Feasibility of Protecting Bamboo Surface by High Temperature Wax Penetration

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The permeability, hydrophobicity, glass transition temperature, crystallinity and chemical composition of bamboo subjected to wax-scalding heat treatment were detected using depth-of-field microscopy, contact angle measurement, thermogravimetric analysis, X-ray diffractometry, and infrared spectrometry. Effects of wax scalding technology and formula on bamboo properties were studied from three aspects: temperature, time, and wax species. The improved wax scalding technology was found to have a good penetration effect and to achieve hydrophobic and mildew-proof effects. At the heat treatment temperature of 180 °C, a good permeation effect was achieved and the hydrophobicity of bamboo was increased. The penetration depth of wax in bamboo within 2 min decreased with the shortening of wax scalding time. However, the infiltration effect was not obvious with the prolonging of time, and increasing preheating treatment will obviously improve the permeability. The effect was the best after preheating and wax scalding at 180 °C for 2 min. Mixed experiments of various waxes showed that the wax had the best penetration effect into bamboo when the ratio of paraffin wax to white wax was 5:4:2, the wax scalding temperature was 180 °C (above which the wax begins to sublimate), and the wax scalding time was 2 min.

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INTRODUCTION

Bamboo is one of the richest biomass resources. The bamboo forest area has been increasing continuously since 1990, and the global total area was about 35 million hm^2 in 2020 (INBAR 2019). The global export trade volume of bamboo and rattan commodities was US$ 1.769 billion in 2010, and it increased by 89.6% to US$ 3.354 billion in 2020, with an annual growth rate of 8.15% (Sharma and Van Der Vegte 2019). Bamboo is widely distributed in Africa, Asia-Pacific, North America, and Europe. Among them, China has the richest bamboo resources in the world, with 500 varieties and an annual output of about 4.1 billion. Due to its strong environmental adaptability, short growth cycle, high yield, light weight, wide application, good mechanical properties, reproducibility, and adaptability, bamboo is widely used in architecture, furniture, interior decoration, household goods, and so on (Humar *et al.* 2017; Tang *et al.* 2019; Chen *et al.* 2020, 2021). However, bamboo has a radial gradient porous structure and a large number of hydroxyl groups, which are significantly affected by the change of environmental humidity. Its cell wall is hydrophilic and very sensitive to moisture (Li *et al.* 2018). Therefore, bamboo products are prone to warping, deformation, and cracking in humid environments or when exposed to sunlight (Li *et al.* 2015). Compared with wood, bamboo contains more nutrients (*e.g.*, sugar, protein, and starch), and that make it vulnerable to fungi, bacteria, and pests (Humar 2013; Gérardin *et al.* 2016; Lee *et al.* 2018). Such vulnerability greatly shortens the service life of bamboo products and hinders the development of the bamboo industry. Hence, it is of great significance to protect the bamboo surface so as to achieve efficient utilization of bamboo resources and improve the dimensional stability, mildew resistance and service life of bamboo products.

Bamboo mildew prevention has been studied through sterilization, removal of nutrients from extracts, and surface protection, such as heat treatment, surface coating, nanotechnology, and bactericide impregnation (Tanasa *et al.* 2021). Impregnation with bactericides is currently the most common and effective treatment method. Inorganic fungicides (such as metal salts, boron compounds, silicates) cannot be fixed and become lost in bamboo, resulting in low stability of outdoor mold. Organic fungicides, including propiconazole, tebuconazole, chlorothalonil, isothiazolone, and carbamate, are known for their environmental friendliness, high efficacy, and resistance to runoff (Zhang *et al.* 2021). Fungicide impregnation has been widely used in the mildew-proof modification of bamboo. However, bamboo is rich in nutrients such as starch and reducing sugar, and the mildew problem of bamboo cannot be fundamentally solved only by soaking with bactericide. High-temperature heat treatment can degrade hemicellulose in bamboo, thus reducing the contents of free hydroxyl and some nutrients in bamboo and further improving the dimensional stability and corrosion resistance of bamboo. Unluckily, heat treatment damages the mechanical properties of bamboo (Wang *et al.* 2016). Despite the rapid development of nanotechnology in bamboo mildew prevention and corrosion protection, the aging, mechanical strength, loss, cost and biological safety of nano-materials are still worthy of attention (Evans *et al.* 2005).

Wax-sealing is a decorative method used for surface treatment of ancient Chinese furniture, playing a unique role in decoration. The wax-sealing technique for ancient furniture can be divided into steps such as wax preparation, wax melting, wax application, and wax sealing. Each step is carried out in order, providing stronger protection for the surface of wooden materials and enhancing their ornamental and artistic qualities (Yue 2010). Waxes are transparent and have no special color. Because of their minimal pollutant content, waxes have been widely used as additives for waterproofing and wood preservatives in the past few years (Schultz *et al.* 2007; Moghaddam *et al.* 2016). Impregnating wood with wax emulsion will reduce the water absorption of wood and the risks of fungi and pests and enhance the dimensional stability of wood (Scholz *et al.* 2010; Brischke and Melcher 2015; Bi *et al.* 2021). Wax coating on the surface, which plays the role of semi-permeable membranes, can prevent the absorption of liquid water, delay the diffusion of water vapor, and maintain the moisture buffering capacity of wood (Chen *et al.* 2020). The waxes commonly used in traditional wax ironing are natural, and they include beeswax and Sichuan wax. After the bamboo wood is scalded with wax, a layer of wax film will be formed on its surface. Wax molecules will seal the wood pores, and separate moisture and air from the bamboo wood, thus improving the bamboo wood stability and protecting from moisture, corrosion and insects (Schulte *et al.* 2020). Due to the influence of the external environment, bamboo will expand after wetting, shrink in hot conditions, and shrink upon coldness, which will lead to wax precipitation.

The research direction of the wax scalding technology is focused on its influence on bamboo properties, wax ratio and wax modification, but the application into bamboo and the influence of wax scalding heat treatment on bamboo properties have not been discussed in depth.

The macrostructure of bamboo is illustrated in Fig. 1(a). Bamboo has a typical unidirectional fiber structure, and its microstructure and super-depth are shown in Fig. 1(b) (c). The process of hot waxing is essentially a fluid seepage process in bamboo cells. The flow of liquid wax is mainly concentrated inside and between vascular bundles, and liquid wax is distributed in the internal tissue structure of bamboo in the form of particles, as shown in Fig. 1(d). Scientists (Mahottamananda *et al.* 2020; Schulte and Hacke 2020) simulated the fluid flow state and water sensitivity near the pits in the interior of wood and bamboo, revealing the influence of wood and bamboo structure on the internal water transport resistance, as shown in Fig. 1(e).

