

Comparative Research between Rhizome and Culm of *Phyllostachys pubescens* Based on Thermal Analysis

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A systematic study was conducted on *P. pubescens* by analyzing thermal characteristics of its rhizome and culm (1.5 m) at different ages, using various analyses such as X-ray diffraction (XRD), differential scanning calorimetry (DSC), and thermogravimetry (TG). For both the rhizome and culm of *P. pubescens*, the relative crystallinity (Cr) of cellulose increased and then appreciably decreased with increasing age, but the rhizome contained about 10% less Cr than culm. However, the glass transition temperature (T_g) of culm (268.8 to 273.8 °C) was higher than in the rhizome (213.9 to 219.4 °C). The extrapolated initial decomposition temperature (T_e) of 1-, 3-, and 5-year-old rhizome were 263.7 °C, 266.1 °C, and 263.9 °C, respectively; the T_e of culms of the same age were 289.7 °C, 282.8 °C, and 283.4 °C, respectively. Variance analysis showed no significant differences in the T_e of either rhizome or culm of different ages. Taking the chemical characteristics into consideration, it was concluded that the rhizome has a stable composition and good heat resistance.

Keywords: Rhizome; Culm; Relative crystallinity; Glass transition temperature; Extrapolation initial decomposition temperature

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INTRODUCTION

As a commercial bamboo species in China, *Phyllostachys pubescens* is cultivated over a large region, mainly in subtropical areas such as the Qinling Mountains and between the Hanjiang and Yangtze River Basins (Jiang 2007; Geng and Wang 2004). The underground portion of *P. pubescens* is composed of rhizome, root, and bud, while the aboveground part consists of culm, branch, and leaf. Bamboo rhizome, an underground culm, is an important component of *P. pubescens* with a huge potential for exploitation and utilization. Its high strength and high tenacity attract tremendous attention when the issue of environmental protection is addressed. The rhizome is traditionally used to make tobacco culms, bamboo-root carvings, and other items (Xiang 2010; Yang *et al.* 2012; Fei *et al.* 2014). There has been systematic research on the anatomical index (Liu *et al.* 2014) and chemical features (Jain *et al.* 1992; Xu *et al.* 2007; Xu *et al.* 2014) of the *P. pubescens* rhizome, but its thermal properties have not been studied.

This study used thermal analysis methods, such as thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) (Liu 2000; Jiang *et al.* 2005; Jiang *et al.* 2008; Xu *et al.* 2014), to investigate the thermal properties (relative crystallinity of cellulose, glass-transition temperature, and extrapolated initial decomposition temperature) of the rhizome and culm (1.5 m from the root) of 1-, 3-, and 5-year-old *P. pubescens*. Combined with the rhizome anatomical properties and chemical composition,

and the study will discuss whether it can be used as heat resistant materials or pulp and paper materials. This data will form a basis for a theoretical research system, provide a basis for the utilization of whole bamboo, and increase the value derived from the rhizome.

EXPERIMENTAL

Materials

Phyllostachys pubescens samples were collected on September 27, 2012 from a northern slope (gradient 30° to 40°), where the east longitude and northern latitude were 118° 01' 22" and 30° 12' 55", respectively, in Tangjiacun village, (Taiping) Jiacunzhen town, Huangshan City, Anhui Province. The bamboos were II operation level with healthy growth and development status but without the topping treatment (Liu and Zeng 2010). The average heights of culm and clear bole were 16.5 m and 7.1 m, respectively, and the average diameter at eyebrow (1.5 m) was 10.96 cm. Bamboo age was determined based on the integrity or degree of decay of the surviving bamboo shoot shell from the mother bamboo, branchlet age marker, and culm color. Samples from 1-, 3-, and 5-year-old *P. pubescens* including rhizome parts (30 cm each) were collected with three replicates each and preserved indoors using pigsty packing in a ventilated corridor for three months before the tests.

Methods

Sampling (30 cm) was carried out on different parts of the rhizome and culm (1.5 m) of bamboo following guidelines in GB/T 15780-1995 (1996). Three samples were taken from every part (rhizome and 1.5 m of bamboo) at each age. For example, in the case of the rhizome, three segments (5 cm each) were sampled (Fig. 1). Fresh samples were dried naturally until they reached air-dried moisture content, sliced into thin, match-sized sections by an axe, and ground using a TL2020 high-speed pulverizer (Beijing DHS Life Science and Technology Co., Ltd., Beijing, China). The powder particle size was such that it passed through a 0.18-mm aperture (80 mesh) but not a 0.15-mm aperture (100 mesh).

