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Effect of High-Temperature Hydrothermal Treatment on Chemical, Mechanical, Physical, and Surface Properties of Moso Bamboo

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Abstract: Bamboo is an ideal material as it is green, fast-growing, and easy to process. However, the low dimensional stability may limit the application of bamboo due to its richness in hydrophilic groups. Thus, an effective and environment-friendly modification is needed to solve the aforementioned problems. This study employed high-temperature hydrothermal treatment for the modification of bamboo to offer technical support to further promote the application of bamboo materials. Bamboo was heated at various temperatures (120–160 °C) for diverse durations (60–120 min), and the chemical composition, mechanical properties, dimensional stability, and surface color were studied. Results revealed that the parenchyma cells of bamboo were deformed and the parenchymal cell lumen without starch granules after treatment at 160 °C for 120 min. The cellulose and hemicellulose content of bamboo decreased, and the lignin content increased in relative terms as the temperature and time of high-temperature hydrothermal treatment continued to rise. The mechanical properties of bamboo declined after high temperature hydrothermal treatment, and the modulus of rupture (MOR) and modulus of elasticity (MOE) of bamboo at 160 °C for 120 min decreased by 47.11% and 16.14%, respectively, compared to untreated bamboo. The swelling test indicated that the dimensional stability of the bamboo was improved, and the swelling ratio of the bamboo was reduced through the high-temperature hydrothermal treatment. The tangential and radial swelling coefficients of bamboo were reduced by 53.28% and 53.59%, respectively, after treatment at 160 °C for 120 min compared to untreated bamboo. The bamboo surface color was darkened after heat treatment, which gives the bamboo better surface decorative properties.

Keywords: high-temperature hydrothermal treatment; bamboo; chemical constituents; color; mechanical properties; dimensional stability

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Citation: Li, X.; Peng, H.; Niu, S.; Liu, X.; Li, Y. Effect of High-Temperature Hydrothermal Treatment on Chemical, Mechanical, Physical, and Surface Properties of Moso Bamboo. *Forests* **2022**, *13*, 712. https://doi.org/10.3390/f13050712

Academic Editor: Antonios Papadopoulos

Received: 13 April 2022
Accepted: 29 April 2022
Published: 1 May 2022

1. Introduction

As an abundant and environment-friendly lignocellulosic material, bamboo exhibits rapid growth and high mechanical strength [1–3]. In the past decades, contemporary industrial technology has vastly enlarged the application of bamboo materials in various fields such as architecture, furniture, flooring, and civil engineering. However, owing to the rich hydrophilic groups, anisotropy, and non-uniformity, bamboo has strong hygroscopicity, resulting in poor dimensional stability [4]. When exposed to humidity changes, the bamboo responds by shrinking and swelling readily [5], resulting in dimensional and form variations and inevitable flaws in bamboo. Poor dimensional stability not only causes distortion and deformation, but also causes cracking, which is a troublesome problem for all products made of lignocellulosic materials, including bamboo products. Therefore, it is of profound significance to explore how to modify bamboo materials available and improve the corresponding dimensional stability to expand the application range of bamboo materials.

Currently, various treatment strategies have been used to enhance the dimensional stability of bamboo, in terms of chemical, biological, or physical treatments. The chemical methods normally involve the addition of acetic anhydride [6], furfuryl alcohol [7], and polyethylene glycol [8]. However, the structure of bamboo components is variable, such as the irregular distribution of density across bamboo cane thickness, which may result in different effects of infiltration and thus generally requires vacuum pressurization for treatment. The biological modification method is mainly treated by adding fungi [9] and enzymes [10]. Although these methods have been applied in small-scale production according to their abilities to ameliorate bamboo performance, these methods have some disadvantages. For example, the process takes a long time and has high costs [11,12]. Physical modification methods include ultrasonic treatment [13], gamma rays irradiation treatment [14], and heat treatment, etc. [15,16]. As reported, ultrasonic processing can further improve the properties of bamboo, but it is not suitable for large-scale production and application [17]. The gamma irradiation technology can provide potent insecticidal and sterilizing effects without significantly degrading the chemical composition of the bamboo [18]. However, high doses of radiation can create a threat to human health [19]. On the contrary, hot treatment is supposed to be low cost, casue less environmental pollution, and is a feasible method of treatment for bamboo, with evidence of altering the surface or chemical composition and increasing the dimensional stability of bamboo [20].