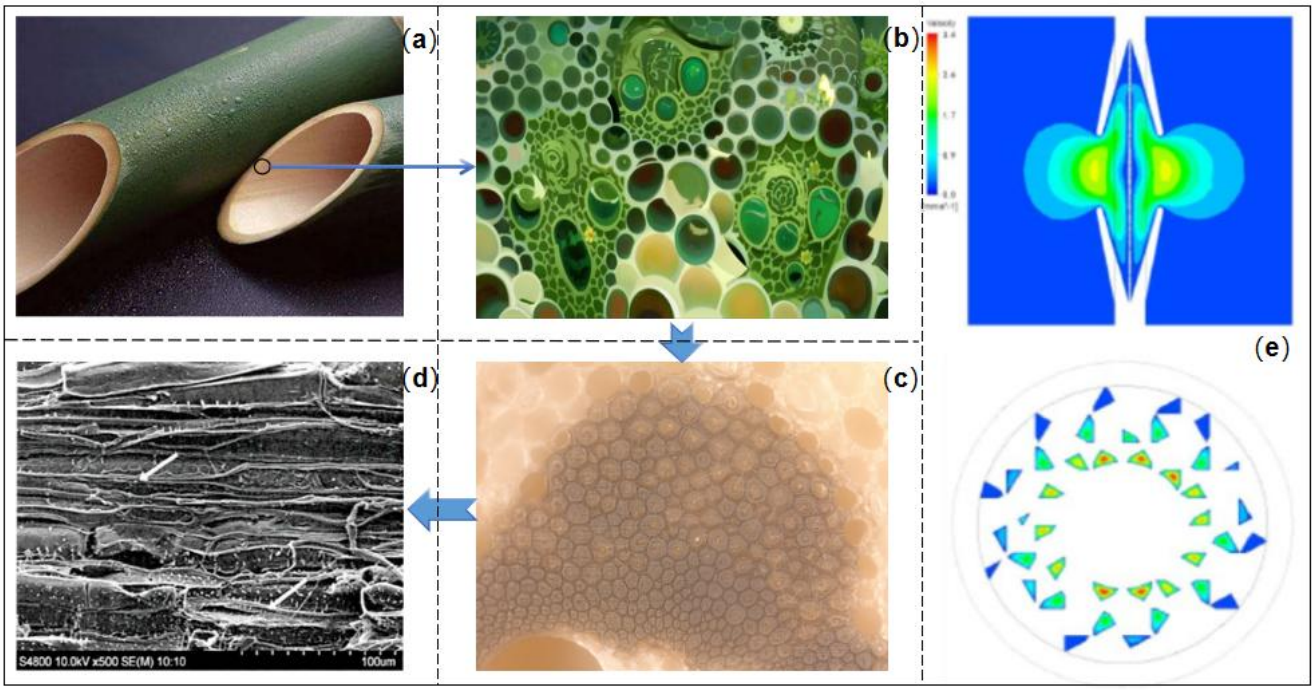

Fig.1. Microscopic seepage structure and simulation of bamboo. (a) Macroscopic structure of bamboo, (b) Microscopic structure of bamboo, (c) Transverse microscopic structure of bamboo, (d) Longitudinal microstructure of bamboo fiber, (e) Simulation of seepage flow near the vascular

To implement the wax-ironing technique on bamboo furniture, this study selected relatively inexpensive paraffin wax, white wax, coarse wax, and soft wax for the wax-ironing treatment of bamboo materials. Through the application in bamboo, a study was made of the wax scalding technology, the wax scalding formula, and the influence of the treatment on bamboo properties. The wax scalding technological parameters, technological process, and wax scalding formula with better physical and chemical properties were obtained by comparing and analyzing the permeability, hydrophobicity, dimensional stability, glass transition temperature, crystallinity, and infrared spectrum of bamboo treated with different technological parameters and wax scalding formulas. The study results will provide a reference for further exploration of the application of wax scalding and heat treatment of bamboo products.

EXPERIMENTAL

Materials

Five-year-old *Phyllostachys pubescens* from Zhejiang Academy of Forestry Science was selected and processed into bamboo slice size 70 × 20 × 5 mm^3.

Paraffin: A mixture of hydrocarbons extracted from certain distillates of petroleum, shale oil, or other asphalt mineral oils. The main component is solid paraffins, which are odorless, tasteless, and white. Its melting point ranges from 50 to 70 °C. It was purchased from Boyuan Chemical Co., Ltd. in Zhengzhou, Henan, China.

White wax: A wax secreted by the larvae of the white wax scale during their growth process. It is solid at room temperature, white in color, and lustrous. Its main component is hexacosanol hexacosanoate, with a melting point of 80 to 85 °C. It was purchased from Xifu Gui Co., Ltd. in Jieyang, Guangdong. China.

Coarse wax: It belongs to polishing and grinding wax, white emulsion, no luster, with a melting point of 60 to 70 °C. It was purchased from Kaiyuan Chemical Co., Ltd. in Zhengzhou, Henan, China.

Soft wax: It is refined using refined microcrystalline wax supplemented with high-quality additives. It exhibits excellent softness, adhesiveness, and water resistance. As a high-performance material widely used in multiple fields, soft wax typically has a melting point ranging from 50 to 70 °C. This characteristic allows soft wax to exhibit different states at different temperatures, opening up possibilities for diverse applications. With an ash content of less than 0.3%, the hardness of soft wax falls between 10 and 30. The hardness can be adjusted by altering the composition and adding additives to meet the needs of different application scenarios. It was sourced from Longwang Chemical Co., Ltd. in Cangzhou, Hebei, China.

Colorant: The colorant is an inorganic pigment, which boasts a rich and vibrant color. It is an oxide of a non-ferrous metal or some insoluble metal salts, insoluble in water but soluble in organic solvents. The colorant offers a beautiful and diverse range of colors, and in this design, a blue colorant was selected. It was purchased from Ruan Fu Co., Ltd. in Hangzhou, Zhejiang Province. The model number is 121207-31-6.

Qualitative Analysis of Four Types of Wax

The GC-MS total ion chromatograms of paraffin, coarse wax, white wax, and soft wax are shown in Fig. 2. The trends for the total ion current intensity of each wax species with temperature or time were basically consistent, indicating that the thermal decomposition process of each wax species was similar (Sotoudehnia *et al*. 2021). According to the trend of ion flow intensity with temperature or time, it can be seen that in the early stage of thermal decomposition, the total ion flow remained basically unchanged for a period of time (paraffin and coarse wax within 5.49 to 35.57 min, white wax within 5.47 to 39.34 min, and soft wax within 5.49 to 33.84 min), indicating that the gas product content remained basically unchanged during this stage. Subsequently, the total ion current intensity of paraffin, coarse wax, and soft wax sharply increased to form a prominent peak group, indicating that the content of gas products increased sharply at this stage, and a violent chemical decomposition occurred, while white wax only had a prominent peak at this stage, indicating that the thermal decomposition gas products of white wax were very few. In addition, the greater the ion current intensity, the more gas products, and the more intense the corresponding thermal chemical decomposition reaction (Song *et al.* 1993). It was confirmed that the heat treatment time and temperature of hot wax have a certain impact on the penetration effect of wax in bamboo.

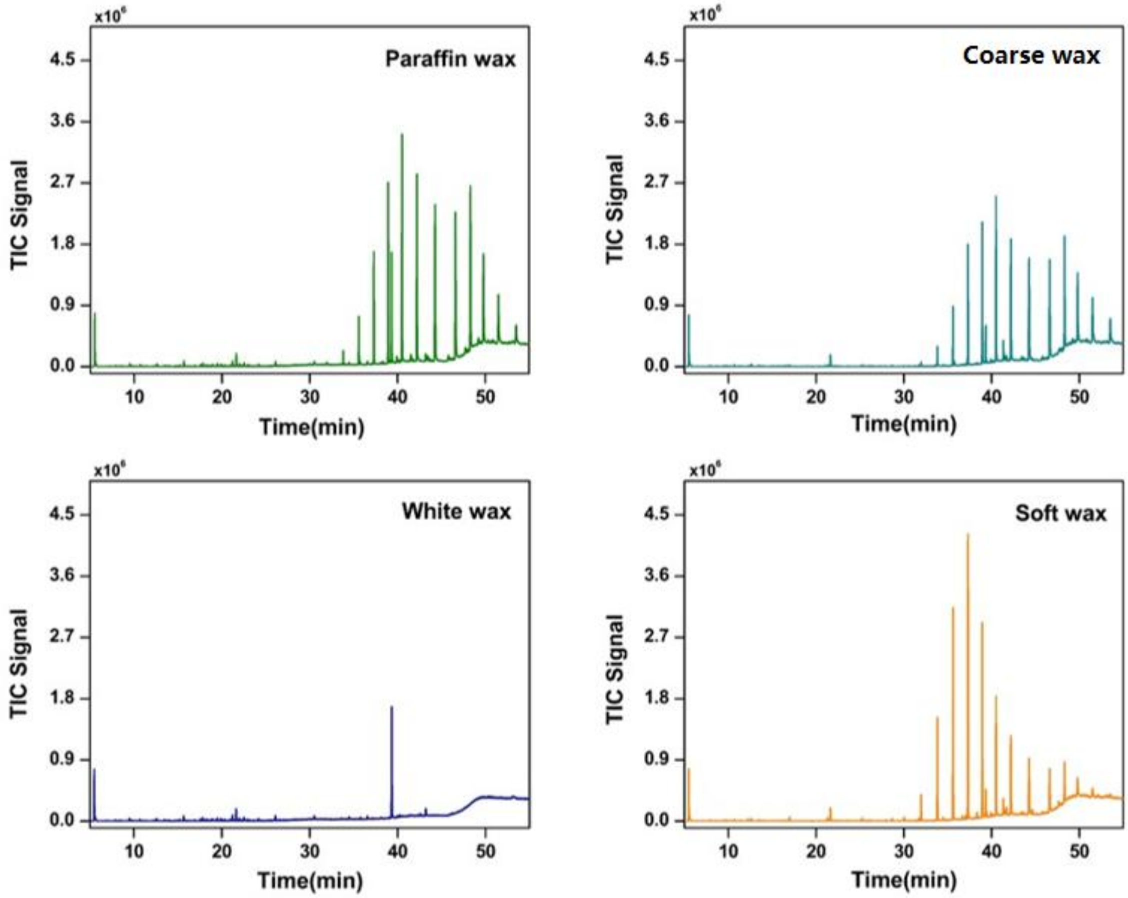

Fig. 2. GC-MS total ions chromatograms of paraffin wax, coarse wax, white wax, soft wax

Tables 1 to 4, respectively, list the gas products after pyrolysis of various waxes identified by GC-MS. The paraffin wax, coarse wax, white wax, and soft wax contained 33, 20, 15, and 31 substances, respectively.

