Improvements in the Physical Properties and Decay Resistance of Bamboo Materials *via* Modification with Boric Acid and Borax

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As a renewable biomaterial, bamboo has been widely used in construction and indoor decoration. While the application of bamboo is limited by its low decay resistance and flame retardancy, the combination of modification with boric acid and borax has been found to be effective for improving bamboo flame retardant. In this study, the decay resistance, mechanical properties, and physical properties of bamboo, both before and after treated being with boric acid and borax (in a 1 to 2 ratio), were analyzed to better utilize the modification solution for bamboo. The results showed that in comparison to the untreated bamboo, the treated bamboo had a strong decay resistance, *i.e.*, greater than 80% resistance effectiveness improvement. In addition, the decay resistance and compression strength of the samples showed a considerable increase, *i.e.*, 21%, after the modification. However, the physical properties *e.g.*, the weight gain, equilibrium moisture content, and dimensional stability and bending properties of the bamboo were less affected by the modification method, which could be improved *via* the combination with another chemical modification agent.

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INTRODUCTION

With the increase in environmental awareness and the decrease in unrealized resources, the application of environmental friendliness and renewable materials has begun to be prioritized in many fields (Serrano-Ruiz *et al.* 2010; Ribé 2018; Liu *et al.* 2021b). As an environmentally friendly material, bamboo has been widely used in many fields, especially as a decorative material for furniture production and indoor decoration, due to its fast material formation, excellent mechanical properties, and high economic efficiency (Yu *et al*. 2015; Li *et al*. 2016; Li *et al.* 2018a; Zhao *et al*. 2019; Li *et al.* 2020). However, the application of bamboo materials is highly limited because of its ease of decay and high flammability, which is caused by the rich starch grains and extractives in the bamboo. Consequently, it is necessary to effectively improve the decay resistance and flame retardancy of decorative bamboo.

To improve the decay resistance and flame retardant performance of bamboo, many methods have been studied. Due to the poor lateral permeability of bamboo, it plays an important role in the use of bamboo, with respects to the function of the surface of the bamboo (Tian *et al.* 2012; Sharma *et al.* 2017; Klemm *et al.* 2018). Impregnation with a synthetic resin, *e.g.*, furfuryl alcohol (Li *et al.* 2019; Li *et al.* 2020b; Liu *et al.* 2020b; Liu *et al.* 2021a), or surface modifications with a nanocoating of ZnO and/or TiO_2 (Li *et al.* 2015; Zhang *et al.* 2017), have been used to improve the durability properties, *e.g.*, the decay resistance of bamboo. For biomass materials, *e.g.*, wood and bamboo, the improvement of the flame retardant performance primarily depends on the corresponding combustion improvement material, which usually contains certain elements, *e.g.*, boron, aluminum, nitrogen, or halogen (Li *et al*. 2017; Wang *et al*. 2017). With concerns about human health and environmental protection, the use of halogen-containing flame retardants has been reduced gradually in many countries (Chen *et al.* 2009). As a result, fire retardants which contain P, N, and B have been used as fire retardants for wood and bamboo due to their positive characteristics, *i.e.*, low smoke, low toxicity, and smoke suppressing properties (Yang *et al.* 2014; Tsapko *et al.* 2020; Wu *et al.* 2021). However, most of the research on improving the decay resistance and flame retardancy of bamboo are independent of each other. Therefore, it is necessary to discover a modifying agent that improves both the decay resistance and flame retardancy of bamboo.

Boron compounds are widely used as flame retardants in wood and bamboo products, which considerably reduces the severity of thermal degradation (Nagieb *et al.* 2011; Wu and Xu 2014; Cavdar *et al.* 2015). In addition to imparting flame retardancy, boron compounds also have several advantages in wood preservation, and are especially effective against fungi and termites (Nagieb *et al.* 2011; Yildirim and Candan 2021). As one of the most widely used boron compounds, boric acid is a good preservative and flame retardant for biomaterials (Pedieu *et al.* 2012; Efhamisisi *et al.* 2016). In addition, the thermal stability of nanocellulose and lignocellulose, which are used in bio-composites, could also be improved by boric acid modification (Zhang *et al.* 2020; Yildirim and Candan 2021).