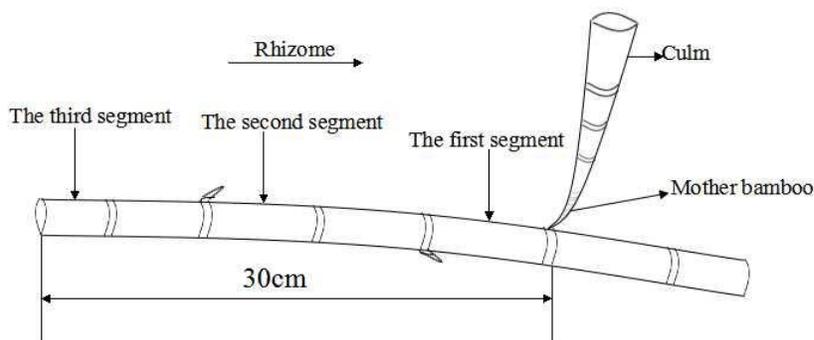


Fig. 1. Schematic diagram of sampling

The relative crystallinity of cellulose was tested using an XD-3 X-ray diffraction meter (Beijing Persee General Instrument Co. Ltd., Beijing, China). A sample of bamboo powder was placed into the sample stand, and the $\theta/2\theta$ linked scan was used. The main testing conditions were as follows: Cu target (λ), 1.54056 Å; step width, 0.01°; voltage,

36 kV; current, 20 mA; scanning speed, 10°/min; and scanning scope range, 10° to 40°. The relative crystallinity of rhizome and culm was tested according to the strength of diffraction pattern, the Segal method (Thygesen *et al.* 2005) was used to calculate whether the relative crystallinity had a maximum peak value (I_{002}) when $2\theta = 22^\circ$ in the scanning curve (Fig. 2) and whether it had a minimum peak value when $2\theta = 18^\circ$ (I_{am}) in the scanning curve. The relative crystallinity (Cr) was calculated as,

$$Cr (\%) = ((I_{002} - I_{am}) / I_{002}) \times 100 \quad (1)$$

where I_{002} is the maximum strength of the diffraction angle of crystal lattice, namely, the diffracted intensity of the crystalline region and I_{am} is the scattering strength of diffraction under an amorphous background (Yang *et al.* 2010; Park *et al.* 2010; López-Simeon *et al.* 2012; Mi *et al.* 2014).

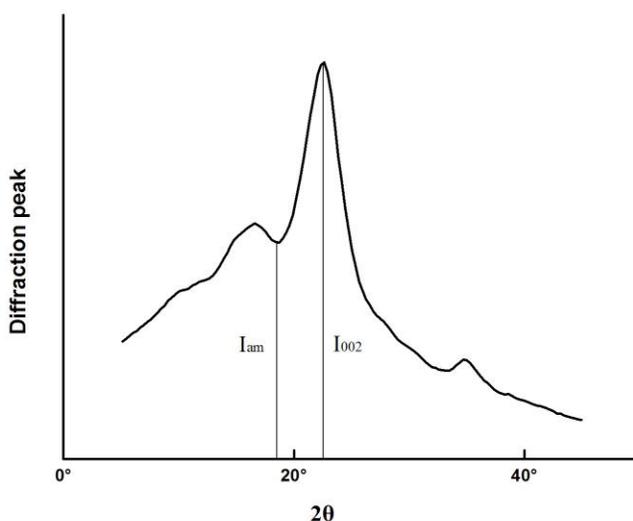


Fig. 2. Typical X-ray diffraction distribution curve of *Phyllostachys pubescens* in 2θ

The glass transition temperature (T_g) was measured and calculated using a DSC 200F3 PC differential scanning calorimeter (Netzsch Group, Bavaria, Germany). The testing conditions were as follows: temperature, 30 °C to 300 °C; heating rate, 10 K/min; atmosphere, air; sample mass, 3 mg to 5 mg (weighed to an accuracy of 0.0001 g). The instrument automatically collected data, which was saved in txt format. Origin software version 8.0 (Originlab Corp., Northampton, USA) was used to generate a DSC curve (Ren *et al.* 2014). The extrapolated initial decomposition temperature (T_e) was measured using a TGA 209F3 PC thermogravimetric analyser (Netzsch Group, Bavaria, Germany). The testing conditions were as follows: temperature, 35 °C to 800 °C with three sections including warming (35 °C to 100 °C), constant temperature (100 °C, 10 min), and warming (100 °C to 800 °C); heating rate, 10 K/min; Al₂O₃ crucible; N₂ shielding gas and sweep gas; and sample mass, 10 mg or less (weighed to an accuracy of 0.0001 g). The instrument automatically collected data, was saved in txt format. Origin software was used to make a TGA curve (Rashmi *et al.* 2007; Li *et al.* 2010). Analysis of multiplicity is widely used for comparison between two groups of all independent multi-samples. Thereby, the thermal properties difference between different ages of bamboo was obtained easily, using analysis methods of the variance. The experimental data were analyzed with SPSS software version 21.0 (IBM, Armonk, NY, USA).