Heat treatment is the treatment of biomass materials in a high-temperature, low-oxygen or even anaerobic environment, which can improve the inherent properties of bamboo without changing its ecological friendliness [21]. Oil heat treatment and steam heat treatment occupy a dominant position when it comes to heat treatment methods [22,23]. By contrast, oil heat treatment can have a negative impact on the environment, and it is very difficult to dispose of the waste. In addition, heating steam requires high energy consumption and special conditions, which have certain limitations. In recent years, hydrothermal treatment of modified bamboo has been recognized as an ecologically friendly, high-efficiency, and feasible method. At present, the effect of hydrothermal treatment on bamboo properties has been studied and analyzed [24,25]. However, research on its mechanism is very limited. Therefore, considering the great influence of high-temperature hydrothermal treatment on bamboo and the actual demand for bamboo application. It is of vital importance to study the mechanism of the effect of high-temperature hydrothermal treatment on the chemical, mechanical, physical, and surface properties of moso bamboo.

This study aimed to explore the influence mechanism of high-temperature hydrothermal treatment on the comprehensive properties of bamboo. Modification of bamboo was performed by the high-temperature hydrothermal method, and the treatment temperature was 120–160 °C, and the treatment time was 60 min, 90 min, and 120 min, respectively. At the same time, the chemical composition, mechanical properties, dimensional stability, and color change of bamboo blocks treated by different processes were analyzed. In addition, the microstructural changes of bamboo after hydrothermal treatment were viewed by scanning electron microscope (SEM). High-temperature hydrothermal treatment has enhanced the dimensional stability of the bamboo blocks. This study provides the support of theory and technology for promoting the production and application of bamboo.

2. Materials and Methods

2.1. Materials

Moso bamboo (*Phyllostachys edulis*) of 6 years old was harvested from Lin'an, Zhejiang, China. The moso bamboo was cut into 160 mm × 10 mm × 8 mm (longitudinal × tangential × radial) samples; the bamboo samples were selected 2 m from the root. The hydrothermally modified bamboo samples were divided into 18 treatment groups, including control group (untreated) samples. Of note, bamboo samples with a certain density range

(0.58–0.64g/cm^3) were selected for high-temperature hydrothermal treatment, to eliminate the influence of density differences on performance.

2.2. High-Temperature Hydrothermal Treatment Method

The bamboo samples were dried in a drying oven at 60 °C until the weight was constant. Subsequently, bamboo was subjected to hydrothermal treatment at different temperatures and times in high-temperature hydrothermal treatment facilities. The temperature of heat treatment was 120 °C, 130 °C, 140 °C, 150 °C, and 160 °C, and the duration of heat treatment was 60, 90, and 120 min, respectively. The processed samples were treated with balanced water content (temperature 20 °C, relative humidity 65%) and then stored in a desiccator. Figure 1 illustrates the process of high-temperature hydrothermal treatment of bamboo samples.

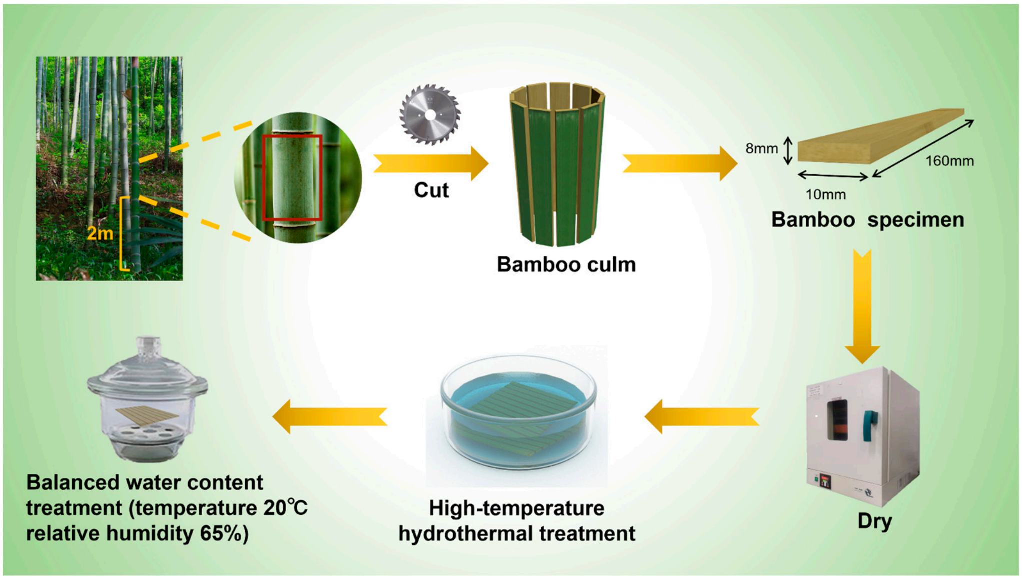

Figure 1. The high-temperature hydrothermal treatment process of moso bamboo specimens.