Boric acid is usually used with borax in most studies, due to their different flame-retardant characteristics (Baysal *et al.* 2007). A modified solution containing boric acid and borax was used to improve the durability and flame retardancy of wood or wood products, and some of the physical and mechanical properties of wood could be improved by varying the proportion of boric acid and borax and the treatment temperatures (Ustaomer and Usta 2012; Palanti *et al.* 2012; Zhang *et al.* 2015; Kol *et al.* 2017; Wu *et al.* 2021). Although boric acid and borax have been used in wood modification for many years, there are only a few reports on its application for improving the flame retardancy of bamboo (Yu *et al.* 2017; Li *et al.* 2020a). To better understand the impact of boric acid and borax on the performance of bamboo, various physical properties, mechanical properties, and decay resistance of bamboo were analyzed in this study.

EXPERIMENTAL

Materials

Moso bamboo (*Phyllostachys pubescens* Mazei ex H. de Lebaie) strips with a regular cross section were taken from Yiyang, Hunan Province, China. The strips were split from newly cut bamboo, following removed bamboo green and bamboo yellow before further testing. The bamboo samples were prepared according to GB/T standard 1928 (2009) (as shown in Table 1) and then dried at a temperature of 103 °C until an oven-dry state was achieved. Boric acid (BA, 99.5%) and borax (BX, 99.0%) were purchased from Aladdin Chemistry Co., Ltd. (Shanghai, China) and were not further purified, *i.e.*, they were used as received. Deionized water was used in all experiments.

Table 1. Information of the Bamboo Samples

Parameter	Size (T × R × L)	Number of Samples/Groups
WPG	20 mm × 5 mm × 20 mm	8
ASE		
EMC	20 mm × 5 mm × 20 mm	8
CS		
MOR	10 mm × 5 mm × 160 mm	12
MOE		
Decay test	20 mm × 5 mm × 50 mm	24

Solution Preparation and Soaking

The modification solution, which contained 1.5 wt.% BA and 3.0 wt.% BX, was prepared at room temperature. The bamboo specimens were soaked in the modification solution for 72 h at room temperature. After soaking, the specimens were dried at a temperature of 60 °C and 80 °C for 2 h, respectively, and later at a temperature of 103 °C until an oven-dried state was achieved.

Physical and Mechanical Properties

The physical properties of all the samples were tested according to Chinese National Standard GB/T 1928 (2009), and the mechanical properties were tested according to Chinese National Standard GB/T 15780 (1995), while the decay resistance was tested by following the method described in the Chinese National Standard GB/T 13942.1 (2009). White rot fungus (*Coriolus versicolor* (CV)) and brown rot fungus (*Gloeophyllum trabeum* (GT)) were used in this study, and the specific testing and computing methods were described in Liu *et al.* (2020b).

The weight percentage gain (WPG) was measured to determine the introduced amount of boric acid and borax and was calculated based on Eq. 1,

$$WPG = \frac{(m_1 - m_0)}{m_0} \times 100\% \tag{1}$$

where m_0 and m_1 are the oven-dried mass of the bamboo before and after modification, respectively.

The anti-swelling efficiency (ASE) was used to judge the dimensional stability of bamboo, and the ASE and equilibrium moisture content (EMC) were tested according to Liu *et al.* (2020a); the ASE was calculated according to Eqs. 2 and 3,

$$\alpha = \frac{(V_{wet} - V_{dry})}{V_{dry}} \times 100\% \tag{2}$$

$$ASE = \frac{(\alpha_0 - \alpha_1)}{\alpha_0} \times 100\% \tag{3}$$

where V_{wet} and V_{dry} are the volume of specimens in a wet and dry state, respectively, and

α_0 and α_1 represent the coefficient of the wet expansion of the untreated and modified wood, respectively.

