

EFFECT OF HEAT TREATMENT ON BAMBOO FIBER MORPHOLOGY CRYSTALLINITY AND MECHANICAL PROPERTIES

HONG YUN, KAIFU LI, DENG YUN TU, CHUANSHUANG HU
CHINA AGRICULTURAL UNIVERSITY, COLLEGE OF MATERIAL AND ENERGY, DEPARTMENT
OF WOOD SCIENCE AND ENGINEERING
SOUTH, GUANGZHOU CITY
CHINA

(RECEIVED MARCH 2015)

ABSTRACT

This study aimed to investigate the fiber morphology, crystalline structure and mechanical properties of heat-treated bamboo. Moso bamboo was treated by superheated steam at 120, 140, 160, and 180°C. Fiber morphology and crystalline structure of heat-treated Moso bamboo were researched by transmission electron microscope and X-ray diffraction. The mechanical properties of heat-treated bamboo were tested in the paper. The result showed that the relative crystallinity and length-width ratio of heat-treated bamboo were increased with increased temperature. The elastic modulus of bamboo was increased as the temperature rose from 120 to 140°C. and then decreased with higher temperature, which was associated with the increase of the crystallinity.

KEYWORDS: Heat treatment, bamboo, length-width ratio, crystallinity, mechanical properties.

INTRODUCTION

Bamboo is mainly planted in Southeast Asia, such as China, Japan, India and other countries (Kleinhenz and Midmore 2001). Bamboo is a renewable resource, and possesses many superior characteristics, such as rapid growth rate, wide application range, short operating cycle, and strong regeneration ability (Embaye et al. 2005; Fu 2003).

High-temperature heat-treatment is a common method of bamboo processing and utilization. Draw lessons from heat treatment of wood, heat treatment of bamboo owns superior characteristics (Kamperidou et al. 2014; Korkut and Budakci 2010). Bamboo is treated at high temperature and humidity conditions by controlling environment parameters. Researcher did a series of studies on the heat treatment technological parameter, method and characteristics

of heat-treated bamboo and so on (Hu 2012), but the research and utilization of bamboo heat treatment is still in primary stage. The previous studies indicate that heat-treated bamboo possesses excellent characteristics, such as fine dimensional stability, fine corrosion prevention, and beautiful colors. However, the mechanical properties of heat-treated bamboo decreased obviously (Bao et al. 2009; Bao 2009; Cheng et al. 2013; Wahab et al. 2005; Ochi et al. 2002). Application of these research results would add value to bamboo industry in glulam furniture and flooring. However, the microscopic and mechanisms study of heat-treated bamboo is seldom reported in the literature.

This paper researched the Moso bamboo, which is an important forest resource and economic crop in south China. The aim of this paper was to analyze the influence of heat treatment on the crystalline structure and the micro fiber morphology of the Moso bamboo. Meanwhile, the bending strength and elastic modulus of heat-treated bamboo were tested. The modified mechanization of the heat treated bamboo would be revealed in this paper, providing the theoretical basis for the further development of bamboo heat treatment.

MATERIAL AND METHODS

Materials

Fifty four-years-old *Phyllostachys edulis* samples without mildew, discoloration and other defects were obtained from Yi Yang, Taojiang region of Hunan province. Each species, with dimensions of 1000 (L) × 20 (T) × 6 (R) mm, was randomly selected from the central part of the third section of the base of the bamboo. The moisture content of samples was regulated to 15 %.

Methods

Bamboo heat-treatment

The bamboo samples were dried to 6 % moisture content in the drying chamber, and then heat-treated in a normal pressurized superheated steam kiln heat treatment equipment (capacity: 30 m³, maximum temperature: 220°C) at four temperature levels: 120,140,160, and 180°C for 2 h. The heater was turned off at the end of soaking time, but the fan and the humidifier remained on until the dry-bulb temperature dropped below 110°C. After that the oven was sealed and cooled to room temperature.