2.3. Characterization

2.3.1. FTIR analysis

The spectra were logged by an infrared spectrometer with a vertex 80v (Bruker Corporation, Karlsruhe, Germany). The thin slices pressed by mixing bamboo powder and potassium bromide were fed into the infrared spectrometer to evaluate the variation of molecular groups of the samples before and after the treatment. All FTIR images were scanned from 4000 cm^{-1} to 1000 cm^{-1} with 4 cm^{-1} resolution.

2.3.2. Chemical Composition Test

In this experiment, the untreated bamboo samples and the bamboo samples after high-temperature hydrothermal treatment were sieved into 40–60 mesh bamboo powder. The National Renewable Energy Laboratory (NREL) method was used [26]. The influence of high-temperature hydrothermal treatment on moso bamboo was investigated by measuring three main chemical elements (cellulose, hemicellulose, and lignin) in moso bamboo.

2.3.3. Mass Loss

Mass loss (ML) was calculated as follows:

$$\mathrm{ML}(\%) = \frac{W_1 - W_2}{W_1} \times 100\% \quad (1)$$

where W_1 is the oven-dried mass of the untreated samples, W_2 is the oven-dried mass of the sample after high-temperature hydrothermal treatment.

2.3.4. Microstructure observation

The surface morphology of untreated and treated bamboo samples was observed by TM3030 (Opton, Beijing, China) scanning electron microscope. The bamboo was cut into small pieces with a smooth surface and dried in an oven at 60 °C. Bamboo samples were glued on the aluminum specimen frame with conductive adhesive and then sprayed with a vacuum coating instrument. The changes of bamboo surface structure and starch granules were viewed by scanning electron microscope.

2.3.5. Mechanical Properties

The mechanical properties of bamboo samples before and after high-temperature hydrothermal treatment were examined in accordance with GB/T 15780-1995 "Testing methods for physical and mechanical properties of bamboo" standard [27]. The bamboo specimens used for mechanical testing were 160 mm × 10 mm × 8 mm (longitudinal × tangential × radial) in size, repeated 12 times for 16 groups. The modulus of rupture (MOR) and modulus of elasticity (MOE) of the specimens were measured using a DNS50 (Sinotest, Jilin, China) electronic universal testing machine.

2.3.6. Dimensional Stability Measurements

The bamboo samples treated with high-temperature hydrothermal were left in a room at 20 °C and 65% RH for 4 weeks to attain equilibrium moisture content (EMC). EMC is calculated by the following formula:

$$\mathrm{EMC}(\%) = \frac{m_1 - m_0}{m_0} \times 100\% \tag{2}$$

where m_1 is the mass of the bamboo samples treated with high-temperature hydrothermal, and m_0 is the oven-dried mass of the samples.

The swelling coefficient was assessed according to the Chinese National Standard GB/T15780-1995. The coefficient of expansion is calculated by the following formula:

$$a(\%) = \frac{S_1 - S_0}{S_0} \times 100\% \tag{3}$$

where a is the swelling coefficient (radial or tangential), S_0 is the sample size before treatment, and S_1 is the sample size after treatment.

2.3.7. Surface Color Analysis

A CM-5 colorimeter (Konica Minolta, Tokyo, Japan) was applied to characterize the surface color of the samples after high-temperature hydrothermal treatment. The color values, L*, a*, b* were recorded at three random positions per sample. The color change is defined by the following formula:

$$\Delta E = \sqrt{(\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})} \tag{4}$$

where ΔE and ΔL^* is color disparity and lightness variation respectively, Δa^* and Δb^* are the chroma differences.

3. Results and Discussion

3.1. Main Chemical Compositions and Mass Loss

In the process of high-temperature hydrothermal treatment of bamboo, the decomposition and polymerization of chemical functional groups were inevitable. The infrared spectra of untreated and high-temperature hydrothermally treated bamboo after 120 min of high-temperature heat treatment at 120 °C, 130 °C, 140 °C, 150 °C, and 160 °C, respectively, are shown in Figure 2. Specifically, the peak at 3400 cm^{-1}, 2900 cm^{-1}, and 1670 cm^{-1} was assigned to O—H stretching absorption, C—H stretching, and C=O stretching, respectively.

High-temperature hydrothermal treatment resulted in the peak intensity at 3400 cm^{-1} being slightly lower, which is attributed to the two free hydroxyl groups in the amorphous region of cellulose combined at a high temperature and the formation of hydrogen bonds by the action of water molecules. In addition, there is a reduction in the peak intensity at 2900 cm^{-1} with rising temperature, owing to the decomposition of cellulose in bamboo [28]. Degradation of hemicellulose is manifested in a decrease in the peak at 1670 cm^{-1}, which was attributed to the acetyl group on the hemicellulose polysaccharide molecule hydrolyzed under hydrothermal conditions to generate acetic acid, and this acetic acid triggered a decline in the number of carbonyl C=O band [23].