The modulus of elasticity (MOE), modulus of rupture (MOR), and parallel-to-the-grain compressive strength (CS) of bamboo were tested according to the Chinese national standard GB/T 15780 (1995). For the CS test samples, they were loaded with a constant loading rate, and the maximum compressive load that could be withstood for greater than 90 s without collapse was recorded.

Characterization

The changes of the micro morphologies of the bamboo samples were tested *via* scanning electron microscopy (SEM). The microstructure of the control and treated samples were imaged with an environmental scanning electron microscope (ESEM-XL 30, FEI Company, Hillsboro, OR) was operated at 7.0 kV in this study followed by being sputter-coated with an ultra-thin layer of platinum.

The functional group characterization of the bamboo samples was conducted *via* Fourier-transform infrared (FTIR) spectroscopy (Nexus670, Nicolet, Madison, WI). The FTIR spectra of the untreated and modified bamboo samples were recorded within the range 400 to 4000 cm^{-1} with an average of 64 scans at a resolution of 4 cm^{-1}.

RESULTS AND DISCUSSION

Physical Properties

The physical properties, *i.e.*, the WPG, EMC, and ASE, of the untreated and treated bamboo, are shown in Table 2. The weight of the treated bamboo samples slightly changed, which was different from the results of the wood modification in other previous studies (Baysal *et al.* 2004, 2007). The different changes in the weight could be explained by the starch grains in the parenchymal cells of bamboo being lost because of the hydrolysis of the acidic mixture solution during the treatment, resulting in less weight in the bamboo materials. Compared with the control group, the equilibrium moisture content of the treated bamboo decreased from 10.11% to 9.15%, which represents a decrease by 9.50% after the modification. The reduction of the EMC was primarily caused by the fact that the boric acid can interact with the hydrophilic groups, *e.g.*, the hydroxyls in the lignins, *via* esterification, thus reducing the hydrophilic groups content in the bamboo (Uddin *et al.* 2017; Chio *et al.* 2019). However, due to the limited hygroscopicity of the boric acid, the EMC of the treated bamboo did not considerably increase (Kartal *et al.* 2007). Table 2 shows the ASE value of the treated bamboo was 7.53%, which indicated that the dimensional stability of the bamboo sample had been slightly improved. This discovery has been confirmed by Toussaint-Douvergne *et al.* (2000), who found a low-cost boron preservation system could improve the anti-swelling efficiency of treated timber. In addition, Baysal *et al.* (2007) reported that the diffusion of a monomer into the cell wall would be limited because of the boron crystal deposition and the polymerization within the cell wall, which would have a negative effect on the efficiency in terms of improving the dimensional stability.

Table 2. Physical Properties Values of the Bamboo Treated *via* a Boric Acid and Borax Mixture

	WPG (%)	EMC (%)	ASE (%)
Control	--	10.11 ± 0.22	--
Treated bamboo	0.64 ± 0.10	9.18 ± 0.14	7.53 ± 2.71

Mechanical Properties

Table 3 provides information on the changes of the mechanical properties of the bamboo upon treatment. The values of the modulus of elasticity and modulus of rupture of the treated bamboo were 9.54 GPa and 135.34 MPa, respectively, which were similar to the untreated bamboo. The results showed that the positive effect of the reduction of hydrolysis in the bamboo on the bending properties was offset by the adverse effects of the acidic effect on the mechanical properties of the bamboo. In addition, compared with the untreated bamboo, the CS of the treated bamboo showed an improvement of 21.56%; this result was supported by Kol *et al.* (2017), who found that the compression strength of the wood increased after being impregnating with borax.