Table 1. Gas Products in Paraffin Detected by GC-MS

PK	Library/ID	CAS	Area Pct	RT	Formula
1	Octane	000111-65-9	3.07	3.65	C_8H_{18}
2	Nonane	000111-84-2	2.27	5.49	C_9H_{20}
3	Eicosane	000112-95-8	2.15	49.25	$C_{20}H_{42}$
4	Octadecane	000593-45-3	5.21	38.75	$C_{18}H_{38}$
5	Tetracosane	000646-31-1	8.48	60.25	$C_{24}H_{50}$
6	Phenol, 2,2'-methylenebis[6-(1,1-dimethylethyl)-4-methyl-	000119-47-1	4.97	39.33	$C_{25}H_{36}O_2$
7	Pentacosane	000629-99-2	11.20	40.52	$C_{25}H_{52}$
8	Hexacosane	000630-01-3	11.48	42.21	$C_{26}H_{54}$
9	Heptacosane	000593-49-7	11.87	49.25	$C_{27}H_{56}$
10	Triacontane	000638-68-6	5.75	49.80	$C_{30}H_{62}$

Table 2. Gas Products in White Wax Detected by GC-MS

PK	Library/ID	CAS	Area Pct	RT	Formula
1	Octane	000111-65-9	23.46	3.65	C_8H_{18}
2	Nonane	000111-84-2	17.25	5.49	C_9H_{20}
3	Phenol, 2,5-bis(1,1-dimethylethyl)-	005875-45-6	4.76	21.63	$C_{14}H_{22}O$
4	Phenol, 2,2'-methylenebis[6-(1,1-dimethylethyl)-4-methyl-	000119-47-1	37.72	39.33	$C_{23}H_32O_2$
5	4-Cumylphenol, trimethylacetate	1000462-39-1	3.93	43.22	

Table 3. Gas Products in Coarse Wax Detected by GC-MS

PK	Library/ID	CAS	Area Pct	RT	Formula
1	Octane	000111-65-9	3.68	3.65	C_8H_{18}
2	Nonane	000111-84-2	2.85	5.49	C_9H_{20}
3	Eicosane	000112-95-8	21.43	56.5	$C_{20}H_{42}$
4	Octadecane	000593-45-3	10.48	40.5	$C_{18}H_{38}$
5	Tetracosane	000646-31-1	17.34	54.6	$C_{24}H_{50}$
6	Pentacosane	000629-99-2	10.19	40.51	$C_{25}H_{52}$
7	Hexacosane	000630-01-3	9.44	42.21	$C_{26}H_{54}$
8	Heptacosane	000593-49-7	10.04	44.28	$C_{27}H_{56}$
9	Octadecane, 1-iodo-	000629-93-6	2.95	53.53	$C_{18}H_{37}I$

Table 4. Gas Products in Soft Waxed Detected by GC-MS

PK	Library/ID	CAS	Area Pct	RT	Formula
1	Octane	000111-65-9	4.17	3.65	C_8H_{18}
2	Nonane	000111-84-2	3.09	5.49	C_9H_{20}
3	Eicosane	000112-95-8	9.60	41.02	$C_{20}H_{42}$
4	Heneicosane	000629-94-7	6.57	36.075	$C_{21}H_{44}$
5	Octadecane	000593-45-3	31.47	36.455	$C_{18}H_{38}$
6	Tetracosane	000646-31-1	12.53	38.94	$C_{24}H_{50}$
7	Pentacosane	000629-99-2	8.13	40.51	$C_{25}H_{52}$
8	Hexacosane	000630-01-3	6.58	42.21	$C_{26}H_{54}$
9	Heptacosane	000593-49-7	5.97	44.27	$C_{27}H_{56}$

From the test results, all four of the wax types were found to be mainly alkanes, in addition to a small amount of alcohols, phenols, and lipids. Among them, the paraffin contained heptacosane (12.2%), hexacosane (11.5%), pentacosane (11.2%), tetracosane (18.9%), hexadecane (12.2%), and 1-IODO- (9.31%). The course wax contained heptacosane (10.0%), hexacosane (9.4%), pentacosane (10.2%), tetracosane (17.3%), and eicosane (21.4%). The soft wax was found to have relatively high contents of phenol, 2,2'-methylenebis [6-(1,1-dimethyl ethyl)-4-methyl- (37.7%), octane (23.5%), nonane (17.2%), octadecane (31.5%), tetracosane (12.5%), and pentacosane (8.1%). The content of phenol, 2,2'-methyl bis [6-(1,1-dimethyl ethyl)-4-methyl-in white wax was as high as 37.7%, while its contents in paraffin wax, coarse wax, and soft wax were only 4.97%, 1.93%, and 1.68%. It is speculated that it is this phenolic substance that leads to the worst penetration effect of ash in bamboo. However, paraffin contains many kinds of alkane compounds, so its chemical stability is better, which may be due to the fact that alkane substances are insoluble in water and have high molecular weight and are not volatile. In addition, only some aniline compounds were detected in paraffin wax, and soft wax contained a small amount of acidic substances. The ash contents of the four types of wax are roughly the same.

Waxes and Dyeing Treatment

Paraffin wax, white wax, soft wax, and coarse wax (each 5 g) were weighed on an electronic balance with an accuracy of 0.01 g. The waxes were put into beakers separately and heated in an electric furnace at 80 to 100 °C. After all waxes were completely transformed into a liquid state, 0.5 g of oil-soluble dye was added into each beaker. The color of the dye was blue. During heating, stirring with a glass rod was continued until the system was evenly mixed. The above process is shown in Fig. 3.

Fig. 3. Wax making process

Waxing Process

Surface pre-treatment

Three steps were taken: coarse grinding, fine grinding, and pneumatic spray gun to remove sawdust. The concave and convex parts on the surface of bamboo pieces were repaired with a utility knife, and 200- and 800-mesh sandpapers were used to polish them along the grain direction until the surface of bamboo pieces was smooth. The debris on the bamboo surface was removed using a pneumatic spray gun and wiped with cotton cloth. After polishing, the bamboo slices had smooth surface and a clear and beautiful texture.

Wax Ironing

The wax ironing process was divided into six groups. The wax species was paraffin (5 g), and bamboo was scalded with a constant-temperature hot air gun and a high-temperature resistant brush. The hot air gun was used to adjust the temperatures of preheating and wax scalding, and the resistant brush was used to dip dyed paraffin liquid and brush it along the grain direction of bamboo. Finally, the wax layer on the surface of bamboo slices was removed with a spatula, and the residual wax and floating wax on the surface of bamboo slices were wiped with cotton yarn. Consequently, the bamboo slices after wax scalding and heat treatment showed natural luster and were even and harmonious. Each experimental parameter was repeated at least three times, and the results were averaged. The process is illustrated in Fig. 4.

After heat treatment, the main components of the cell wall undergo chemical reactions, with intense thermal and oxidative degradation reactions. Heat treatment reduces the relative content of cellulose and hemicellulose, decreases material density, and simultaneously, high temperatures harden the surface of bamboo, making it brittle, thus leading to a decrease in the overall strength of bamboo. Different treatment media can all reduce the strength of bamboo to some extent (Yang *et al.* 2016.), but the elastic modulus may increase due to the formation of a shell-like layer on the bamboo surface at high temperatures (Shao *et al.* 2003). 200 °C is considered the critical temperature for changes in the elastic modulus of bamboo. Below 200 °C, the elastic modulus is less affected, while above this temperature, it decreases rapidly (Li *et al.* 2015.). Based on the above analysis, the preheating temperature and wax-scalding temperature were selected as shown in Table 5. Each experimental condition should be repeated at least three times.