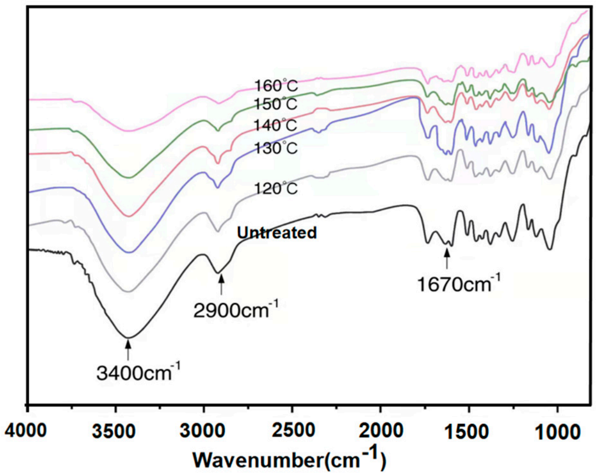

Figure 2. FTIR spectra for bamboo samples.

Figure 3 shows the influence of different temperatures of high-temperature hydrothermal treatment on cellulose, hemicellulose, and lignin content in bamboo samples. Figure 3 reveals the significant changes in the cellulose and hemicellulose contents of the bamboo samples under hydrothermal conditions at 120–150 °C. Nevertheless, when the temperature was 160 °C, the main chemical composition of bamboo changed significantly. The overall trend of cellulose and hemicellulose content dropped with increasing temperature and time, while the relative content of lignin rose [29]. The hemicellulose content of bamboo samples decreased by 71% compared with untreated bamboo samples after high-temperature hydrothermal treatment at 160 °C for 2 h, indicating that hemicellulose degradation started [30]. Hemicellulose had a lower molecular weight and more branched chain structure, which was less stable and easier to be degraded than other chemical substances in high-temperature hydrothermal treatment. At an elevated temperature of 130 °C, the cellulose content of the bamboo gradually rose compared to untreated bamboo, which was primarily due to the loss of volatile substances. Furthermore, the cellulose content obviously dropped as the temperature rose to 160 °C on account of the hydrolysis of cellulose. Under high-temperature hydrothermal treatment conditions, the cellulose decline was caused by the decomposition of acetyl groups in hemicellulose to produce acetic acid, which can catalyze the hydrolysis reaction on cellulose [31]. The lignin relative content rose as the content of cellulose and hemicellulose reduced compared to untreated bamboo due to the good thermal stability of lignin or lignin condensation and cross-linking reactions.

Influence on the mass loss of bamboo by high-temperature hydrothermal treatment is presented in Table 1. As the results indicated, the mass loss increased with the prolongation of temperature and time. From the table, it could be seen that the mass loss was not significantly affected by treating the bamboo samples in hydrothermal at 120 to 150 °C because the main chemical components in the bamboo were not completely degraded, and the mass loss at this time was primarily caused by the vaporization of overall moisture and part of the bamboo extract. It was noticed that the mass loss of bamboo timber significantly rose at a further temperature rise of 150 °C to 160 °C, which indicated that the chemical composition of the bamboo had started to degrade [32].

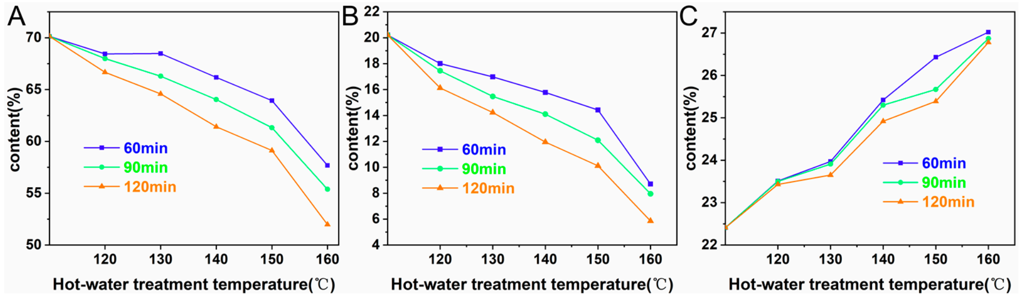

Figure 3. Influence of hydrothermal treatments on the main chemical composition of bamboo samples: (**A**) Cellulose; (**B**) Hemicellulose; (**C**) Lignin.

Table 1. Mass loss of bamboo samples.