Table 3. Mechanical Properties Values of Bamboo Treated *via* a Boric Acid and Borax Mixture

	MOE (GPa)	MOR (MPa)	CS (MPa)
Control	9.31 ± 0.84	128.76 ± 10.92	59.52 ± 2.41
Treated bamboo	9.54 ± 0.71	135.34 ± 7.43	72.35 ± 1.77

Decay Resistance Properties

The damage to bamboo *via* decay fungi can be evaluated *via* the mass loss percentage. The average mass loss percentages of the control and treated bamboo after erosion by GT fungus and CV fungus are shown in Table 4. For the decay resistance, the mass loss percentages of the control bamboo after erosion by the white rot fungi and brown rot fungi were 62.6% and 55.6%, respectively, which indicated that the untreated bamboo was seriously decayed after the decay test. However, the mass loss percentages of the treated bamboo were only 12.5% and 9.6%, respectively. These results showed that the protection could control bamboo-destroying fungus and resulted in bamboo materials with excellent resistance to decay. This is due to the toxic effect of the boric acid and borax on the bamboo biology, which would destroy the appropriate environment for the survival of decay fungi (Wu *et al*. 2021).

Table 4. Mass Loss of the Treated Bamboo after the Decay Test

	Mass Loss (%)	
	Coriolus versicolor (CV)	*Gloeophyllum trabeum* (GT)
Control	62.57 ± 9.86	55.62 ± 11.53
Treated bamboo	12.74 ± 1.65	9.64 ± 1.46
Note: The mass loss rate refers to the ratio between the mass differences of the sample before and after it is corroded *via* decay and the mass of the sample before it is corroded by decay		

Microscopic Morphology

Figure 1 displays the SEM images of the cross sections of the untreated and modified bamboo samples. As shown in Fig. 1a, there were a larger amount of starch grains located in the cavity of the parenchymal cells of the control bamboo, which provided nutrients for the growth of the mold and decay fungi. However, it could be observed that the number of grains in the parenchymal cells was considerably reduced after the modification (Fig. 1c), which was primarily due to the hydrolysis of the acidic mixture modification solution during the long soaking treatment. This phenomenon led to the weight of the bamboo samples slightly increasing and destroying the living environment of the mold and decay fungi.

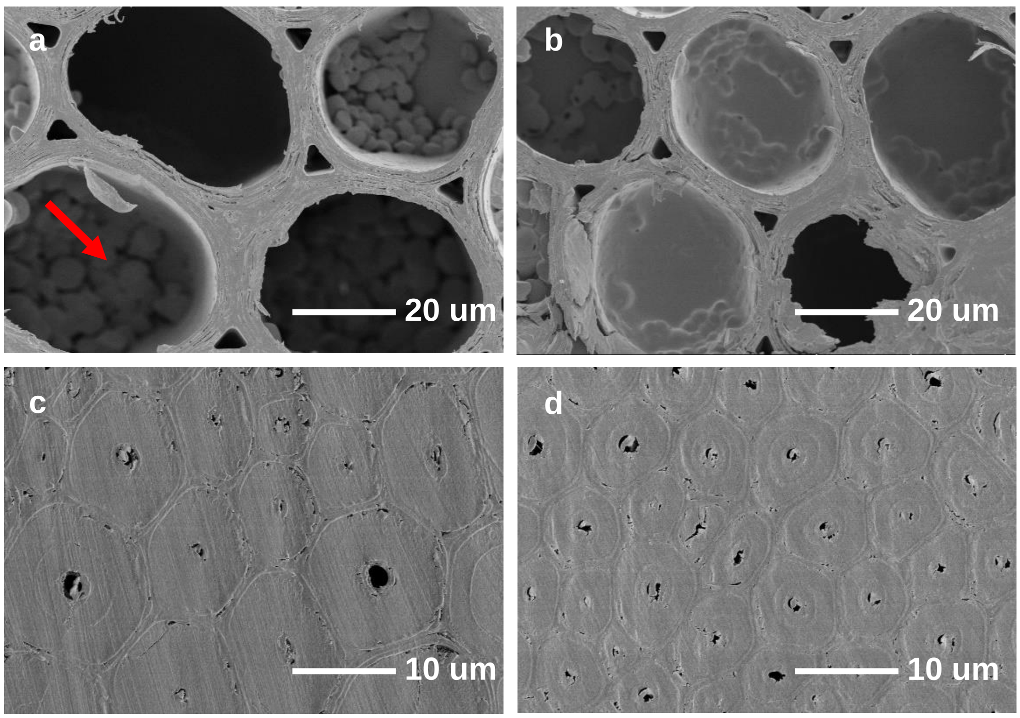

Fig. 1. SEM micrographs of the microtomed cross-sections: parenchymal cells of the (a) untreated bamboo and (b) treated bamboo; fibers of (c) untreated bamboo and (d) treated bamboo

