

Investigation of the Physical and Mechanical Properties and Chemical Composition of *Bambusa rigida* Before and After Accelerated Aging

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The differences in the physical and mechanical properties and chemical composition of *Bambusa rigida* bamboo before and after accelerated aging tests were comparatively investigated. The results revealed that the aged specimens had lower physical and mechanical properties than the controls. The differences in chemical composition provided evidence that the reduction in physical and mechanical properties was related to the loss of low-molecular weight substances, such as extractives and inorganic matter, and the depolymerization of the carbohydrates cellulose and hemicellulose. Lignin caused the main resistance to the accelerated aging test because the aged specimens had relatively high Klason lignin content. Significant differences ($p < 0.05$) in surface color between the control and aged specimens were observed, and variations in bamboo properties among culm heights were also evaluated in this study. The results showed that basic density and mechanical properties for both the control and aged specimens increased with increasing culm height, while the volume shrinkage showed an inverse trend.

Keywords: *Bambusa rigida*; Accelerated aging; Chemical components; Physical and mechanical properties

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INTRODUCTION

Bamboo is one of the most important raw materials for housing, bridge construction, and a number of other uses due to its high strength-to-weight ratio, straightness, and rapid growth rate. It can be regarded as the best possible alternative to replace timber in the future. Information on physical and mechanical properties of bamboo is important in assessing its suitability for various end-products (Sattar *et al.* 1990). For example, basic density is considered to be an important factor in determining the suitability of bamboo for bamboo floor application and chemical treatment processes. Moreover, mechanical properties including shear strength, compressive strength, modulus of rupture, and modulus of elasticity are also key parameters defining the structural materials made from bamboo.

Accordingly, the physical and mechanical properties of woody materials can be changed after aging. Methods including long-term (outdoor exposure test) and short-term tests (accelerated aging test) have been used in evaluating the change in wood properties of woody materials. The accelerated aging test, compared to the outdoor exposure test, is a much more applicable method because it can be finished in a short duration and

conducted in an interior space. According to the literature (Kojima and Suzuki 2011), the bending properties of wood-based panels with accelerated aging treatment is the same as that with 5 years of outdoor exposure.

Recent achievements in techniques for using wood as a structural engineering material have stimulated certain studies focused on evaluating bamboo as a raw material for manufacturing bamboo-based structural materials. However, bamboo properties such as anatomical and chemical characteristics have been reported to differ with regard to species, age, location, and portion; such differences can significantly affect its processing procedures and the performance of end products (Hisham *et al.* 2006). Although pilot-scale evaluation of wood and bamboo as structural materials have been previously studied using accelerated aging treatment (Kajita *et al.* 1991; Kojima *et al.* 2009; Kojima and Suzuki 2010; 2011; Tomak *et al.* 2012), there are still limited studies on the evaluation of bamboo properties with respect to aging. Thus, in this present study, the properties of *Bambusa rigida* bamboo, the most commonly distributed bamboo species in Sichuan, China, was investigated before and after accelerated aging treatment. The objective of this study was to provide a primary understanding of the effect of accelerated aging on bamboo properties.

EXPERIMENTAL

Materials

Four-year-old bamboo (*Bambusa rigida*) was harvested from a bamboo plantation near Yibin, Sichuan, China. The bamboo culms were randomly selected and cut at about 10 cm above the ground. Bamboo branches and tops of the culms were removed, then transported to the laboratory and allowed to air-dry at 22 °C and 65% relative humidity for 60 days. Meanwhile, the culms were subdivided into three groups (base, middle, and top), with each stalk in the group containing 8 internodes.

Methods

Accelerated aging test

The accelerated aging test was carried out in accordance with a standard method (ASTM D 1037-89 1999). This procedure consists of 6 cycles of the following sequence: water soak for 1 h at 50 °C, steam at 95 °C for 3 h, freeze at -12 °C for 20 h, dry at 100 °C for 3 h, steam at 95 °C for 3 h, and dry at 100 °C for 18 h. After this accelerated aging procedure, the specimens were conditioned at 22 °C and 65% relative humidity for 6 weeks prior to testing.