	Mass Loss (%)		
	60 min	**90 min**	**120 min**
120 °C	4.43	5.01	5.46
130 °C	5.61	6.27	6.97
140 °C	6.15	8.27	9.34
150 °C	7.40	8.81	10.27
160 °C	10.83	16.03	23.51

The possible influence mechanism of high-temperature hydrothermal treatment at 160 °C duration for 120 min on the bamboo chemical composition is shown in Figure 4. In the treated bamboo samples, the cellulose and hemicellulose content was decreased and the relative content of lignin was increased compared with untreated bamboo.

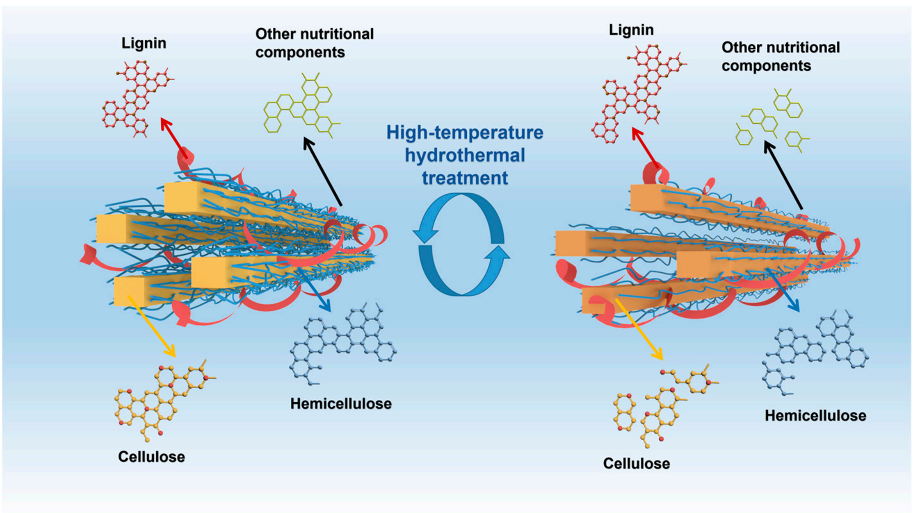

Figure 4. Schematic diagram of chemical composition changes of moso bamboo under high-temperature hydrothermal treatment.

3.2. Microstructure

The microstructures of untreated and hydrothermally treated bamboo are displayed in Figure 5. For untreated samples, the cross-sectional images showed complete vascular bundles and parenchyma cells (Figure 5A). Vascular bundles were surrounded by parenchyma cells, and starch granules were displayed in the cell lumen. The microstructure of bamboo after high-temperature hydrothermal treatment at 140 °C for 120 min is shown in Figure 5B. The parenchymal cell wall was delaminated and the parenchymal cells did not show obvious deformation and distortion. In addition, starch granules were not seen in the cell cavity. Figure 5C shows the microstructure of the bamboo cross-section after high-temperature hydrothermal treatment at 160 °C for 120 min. The thin-walled cells of bamboo were deformed and the fiber walls were delaminated; additionally, the starch granules were also not seen in the cell lumen, which indicated that the bamboo components were degraded [33,34].

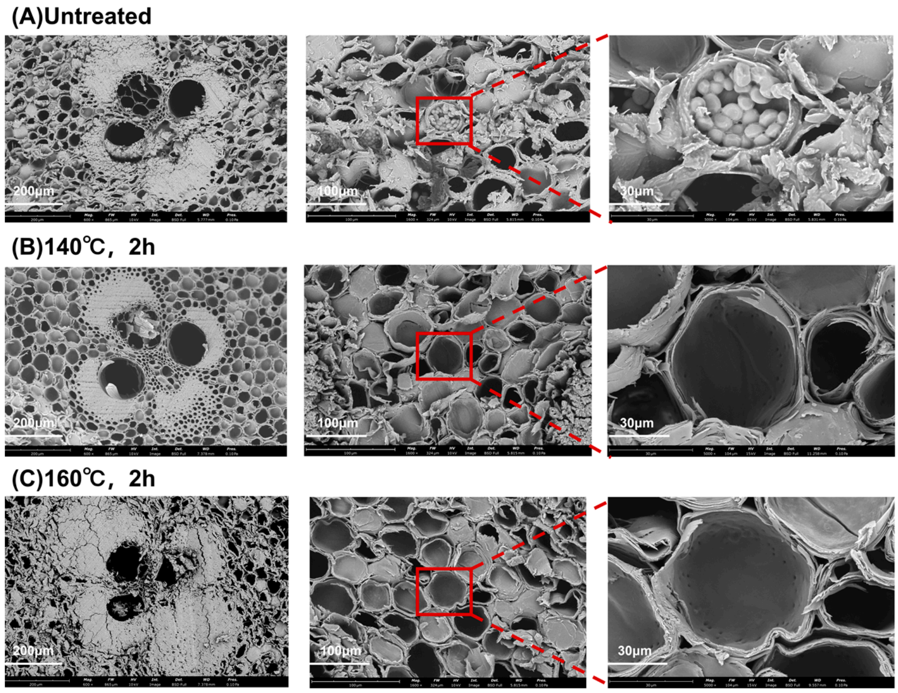

Figure 5. SEM images of bamboo samples hydrothermal treated at different temperatures for 2 h.

