Effect of Vacuum Freeze-drying on Enhancing Liquid Permeability of Moso Bamboo

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Permeability has been proven useful and important in the application and basic research of biomaterials, such as anti-mildew, dyeing, or other impregnated modification, especially in the study of bamboo. However, the traditional methods of improving bamboo's permeability are chemical treatments and destructive physical treatments. This study proposed an innovative way to produce more porous bamboo with effective penetration *via* the vacuum freeze-dried method. The greatest advantage of this method is that the original form of bamboo was preserved according to the three-phase principle of water. From scanning electron microscopy (SEM) and mercury intrusion porosimetry (MIP), the pore characteristics and microstructure of moso bamboo were characterized, the porosity of bamboo increased to 73%, a microporous bamboo was formed, and the liquid penetration of bamboo improved noticeably. Meanwhile, the vacuum freeze-dried method turned bamboo into a mould-preservation biomaterial that effectively removed starch grains. Moreover, testing the mechanical properties showed that the vacuum freeze-dried method did not have a noticeable impact on bamboo's mechanical properties, although it had a remarkable impact during later-stage processing and utilization. More importantly, this work provided a good example with which to expand high-value applications of bamboo resources.

Keywords: Permeability; Moso bamboo; Vacuum freeze-dried method; Mechanical properties

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INTRODUCTION

With the shortage of woody resources, non-wood resources are garnering increased attention. Bamboo, as a sustainable biological material, has been stated to be the best substitute for wood (Atanda 2015). In East Asia, the utilization of bamboo has been intensively investigated from its raw form to processing, in areas such as bamboo crafts and other building materials (Yan *et al.* 2017). However, bamboo is readily infected with mould fungi due to its high content of sugar, starch, and protein, which greatly limits the applications of bamboo in furniture and building materials (Feng *et al.* 2010). To improve the durability and value of bamboo products, chemical preservation and modification have become an indispensable part of bamboo processing (Chen *et al.* 2017). Due to the conspicuous heterogeneity of bamboo (Dixon and Gibson 2014), the distribution of the main tissue system (the vessel, sieve tube, and parenchyma cells) in bamboo is longitudinal, with no horizontal transmission system (Huang *et al.* 2015). Furthermore, it has low permeability compared with wood, and the relative poor volume of bamboo is also far less than that of wood (bamboo: 47%, wood: 67%) (He *et al.* 2017). Hence, it is difficult for pharmaceuticals to be impregnated into bamboo. Therefore, improving permeability has become an important aspect in bamboo processing.

Previous studies have revealed that several methods are effective for improving wood's permeability (Hill 2006; Jiang *et al.* 2006; He 2014; He *et al.* 2016). It is reasonable to consider that such methods can also be used to modify the permeability of bamboo, as bamboo is also a biologically porous material like wood. Microwave treatment is effective for enhancing the liquid permeability of wood as high steam pressure generated within wood cells would result in the destruction of its microstructure (Jiang *et al.* 2006; Torgovnikov and Vinden 2009; He *et al.* 2014, 2016). Solvent treatment with ethanol is used to improve bamboo permeability by dissolving corresponding components from bamboo (Sun *et al.* 2011). Superheated steam to improve permeability is also effective, but the pit membrane and basic organization of the wood are destroyed, which affects the mechanical properties (Bao and Zhou 2017). Moreover, drilling techniques and mechanical incising methods have been reported (Emaminasab *et al.* 2015; Taghiyari 2015). In summary, an improved permeability is accompanied by degradation in mechanical properties or weight loss resulting from extractive dissolving. Freeze-drying is a dehydration process typically used in food processing. It freezes the material and then reduces the surrounding pressure to allow the frozen water in the material to sublimate directly from the solid phase to the gas phase (Fig. 1). At the same time, extractives in the material may also evaporate along with water. Additionally, consecutive pore paths formed as evaporated water or extractives break through the originally closed pores. Thus, liquid flow in the treated material is facilitated, which means that the liquid permeability is improved. As Lu *et al.* (2005) had investigated, after freeze-drying treatment, the wood's liquid permeability increased as low pit-aspiration ratio and cracks of pits membrane of some bordered pits were found. Such studies have verified that freeze drying is an important means to improve the permeability for both wood and bamboo.

During the process of vacuum freeze-drying, bamboo immersed with water would be frozen first, then water inside it would sublimate directly from solid phase under vacuum conditions. As a result, the dry bamboo had a high surface area and more porous structure (Fig. 2). Microstructural changes contribute to the improvement of liquid permeability for the freeze-dried wood or bamboo. A scanning electron microscope (SEM) was widely used to identify the microstructural changes after treatment. Furthermore, changes in the microstructure would result in the variation in porosity and pore diameter distribution. Methods for measuring the porous parameters of wood include mercury intrusion porosimetry (MIP) (Schneider 1982, 1983; Ding *et al.* 2008; Plötze and Niemz 2011), gas adsorption isotherms, solute exclusion (Berthold *et al.* 1997), as well as nuclear magnetic resonance (Furo and Daicic 1999). The MIP method can be used to measure macro- and mesopores in the range of 1.8 nm to 58,000 nm (Plötze and Niemz 2011). As the pore radii of different kinds of cells in bamboo are key in this range (the radius of a sieve tube is approximately 50 μm, the radius of parenchyma cells is approximately 47 μm, the radius of vessels is approximately 15 μm to 200 μm, and the radius of fibre cells is approximately 9.3 μm), the MIP method is suitable for examining the porosity and pore size distribution of bamboo before and after freeze drying.