Determination of physical properties

The studies of physical properties were carried out according to methods outlined by Kamruzzaman *et al.* (2008). A 2.5 cm section containing the rings was obtained from the middle portion of each internode at the base, middle, and top. The density (D , g·cm⁻³) was determined on the basis of oven-dry weight and green volume. Volumetric shrinkage (S , %) was estimated on air and oven dry samples. The samples for basic density and volumetric shrinkage were oven dried at 103±2 °C until constant weight was obtained. The green volume of samples was determined using the water displacement method.

Determination of mechanical properties

The studies of the mechanical properties were carried out according to methods outlined by Tran (2010) and Zhang *et al.* (2013). Mechanical properties, including shear strength (SS, MPa), compressive strength (CS, MPa), modulus of rupture (MOR, MPa), and modulus of elasticity (MOE, GPa), were determined using a universal testing machine (Reger, RGM-4100, China). A size of 20 mm × 20 mm × culm wall thickness for CS and 200 mm × 12 mm × culm wall thickness for bending tests were used in the present study, and the samples of SS were made as shown in the Fig.1. SS, CS, MOR, and MOE were calculated by formula (1), (2), (3) and (4), respectively. CS and SS were measured by loading the specimen at a constant rate of 0.5 mm/min until the maximum load was reached or when failure occurred. Free span length of 150 mm and loading in culms wall radial direction were required for the MOR and MOE tests, the bending tests were conducted under three-point bending device at the cross head vertical speed of 10mm/min. Thirty replicates were carried out for each sample.

$$SS(MPa) = \frac{P_{\max}}{hL} \quad (1)$$

$$CS(MPa) = \frac{P_{\max}}{bh} \quad (2)$$

$$MOR(MPa) = \frac{3}{2} \times \frac{P_{\max} \times l}{bh^2} \quad (3)$$

$$MOE(GPa) = \frac{1}{4} \times \frac{\Delta p \times l^3}{\Delta f \times bh^3} \quad (4)$$

In these equations P_{\max} is maximum load at which the sample fails (N), L represents length of shear surface, b represents the width (mm), h represents the depth (culms wall thickness, mm), l is the free span (mm), and $\frac{\Delta p}{\Delta f}$ is the slope of the elastic zone (N/mm).

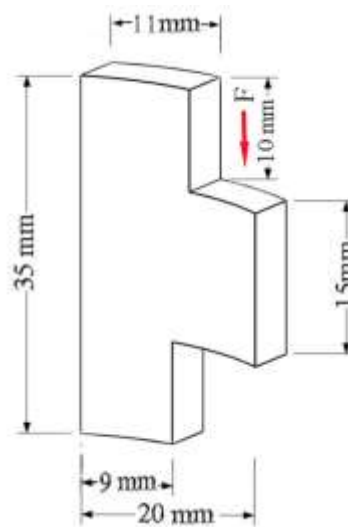


Fig. 1. Physical dimension of SS test specimen

Determination of anatomical properties

Sample blocks were boiled in distilled water with microwave heating for 2 to 3 hours until soft. The softened blocks were sliced into 30 μm sections on a sliding microtome. The cross-sections were stained with 0.1% safranin-o and dehydrated through an alcohol series, and then mounted on a slide with a cover slip. The air-dried slides were examined on a digital photomicroscope (Olympus DP20), and the anatomical properties analyzed by Image-Pro Plus (Media Cybernetics, version 6.0). The vascular bundle frequency was determined by counting the vascular bundle numbers on a section as images per mm^2 . The diameter of the vascular bundles in the radial and tangential planes was measured across the culm wall thickness. The fibrous tissue proportion was calculated by the area-method, *i.e.* the ration of fibrous tissue area to given area across culm wall thickness. Six replicates were carried out for each sample.

