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The mechanical properties and thermal conductivity of bamboo with freeze–thaw treatment

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Abstract
The aim of this research was to investigate the effect of freeze–thaw treatment on bamboo with different initial moisture content (water-saturated, air-dried and oven-dried). Bamboo (*Phyllostachys pubescens*) were treated with two freeze treatments and its microstructure, chemical composition, mechanical properties and thermal conductivity were characterized by field emission scanning electron microscopy (FE-SEM), Fourier-transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), mechanical testing machine and thermal conductivity tester, respectively. The results showed that the freeze–thaw treatment had little influence on the microstructure of bamboo, the chemical composition content and the cellulose crystalline structure of bamboo were also not altered. The crystallinity index was found to increase with the increase of initial moisture content. The bending strength and elastic modulus of the treated bamboo increased, the extent of the increase was dependent on the initial moisture content and the freezing temperature. The thermal conductivity of the treated bamboo increased remarkably, which might be possibly determined by the cellulose crystallinity, moisture content, and density of bamboo.

Keywords: Bamboo, Freeze–thaw treatment, Mechanical properties, Thermal conductivity

Introduction
With the increasing concerns about low carbon development, renewable biomass materials have attracted growing attention [1]. Bamboo, as a biomaterial with high mechanical properties and effective reduction of greenhouse gases [2], has been applied in architecture, interior, furniture, etc. Although bamboo has been long known as a building material with a long history, to date, it still has not widely accepted as the main material for architecture due to the lack of unified and reliable design standards [3]. Bamboo needs to be further explored intensively and extensively to provide comprehensive data with an expectation for making the design standards of bamboo. The material properties of bamboo are often affected by the moisture and the environmental temperature, especially when used outside in extremely cold environments [4]. Previous researches showed that the moisture content and the temperature were two important factors influencing wood mechanical properties [5, 6]. The mechanical properties of wood with an initial moisture content of 8–9% increased when treated below ambient temperatures [7]. De-Geer et al. found that the modulus of elasticity (MOE) and bending strength of wood lumbers increased when the treatment temperature decreased [8]. The bending strength and MOE of wood increased by 34% and 38%, respectively, when the treatment temperature decreased from 40 to – 40 °C [9]. Previous studies indicated that the mechanical properties of wood increased when treated at low temperature, because the polymers like cellulose and the lignin in wood would be close to each other with the decrease of temperature.

Unlike wood that comprises vessel, tracheid, wood fiber, wood parenchyma cell and wood ray, bamboo

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mainly consists of vascular bundles and matrix [10–12]. The density gradually decreased and formed a gradient structure as the distribution of vascular bundle decreased from the outer layer to the inner layer of bamboo [13, 14]. This unique structure of bamboo has a significant impact on its mechanical properties. However, there was limited information on the changes of mechanical properties and physicochemical of bamboo under low temperature. In this regard, the studies on the change in materials properties of bamboo under freeze–thaw treatment might be important for its development and utilization in outdoor applications, particularly for the use in extremely cold environments.

This research investigated the effects of freeze–thaw treatment on bamboo with different initial moisture contents. The microstructure, chemical composition, mechanical properties and thermal conductivity of bamboo were characterized by field emission scanning electron microscopy (FE-SEM), Fourier-transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), mechanical testing machine and thermal conductivity tester.

Materials and methods

Materials

Three-year-old Moso bamboo (*Phyllostachys pubescens*) was collected in August from Zhejiang province, China, because 3- to 5-year-old bamboo was usually produced into laminated bamboo, flattened bamboo, bamboo scrimber used in building and furniture. The bamboo culms at a height of 2–4 m from the base were obtained. The sample sizes are shown in Fig. 1. According to the gradient structure of bamboo, the parts close to outer layer and inner layer of bamboo were separated for FE-SEM observation, FT-IR, XRD and thermal conductivity analysis. Bamboo was cut into 20 mm in length and width and 3 mm in thickness. For the mechanical properties test, the bamboo was cut into 160 mm in length, 10 mm in width and the thickness of the sample was that of the bamboo wall.

Sample preparation

The samples were divided into six groups according to three moisture contents and treated by two kinds of freeze–thaw processes. C0 was the control sample, T and F were referred to – 20 °C and – 40 °C, respectively. The number 1, 2, 3 were referred to three different moisture contents, respectively. The freeze–thaw treatment parameters are shown in Table 1.