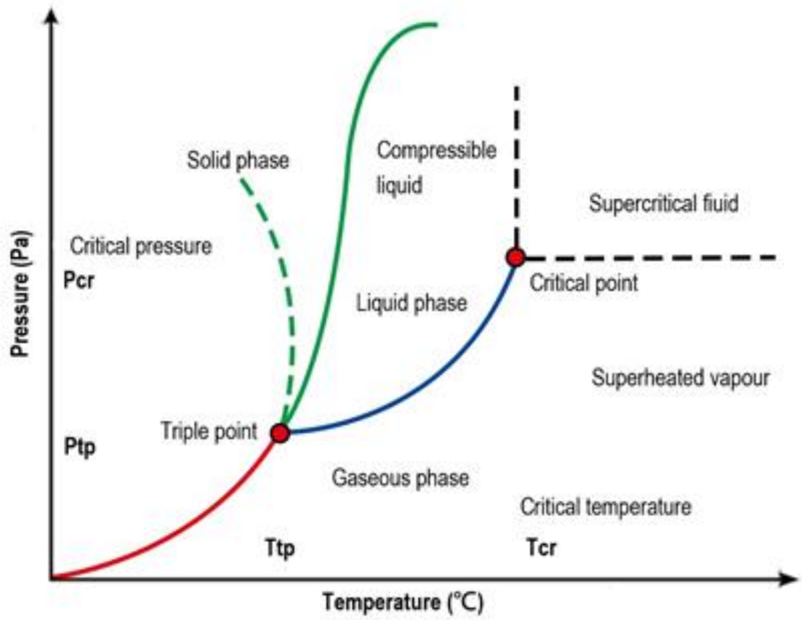

Fig. 1. Three-phase diagram of water (Xu *et al.* 1994; Han 2007)

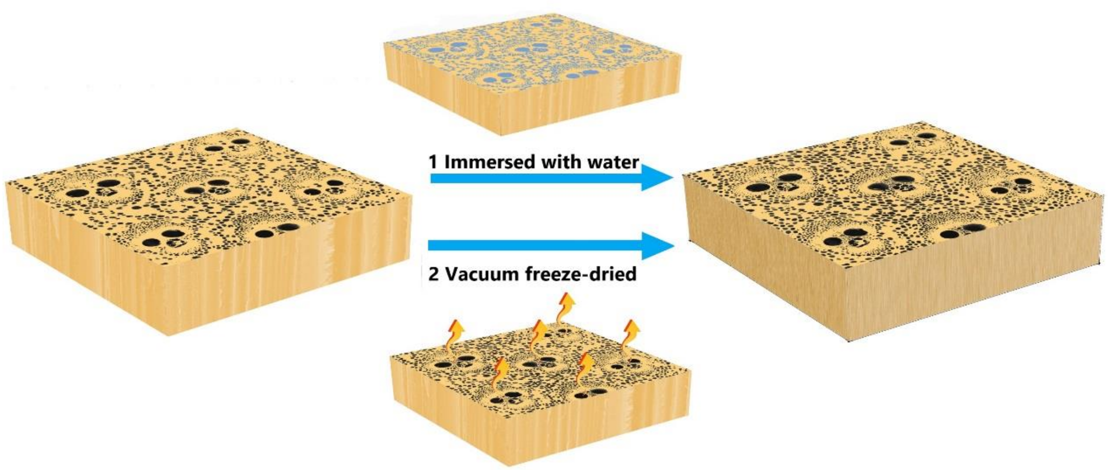

Fig. 2. Preparation of porous bamboo by vacuum freeze-drying

In the present study, the freeze-drying method was used to improve the liquid permeability of bamboo. Microstructure changes in the treated bamboo were verified by SEM examination. A comparison of pore parameters was performed with the MIP method to measure the porosity and pore size distribution of bamboo before and after freeze-drying. The mechanism of permeability improvement through freeze-dried bamboo was analysed. Moreover, the volume expansion rate and water absorption rate tests were conducted to verify the effects of freeze drying on liquid permeability improvement, while mechanical property tests of modulus of elasticity (MOE) and modulus of rupture (MOR) were conducted to evaluate the effects of freeze drying on the mechanical strength of bamboo.

EXPERIMENTAL

Materials

Specimen preparation

Moso bamboo (*Phyllostachys pubescens*), aged 5 years, was obtained from Anji City, Zhejiang Province, China. From the internodes between 1.5 m and 2.0 m from the base of the bamboo culm, three types of bamboo specimens (S1, S2, and S3) were prepared for experimental treatment and testing (Fig. 3). These specimens were: S1, of 160 mm (length) × 20 mm (width) × 5 mm (thickness) for mechanical properties test; S2, of 50 mm (length) × 20 mm (width) × 5 mm (thickness) for the MIP and SEM analyses; and S3, of 40 mm (length) × 20 mm (width) × 8 mm (thickness) for other physical performance tests.

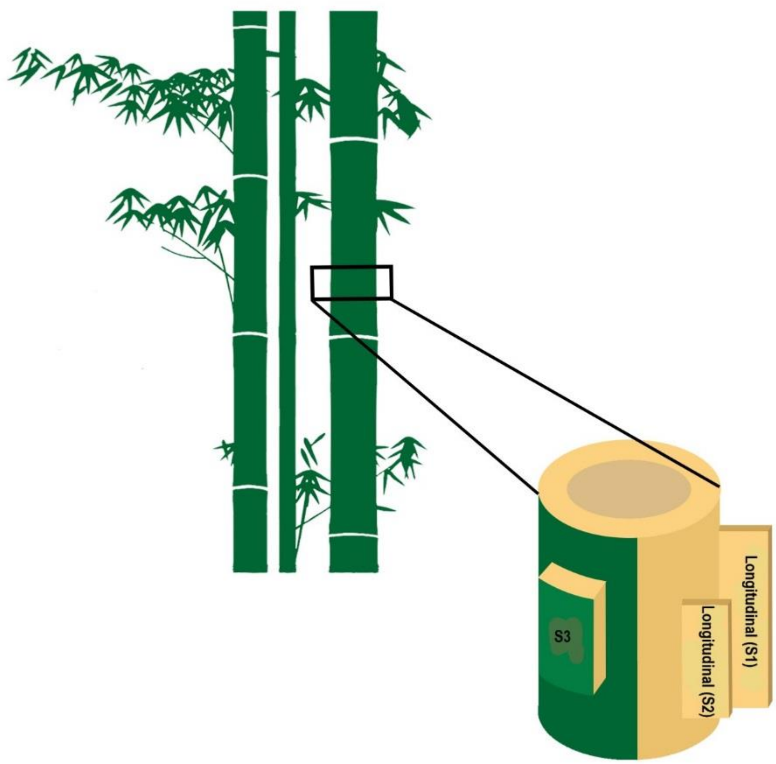

Fig. 3. Preparation of the specimens

Methods

Vacuum freeze-drying and control processing

The experimental processes are displayed in Fig. 4. Each type of specimen was immersed in water until the equilibrium of water absorption was attained; then the material was divided into two groups. One was pre-treated with the vacuum freeze-dried method, while the other was oven-dried as the control group (Fig. 4a). The vacuum freeze-dried method was performed using the vacuum freeze-drying machine (FD-5; JingFu, Shanghai, China) to dry the bamboo specimens. The pre-frozen bamboo temperature was -55 °C, and the vacuum degree was 4 Pa. The oven-dried method was performed in accordance with the national standard (GB/T 15780 (1995). After treatment, all of the specimens were sealed in the vacuum environment to isolate air, especially water.