Chemical analysis

Air-dried control and aged specimens from the base, middle, and top portions of the stalk were reduced to particles using a Wiley mill and then screened to harvest the particles that passed through a 40-mesh sieve and were collected on a 60-mesh sieve. The particles are then dried to a constant weight in an oven at 80 $^{\circ}\text{C}$, and the chemical components were assayed. The holocellulose, alpha-cellulose, Klason lignin content, hot-water extracts, alcohol-toluene extracts, 1% NaOH solubility, and the ash contents of the control and aged specimens were determined according to ASTM standards D1104-56 (1971), D1103-60 (1971), D1106-96 (1996), D1106-96 (1996), D1107-96 (1996), D1109-84 (2001), and D1102-84 (2001), respectively. The hemicellulose content was determined in accordance with the method reported by Zhang *et al.* (2012).

Color measurement

The color measurement was conducted using a TCP 2-B color meter (Beijing Ao Yi Ke Photoelectric Instrument Co., Ltd) using CIE10 $^{\circ}$ field spectral tristimulus values. The color measurement standard light source was D65, which is produced by Philips. The color temperature of light source is 6500K \pm 200K, and the rendering index is greater than 96. The diameter of measuring window was 20 mm. Color parameters (L^* , a^* , b^*) according to the CIE LAB color system were obtained directly from the color meter and were used for color evaluation. The brightness difference (ΔL^*), the difference of a^* component (Δa^*), the difference of b^* component (Δb^*), the color difference (ΔE^*), the chroma difference (ΔC^*), and the hue difference (ΔH^*) were calculated with the following formulas,

$$\begin{aligned}\Delta L^* &= L_a^* - L_o^* \\ \Delta a^* &= a_a^* - a_o^* \\ \Delta b^* &= b_a^* - b_o^* \\ \Delta E^* &= [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \\ \Delta C^* &= C_a^* - C_o^* \\ \Delta H^* &= [(\Delta E^*)^2 - (\Delta L^*)^2 - (\Delta C^*)^2]^{1/2}\end{aligned}$$

where L^* , a^* , and b^* describe the chromatic coordinates on white-black, green-red, and blue-yellow axis, respectively. C^* is chroma, *i.e.*, $C^* = [(\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$. L_o^* , a_o^* , and b_o^* are the control reference, and L_a^* , a_a^* , and b_a^* are the accelerated aging treated sample, respectively.

Statistical analysis

Statistical analysis was carried out using SAS (version 9.1, SAS Institute, Cary, NC). The mean and the standard deviation were calculated, and analysis of variance (ANOVA) was performed to determine significant differences ($\alpha=0.05$) among the samples.

RESULTS AND DISCUSSION**Effect of Accelerated Aging on Bamboo Properties***Physical and mechanical properties*

The physical and mechanical properties of both the control and aged specimens from three culm height portions are shown in Table 1. The aged specimens had lower basic density and volumetric shrinkage than the controls. However, the differences between them were not significant over the ANVOA results. A similar reduction in density was also found in a study by Salim *et al.* (2010), in which *Semantan* bamboo was treated with oil heating. Compared to the control specimens, the mechanical properties of the aged specimens were lower. According to one way ANOVA, the differences in mechanical properties between the control and aged specimens were not significant ($p<0.05$) at the base portion, while that for the middle and top were not significant. To further clarify the effect of the accelerated aging treatment on the physical and mechanical properties of bamboo, an analysis of chemical components for both control and aged specimens was conducted.