RESULTS AND DISCUSSION

Variation Analysis of the Relative Crystallinity of Cellulose in Rhizome and Culm

Relative crystallinity is an important parameter describing the super-molecular structure of cellulose, and it is closely linked to the contents of lignin, cellulose, and hemicellulose. During the generation of bamboo materials, the changing tendency of the relative crystallinity of cellulose is consistent with the contents of the three elements. They present a fluctuating difference with the increase of bamboo age (Yang *et al.* 2010; Liu *et al.* 2014).

Table 1. Descriptive Statistics of Thermal Characteristics of *P. pubescens* at Different Ages

Characterization Parameter	1-year Rhizome	3-year Rhizome	5-year Rhizome	1-year Culm	3-year Culm	5-year Culm
Cr (%)	43.8	44.3	43.7	52.3	56.7	55.2
T_g (°C)	213.9	219.4	218.3	268.8	269.9	273.8
T_e (°C)	263.7	266.1	263.9	289.7	282.8	283.4
T_5 (°C)	241.7	258.6	238.6	242.1	255.2	243.3
T_{50} (°C)	347.9	348.7	343.7	342.6	334.8	341.8
T_{max} (°C)	342.0	344.6	343.3	341.5	331.5	345.5

Note: Cr: relative crystallinity of cellulose; T_g : glass-transition temperature; T_e : extrapolated initial decomposition temperature of the high polymer; T_5 : temperature at 5% quality loss; T_{50} : temperature at 50% quality loss (the half decomposition temperature of the high polymer); T_{max} : temperature at the maximum weight loss rate

Change in Relative Crystallinity of *P. pubescens* Rhizome at Different Ages

The Segal method was used to calculate the relative crystallinity of cellulose of rhizomes at 1, 3, and 5 years of age. The results were (Table 1) 43.8%, 44.3%, and 43.7%, respectively. From the result of variance analysis (Table 2), the effect of different bamboo ages on the relative crystallinity of cellulose was not clear.

Table 2. Analysis of Variance of Thermal Characteristics between Rhizome and Culm

	Relative Crystallinity		Glass-Transition Temperature		Initial Decomposing Temperature	
	Rhizome	Culm	Rhizome	Culm	Rhizome	Culm
F	1.002	41.752	1.001	35.934	9.307	79.028
P	0.382	0.001	0.405	0.131	0.626	0.001
Number of samples	27	27	27	27	27	27

Note: The statistical significance was at the 95% confidence level.

Difference in Relative Crystallinity of Cellulose between Rhizome and Culm

The crystallinity of culms 1.5 m away from the ground after 1, 3, and 5 years were 52.3%, 56.7%, and 55.2%, respectively (Table 1). With increasing bamboo age, the

changing trend was the same as that of the rhizome, but the fluctuation was great. The variance analysis ($P = 0.001 < 0.01$) indicated that the effect of different bamboo ages on the crystallinity of cellulose was clear. The relative crystallinity of cellulose of the rhizome of the same age was approximately 10% lower than in the culm. The relative crystallinity of cellulose was positively related to the contents of cellulose and hemicellulose. They presented a fluctuating difference with the increase of bamboo age. Previous research has shown that the cellulose and hemicellulose contents of the rhizome are less than those of the culm (Yang *et al.* 2010; Mi *et al.* 2014; Xu *et al.* 2014). This result could reflect the material heterogeneity between the rhizome and the aboveground part of bamboo.

DSC of Rhizome and Culm

DSC is a technology that is used to measure the relationship of transport material and reference material power difference with temperature controlled by a procedure. The glass-transition temperature (T_g) is measured and calculated based on the DSC curve; it is an important intrinsic attribute of amorphous polymer materials. The physical properties (such as heat capacity, coefficient of thermal expansion, elastic modulus, and refractive index) of a high polymer change significantly under an insignificant change in the T_g . The change between the glassy state and the elastomeric state is called glass transition, and its corresponding transformation temperature is the T_g (Back and Salmen 1982). Generally, the thermal softening point of lignin in the dry state ranges from 130 °C to 205 °C, the thermal softening point of hemicellulose ranges from 150 °C to 220 °C, and the thermal softening point of cellulose ranges from 200 °C to 250 °C. Faix *et al.* (1988) assumed that lignin structure changes at 47 °C, but the quality loss changes from approximately 180 °C to 200 °C (Back and Salmen 1982; Faix *et al.* 1988).