Evaluation of permeability

To evaluate the changes in permeability, samples (control and treated samples respectively) were soaked in water for 100 h, the volume expansion rate (VER) and water absorption rate (WAR) were calculated based on the equation displayed in Fig. 4b. V1 is the volume (mm^3) before the specimens were immersed in water and V2 is the volume (mm^3) after being treated; W1 is the weight (g) of the treated specimens, and W2 (g) is the weight of the treated specimens after water absorption.

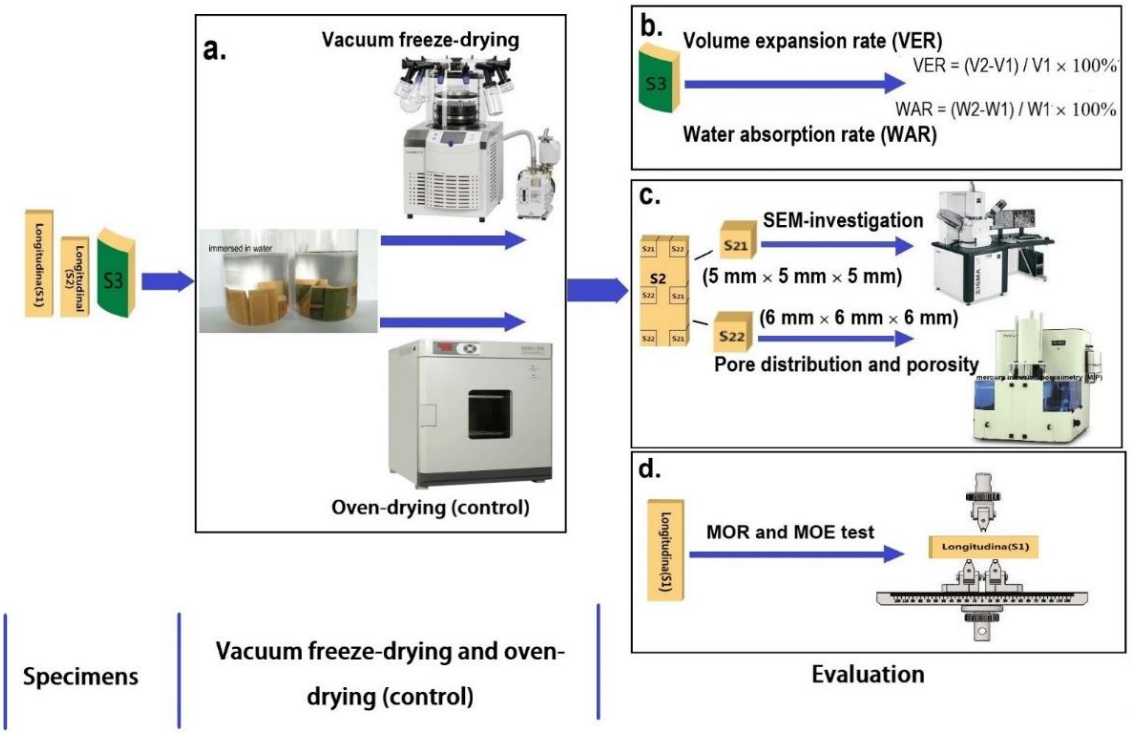

Fig. 4. Overview of the experiment

Observation of bamboo's microstructure

Scanning electron microscopy (Hitachi S3400, Fukushima, Japan) was used to observe the microstructure of bamboo (vessels, parenchyma cells, fibre cells, pits, *etc.*). The samples obtained from S2, as shown in Fig. 4c, were coated with gold prior to observation (Anji, Zhejiang, China).

MIP and main properties

The characterisation process was performed using an automated mercury porosimeter (AutoporeTM IV 9500; Norcross, GA, USA) to force mercury into the pores of the bamboo samples as shown in Fig. 4c. Measurements of total intrusion volume, total pore surface area, pore size, and distribution were all available. The pore distribution was determined with the Washburn equation (Peng *et al.* 2015). Furthermore, some studies (Ilic 1995) found that water, frozen inside the cell, would impart a compressive stress to the cell wall, which may have an influence on mechanical strength; thus, the mechanical properties of wood in timber research has been investigated. Therefore, the MOR and MOE were used to explore the effects of the freeze-dried method on the mechanical properties of bamboo

(Fig. 4d) according to the national standard GB/T 15780 (1995). The data were analysed for each property, to determine the MOE effect and MOR effect separately through an analysis of variance (ANOVA), and further using the Least Significance test using SPSS Statistics 19.0 (SPSS Inc., Chicago, IL, USA).

RESULTS AND DISCUSSION

Verification of Permeability

The freeze-dried method is important for food storage (Valarmathi *et al.* 2017), because it can improve the rehydration of food (water absorption); therefore, the authors used the water absorption rate to characterize bamboo's liquid penetration. The influence of VER and WAR of the bamboo samples, treated with vacuum freeze drying are shown in Figs. 5 and 6. As shown in Fig. 5, the volume expansion rate of the treated bamboo was positive, different from the negative growth of the control bamboo. Meanwhile, the water absorption rate of the control and treated bamboo increased when the soaking water time increased from 1 h to 100 h, but the water absorption rate of the treated bamboo was higher than that of the control bamboo. The rapid increase in the water absorption rate of the treated bamboo may have been due to multiple factors. Water, frozen inside the bamboo, leads to the expansion of bamboo, which was already verified with the volume expansion rate. When frozen water had sublimated, the original location of it formed the pores, thereby it raised the porosity of the bamboo. However, sublimation broke through part of the pits and holes under vacuum conditions, which was verified in the SEM analysis.