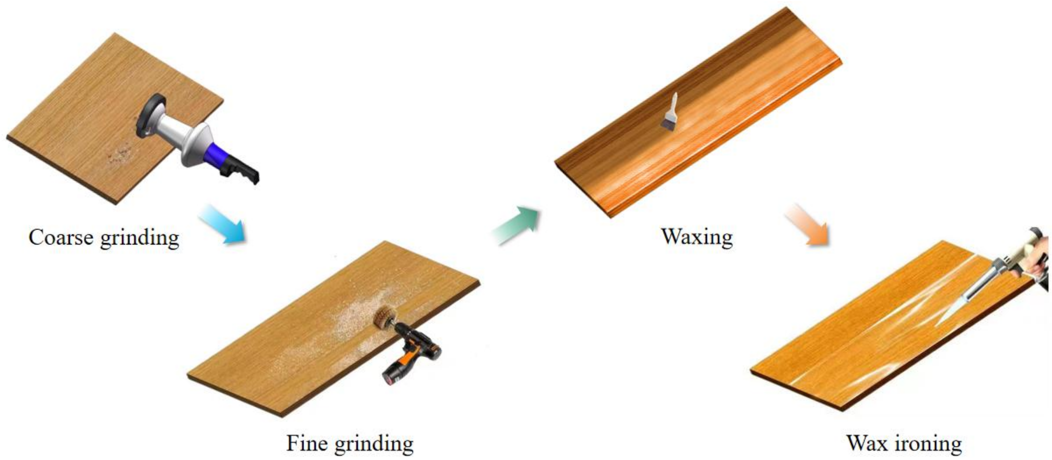

Fig. 4. The process of waxing bamboo

Table 5. Research Scheme on the Influence of Wax Scalding Process on the Penetration Depth of Wax in Bamboo

Scheme	Preheat Temperature (°C)	Preheat Time (min)	Wax Ironing Temperature (°C)	Wax Ironing Time (min)
A	Contrast group	-	180	2.0
B	180	2.5	180	2.0
C	180	2.5	180	0.5
D	180	2.5	180	4.0
E	120	2.5	120	2.0
F	250	2.5	250	2.0

As shown in Table 5. This study aims to obtain the optimal process and materials for the wax-ironing process by adjusting various process parameters. It provides a theoretical and scientific basis for the application of wax-ironing technology for bamboo materials.

Characterization

Permeability and cold-heat cycle precipitation test

A VHX-7000 ultra-depth-of-field microscope (Keens Company) was used to test and observe the penetration effect of wax in bamboo. The middle of each wax-treated specimen was cut horizontally, and then the fresh cross-section was cut with a knife and observed under the microscope. The sample was placed at -18 °C for after 2 h. After the surface was leveled, the precipitation of wax on the shovel was observed.

Hydrophobic Analysis

Contact angles of bamboo surface after wax scalding and heat treatment were measured using an OCA20 video optical contact angle instrument (Germany), and its hydrophobicity was analyzed. On the flat side of each wax-scalding sample, five different points were selected to be measured with distilled water, and the droplet volume was measured to be 5 µL. After the distilled water dropped on the sample surface, it was left for 300 s. The contact angles at 0, 10, 20, and 300 s were recorded.

Temperature Analysis of Glass Transition

The glass transition temperature of bamboo was measured using a vacuum sealed high-precision thermogravimetric analyzer (NITCH TG 209 F1 Libra, Germany). Bamboo debris were scraped with a knife after wax-scalding at different temperatures, and about 10 mg of bamboo debris was collected. The test was carried out under the starting temperature of 50 °C, ending temperature of 260 °C, and heating rate of 10 °C/min.

Crystallinity Analysis

A D8 ADVANCE X-ray diffractometer (XRD, Brook Company) was used to test the changes in crystallinity of wax-scalded bamboo at different temperatures. Bamboo slices after wax-scalding heat treatment at different temperatures were sawed with a small saw, and about 10 mg of bamboo slices were collected. Then the bamboo slices were coated on a glass slide and dried at the test rate of 2 °/ min. After the test, data were recorded. The relative crystallinity CrI was calculated by referring to Segal method,

$$C_{rI} = \frac{I_{002} - I_{AM}}{I_{002}} \times 100\% \tag{1}$$

where I002 (2θ=22.7°) is the highest peak intensity, which is the diffraction intensity of the cellulose crystallization region, and IAM (2θ=18°) is the diffraction intensity of amorphous region in cellulose.

The size of crystallization area D002 was calculated according to Scherer method,

$$D_{002} = \frac{K\lambda}{B_{hkl} \times \cos\theta} \tag{2}$$

where *K* is the diffraction constant of 0.89, λ is the incident wavelength of 0.154 nm, Bhkl is the half width of the diffraction peak, and θ is the diffraction angle. The calculation method of crystal layer spacing *d* is as follows:

$$d = \frac{\lambda}{2\sin\theta} \tag{3}$$

Chemistry Change Analysis

Bamboo slices were sawn with a small saw after wax-scalding heat treatment at different temperatures, and 10 mg of bamboo slices were collected. Then the changes of infrared light absorption rate were tested by a potassium bromide tabletting method and a VERTEX－80V infrared spectrometer. The chemical changes of bamboo after wax-scalding heat treatment were analyzed.

GC-MS Analysis

Dilute paraffin, coarse wax, soft wax, and white wax were dissolved in n-hexane solution to form a solution concentration of 0.10 g/L and the chemical composition of each wax was analyzed using an Agilent 19091S-433. HP-5ms chromatographic column (30 m × 250 µm × 0.25 µm). The carrier gas was helium, with a flow rate of 1 mL/min and a sample inlet temperature of 280 °C. The temperature programmed conditions for the chromatographic column were: maintain at 50 °C for 0.5 min, increase the temperature from 50 to 250 °C at 5 °C /min, maintain for 5 min, and maintain at 10 °C/min to 280 °C for 10 min, Injection volume: 1 μL, no split injection.

RESULTS AND DISCUSSION

Effect of Wax Scalding Process on Penetration Depth

The penetration depth of cold-cycle wax and the penetration effect of paraffin on bamboo under different processing techniques are shown in Table 6 and Fig. 5, respectively.

Table 6. Wax Precipitation on Bamboo Surface During Cold and Hot Cycling

Scheme	Penetration Depth (mm)	Cold and Hot Precipitation	Standard Deviation
A	0.5	No	0.008
B	0.9	Yes	0.01
C	0.6	Yes	0.004
D	1.0	Yes	0.006
E	0.4	Yes	0.002
F	0.7	Yes	0.004

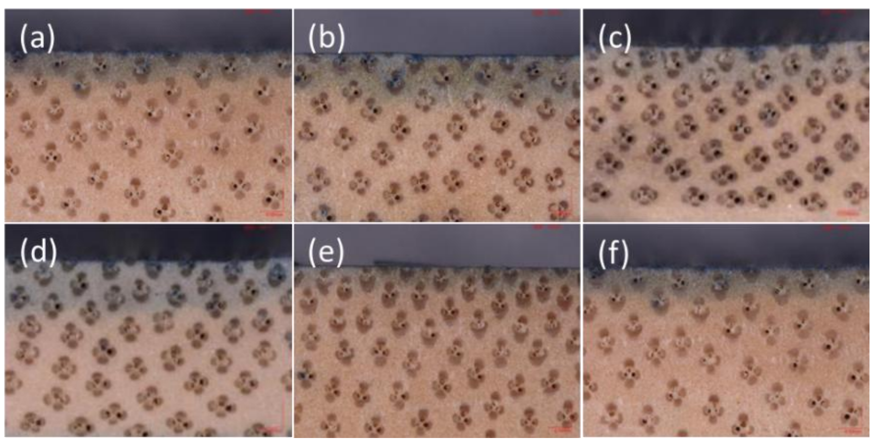

Fig. 5. The penetration effect of paraffin wax in bamboo under different processing technologies: (a) No preheating, wax scalding at 180 °C and for 2 min, The preheating time of (b), (c), (d), (e) and (f) was 2.5 min, and the preheating and wax scalding temperatures were 180, 180,180, 120 and 250 °C respectively, The wax scalding time was 2, 0.5, 4, 2 and 2 min respectively.