Difference in Glass-transition Temperature of Rhizomes of Different Ages

Table 1 and Fig. 3 show that the T_g of the rhizome ranged from 268.8 °C to 273.8 °C. The glass-transition temperature increased with increasing bamboo age. The result of variance analysis ($P = 0.405 > 0.05$) indicated that bamboo age has a slight influence on the T_g of the rhizome.

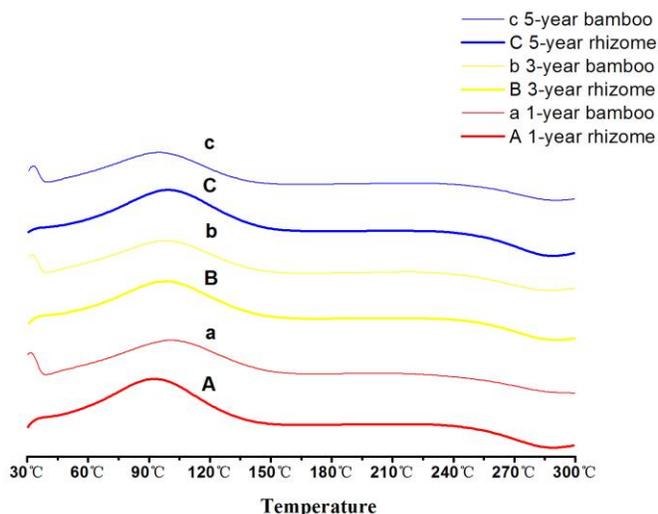


Fig. 3. Plots in DSC between rhizome and culm of *P. pubescens* at different ages

Difference in Glass-Transition Temperature between Rhizome and Culm

Table 1 and Fig. 3 indicate that the glass-transition temperature of the culm ranged from 213.9 °C to 219.4 °C, and it first increased and then slightly decreased with increasing bamboo age. The result of variance analysis ($P = 0.131 > 0.05$) showed that bamboo age had a minimal influence on the T_g of the culm. However, the T_g of the rhizome was approximately 50 °C higher than that of the culm. Studies have shown that the T_g has a positive correlation with Cr (Leigh 1995). During the maturation of bamboo, the constant thickening of the cell wall inevitably led to the accumulation of basic cell wall substance. Lignification occurred with the thickening of the cell wall; namely, lignin, cellulose, and hemicellulose simultaneously deposited with the same increased quality percentage. The cellulose content expressed by quality percentage did not change obviously. The cellulose content of the rhizome was less than that of the culm (Yang *et al.* 2012); thus, the rhizome has better heat-resistant quality.

TGA of Rhizome and Culm

The thermal stability of a high polymer refers to its ability to maintain its chemical structure or keep stability under high temperature. Over a certain temperature, the high polymer undergoes thermal decomposition. The backbone of the macromolecule breaks, leading to reduced molecular weight and changes in chemical structure, physical properties, and mechanical properties. TGA is an important way to examine the heat stability of polymers and to test the temperature when the sample weight decreases because of the generation of volatile products. The extrapolated initial decomposition temperature of the high polymer is the crossing point between the tangent and the baseline in the reducing segment of the TGA curve. Given its best repeatability, the temperature at this point is used to express the heat stability of materials. Research has shown (Lee and Via 2010) that the cumulative weight loss of a pure bamboo powder sample at a final temperature of 700 °C was mainly divided into the following three stages: (1) when the sample reached approximately 260 °C from the normal temperature, the absorbed water of bamboo material was evaporated; (2) from 260 °C to 370 °C, the polymer, mainly hemicellulose, degraded; and (3) from 370 °C to 700 °C, the sample presented partial weight loss (mainly the decomposition of lignin), and pyrolysis was basically stable (Li *et al.* 2006).