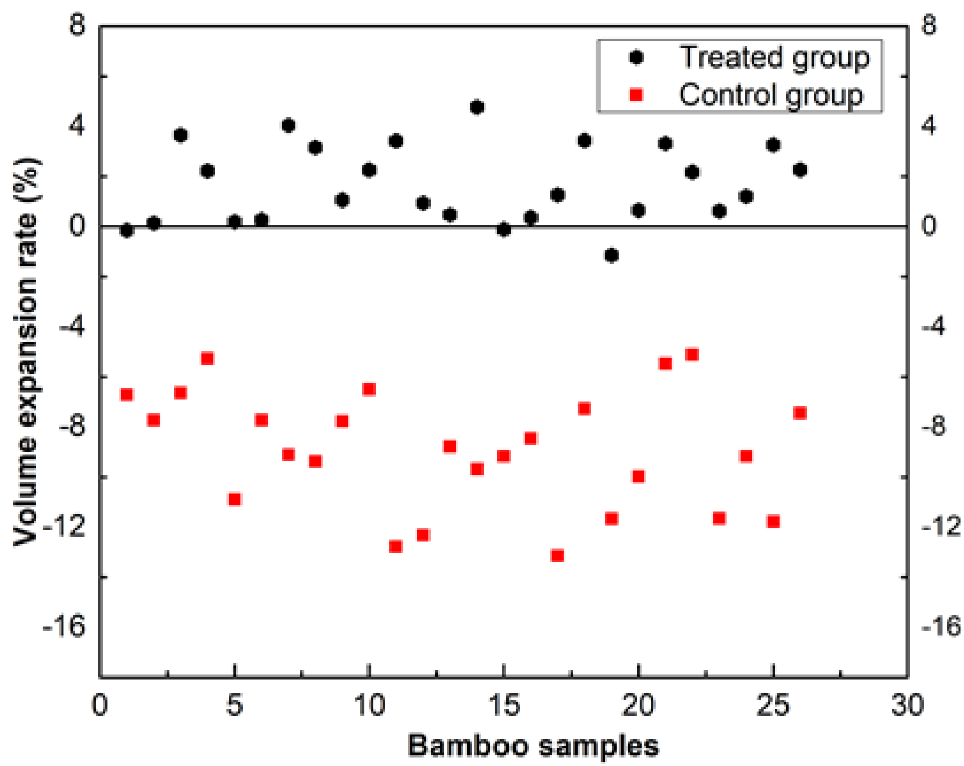

Fig. 5. Volume expansion rate of bamboo samples for C (control) and T (treated) bamboo

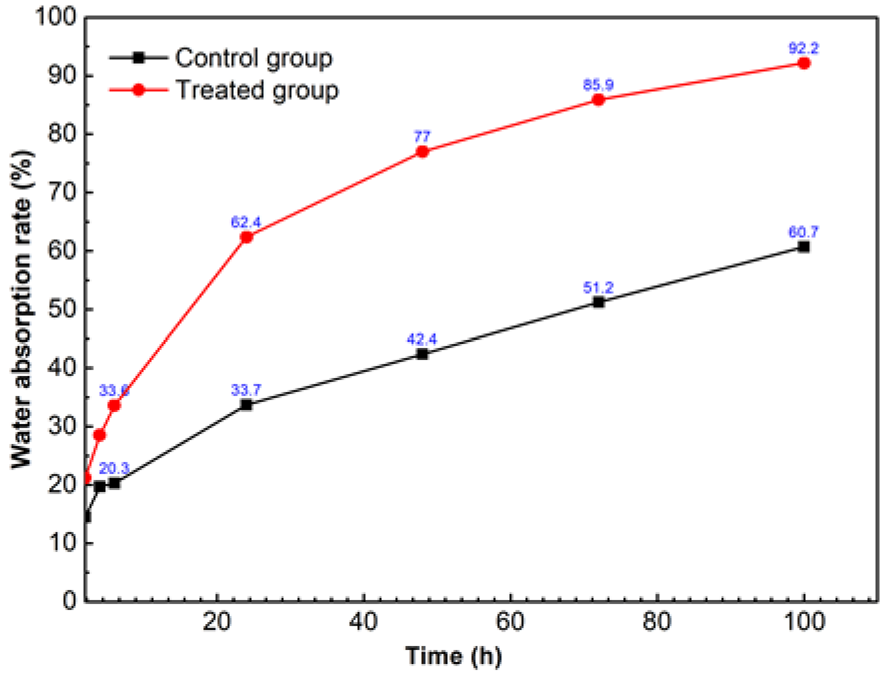

Fig. 6. Water absorption rate of bamboo samples for C (control) and T (treated) bamboo

Analysis of Microstructure

In the field of organic chemistry and materials science, there is a saying: Structure can reflect the properties of materials (Jain *et al.* 1992; Nogata *et al.* 1995). Bamboo belongs to the grass family Gramineae (Zhang *et al.* 2017). Vascular bundles and parenchyma ground tissues are the main structures of bamboo, with fibre cells encircling the vascular bundle vessels as shown in Fig. 7. The authors' previous study revealed that bamboo was less permeable than normal wood because of some differences in the anatomical features, such as organizational structure and pore size (Emaminasab *et al.* 2016), which affect fluid flow paths. Figure 7T1 displays the difference with the control bamboo (Fig. 7C1). Vertical to the vessel's wall, there were splits after the bamboo had been freeze-dried. Furthermore, the comparison of Figs. 7C2 and T2 showed another result: with vacuum freeze-drying, starch grains were removed from the parenchyma cells, which means that the treated bamboo may contain a certain degree of mold preservation. All the results reflected that water, frozen in bamboo, caused a pressure upon the vessel and cell walls, with the sublimation of ice inside the cell lumens, which caused starch grains to be removed and some pores to open.