Compared with bamboo without preheating (Fig. 5(a)), bamboo preheated at 180 °C (Fig. 5(b)) was able to make the capillary pores on the surface open, which was beneficial to the penetration and filling of paraffin. However, bamboo without preheating was not fully opened, so paraffin could hardly penetrate. When bamboo was preheated at 180 °C, its mechanical properties were less lost, and its dimensional stability was more improved. Through comparative analysis of bamboo wax scalding for 0.5, 2, and 4 min (Fig. 5(c), 5(b), 5(d)), the wax scalding time had certain influence on the paraffin penetration depth in bamboo, and the longer wax scalding time led to a larger penetration depth of paraffin in bamboo. When bamboo was preheated and waxed at 120, 180, and 250 °C (Fig. 5(e), 5(b), 5(f)) respectively. The waxing temperature of 180 °C was the best for paraffin penetration in bamboo.

As described above, this treatment was able to fully open the surface capillary tubes, which was more conducive to the penetration and filling of paraffin. In addition, when treated with hot wax at 180 °C, bamboo had a more stable structure and is less prone to precipitation during use.

Formula of Hot Wax

Effects of temperature on penetration depth of wax in bamboo

Table 7 and Fig. 6 show the penetration effect of wax in bamboo was the worst at 120 °C, and the penetration effect was the best at 180 °C for paraffin wax and soft wax, and at 250 °C for coarse wax and soft wax.

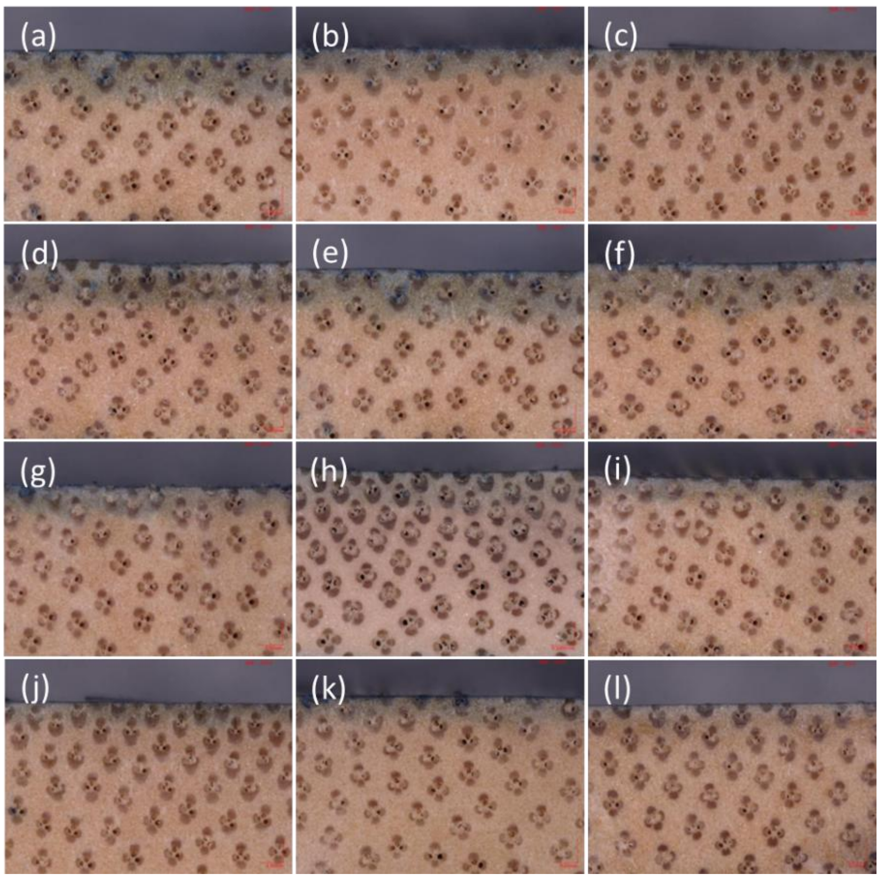

Fig. 6. Penetration effect of different wax scalding species in bamboo at different wax scalding temperatures: paraffin wax at (a) 180, (b) 250,(c) 120 °C , coarse wax at (d) 180, (e) 250, (f) 120 °C , and (g) soft wax at 180 °C

Table 7. Comparison of Penetration Depth of Different Wax Scalding Species at Different Wax Scalding Temperatures

Waxes	Penetration Depth (mm)			Standard Deviation
	120 °C	180 °C	250 °C	
Paraffin	0.4	0.9	0.7	0.008
Coarse wax	0.8	1.0	0.9	0.006
Soft Wax	0.3	0.5	0.8	0.006
White Wax	0.2	0.3	0.4	0.002

When the temperature is too low, the pores of bamboo cannot be fully opened, and the infiltration effect is poor. When the temperature is too high, the wax will sublimate, which will reduce the content of scalding wax and affect the scalding effect (Akgün *et al.* 2021). The boiling point is about 300 °C for both paraffin wax and soft wax and is about 500 °C for both coarse wax and soft wax (Dubas and Schlenoff 2001). The waxes with low boiling point are easy to handle, but they easily sublimate. By contrast, the waxes with high boiling point are relatively stable, but too high temperature consumes too much energy, which is unsuitable for industrial production. Generally, wax with small molecular weight has strong permeability, but it is easily lost through vaporization, and wax with large molecular weight is difficult to handle but relatively stable (Enamul *et al.* 2021). After comprehensive consideration, it is recommended to blanch bamboo with paraffin at 180 °C.

Formula Optimization of Hot Wax

The mixture of paraffin wax and soft wax demonstrated the best penetration effect. Moreover, the coarse wax showed a better penetration effect when scalded alone, and it is suitable for industrial production because of its low price (Bhushan and Jung 2011). To use their respective advantages to increase the long-term effect and spectrum, the authors organized several wax combinations to heat bamboo, as shown in Fig. 7.

Comprehensive analysis and preliminary exploratory experiments indicated that when the proportion of paraffin, white wax, and coarse wax was 5:4:2, the wax scalding temperature was 180 °C and the wax scalding time was 2 min, the wax exhibited the best penetration effect in bamboo and the penetration depth could reach 1.5 mm. The osmotic effect is shown in Fig. 7 (a) transverse cells and (b) radial cells.

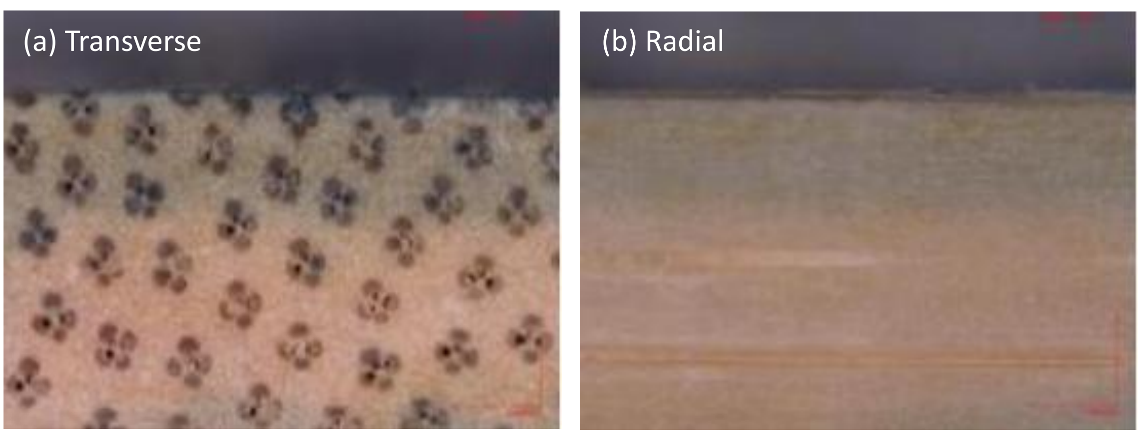

Fig. 7. The effect of 5:4:2 wax scalding on paraffin wax, white wax, and coarse wax

Effect of Wax Scalding Treatment on hydrophobicity

Hydrophobicity analysis of bamboo at different wax scalding temperatures

Table 8 and Fig. 8 show that the contact angle of untreated bamboo was 90.2 when it just touched the material surface, but it decreased to 89.7 after standing for 300 s. This indicated the surface hydrophobicity of the untreated bamboo was poor. After the heat treatment of wax scalding, the contact angle was greatly enlarged, and the maximum was 106.4. Under the action of heat, a part of wax molecules will fill in the cell cavity after condensation reaction, and most of them will remain on the material surface (Paso *et al.* 2009), which will prevent water molecules from entering, thus greatly improving its hydrophobicity and making the bamboo surface unwettable.