Microstructure and chemical composition analysis

The field emission scanning electron microscope (FE-SEM, XL30 ESEM FEG, FEI Company, USA) was used to observe the cross-section of the outer and inner layer of bamboo after freeze–thaw treatment. Each sample tested one specimen. The FT-IR spectra of the samples were obtained with a spectrometer (VERTEX 80 V, Bruker, German) within the range of 4000–500 cm^{-1} at a resolution of 4 cm^{-1} and 64 scans. Two specimens were tested for each sample. All samples were cut into a length and width of 19 mm × 19 mm for XRD testing, three replicates were measured for each sample. The crystalline structure of cellulose untreated and treated samples was measured by an X-ray diffractometer (XRD, Ultima IV, Rigaku, Japan) with a CuKα radiation source, the scan speed was 10°/min and the scan range was 5°–45°. The crystallinity index (CrI) of samples was calculated by the following formula (1):

$$\mathrm{CrI} = \frac{I_{200} - I_{\mathrm{am}}}{I_{200}} \times 100\%, \tag{1}$$

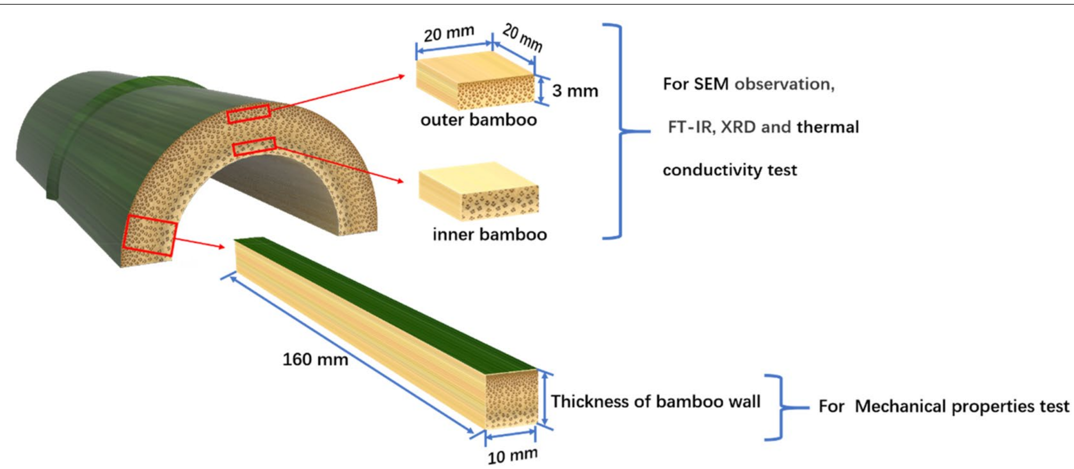

Fig. 1 Sample preparation from different parts of bamboo culm

Table 1 The parameters of freeze–thaw treatment

Treatment condition	Sample initial state	Treatment		
		Freezing treatment	Place at room temperature	Heat treatment
C0	Air-dried	–	–	–
T-1	Water-saturated	− 20 °C/24 h	About 28 °C/24 h	60 °C/24 h
T-2	Air-dried			
T-3	Oven-dried			
F-1	Water-saturated	− 40 °C/24 h	About 28 °C/24 h	60 °C/24 h
F-2	Air-dried			
F-3	Oven-dried			

Water-saturated: the sample was placed in the water for 24 h, the moisture content of the samples from the inner layer and the outer layer of bamboo was about 65% and 80%, respectively; air-dried: the sample was dried in the oven at 60 °C until the weight was stable and the moisture content of the samples was about 8–12%; oven-dried: the sample was dried in the oven at 103 °C until the weight was stable and the moisture content of the samples was 0%

The parameters of the freeze–thaw treatment were set according to the ASTM D 6662-2007

where I_{200} is the the maximum intensity of the (200) diffraction peak, and I_{am}, the amorphous diffraction intensity.

Mechanical properties and thermal conductivity

The bending test was conducted according to GB/T 15780-1995 [15] and performed by a universal testing machine (AGS-X, Shimadzu, Japan) with a strain rate of 10 mm/min. Five effective specimens were tested for each sample. The final moisture content of untreated and free–thaw-treated bamboo strips for bending strength and elastic modulus test was 10% and 5–8%, respectively. The thermal conductivity of bamboo was measured with a thermal conductivity tester (TCI-2-A, C-Therm, Canada), the test temperature was 25 °C. In the testing process, a single-side thermal reflection probe contacting the interface of the sample was used to provide a transient heat source for the sample, and then the thermal conductivity of the sample was directly measured and analyzed by using its equipped data model. Three specimens were tested for each sample. The thermal conductivity of samples was calculated by the following formula (2):

$$k = \frac{\mu^2}{\rho C_P}, \quad (2)$$

where k is the thermal conductivity (W/(m K)); μ, the thermal effusivity (W/(m^2 K)); ρ, the density (kg/m^2), and; C_p is the specific heat capacity (J/(kg K)).