Figure 8 shows the comparison of the vascular bundle vessels around the treated and control bamboo. The vessels in Figs. 8C1 and T1 were protoxylem vessels, and in Figs. 8C2 and T2 the vessels were primarily phloem vessels (sieve tube). There were more pits on the protoxylem vessel walls (Fig. 8a), in which the main type of pits was alternate pitting. As bamboo was treated, the pits were enlarged, and the vessel walls collapsed (Fig. 8b). Both oven-dried and vacuum freeze-dried methods had affected the structure of primary phloem vessels (Figs. 8C2 and T2), but the effect of vacuum freeze drying on the sieve tube was more obvious. The size of the sieve holes on the sieve tube walls increased, and the pore diameter of the sieve tube expanded to 70 μm (Fig. 8d), larger than that of the sieve tube in Fig. 8c. Moreover, the parenchyma cells around the vessels of the treated bamboo were extruded when water inside the vessels swelled (Fig. 8T1). All these phenomena proved that the structure of bamboo became looser and that porosity increased, which was consistent with the results of Fig. 9T.

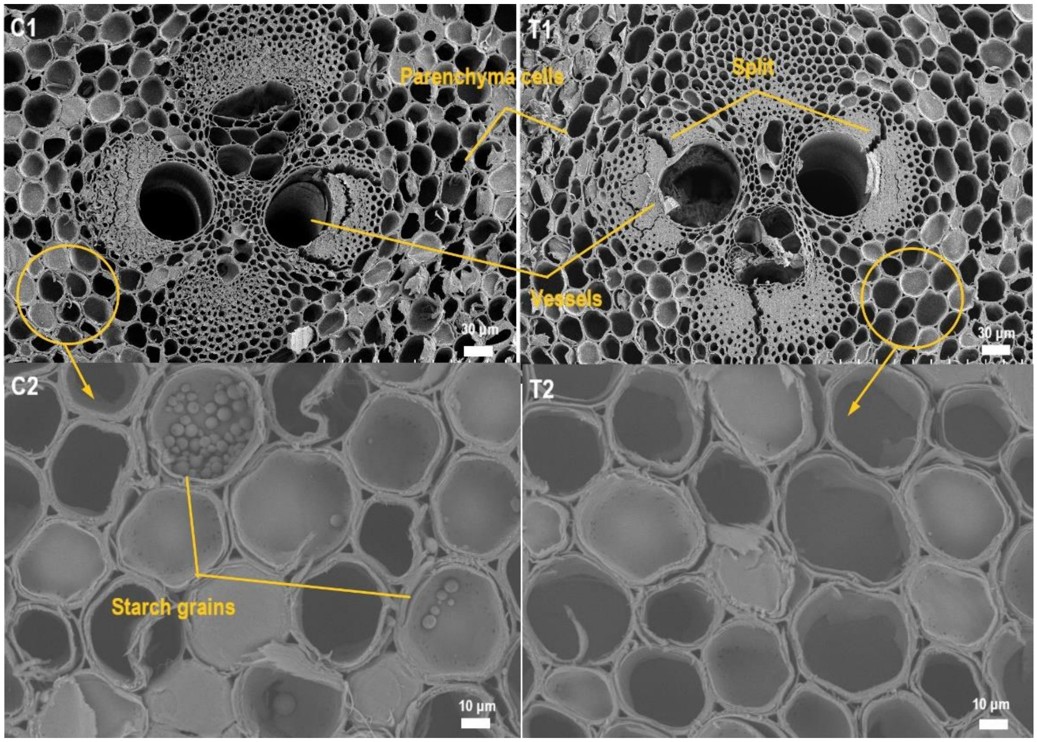

Fig. 7. Overall comparison of the vascular bundle tissue for control (C1/C2) and treated (T1/T2) bamboo

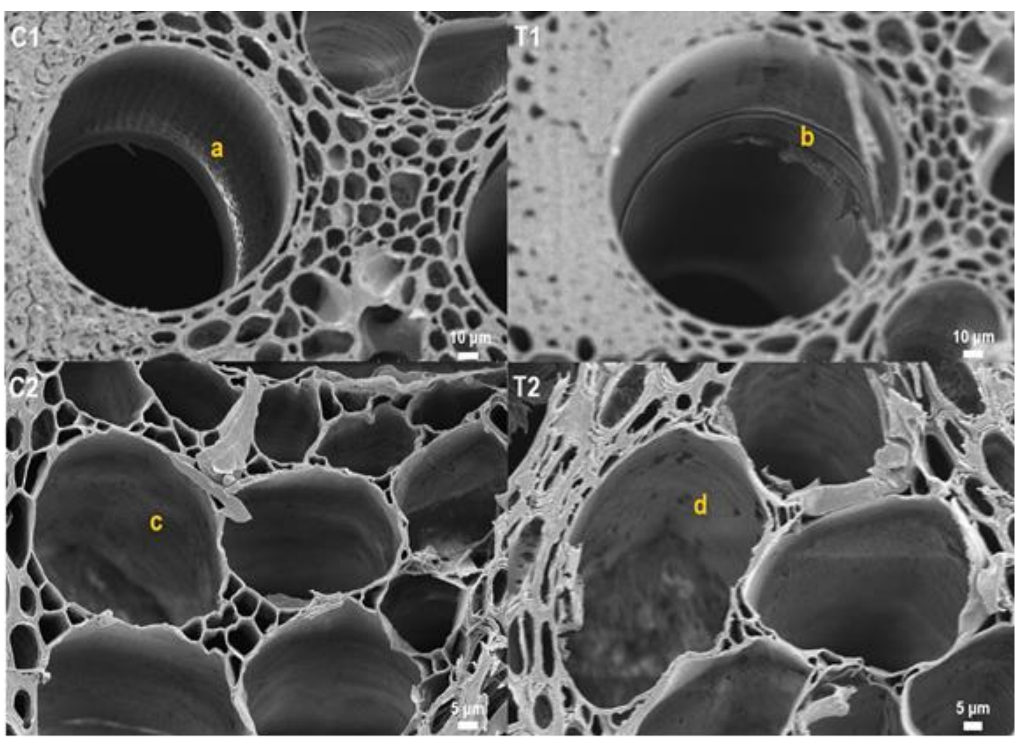

Fig. 8. Single comparison of the vascular bundle vessels for control (C1/C2) and treated (T1/T2) bamboo

Figure 9 displays the comparison of parenchyma cells in the treated and control bamboo. Upon the parenchyma cell walls, there were more single pits as shown in Fig. 9C and 9T, distributed in the transverse wall (d,f) and longitudinal wall (a,b). On the longitudinal wall of the parenchyma cells, most of the pits were closed, as shown in Fig. 9a of the control bamboo. After freeze drying, the pits were opened, and even the pore size had increased to 5 μm, compared with 2 μm in Fig. 9C. The result was also consistent with the data of total pore area in Table 1 below. With the increase in porosity, the pore area increased to 93.9 cm^2/g and microporous bamboo was formed.