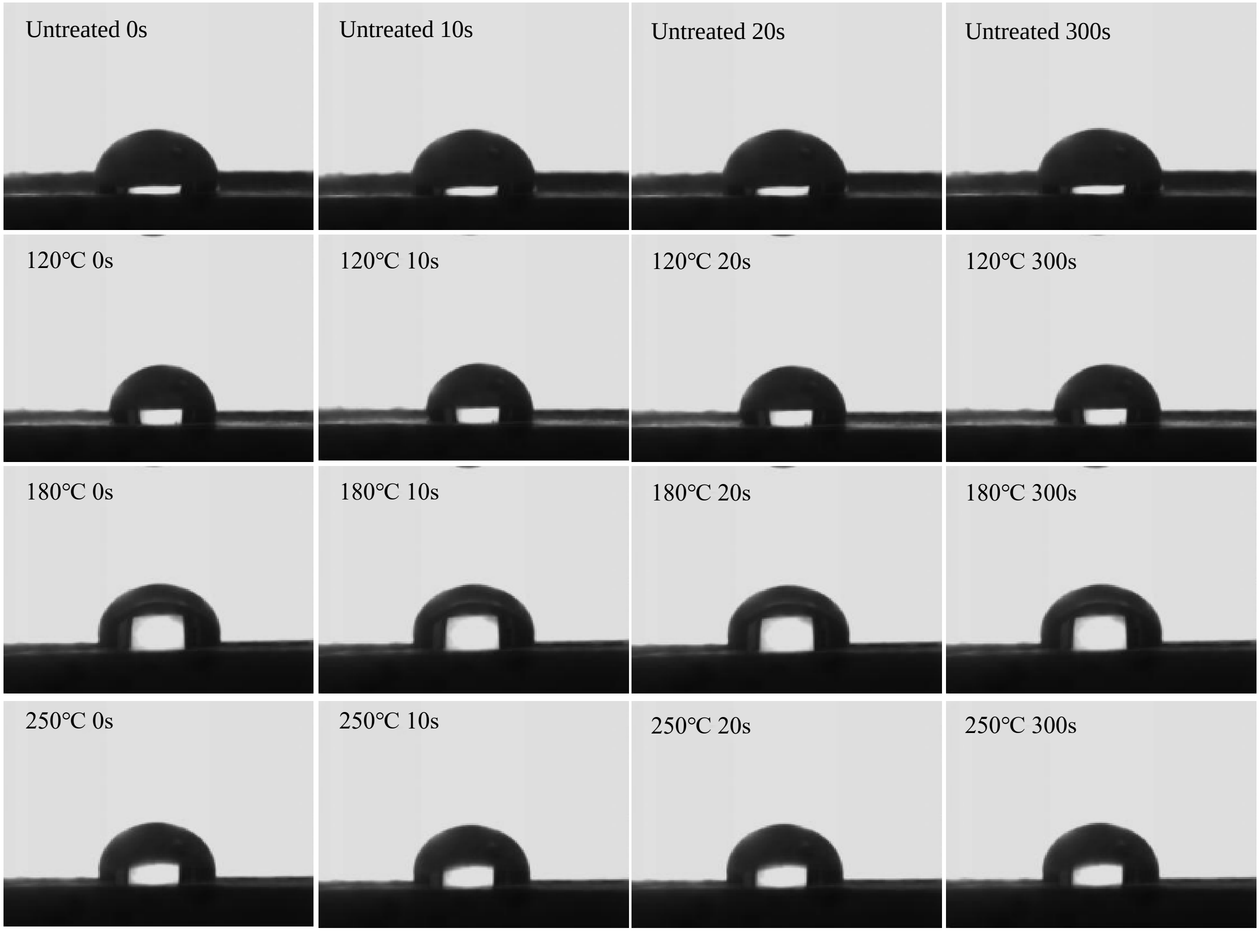

Fig. 8. Comparison of hydrophobicity of bamboo heat-treated at different wax scalding temperatures under different time gradients

Table 8. Contact Angle Degrees of Bamboo Heat-treated at Different Wax Scalding Temperatures under Different Time Gradients

Treating temperature	0 s	10 s	20 s	300 s
Untreated	90.2 °	90.2 °	90.2 °	89.7 °
120 ℃	97.9 °	97.9 °	97.9 °	97.4 °
180 ℃	106.4 °	106.4 °	106.4 °	105.5 °
250 ℃	94.4 °	94.4 °	94.4 °	93.8 °

The hydrophobicity of bamboo is enhanced after wax scalding, and especially its hydrophobic effect is optimized when the preheating temperature and wax scalding temperature reach 180 °C. This is attributed to the fact that the vascular bundle can be opened better at 180 °C, which makes the wax molecules more fully filled and prevents the entry of water molecules. Moreover, paraffin contains substances that can shield hydrophilic groups (Li *et al.* 2015), which make the specimen surface unwettable, and the average contact angle after treatment is between 95 and 107. In addition, the contents of hydrophilic groups such as hydroxyl and carbonyl in bamboo decreased significantly after high-temperature wax scalding treatment (He *et al.* 2022). The contact angle of the specimen only slightly decreased with time at various treatment temperatures, and tended to be stable as a whole, showing a long-term hydrophobic effect.

Effect of Wax Type on Hydrophobicity of Bamboo

The surface hydrophobicity test results of bamboo treated with different wax species are shown in Table 9 and Fig. 9. The initial contact angles of bamboo treated with paraffin wax, white wax, coarse wax, and soft wax were 106.5, 75.8, 102.5, and 101.3 ° respectively, but they decreased to 105.5, 74.6, 102.1, and 99.9 ° respectively after standing for 300 s. Clearly, the hydrophobicity was the most obvious in the bamboo treated with paraffin wax, and it was the lowest for the bamboo treated with white wax. However, the changes in contact angles of bamboo treated with the four types of waxes were not obvious with time, and the overall trend was stable, which indicates a long-term hydrophobic effect. Therefore, the proportion of paraffin in the improved wax scalding formula shall be increased.

Table 9. Comparison of Hydrophobicity of Different Wax Species at Different Time Points

Waxes	0 s	10 s	20 s	300 s
Paraffin	106.4 °	106.4 °	106.4 °	105.5 °
White wax	75.8 °	75.8 °	75.7 °	74.6 °
Coarse wax	102.5 °	102.5 °	102.5 °	102.1 °
Soft wax	101.3 °	101.3 °	101.3 °	99.9 °