Results and discussion

Microstructure

The microstructure of the cross-section of the treated inner and outer bamboo is shown in Fig. 2 and Additional file 1: Fig. S1. The change of microstructure in outer bamboo and inner bamboo was similar. There was no significant change in the microstructure of fibers and parenchyma cells with different moisture contents after freeze–thaw treatment. The parenchyma cells in untreated bamboo had a round cell cavity and massive starches. After freeze–thaw treatment, the shape of parenchyma cells did not change, but the number of starches reduced dramatically. It can be seen from T-2 and T-3 that there were only a few parenchyma cells containing a large amount of starch. In addition, there was almost no starch in bamboo treated with T-1. The number of starches reduced more pronouncedly and even disappeared when treated at – 40 °C, especially in the F-1and F-2. A previous study showed that the content of amylose in noodles decreased significantly during 10-day frozen storage (− 18 °C), which was probably attributed to the growth and diffusion of ice crystals, resulting in the starch granules being damaged mechanically. Another reason was that the freeze process resulted in the increase of α-amylase activity, which led to the rapid degradation of starch [16]. Meziani et.al revealed that lower temperature, i.e., – 40 °C, would lead to phase change and breakdown of starch [17]. In addition, moisture content was another crucial factor for starch retrogradation. The cross-linking entanglement and crystal rearrangement of starch molecules depend on the moisture content [18], the hydrogen bonds between starch molecules and water molecules were easy to break, and stable hydrogen bonds could be formed between starch molecules. When the moisture content of starch is at a high level, the chances of cross-linking entanglement and polymerization of starch molecules decreased, which hindered the crystal rearrangement of starch molecules [19].

Chemical compositions

The FT-IR spectra of freeze–thaw bamboo are displayed in Fig. 3. The band at 1730 cm^{-1} was attributed

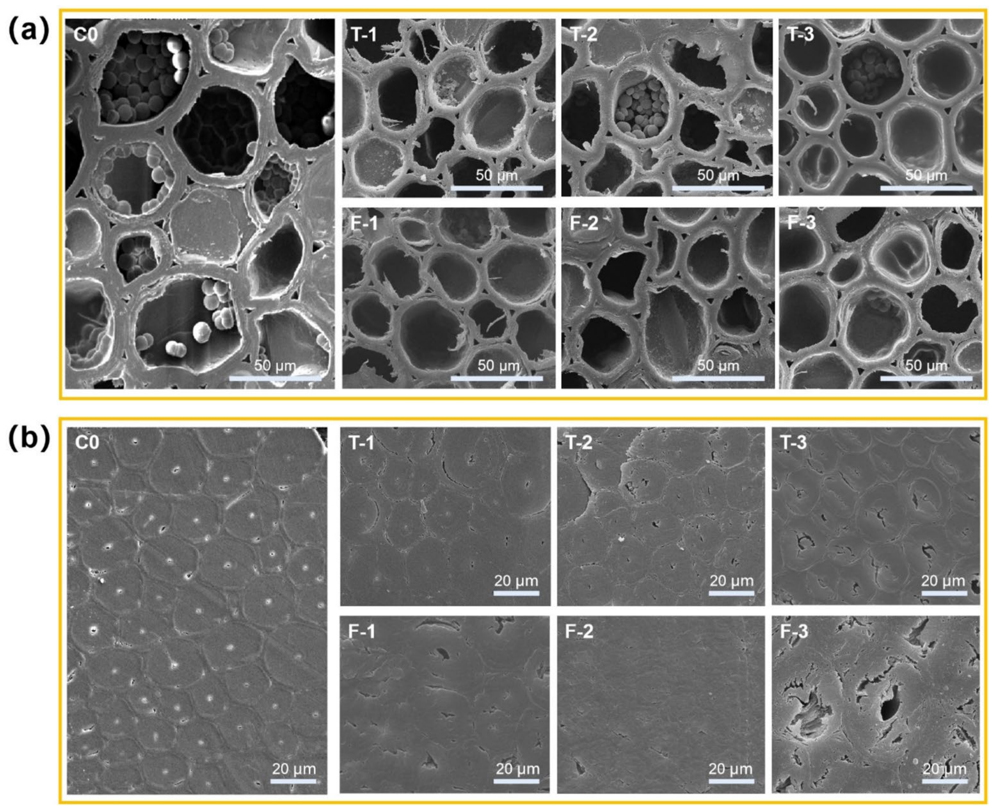

Fig. 2 Microstructure of freeze–thaw-treated **a** parenchyma cells and **b** fibers of inner layer bamboo