3.3. Mechanical Properties

Figure 6A indicates the variation of modulus of rupture (MOR) of bamboo samples before and after high-temperature hydrothermal treatment. The MOR of bamboo showed a declining trend with the increase in temperature and extension of time. In comparison to the untreated samples, when the heat treatment temperature was raised to 120 °C, 130 °C, 140 °C, 150 °C, and 160 °C for 120 min duration, the decreases of MOR were 15.60%, 17.77%, 24.32%, 34.66%, and 47.11%, respectively. According to previous studies, cellulose and hemicellulose in bamboo began to gradually undergo thermal decomposition under heat treatment conditions and caused some mass loss in the bamboo samples, further leading to a decline in the MOR of bamboo [35]. As the temperature and time prolonged, the hemicellulose degradation was more intense, which led to weakened adhesion between the cellulose and lignin, which in turn affected the cell wall strength. Consequently, after high-temperature hydrothermal treatment, the major chemical composition of the bamboo material was markedly changed, thus leading to the dramatic change of the MOR.

Figure 6B revealed the influence of high-temperature hydrothermal treatment on the chordwise modulus of elasticity (MOE) of the bamboo samples. The MOE was reduced by 16.14% after treatment at 160 °C for 120 min compared to untreated bamboo. It was found that the changes in MOE of bamboo were related to the absorption of water and the thermal decomposition of the chemical composition of bamboo in heat treatment [20]. Absorption of water played a crucial role when the heat treatment temperature was up to 140 °C, and raised the MOE of bamboo to a certain extent. Subsequently, the pyrolysis of cellulose and hemicellulose intensified, leading to a lower degree of cellulose polymerization and disruption of hydrogen bonds, and the MOE of bamboo was noticed to gradually reduce [36,37].

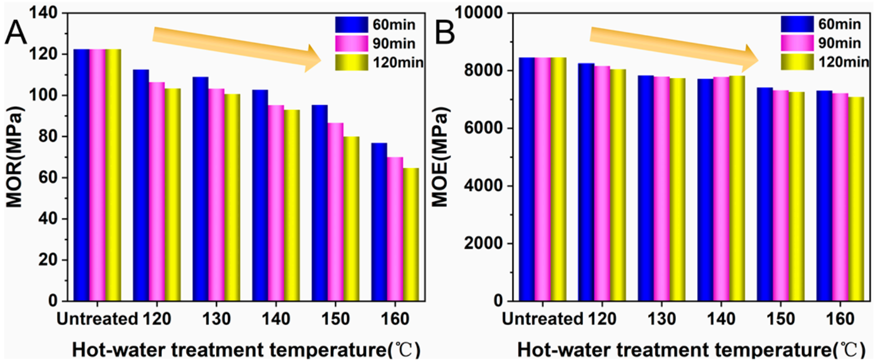

Figure 6. (**A**) Influence of different hydrothermal treatments on MOR of samples; (**B**) Influence of different hydrothermal treatments on MOE of samples.

3.4. Dimensional Stability

Figure 7A illustrates the variation of the equilibrium moisture content (EMC) of bamboo samples before and after the high-temperature hydrothermal treatment. It was observed that the EMC of bamboo was not significantly affected in hydrothermal treatment at temperatures below 140 °C. The EMC content of the bamboo samples treated with 160 °C for 120 min was 30.75% less than the untreated samples. It is well known that hemicellulose has a strong hygroscopic ability as one of the three main chemical components of bamboo, which can be thermally decomposed at 160 °C [20]. Therefore, the thermal degradation of hemicellulose was responsible for the reduction in EMC of bamboo. Additionally, as the temperature rises, the migration rate of bound water on the surface of bamboo improved, resulting in a reduction in free hydroxyl groups present in cellulose and hemicellulose, resulting in reduced moisture absorption and EMC content of bamboo [38].