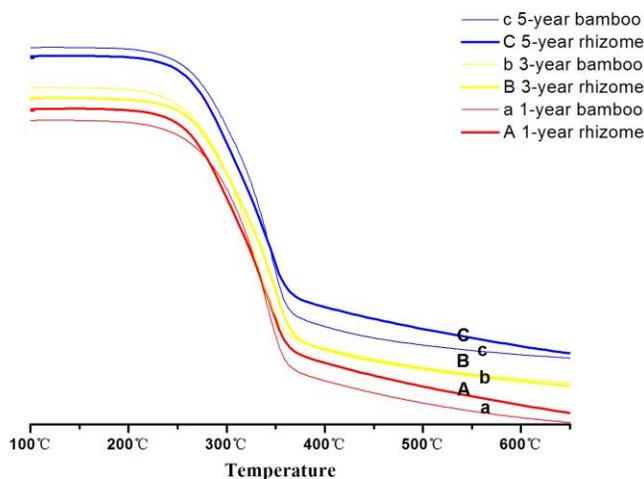


Fig. 4. Plots in TGA between rhizome and culm of *P. pubescens* at different ages

Change in Thermogravimetric Temperatures for Different Ages

Table 1 and Fig. 4 show that the extrapolated initial decomposition temperatures (T_e) of rhizomes aged 1, 3, and 5 years old were 263.7, 266.1, and 263.9 °C, respectively. When the quality loss was 5% (T_5), the temperatures were 241.7, 258.6, and 238.6 °C. At 50% quality loss (T_{50}), the temperatures were 347.9, 348.7, and 343.7 °C. The temperatures at the maximum weight loss rate were 342.0, 344.6, and 343.3 °C. In terms of T_{50} and T_{max} , no significant difference existed for different ages of the rhizome, and the temperature was approximately 340 °C. The result of variance analysis ($P = 0.626 > 0.05$) indicated that bamboo age had minimal influence on the rhizome T_e , but it was the highest for the 3-year-old rhizome. Thus, the 3-year-old rhizome had the best heat stability.

Thermogravimetric Difference between Rhizome and Culm

Table 1 and Fig. 4 show that for the culm of 1, 3, and 5 years of age, T_e was 292.3, 282.8, and 283.4 °C, respectively; T_5 was 242.1, 255.2, and 243.3 °C, respectively; T_{50} was 342.6, 334.8, and 341.8 °C, respectively; and T_{max} was 341.5, 331.5, and 345.5 °C, respectively. According to the result of variance analysis ($P = 0.001 < 0.01$), bamboo age had a great influence on T_e of the rhizome. Under the same age, T_e of the rhizome was less than that of the culm; T_e was related to material crystallinity such that high crystallinity of cellulose denotes high T_e (Shao *et al.* 2012). In terms of T_{50} and T_{max} , no great difference existed for different ages of rhizome and culm.

CONCLUSIONS

1. The Cr of 1-, 3-, and 5-year-old rhizomes was 43.8%, 44.3%, and 43.7%, respectively; the Cr of 1-, 3-, and 5-year-old culms was 52.3%, 56.7%, and 55.2%, respectively. With increasing bamboo age, the Cr of the rhizome and culm first increased and then decreased slightly, whereas that of the culm greatly fluctuated and has a significant difference. The Cr of rhizomes of the same age was approximately 10% less than that of culms. The Cr is closely linked to the contents of lignin, cellulose, and hemicellulose. According to our previous study about the chemical composition of same age rhizome and culm of bamboo, the contents of cellulose, and hemicellulose of rhizome is all lower than in the culm.
2. The T_g of 1-, 3-, and 5-year-old culms ranged from 268.8 °C to 273.8 °C, whereas the T_g of rhizomes ranged from 213.9 °C to 219.4 °C. At the same age, the T_g of rhizomes was lower than that of culms. However, at different ages, there was no clear difference in T_g between rhizome and culm.
3. For T_e , there was a difference between rhizome and culm. For 1-, 3-, and 5-year-old rhizomes, T_e was 263.7, 266.1, and 263.9 °C, respectively. The T_e of the 3-year-old rhizome was the highest. For culms, T_e was 292.3, 282.8, and 283.4 °C. At the same age, the T_e of rhizomes was less than that of culms. At different ages, there was no clear difference found in the T_e of rhizomes, whereas a great difference was observed in T_e culms.
4. Along with our previous study on the fiber characteristic and chemical differences of rhizome at different ages, the related results obtained in this paper show that the rhizome is most suitable for use as heat resistant materials. Also to some extent, the

finding provide a theoretical basis for effective utilization of rhizome as well as enhancement of its value.

ACKNOWLEDGMENTS

This study was kindly supported by the key laboratory of Wood Science and Technology of Anhui Agricultural University in China under the “The Twelfth Five-Year Alternative Plan of the National Science and Technology in Rural Areas” “Construction of the Basic Database for Bamboo and Rattan Germplasm Resources” No. 2015BAD04B03.

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Article submitted: December 29, 2015; Peer review completed: March 4, 2016; Revised version received and accepted: March 20, 2016; Published: March 30, 2016.

DOI: 10.15376/biores.11.2.4369-4378