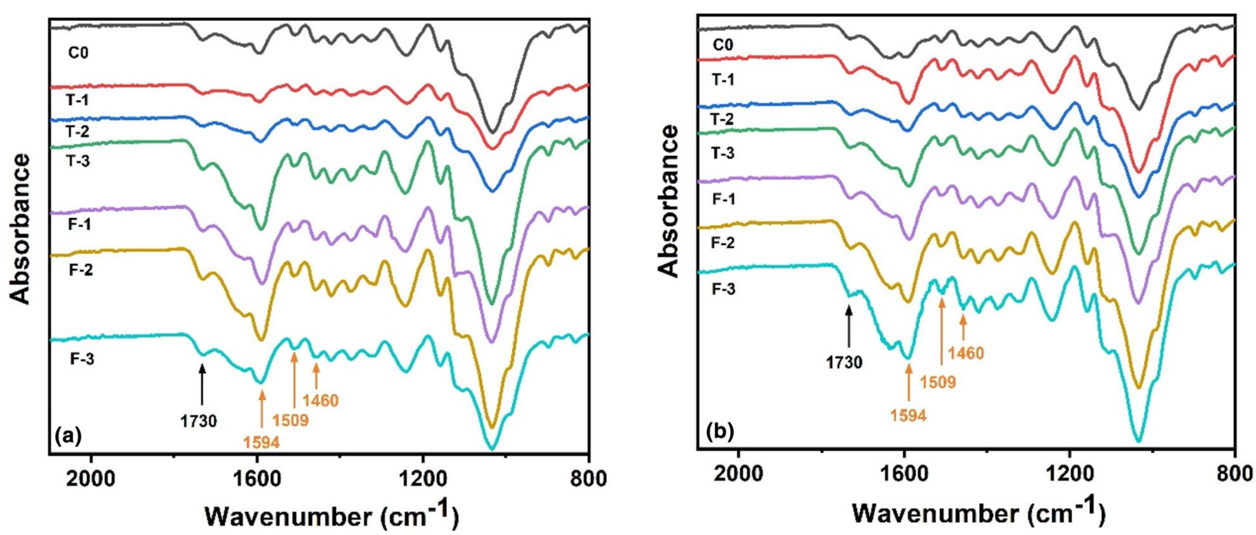

Fig. 3 FT-IR spectra of bamboo after freeze–thaw treatment with different moisture content. (**a** From the outer layer of bamboo, **b** from the inner layer of bamboo.)

to the C=O vibration of hemicellulose in bamboo [20]. The characteristic bands of lignin were at 1594 cm^{-1}, 1509 cm^{-1} and 1460 cm^{-1}, respectively (aromatic skeleton vibration) [21]. The absorption band did not change significantly in both inner and outer bamboo regardless of the difference in their moisture content or freeze–thaw treatment, indicating that the freeze–thaw treatment had little impact on the chemical composition of bamboo.

Figure 4 shows the XRD patterns and crystallinity index of cellulose in freeze–thaw-treated bamboo. The peaks at 2θ around 16°, 22° and 34° in the XRD patterns of untreated bamboo were ascribed to (10_), (1200) and (040) reflection of the crystalline structure of typical cellulose I [22]. Similar XRD patterns of all treated bamboo were observed as the untreated samples, suggesting the similar cellulose I. The crystallinity index of outer and inner bamboo is presented in Fig. 4c and the ANOVA analysis is shown in Additional file 1: Tables S1–S4. The CrI of cellulose of the water-saturated bamboo with freeze–thaw treatment was the highest. Moreover, the ANOVA analysis showed the moisture content ($P_{\text{inner bamboo}} = 0.026 < 0.05$) had a significant effect on the CrI of inner layer bamboo, but

