamboo.

Loss factor (tan δ) measurements provided information on transitions of the material. At low MC (0 and 8%) there was no detectable transition during the tested temperature range (30–150 °C) (Fig. 9). Previous study shows that dry wood exhibits only very weak secondary relaxations between −150 and 150 °C, because under this condition the in situ T_g values for hemicellulose, lignin and amorphous cellulose occur at above 150 °C [23]. The bamboo materials with 12 and 16% MC exhibited two transitions: one transition centred around 100 °C (α_1); and the other between 30 and 60 °C (α_2). Similar results have been previously observed by Liu et al. [22] who found that α_1 transition occurred around 80 °C and α_2 transition around 30 °C for moso bamboo with 8 and 12% MC. The α_1 and α_2 transitions are probably linked to the glass–rubber transition of lignin and hemicelluloses, respectively. The presence of water has been found to plasticize cell wall components, causing a reduction of energy required to initiate chain mobility. Consequently, the precise temperature of α transitions is expected to vary with MC. However, in this study the temperature of α transitions kept almost unchanged as moisture level increased from 12 to 16%. The sensitivity of these transitions to moisture complicated the DMA analysis since the relative humidity of the sample chamber is difficult to control. It should be noted that the α_1 transition of sample with 16% MC occurred in a broader range than 12%, most likely due to the loss of moisture in the sample during the scan. It has been reported in the literature that moisture loss can lead to a broadening of the tan δ response, an effect which would be most pronounced for samples with a transition around 100 °C [20].

Fig. 9 Loss factor (tan δ) of bamboo samples with different moisture contents: **a** 0%, **b** 8%, **c** 12%, **d** 16%. *Note* α_1 refers to the transition centred on 90–110 °C and α_2 refers to the transition centred on 30–60 °C, respectively

Conclusion

For manufacturing profiled bamboo components that can be used in structural or engineered products, it is essential to understand the deformation and softening behaviour of curved bamboo. The presented work investigated the influence of variables including thickness, MC, hot-press temperature and time on the compression of curved moso bamboo during hot-press molding. Experimental results indicated that temperature had the most prominent effect on the compression and recovery ratios of bamboo, followed by time, thickness and MC. Generally, pressing at higher temperature (180 and 210 °C) for longer time (30 and 40 min) resulted in higher compression ratio, as well as lower recovery ratio after equilibrium. This is related to the preferential thermal degradation of cellulose and hemicelluloses. Keeping MC of bamboo above 8% would facilitate compression and subsequent fixation, attributed to the plasticizing effect of water. No significant difference for the glass–rubber transition temperatures of cell wall components was observed as moisture level increased from 12 to 16%. This is probably due to that the DMA equipment used in this research lacks relative humidity control of the sample chamber.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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