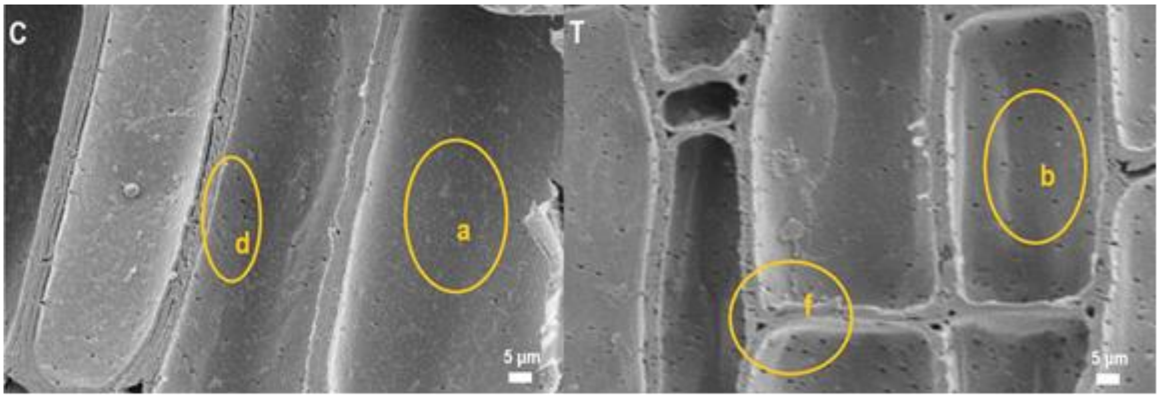

Fig. 9. Single comparison of the parenchyma cells for control (C) and treated (T) bamboo

Porosity, Pore Size, and Distribution of Bamboo

Bamboo is a natural polymer composite, with a multi-scale hierarchical structure. Similarly, pores in bamboo have multi-scale distribution from microns to nanometres (Wang *et al.* 2014). From previous literature, the determination of the pore size distribution is primarily divided into three categories: Plotze *et al.* (2011) divided pores into macropores (58 μm to 0.5 μm), mesopores (500 nm to 80 nm), and microspores (80 nm to 1.8 nm). The International Union of Pure and Applied Chemistry (IUPAC) classifies the pore sizes of porous materials further into three types: micropores (< 2 nm), mesopores (2 nm to 50 nm), and macrospores (> 50 nm) (Fengel 1969). To facilitate the exchange in different areas, this paper divided the pores into microspores (< 2 nm), mesopores (2 nm to 50 nm), and macrospores (> 50 nm).

Table 1. MIP Test Results of Moso Bamboo

Samples	Total Intrusion Volume (mL·g^{-1})	Median Pore Diameter (nm)	Total Pore Area (cm^2·g^{-1})	Bulk Density (g·cm^{-3})	Porosity (%)
Control Group	0.646	44.3	73.6	0.752	48.54
Treated Group	2.091	101.5	93.9	0.351	73.48

Table 1 shows the results derived from the MIP measurement, in which the total intrusion volumes were 2.09 mL/g and 0.65 mL/g for the treated group and control group, respectively. The porosity of bamboo reached 73% (control group: 48%). This conclusion indicated that the total pore volume for the former was larger than that of the latter, demonstrated by principles of physics. The total pore area and median pore diameter also revealed that the total micro-voids for vacuum freeze-dried bamboo clearly increased.