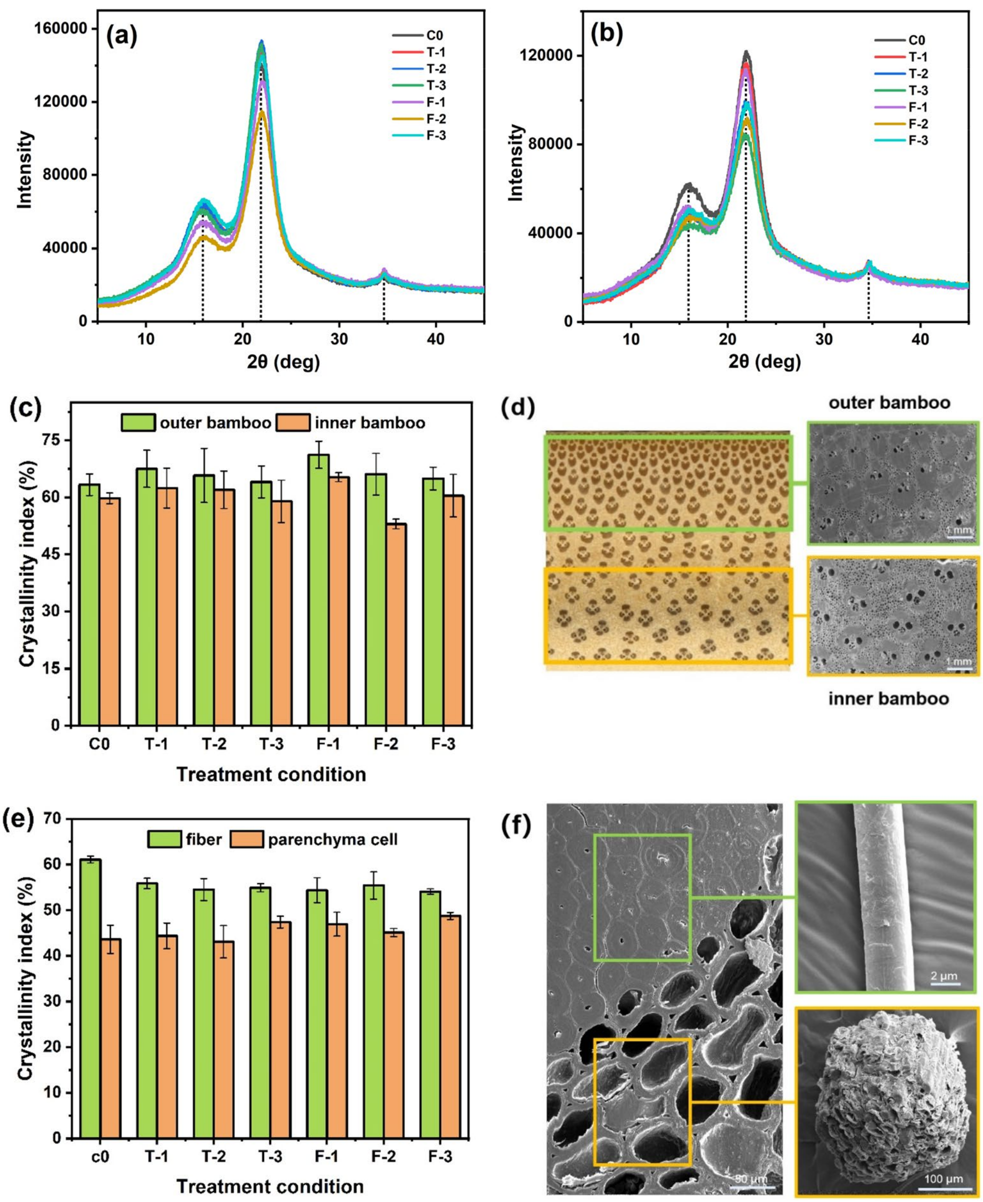

Fig. 4 XRD patterns and crystallinity index of bamboo after freeze–thaw treatment with different initial moisture content. (**a** XRD patterns from the outer layer of bamboo; **b** XRD patterns from the inner layer of bamboo; **c** CrI of the outer and inner layer of bamboo; **d** the difference of fibers and parenchyma cells content of outer and inner bamboo; **e** CrI of fiber and parenchyma cell; **f** the microstructure of fiber and parenchyma cells from bamboo.) (The error bars refer to the standard deviation which reflects the degree of dispersion of the value relative to the mean.)

the temperature affected little on both inner and outer layer bamboo. It indicated that the higher the moisture content was, the higher the CrI of inner bamboo. The outer layer of bamboo contained more fibers and fewer parenchyma cells compared with the inner layer of bamboo (Fig. 4d). To get more insight into the difference in crystallinity, the fibers and parenchyma cells were separated mechanically from bamboo and treated with the same methods. The CrI of the freeze–thaw-treated bamboo fibers and parenchyma cells with different moisture content is shown in Fig. 4e. The CrI of fibers was much higher than that of parenchyma cells, which may account for that the CrI of outer bamboo was higher than that of inner bamboo. After freeze–thaw treatment, the CrI of outer bamboo slightly increased, the extent of increase varied depending on the moisture content.