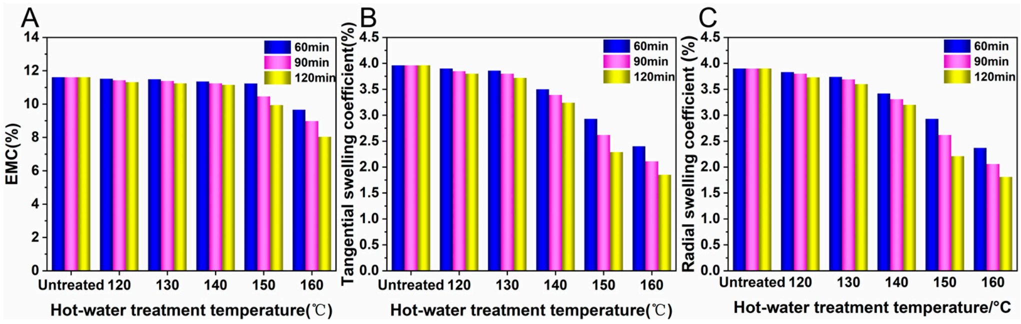

Figure 7. (**A**) Influence of different hydrothermal treatments on EMC; (**B**) Influence of different hydrothermal treatments on tangential swelling coefficient; (**C**) Influence of different hydrothermal treatments on radial swelling coefficient.

The dimensional stability of bamboo is related to the quality and service life of bamboo products, and the swelling coefficient in tangential and radial directions is the key index to evaluate the dimensional stability. Figure 7B,C show the variation of the swelling coefficient in tangential and radial directions of the bamboo samples before and after the treatment. Compared with untreated bamboo, the tangential swelling coefficients were decreased by 4.04%, 6.06%, 18.18%, 42.17%, and 53.28%, respectively, and the radial expansion coefficients were decreased by 4.36%, 7.69%, 17.95%, 43.33%, and 53.59%, respectively, after 120 °C, 130 °C, 140 °C, 150 °C, and 160 °C treatment for 120 min. This implied that the samples obtained the optimum dimensional stability by treatment at 160 °C for 120 min. This was attributed to the decrease in hemicellulose and cellulose in bamboo after high-temperature treatment, resulting in the reduction in hydrophilic groups and free hydroxyl groups, so as to lower hygroscopic properties and higher dimensional stability of the bamboo [39]. Meanwhile, due to the structure and anisotropy of bamboo itself, the variation of the size and volume of the bamboo cell wall could affect the dimensional stability. When bamboo absorbs or loses water, the water layer becomes thinner between the adjacent microfibrils and microcrystals in the amorphous zone of the cell wall and vice versa, which leads to variations in the size and volume of the cell wall, and therefore affects the difference in appearance, size, and volume of the whole bamboo.

3.5. Surface Color

Figure 8 demonstrates the impact of high-temperature hydrothermal treatment on the surface color of bamboo. The corresponding change values of L* were reduced by 18.02%, 24.67%, 29.63%, 40.08%, and 43.59% at 120 °C, 130 °C, 140 °C, 150 °C, and 160 °C duration of 120 min, respectively, compared to the untreated bamboo. This indicated that the L* value of the bamboo gradually reduced upon high-temperature hydrothermal treatment, signifying that the color of the bamboo darkened upon high-temperature hydrothermal treatment. Furthermore, the green-red (a*) and blue-yellow (b*) color coordinates also varied under different conditions. It could be observed that the value of a* in the bamboo samples was reduced by 40.68% after heat treatment at 160 °C for 120 min compared to untreated samples, showing that this bamboo had an obvious tendency of turning greenish. Based on the measured value of b* of the bamboo samples, the b* value decreased by 61.41% after the 160 °C duration of 120 min in comparison with the untreated bamboo, meaning that the surface color of the treated bamboo had a tendency to turn blue. Notably, the color change b* of the bamboo samples was more than the a* value after 120 min treatment at 160 °C, suggesting that the effect of yellowing on bamboo samples was greater with high-temperature hydrothermal treatment.