In addition, the fiber and parenchymal cells of the bamboo still maintained a complete cell wall structure and form, as shown in Fig. 1c and 1d, which indicated that the decrease of some of the mechanical properties of the bamboo was limited after the modification with the BA and BX mixture.

The FT-IR spectra of the control and treated bamboo are presented in Fig. 2. In the spectra of the treated bamboo (Fig. 2), the intensity of the bands located at 1380, 1340, and 810 cm^{-1} underwent a considerable increase in intensity, which were assigned to the stretching vibrations of the boric acid hydroxyl groups, the B-O-B stretching vibrations, and the stretching and bending vibrations of the B-O in BO_4-tetrahedra (Feng *et al.* 2011; Zhang *et al.* 2020). In addition, the absorption band between 1734 and 1745 cm^{-1}, which is assigned to the carbonyl group of boric acid, showed a considerable enhancement and widening (Nagieb *et al.* 2011). These results demonstrated that the boric acid and borax was successfully introduced into the bamboo, and the esterification between the boric acid and wood component was established. With further analysis, the characteristic bands of a benzene ring at 1600, 1500, and 1450 cm^{-1} were enhanced by the valence vibrations bands of B-O-B and B-O-C, which indicated the presence of the self-polymerization of the boric acid in the treated bamboo (Zhang *et al.* 2020).

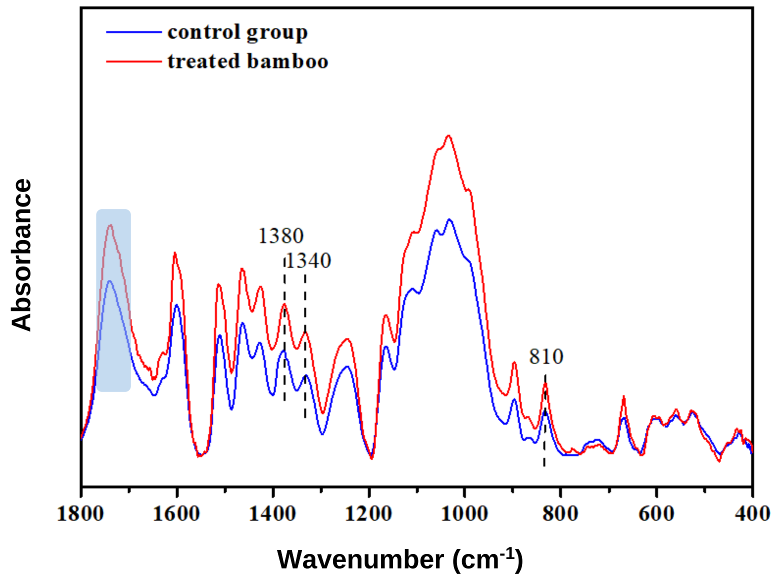

Fig. 2. FT-IR spectra of the control and treated bamboo

CONCLUSIONS

1. The modification of bamboo with boric acid and borax considerably improved its decay resistance; the mass loss percentages of the treated bamboo after erosion by white rot fungi and brown rot fungi were 12.5% and 9.6%, respectively. This showed excellent resistance to decay compared to the untreated samples, the treatment resulting in a greater than 80% resistance effectiveness.
2. The combination impregnation with boric acid and borax had a small influence on some of the physical properties of bamboo, *e.g.*, the equilibrium moisture content (EMC) and dimensional stability.
3. The modulus of elasticity and modulus of rupture of the bamboo only exhibited slight changes after the modification with boric acid and borax. The compression strength of the samples showed a considerable increase after impregnation with the boric acid and borax mixture.

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