Mechanical properties

Figure 5 shows the bending strength and elastic modulus of bamboo after freeze–thaw treatment with different moisture contents. Both the bending strength and elastic modulus increased after freeze–thaw treatment, and the lower the treatment temperature was used, the higher bending strength was achieved. Also, the ANOVA analysis is presented in Additional file 1: Tables S5–S7. It showed that the temperature ($P=0.003<0.05$) had a significant effect on the bending strength. The previous research indicated that the molecule of cellulose and lignin approached each other much tighter and the connection became stronger when the temperature decreased, which led to the increase of strength [9]. Therefore, the bamboo slivers treated at the lowest temperature have the highest strength. However, the elastic modulus of bamboo strips with freeze–thaw treatment at − 20 °C was higher than that treated at − 40 °C. In addition, the moisture content had a significant influence on

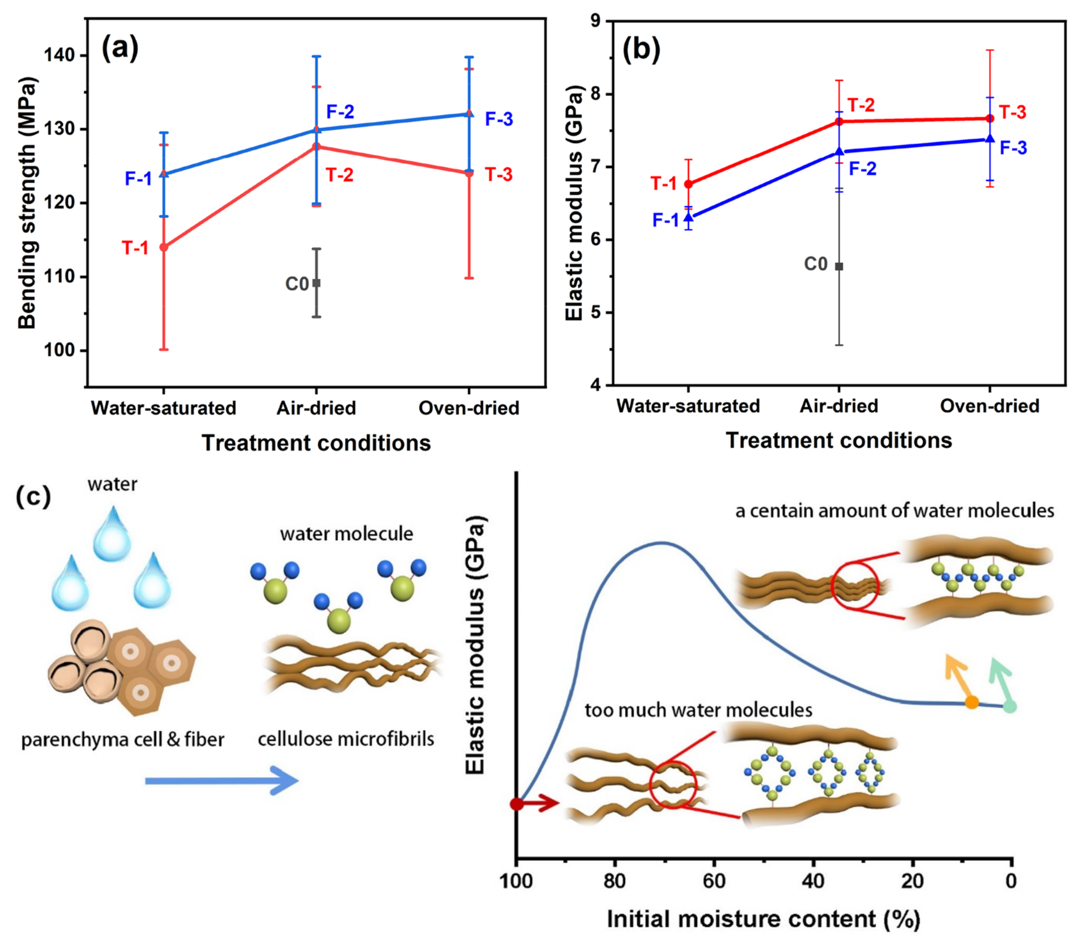

Fig. 5 The change of bending strength and elastic modulus of bamboo after freeze–thaw treatment with different moisture content. (**a** bending strength, **b** elastic modulus, **c** schematic diagram of water entering cell wall under different moisture contents [23].) (The points are the mean values and the error bars refer to the standard deviation.)