XRD analysis of heat-treated bamboo

Samples and control specimens were crushed separately to 80 meshes, and then were dried in drying oven at 100±3°C. The dried samples were pressed into thin slices to determine their X-ray diffraction characteristics by a XRD-6000 diffractometer (Shimadzu Corp. Japan). The relative crystallinity was calculated followed a previous study (Yang et al. 2010). The relative crystallinity was calculated using Eq. 1,

$$C_r I = \frac{I_{002} - I_{am}}{I_{002}} \times 100\% \quad (1)$$

where: $C_r I$ - the relative crystallinity (%),
 I_{002} - the maximum intensity of the (002) lattice diffraction angle (arbitrary units),
 I_{am} - the scattering intensity of the non-crystalline background diffraction. When the 2θ angle is close to 18°, I_{002} and I_{am} have the same units.

Fiber characteristics analysis of heat-treated bamboo

Heat-treated samples (at different temperature levels: 120, 140, 160, and 180°C) were separated into bamboo fibers using potassium chlorate and nitrate (Chen et al. 2011). Fifty complete bamboo fibers were selected randomly from the samples and the length and width of each fiber were measured by a digital biological microscope system (Leica Corp. Germany).



Fig.1: The measurement of length and width of bamboo.

Mechanical properties test of heat-treated bamboo

The elastic modulus and bending strength of heat-treated bamboo were examined using a universal mechanical testing machine (Shimadzu Corp. Japan), in accordance with the GB/T 15780 (1995) standards.

RESULTS AND DISCUSSION

Crystalline characteristics of heat-treated bamboo

The XRD characteristics of heat-treated bamboo are presented in Tab. 1. The results show that the relative crystallinity of bamboo fibers increased with increasing temperature. The relative crystallinity respectively increased 3.35, 4.50, 5.30 and 5.67 % (at 120, 140, 160, 180°C) than untreated bamboo. The increase in crystallinity was caused by the hydroxyls of the bamboo amorphous region lost water through condensation and produce ether bond at certain temperatures (Sun et al. 2013). Therefore, the arrangement of the micro fibrils was more orderly and closer to the crystalline region, resulting in increase of relative crystallinity. However, there were also studies showed that the increasing trend of relative crystallinity declined.

Tab. 1: Crystalline characteristics of heat-treated bamboo.

Temperature(°C)	002 Crystal plane Angle (°)	I_{002}	I_{am}	Relative crystallinity (%)
120	22.1	4158	2426	41.65
140	21.9	4930	2818	42.80
160	22.3	4122	2324	43.60
180	22.0	5148	2884	43.97
Untreated Bamboo	22.0	4226	2606	38.30

Fiber characteristics of heat-treated bamboo

Tab. 2 shows the results of the length and width of the heat-treated bamboo fibers.

Tab. 2: Fiber characteristics of heat-treated bamboo.

Temperature(°C)	Length of fibers (μm)	Width of fibers (μm)	Length-width ratio of fibers (%)	Replicates	STDEV
120	2187.277	15.698	169.19	50	31.7
140	2043.378	12.150	175.16	50	21.9
160	2256.235	12.906	174.82	50	36.8
180	2321.377	17.028	176.89	50	25.3
Untreated Bamboo	1683.744	14.544	129.25	50	35.5

Tab. 3: Analysis of variance.

Source	SS	df	MS	F	Sig.
Temperature	258493.30	4	64623.32	2.26	*
Error	7002718.56	245	28582.52		
Sum	7261211.85	249			

$F_{0.10}(4, 245)=1.94$

The results showed that the length-width ratio of untreated bamboo fibers was 129.25, but it increased obviously through the heat treatment at length-width ratio were increased by 30.9, 35.5, 35.3 and 36.9 %. The temperature had obvious effect on the length-width ratio of fibers as showed in Tab. 3. The reduced cavity material and water caused shrinkage in the wood fibers during the heat treatment. Moreover, the hydroxyls of the bamboo molecular chain lost water through condensation at certain temperatures. Therefore, the arrangement of the micro fibrils was more orderly in treated bamboo than in untreated bamboo (Sun and Li 2010). According to the bamboo fiber multilayer structure model raised by Parameswaran and Liese, the micro fibrils was arranged in axial and transverse alternating direction in the structure of the secondary cell wall, as shown in Fig. 2 (Pei 2012). Meanwhile, axial micro fibrils accounted for greater proportion in the multilayer structure of bamboo micro fibrils, therefore the rearrangement process makes the axial size of fibers increase. According to the Tab. 1, the crystallinity of the bamboo increased through the heat treatment, and there is a trend of increase with the temperature. In the future studies, we will intensive study the relativity of the crystallinity and the fiber sizes of the heat-treated bamboo.