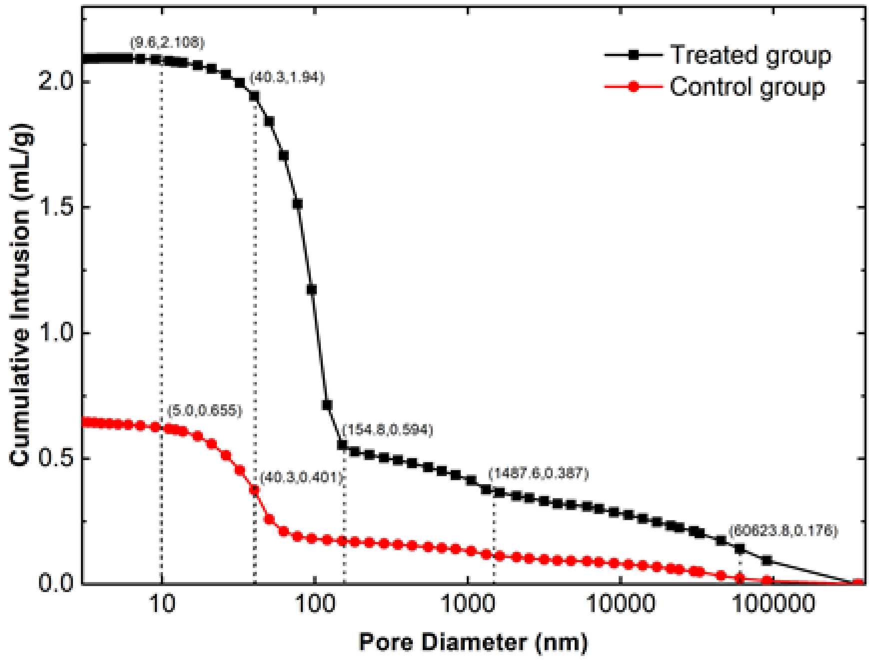

Fig. 10. Cumulative pore volume as a function of the pore diameter of bamboo

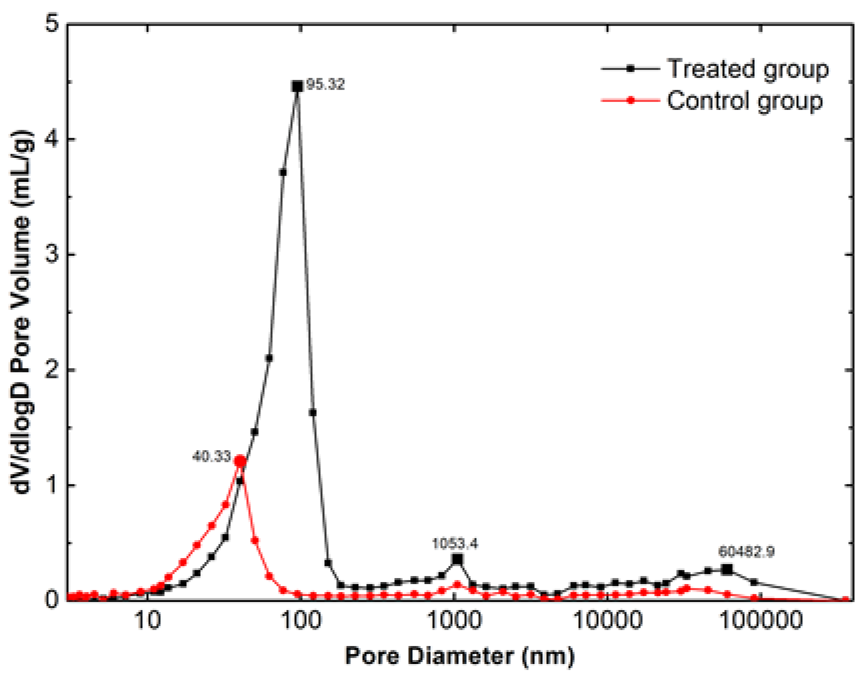

Fig. 11. Log-differential intrusion as a function of the pore diameter of bamboo

Figure 10 displays the relationship between the pore diameter and the mercury intrusion volume. After being treated, the cumulative pore volume of bamboo was approximately three times larger than that of the control group. This result was also consistent with Table 1. Taking the logarithmic integral of the mercury intrusion curve, the change rate in pore volume in the corresponding aperture is shown in Fig. 11. For the treated group, the volume in the range of 40 nm to 154.8 nm and 1.5 µm to 60.6 µm pores were 1.5 mL·g^{-1} and 0.2 mL·g^{-1}, respectively. They were larger than that of the control group. According to the research of Thygesen *et al.* (2010) and Griffin (1977), the macrospores might be vessels, parenchyma cells, fibre cells, pits, or other small voids. The mesopores might be cell wall (dried), and the microspores might be cell wall (wetted), inter-micro fibril spaces (Griffin 1977; Thygesen *et al.* 2010). Moreover, compared with that of the control group, the median pore diameter of the treated group increased to 101.5 nm (Table 1 and Fig. 11). These results indicated that the pore volume and distribution of bamboo raised the total, after being vacuum freeze-dried. Nevertheless, Fengel (1969) found that mesopores especially pores in the cell wall of the wood, would close during the drying process. The actual results were consistent with the control group, in that the pore volume in the 9.6 nm to 40 nm range had decreased (Fig. 11).

The MIP measurement results were based on intrusion-extrusion curves. Figure 12 shows the cumulative pore volume as a function of the pressure of one intrusion-extrusion cycle. The intrusion curve was used to obtain the information on pore size distribution for bamboo, and the extrusion curve could indicate the change in inkwell holes (Yang *et al.* 2013), which caused mercury stagnation when exited from the bamboo pores. According to the curve in Fig. 12, the disparity of cumulative intrusion mercury between the treated and control group was particularly large, which coincided with the results in Fig. 10. It indicated that the pore volume of the vacuum freeze-dried bamboo was three times higher than the control bamboo. Therefore, a more porous bamboo resulted from the treatment.

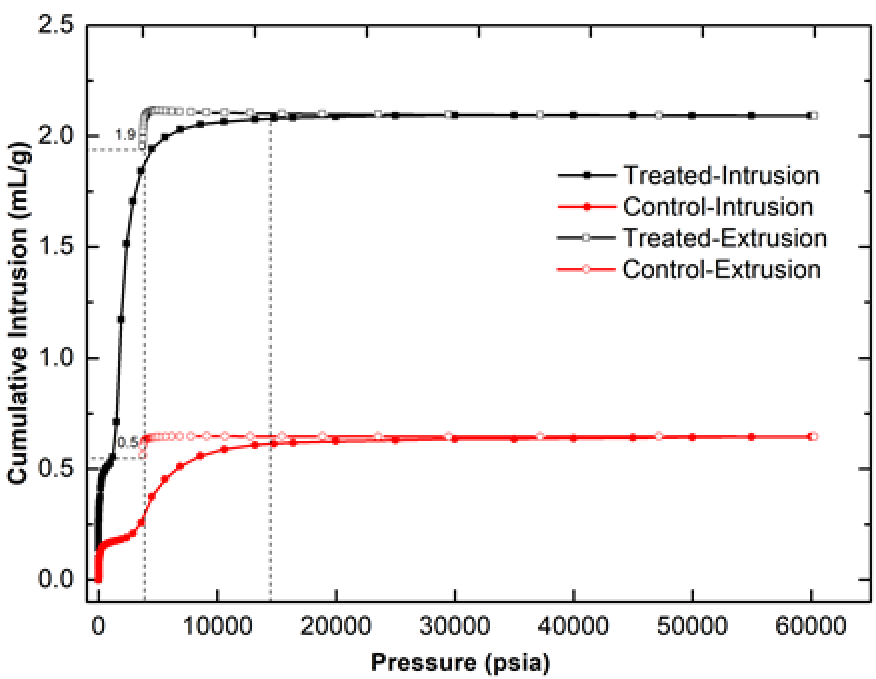

Fig. 12. Cumulative pore volume as a function of the pressure of one intrusion-extrusion cycle

With the extrusion curve, when the initial pressure was reached, all the intrusion-extrusion cycle of the treated and control bamboo did not close, and the path followed by the extrusion curve was not the same as the intrusion path (Fig. 12). At any given pressure, the volume indicated on the extrusion curve was greater than that on the intrusion curve. Moreover, the cumulative extrusion mercury of treated samples was higher than the control samples, which could indicate that some of the inkwell holes in the vacuum freeze-dried bamboo have opened.