the mechanical properties of bamboo strips with freeze–thaw treatment. The ANOVA analysis in Additional file 1: Tables S8, S9 shows both the moisture content ($P=0.041<0.05$) and the temperature ($P=0.001<0.05$) have a significant effect on the elastic modulus, but the temperature plays a more critical role. However, there was no significant change between − 20 and − 40 °C ($P=0.173>0.05$) (Additional file 1: Table S10). The elastic modulus of bamboo strips was greatly influenced by low temperature, but it was not that the lower the temperature, the greater the elastic modulus. The mechanical properties of bamboo strips with less moisture content increased more significantly after freeze–thaw treatment. The bending strength and elastic modulus of water-saturated bamboo strips (T-1 and F-1) were the lowest in comparison with those air-dried and oven-dried ones. It might be possibly ascribed to that a large number of water molecules diffused into the cell wall, the water molecules not only formed hydrogen bonds between cellulose molecules, but continued to form hydrogen bonds with the molecules that had already formed hydrogen bonds. Moreover, it led to the increase of weak bonding among water and water in cellulose molecule chains, which would weaken the intermolecular forces and eventually result in a decreased elastic modulus [23].

Thermal conductivity

The thermal conductivity of the outer and inner layer of bamboo after freeze–thaw treatment is shown in Fig. 6. The thermal conductivity of untreated outer and inner bamboo was almost the same. This suggested that bamboo material has good thermal insulation, which is similar to wood [24]. The thermal conductivity of all samples increased except T-1 of outer bamboo and T-2 of inner bamboo. The thermal conductivity of oven-dried bamboo strips (T-3 and F-3) was higher than that of water-saturated (T-1 and F-1) and air-dried samples (F-2) except the T-2. The results of ANOVA analysis are presented in Additional file 1: Tables S11–S14. It indicated that the moisture content ($P_{\text{outer bamboo}}=0.000<0.05$; $P_{\text{inner bamboo}}=0.035<0.05$) has significant effects on thermal conductivity, but the temperature was not related. The density of bamboo after freeze–thaw treatment is presented in Fig. 6b. The results showed that the density of outer bamboo did not change significantly, while the density of inner bamboo decreased slightly. For outer bamboo, the density of T-1 increased a little, T-3 remained unchanged and the rest of the samples decreased slightly but was in the error bar range of the C0 except for F-1. For inner bamboo, T-1and F-1 decreased more obviously than other bamboo specimens.

The thermal conductivity was influenced by many factors including crystallization area, moisture content and density [25, 26]. The thermal conductivity would increase when the crystalline area increase as the transport of heat in all non-metals (no free electrons) was by the flow of lattice vibrational energy [27, 28]. The CrI of cellulose in water-saturated bamboo strips (T-1 and F-1) with freeze–thaw treatment increased, which was possibly one of the reasons why the thermal conductivity of water-saturated bamboo strips increased. The final moisture content of treated bamboo in our study was from 5 to 8%, and the moisture content of untreated bamboo was about 10%. It was reported that