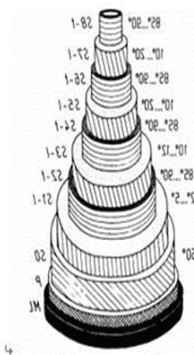


Fig. 2: Bamboo fiber multilayer structure model (Pei 2012).

Mechanical properties of heat-treated bamboo

Heat treatment changed the crystalline structure and length-width ratio of the bamboo fiber. Meanwhile, the mechanical properties of bamboo had the corresponding changes.

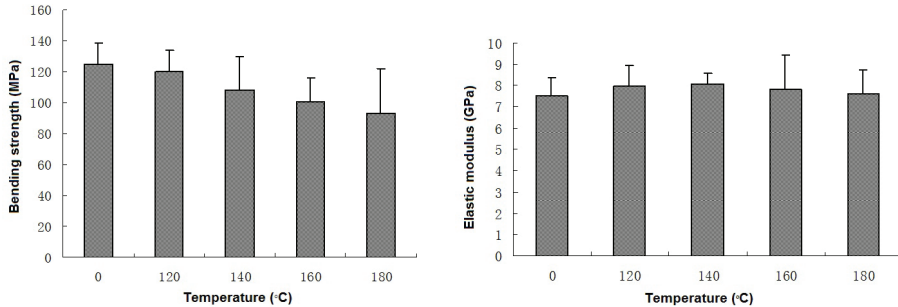


Fig. 3: Effect of temperature on bending strength. Fig. 4: Effect of temperature on elastic modulus.

Fig. 3 show that the bending strength declined after the heat treatment. The bending strength decreased by 4.16, 15.74, 23.76 and 34.41 % compared with the untreated bamboo. Because partial hemicellulose (play adhesive role) decomposed into acetic acid when the temperature reached 120°C, and the acetic acid decomposed (Li 2002) Fig. 4 show that the average of elastic modulus was increased compare to the untreated bamboo. The elastic modulus increased by 5.72, 7.45, 3.72 and 0.93 % compared with the untreated bamboo. New chemical bond was formed in the process of heat treatment, and which has greater energy than hydrogen bond (Windeisen et al. 2009). Therefore the inflexibility increased in the range of elasticity of the bamboo. This result is consistent with previous research results (Zhang et al. 2013; Lin et al. 2012). The elastic modulus were 7.96 and 8.1 GPa when the heat treatment temperature were 120 and 140°C respectively. However, the elastic modulus decreased as the temperature rose form 160 to 180°C. There are some relationship between the increase of crystallinity and elastic modulus. Overall, the crystallinity of bamboo fiber showed an increase tendency through heat treatment, as shown in Tab. 1. In this process, partial amorphous body converted into crystal and made the the rigidity of materials increased slightly. But at the same time, the elastic modulus is also influenced by other factors such as extraction of volatile, semi cellulose degradation and so on. The degree of material volatilization and degradation is different temperature. Therefore appear the tendency first increased and then decreased.

CONCLUSIONS

1. According to XRD diffraction spectrum, heat treatment had no clear effect on the position of the cellulose crystalline region. However, the crystallinity showed a gradual increase with heat- treated temperature increasing.
2. The length-width ratio of bamboo fibers had increased obviously after heat treatment. This result will improve the usability of the bamboo fibers.
3. The elastic modulus of the bamboo increased as the heat-treated temperature ranging form 120 to 180°C. However, the bending strength decreased because of the volatilization of the extractives in bamboo.

ACKNOWLEDGMENTS

This study was supported by the Department of Education Nursery Project of Guangdong Province (Grant No. 2013LYM_00030003) and the Outstanding Young Teacher Specific Program of Guangdong Provincial Colleges and Universities (No. Yq 2013029), the Department of Education of Guangdong Province.