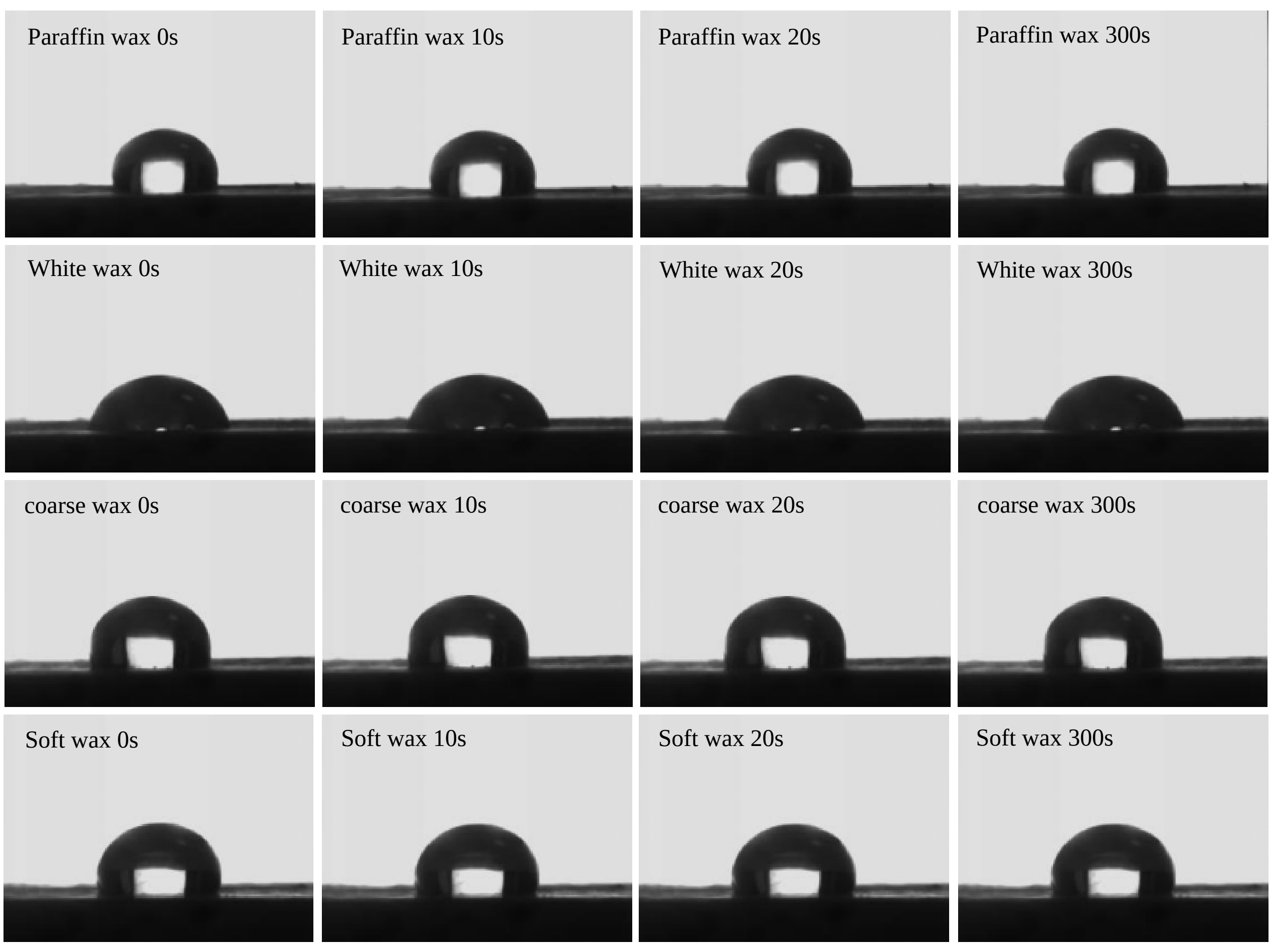

Fig. 9. Comparison of hydrophobicity of different wax scalding wax species at different time points

Effect of Wax Scalding on Glass Transition Temperature of Bamboo

The thermogravimetric and mass change rates of bamboo at different wax burning temperatures are listed in Table 10 and shown in Fig. 10.

Table 10. Changing Rate of Bamboo Weight at Different Wax Scalding Temperatures

Type	Wax ironing temperature (°C)	Weight change(wt%)	Temperature (°C)
Untreated	Room temperature	14.62	183.7767
Thermal treatment	120	17.38	179.2172
Thermal treatment	180	12.43	177.7567
Thermal treatment	250	16.19	181.3794

Figure 10 shows the TG and DTG curves of bamboo at different wax scalding temperatures.

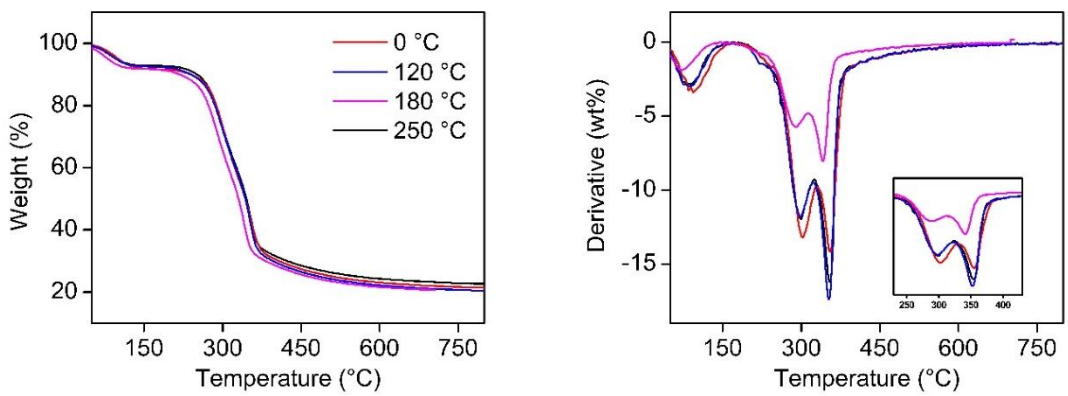

Fig. 10. Comparison of thermogravimetric and mass changing rate of bamboo at different wax burning temperatures

Three thermal degradation stages were observed. The first stage from 50 to 200 °C is caused by the evaporation of water molecules inside the bamboo cell wall. Hemicellulose and some unstable components are decomposed in the second stage at about 200 to 300 °C. Then in the range of 300 to 500 °C, the C-C and C-O bonds of cellulose and lignin are broken, and the weight loss is the fastest near 350 °C. After 600 °C, bamboo components are completely pyrolyzed (Lou *et al.* 2021; Zhang *et al.* 2023). Results showed that the changing rate of bamboo quantity after heat treatment at 180 °C was the smallest. Thus, it can be confirmed the glass transition temperature of this test material was about 180 °C, and it was little affected by different temperature gradients. This result further indicates the most suitable wax-scalding temperature is 180 °C, which is consistent with the previous analysis results of bamboo hydrophobicity at different wax-scalding temperatures.

Effect of Wax Burning on Crystallinity of Cellulose of Bamboo Surface

Table 11 lists the relative crystallinity of heat-treated bamboo, the crystal size of D002, and the highest peak. With the increase of wax scalding temperature, the crystallinity of cellulose in bamboo also increased, indicating the proportion of amorphous region in bamboo cellulose gradually decreases and the proportion of crystalline area slowly increases (Shukla *et al.* 2022). This is because hydroxyl groups in the amorphous area of cellulose undergo polycondensation during the heat treatment, and water is removed to generate ether bonds, which make the arrangement of microfibrils in this area more orderly and closer to the crystalline area (Yun *et al.* 2016). In addition, the crystallinity increased from 0.7% to 1.9% and 3.9%, indicating the degradation reaction of the amorphous region became intense with the increase of heat treatment temperature, and the increasing rate of crystallinity of cellulose was higher.

The size of crystallization zone D002 decreased with the increase of heat treatment temperature, suggesting heat treatment resulted in a smaller crystallization size of bamboo cellulose, a larger interlayer spacing, and a loose crystal structure. Obviously, the sharpness and width of the 2θ peak of bamboo treated with wax at 180 °C outperformed those of the samples treated with other conditions. Hence, it can be confirmed the bamboo treated at 180 °C had higher crystallinity and hydrothermal stability, which is consistent with the analysis results of different wax-scalding temperatures on bamboo hydrophobicity and glass transition temperature. Under high temperature and water vapor conditions, the structure cannot be easily damaged and can maintain sufficient balanced activity (Yun *et al.* 2016).

Table 11. Crystallinity of Bamboo at Different Wax Burning Temperatures

Wax ironing temperature (°C)	Crystal plane angle	Crystallinity	D_{002} (nm)	Relative crystallinity (%)	*d*(nm)
Room temperature	22.51	689	3.53	24.9	0.207
120	22.67	1021	3.39	25.6	0.207
180	22.78	1189	3.02	27.5	0.210
250	22.86	1153	2.87	31.4	0.210

The variation curves of XRD intensity of bamboo treated with different wax scalding temperatures are shown in Fig. 11. Clearly, the pattern of bamboo treated with wax scalding was similar to that of untreated bamboo. Specifically, bamboo that has undergone low-temperature treatment does not exhibit peak formation or peak disappearance to the same extent, which is a typical cellulose I pattern (Zhang *et al.* 2020), indicating the thermal treatment does not change the crystalline morphology of bamboo cellulose.