Mechanical Properties

Table 2. Average Values and Variances of Mechanical Properties of Bamboo

Mechanical Properties	Species	N	Mean Value	Standard Deviation	Coefficient of Variation
Modulus of Elasticity/MPa	Control Group	12	11313.01	580.950	5.135
	Treated Group	12	11035.16	517.173	4.687
Modulus of Rupture/MPa	Control Group	12	137.434	6.120	4.453
	Treated Group	12	134.456	11.146	8.290

Table 3. Variance Analysis of Mechanical Properties of Bamboo

Mechanical Properties	Deviation Resource	Degree of Freedom	Sum of Deviation Square	Mean sum of Square	F	P-value	F Crit
Modulus of Elasticity	Inter-group Intra-group Total	1 24 25	270204.957 4234796.415 4505001.373	270204.957 352899.701	0.766	0.399	4.747
Modulus of Rupture	Inter-group Intra-group Total	1 24 25	31.052 1131.916 1162.968	31.052 94.326	0.330	0.577	4.747

Note: Significance level: * P < 0.05, ** P < 0.01, *** P < 0.001, NS – Non significant

The material properties of bamboo play a fundamental role in its processing and utilization, especially in the research on the basic mechanical performance of treated bamboo, which has great significance during later-stage processing and utilization. Bamboo's mechanical properties refer to its resistance against external mechanical forces, among which two main properties, *i.e.* MOR and MOE, serve as important indicators of its mechanical quality. Figure 13 presents the effect of treatment and control on MOR and MOE, and all the MOR and MOE results of the control and treated groups are respectively shown in Table 2. The MOE of the former group reached 11,300 MPa on average, which was slightly higher than the latter's 11,000 MPa. As displayed in Table 3, the discrepancy in the average MOR between 137,000 MPa and 134,000 MPa was not obvious. Furthermore, results from one-way ANOVA indicated that neither MOE nor MOR of these two groups reached a conspicuous level. Accordingly, the authors can conclude that the vacuum freeze-drying process did not have a noticeable impact on bamboo's mechanical property and equally maintained a high quality in both the control and treated group.

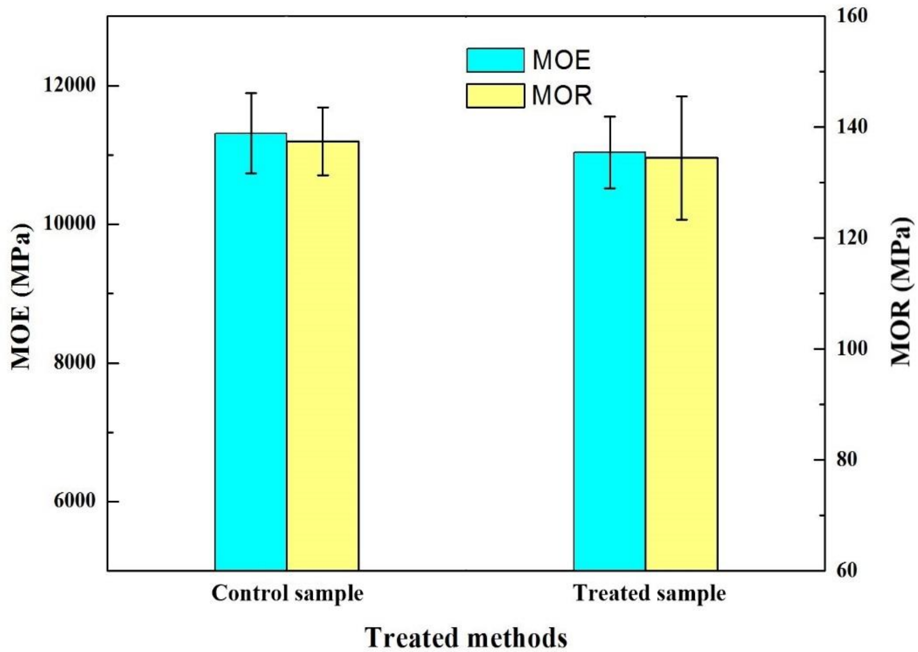

Fig. 13. Comparative values of mechanical properties of bamboo from control and treated state

CONCLUSIONS

This study revealed that the effect of vacuum freeze drying on the permeability of bamboo was obvious without a significant decrease in mechanical properties. The better liquid penetration after freeze drying was attributed to the porous structure of the bamboo formed during the treatment process.

1. Micro-checks were found at part of the pits and holes in the cell walls of the vessel and parenchyma cells under vacuum conditions enlarged. Meanwhile, some pores were opened, thereby raising the porosity of the bamboo. Furthermore, starch grains were removed with the sublimation of ice inside the cell lumens indicating that the treated bamboo will have a good mildew resistance. These microstructural changes were prime factors accounting for the permeability improvement of moso bamboo.
2. The MIP test provided a positive correlation between microstructural changes and liquid permeability. Corresponding to the destruction of pits, the pore diameter enlarged in the pit opening diameter range (control sample: 40.3 nm; treated sample: 95.3 nm), and the porosity of bamboo increased to 73%. Liquid flow was much easier through bamboo, which was reflected in the increase in cumulative pore volume in the pore diameter range of bamboo. The cumulative pore area changes indicated that microporous bamboo were generated after treatment, which also contributed to the liquid permeability improvement.
3. There were splits in the vessel's wall after the bamboo was frozen, and the weak part of the bamboo cell tissue was slightly destroyed but it did not have a noticeable impact on bamboo's mechanical property. The treated microporous bamboo maintained a high quality mechanical property.

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