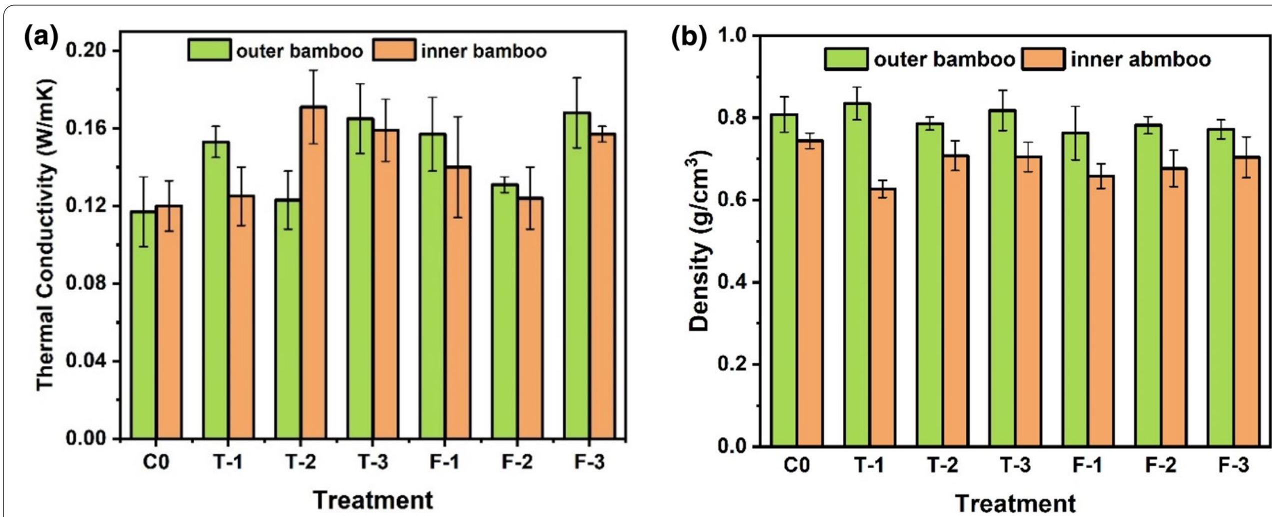

Fig. 6 Thermal conductivity and density of outer and inner bamboo after freeze–thaw treatment with different moisture content. (The error bars refer to the standard deviation which reflects the degree of dispersion of the value relative to the mean.)

the thermal conductivity of bamboo plywood reduced as the moisture content decreased gradually from 100 to 0% [29]. While the thermal conductivity of treated bamboo with lower, final moisture content was higher than that of untreated samples. The material with lower density had lower thermal conductivity [30], but the treated bamboo with the lowest density did not have the lowest thermal conductivity in our study. It indicated that the thermal conductivity was the result of the combined influence of many factors.

Conclusions

In this study, bamboo with different moisture content was subjected to two freeze–thaw treatments to investigate the change in its material properties in terms of the microstructure, chemical composition, mechanical properties, and thermal conductivity. The conclusions are summarized as follows:

1. The freeze–thaw treatment had no obvious effect on the microstructure of bamboo, but the starch content in parenchyma cell of treated bamboo decreased when the temperature decreased. The starches in parenchyma cells of water-saturated bamboo disappeared regardless of the freezing temperature used.
2. There was no significant change in the chemical composition and the cellulose crystalline structure in bamboo with freeze–thaw treatments. The CrI of cellulose in bamboo was affected by the initial water content and freezing temperature. The CrI of bamboo was not only determined by the content of fibers and parenchyma cells, but also closely associated with bamboo structure.
3. Both the initial moisture content and the freezing temperature influenced the bending strength and elastic modulus of bamboo. The bending properties increased significantly with the reduction of the initial moisture content of bamboo. The freezing temperature had a different influence on the bending strength and elastic modulus. The bending strength of bamboo treated at − 40 °C was the highest, while the elastic modulus was highest when treated at − 20 °C.
4. The thermal conductivity of bamboo with freeze–thaw treatment was higher than that of untreated samples. The thermal conductivity was determined by the combined effects of the final moisture content, the crystal structure of cellulose, and the density of treated bamboo.

Abbreviations

CrI: Crystalline index; FT-IR: Fourier-transform infrared spectroscopy; SEM: Scanning electron microscopy; XRD: X-ray diffraction.

Supplementary Information

The online version contains supplementary material available at https://doi.org/10.1186/s10086-021-01998-0.

Additional file 1: Figure S1. Microstructure of freeze-thaw treated (a) parenchyma cells and (b) fibers of outer layer bamboo. **Table S1.** One-way variance analysis of CrI of outer bamboo with different temperature. **Table S2.** One-way variance analysis of CrI of outer bamboo with different moisture content. **Table S3.** One-way variance analysis of CrI of inner bamboo with different temperature. **Table S4.** One-way variance analysis of CrI of inner bamboo with different moisture content. **Table S5.** One-way variance analysis of bending strength of bamboo strips with different temperature. **Table S6.** One-way variance analysis of bending strength of bamboo strips with different moisture content. **Table S7.** Multiple comparisons of temperature. **Table S8.** One-way variance analysis of elastic modulus of bamboo strips with different temperature. **Table S9.** One-way variance analysis of elastic modulus of bamboo strips with different moisture content. **Table S10.** Multiple comparisons of temperature. **Table S11.** One-way variance analysis of thermal conductivity of outer bamboo with different temperature. **Table S12.** One-way variance analysis of thermal conductivity of outer bamboo with different moisture content. **Table S13.** One-way variance analysis of thermal conductivity of inner bamboo with different temperature, **Table S14.** One-way variance analysis of thermal conductivity of inner bamboo with different moisture content.

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Authors' contributions

WJY was the major contributor in data analysis and writing the manuscript. WXK and XX helped with the experiment and analyzed the data partly. LCP helped with analyzing the data partly. FBH financed the research. CH designed the experiment and participated in the writing. All authors read and approved the final manuscript.

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Availability of data and materials

All data generated or analyzed during this study are included in this published article and its additional information files.

Declarations

Competing interests

The authors have declared no conflict of interest.

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