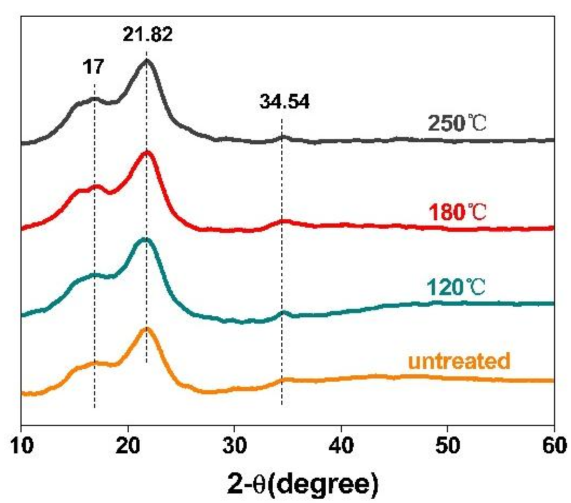

Fig. 11. Comparison of crystallinity of bamboo at different wax scalding temperatures

The structure of bamboo treated in this way was more stable, such that it can effectively reduce wet swelling, dry shrinkage, thermal swelling, and cold shrinkage, and it can also better reduce wax precipitation during scalding and improve the quality of bamboo wax scalding.

Effect of Wax Scalding Treatment on Hydroxyl Content

The results of chemical changes in wax-scalded bamboo tested with an infrared spectrum analyzer are shown in Fig. 12. The chemical changes in the scalding temperature of different bamboo materials treated with wax at 120, 180, and 250 °C were analyzed according to the wave number and absorption sharpness of infrared light. The test results are shown in Table 12.

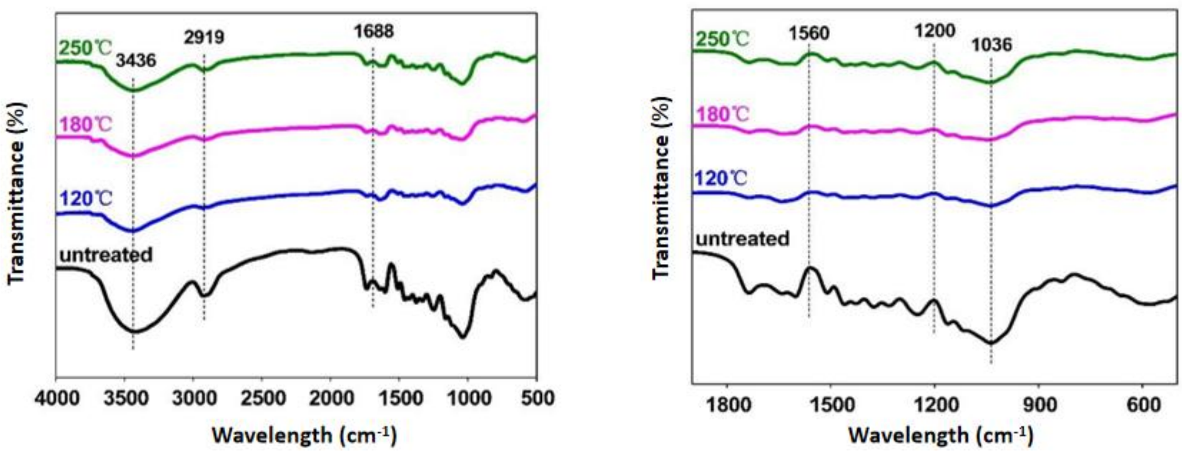

Fig. 12. Infrared spectra of bamboo at different wax scalding temperatures

The elongation vibration peak of hydroxyl group (OH) was at 3436 cm^{-1}, indicating that the hydrogen bond composition in bamboo had changed and the number of hydroxyl groups in bamboo had decreased after wax scalding. The peak at 1688 cm^{-1} stands for the stretching vibration of carbonyl group (C=O) in non-conjugated acetyl group, and the peak at 1560 cm^{-1} reflects the stretching vibration of benzene ring in lignans. These two peaks were weakened at different wax scalding temperatures, indicating the number of hydrophilic and carbonyl groups in bamboo decreased after wax scalding treatment. The main reason for this decrease is that first, the free hydroxyl groups between cellulose molecular chains in bamboo are polycondensed under the high temperature treatment of scalding wax, which removes water and forms ether bonds, resulting in a significant decrease in the number of free hydroxyl groups (Wang *et al.* 2016; Wang *et al.* 2020). Second, under the action of hydrothermal treatment, the acetyl group on the polysaccharide molecular chain of hemicellulose with the worst thermal stability is hydrolyzed to acetic acid, which reduces the number of carbonyl groups (Wei *et al.* 2021). At the same time, under the action of acetic acid, lignin in bamboo reacts, which reduces the number of hydroxyl groups with strong water absorption and increases the number of carbonyl groups. In other words, carbonyl groups with weak water absorption replace hydroxyl groups with strong water absorption (Wei *et al.* 2021). It is precisely because of the combined effect of the above factors that the content of water-absorbing groups in bamboo decreases after wax-scalding heat treatment (Evans *et al.* 2005). Additionally, the peak at 1200 cm^{-1} is the C-O stretching vibration of O=C-O in hemicellulose and ester side chains, and the peak at

1036 cm^{-1} is the asymmetric stretching vibration absorption of C-O-C in lignin and cellulose. During wax scalding, the intensity of these two peaks will decrease after heat treatment, which shows that the number of hydrophilic C-O in bamboo will drop during heat treatment. The water molecules in bamboo cell polymer are mainly OH, C = O, C = O and C-O, while the water in monolayers is mainly composed of these active sites. In summary, when the hydroxyl groups on the surface of bamboo after waxing are reduced, the water contact angle increases, resulting in a stronger "lotus effect". Water molecules find it more difficult to spread and wet the surface, thereby enhancing hydrophobicity. At the same time, the hydroxyl groups inside the material are prone to adsorb water molecules or polar pollutants from the air. Reducing hydroxyl groups can reduce surface adsorption and maintain smoothness.

Table 12. Attribution of Main Absorption Peaks in Infrared Spectra of Bamboo at Different Wax Burning Temperatures

Wavenumber (cm^{-1})	Attribution and Explanation of Absorption Band
3436	O-H stretching vibration
2919	C-H stretching vibration
1688	C=O stretching vibration (Hemicellulose xylan acetyl group CH_3-C=O)
1560	Extension vibration of benzene ring (lignin)
1200	C—O stretching vibration (O = C-O in the side chain of hemicellulose and ester)
1036	Asymmetric telescopic vibration of c-o-c (Lignin and cellulose)

Hot wax treatment leads to a decrease in the content of hydrophilic functional groups, and the decrease of active sites results in a decline in water adsorption of monolayer in bamboo. Due to the decrease of active center, multilayer-adsorbed water will affect the adsorption capacity of water between polymer layers, thus improving the hydrophobicity of bamboo. Because there are abundant hydrophilic groups in the cell wall of bamboo, including carbonyl and especially hydroxyl, water molecules can easily enter the cell wall of bamboo when the external humidity changes. As a result, the bamboo will shrink and swell, and finally bend and deform, which will affect the performance of the bamboo (Zolghadr *et al.* 2022; Yu *et al.* 2023).

CONCLUSIONS

1. After hot wax treatment, the surface of bamboo thermoplastically deforms, which makes its pores fixed, and the wax penetration depth in bamboo is enlarged at high temperature. The heat treatment of scalding wax at 180 ℃ can fully open the capillary pores to achieve the penetration and filling of wax, thereby increasing the hydrophobicity of bamboo.
2. Under the condition of wax heating at 180 ℃, the bamboo had a more stable structure and higher hydrothermal stability, and the wet swelling, thermal swelling and cold shrinkage were reduced, indicating that it did not easily precipitate during use. The wax scalding time within 2 min influenced the wax penetration effect in bamboo, and the change was small between 2 and 5 min. The best effect was obtained at the wax scalding time of 2 min.

3. Paraffin contains substances that can shield hydrophilic groups, and the effect of scalding wax was the most obvious in improving hydrophobicity, which has long-term hydrophobicity. When the proportion of paraffin, white wax, and coarse wax was 5:4:2, the penetration effect of wax in bamboo was the best after wax scalding at 180 ℃ for 2 min.
4. X-ray diffractometry and infrared spectrometry were used to analyze the changes in crystallinity and chemical properties of bamboo after wax-scalding at different temperatures. Results further confirm wax-scalding treatment will lead to higher bamboo crystallinity and reduce the content of hydrophilic functional groups.
5. The protection technology of hot wax treatment has been studied to improve weather resistance in outdoor protection of bamboo products. The findings provide a theoretical basis for studies on the mildew resistance, weather resistance, loss resistance, aging resistance and industrial continuous production of bamboo products treated with hot wax.

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