REFERENCES

1. Bao, Y.J., Jiang, X.S., Cheng, D.L., Fu, S.W., 2009: The effects of heat treatment on physical-mechanical properties of bamboo. *Journal of Bamboo Research* 28(4): 50-53.
2. Bao, Y.J., 2009: Research on the main chemical components, physical and mechanical properties of bamboo after thermal treatment. Nanjing Forestry University, 50 pp.
3. Cheng, D.L., Jiang, S.X., Zhang, Q.S., 2013: Effect of hydrothermal treatment with different aqueous solutions on the mold resistance of Moso bamboo with chemical and FTIR analysis. *Bioresources* 8(1): 371-382.
4. Chen, H., Wang, G., Cheng, H.T., 2011: Properties of single bamboo fibers isolated by different chemical methods. *Wood and Fiber Science* 43(2): 111-120.
5. Embaye, K., Weih, M., Ledin, S., Christersson, L., 2005: Biomass and nutrient distribution in a highland bamboo forest in southwest Ethiopia: Implications for management, Forest ecology and management 204(2-3): 159-169, DOI: 10.1016/j.foreco.2004.07.074.
6. Fu, J.H., 2003: The types and application of bamboo charcoal. *World Bamboo and Rattan* 1(03): 19-20.
7. GB/T 15780 - 1995: Testing methods for physical and mechanical properties of bamboos.
8. Hu, G.Y., 2012: Study on dynamic viscoelasticities of heat-treated bamboo. Zhejiang, A & F University.
9. Kamperidou, V., Barboutis, I., Vasileiou, V., 2014: Influence of thermal treatment on mechanical strength of scots pine (*Pinus sylvestris* L.) wood. *Wood Research* 59(2): 373-378.
10. Kleinhenz, V., Midmore, D.J., 2001: Aspects of bamboo agronomy. *Advances in Agronomy* 74: 99-153.
11. Korkut, S., Budakci, M., 2010: The effects of high-temperature heat-treatment on physical properties and surface roughness of rowan (*Sorbus aucuparia* L.) wood. *Wood Research* 55(1): 67-78.
12. Li, J., 2002: *Wood Science*. Second edition, Higher Education Press, Beijing.
13. Lin, Y., Shen, Y.C., Yu, L., Liu, H.Z., Li, Y.J., 2012: Study of physical-mechanical properties of bamboos through high temperature heat treatment. *Forestry Machinery & Woodworking Equipment* 40(8): 22-24.
14. Ochi, S., Takagi, H., Niki, R., 2002: Mechanical properties of heat-treated natural fibers. *Journal of the Society of Materials Science, Japan*, 51(10): 1164-11684.
15. Pei, J.C., 2012: *Lignocellulosic chemistry*. Fourth edition, China Light Industry Press, Beijing.
16. Sun, R.H., Li, X.J., Liu, Y.Y., Hou, R.G., Qiao, J.Z., 2013: Effects of high temperature heat treatment on FTIR and XRD characteristics of bamboo bundles. *Journal of Central South University of Forestry & Technology* 33(2): 97-100.
17. Sun, W.L., Li, J., 2010: Analysis and characterization of dimensional stability and crystallinity of heat-treated *Larix* spp.. *Scientia Silvae Sinicae* 46(12): 114-118.

18. Wahab,R., Mohamad,A., Samsi, H.W., Sulaiman, O., 2005: Effect of heat treatment using palm oil on properties and durability of Semantan bamboo. Journal of Bamboo and Rattan 4(3): 211-220, DOI: 10.1163/156915905774310034.
19. Windeisen, E., Bachle, H., Zimmer, B., Wegener, G., 2009: Relations between chemical changes and mechanical properties of thermally treated wood. Holzforschung 63(6): 773-778, DOI: 10.1515/HF.2009.084.
20. Yang, S.M., Jiang, Z.H., Ren, Q.H., Fei, B.H., 2010: Determination of cellulose crystallinity of bamboo culms with X-ray diffraction spectrum. Journal of Northeast Forestry University 38(8): 75-77.
21. Zhang, Y.M., Yu, Y.L., Yu, W.J., 2013: Effect of thermal treatment on the physical and mechanical properties of *Phyllostachys pubescens* bamboo. European Journal of Wood and Wood Products 71(1): 61-67, DOI: 10.1007/s00107-012-0643-6.

HONG YUN, KAIFU LI,* DENG YUN TU, CHUANSHUANG HU
CHINA AGRICULTURAL UNIVERSITY
COLLEGE OF MATERIAL AND ENERGY
DEPARTMENT OF WOOD SCIENCE AND ENGINEERING
SOUTH, GUANGZHOU CITY
CHINA
Corresponding author: kfli@scau.edu.cn