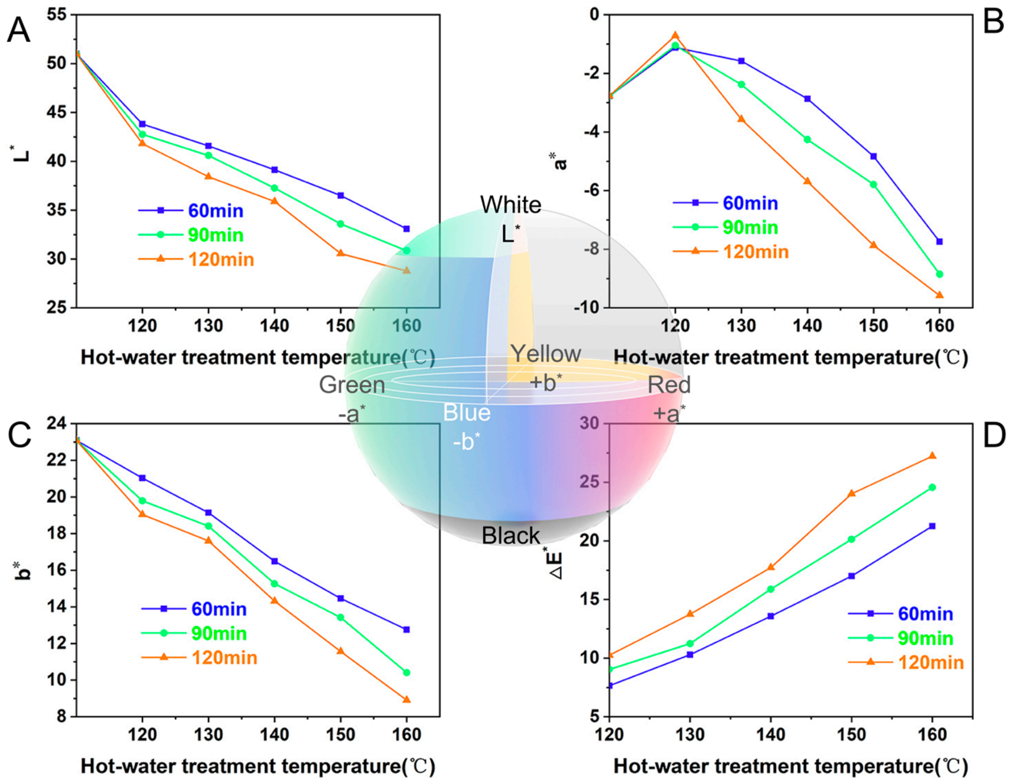

Figure 8. (**A**) Influence of different hydrothermal treatments on L*; (**B**) Effect of different hydrothermal treatments on a*; (**C**) Effect of different hydrothermal treatments on b*; (**D**) Effect of different hydrothermal treatment on ΔE*.

It is seen from Figure 8D that the total color difference (ΔE*) of the bamboo samples increases with rising temperature and time, and ΔE* is a comprehensive indicator of the color variation of bamboo before and after high-temperature hydrothermal treatment. To some extent, the color change of bamboo was attributed to the thermal decomposition of hemicellulose, which led to the decomposition of oxygen-containing groups, resulting in the loss of carbon content [40]. Moreover, during the heat treatment, lignin underwent side chain condensation reactions that produced new β-β, β-5 condensed structures, and conjugated double bonds, causing a shift in the UV absorption band and further darkening the color of the bamboo [41]. At the same time, quinone and other degradation products from high-temperature hydrothermal treatment also caused color changes in the bamboo.

4. Conclusions

In this study, bamboo was modified by high-temperature hydrothermal treatment. The effects of various temperatures (120 °C–160 °C) for diverse durations (60–120 min) on the chemical composition, mechanical properties, dimensional stability, and surface color of bamboo were systematically studied. The SEM images showed that the parenchyma cells of the bamboo were deformed and distorted after high-temperature hydrothermal treatment, and the parenchymal cell lumen without starch granules. As the high-temperature hydrothermal treatment continued, the content of cellulose and hemicellulose in bamboo reduced and the relative content of lignin rose. In addition, the mass loss rate of bamboo also rose.

At the same time, both MOR and MOE values of bamboo were reduced after high-temperature hydrothermal treatment. The MOR and MOE of bamboo samples were reduced by 47.11% and 16.14%, respectively, after treatment at 160 °C for 120 min compared to untreated bamboo. The swelling coefficient of the bamboo was lowered upon high-temperature hydrothermal treatment. The tangential and radial swelling coefficients of bamboo were reduced by 53.28% and 53.59%, respectively, at 160 °C for 120 min compared to untreated bamboo, indicating that the dimensional stability was optimal at this time. Finally, treatment temperature and time also had remarkable effects on the bamboo surface color. That is to say, the surface color of the bamboo gradually deepens as the hydrothermal temperature rises.

Author Contributions: X.L. (Xiaoran Li) and S.N. designed the experiments; H.P. performed the field observation and recording; X.L. (Xiaoran Li) and S.N. performed high-temperature hydrothermal treatment experiments; H.P. assisted in equipment maintenance and sample collection; X.L. (Xiaoran Li) and X.L. (Xiaorong Liu) are major contributors in writing the manuscript; Y.L. provided experimental instruments; All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the National Natural Science Foundation of China (Nos. 31971740), Jiangxi Forestry Bureau Forestry Science and Technology Innovation Special Project (No. 202134), Nanping Science and Technology Plan Project (N2020Z001), Zhejiang Provincial Key R&D Program Project (2019C02037).

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

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