Table 1. Physical and Mechanical Properties of Control and Aged Specimens of *Bambusa rigida*

Items	Base		Middle		Top	
	Control	Aged	Control	Aged	Control	Aged
D ($\text{g}\cdot\text{cm}^{-3}$)	0.60±0.04 ^a	0.59±0.03 ^a	0.69±0.02 ^a	0.67±0.06 ^a	0.79±0.07 ^a	0.75±0.05 ^a
S (%)	15.78±0.61 ^a	14.04±0.63 ^a	14.32±0.53 ^a	13.89±0.51 ^a	12.50±0.47 ^a	11.26±0.58 ^a
SS (MPa)	11.50±0.20 ^a	8.71±0.63 ^b	12.26±0.67 ^a	9.71±0.45 ^a	13.07±0.32 ^a	11.89±0.72 ^a
CS (MPa)	69.10±3.02 ^a	58.17±3.73 ^b	82.34±2.67 ^a	72.70±4.35 ^a	87.87±2.21 ^a	85.32±2.14 ^a
MOR (MPa)	170.34±12.61 ^a	131.23±12.39 ^b	204.58±7.67 ^a	189.12±11.33 ^a	214.32±6.30 ^a	205.55±6.60 ^a
MOE (GPa)	15.50±1.93 ^a	12.49±1.75 ^b	17.70±1.30 ^a	16.92±0.93 ^a	19.57±0.84 ^a	18.35±0.93 ^a

The values represent the mean \pm SD

Values followed by the same letter in the same row are not significantly different at $p<0.05$

Chemical composition

The main chemical composition of *Bambusa rigida* bamboo before and after the accelerated aging treatment is presented in Table 2. As shown in Table 2, the hot-water extractives, toluene-alcohol extractives, and ash content for the aged specimens were much lower than those for the controls. A significant difference ($p<0.05$) in the toluene-alcohol extractive for the base portion specimens was found between the control and aged specimens. This suggests that the low-molecular weight components in aged specimens, such as tannins, gums, silicates, and starches, were purged by hot water during the water and steam cycles, which may contribute to the reduction in density. Also, alpha-cellulose

for the aged specimens was lower than that for the control specimens, and while not significantly different ($p < 0.05$), this does suggest that cellulose was not significantly degraded by aging cycles. In terms of hemicellulose, the reduction was higher than that for cellulose (4.49% and 5.87% for cellulose and hemicellulose, respectively), which can be attributed to the fact that cellulose is much more stable toward acid hydrolysis than hemicellulose. It was interesting to find that the reduction in carbohydrates (cellulose and hemicellulose) with aging treatment was consistent with a decrease in the mechanical properties. The depolymerization of hemicellulose was greater than that of cellulose from the base to the top portion of aged specimens.

According to the literature (Kocaefe *et al.* 2008; Tuong and Li 2011; Yildiz *et al.* 2011), the degradation of hemicellulose, which was caused by thermal treatment, could contribute to the increase in dimensional stability. In this present study, the reduction in hemicellulose content induced by heating and steaming treatments involved in the accelerated aging cycles contributed a lot to the decrease in volumetric shrinking. Because the shear strength closely corresponded to the parenchyma cell wall components, and hemicellulose is the main ingredient of parenchyma cell walls (Esteves *et al.* 2007; Hsu *et al.* 1988; Yildiz 2002), it can be concluded that the lower SS for aged specimens may also be related to the loss of hemicellulose. Meanwhile, changes in CS, MOR, and MOE also reflect the loss of fiber strength parallel or perpendicular to the long axis along bamboo culms, and the fiber strength correlates with the cellulose content (Zhang *et al.* 2013). Thus, the reduction in CS, MOR, and MOE may be due to the loss of cellulose in the aged specimens.

Based on these results, it can be easily understood that the reduction in mechanical properties with accelerated aging has a positive relationship with the loss of carbohydrate components. The change of Klason lignin before and after accelerated aging was opposite to those of cellulose, hemicellulose, and the extracts. As shown in Table 2, the lignin content for the aged specimens was much higher than the controls. This result was expected due to the degradation of the carbohydrates or the weight loss of extractives and inorganic elements, and therefore, the (relative) lignin content increased.

Table 2. Chemical Components of Control and Aged Specimens of *Bambusa rigida*

Chemical components	Base		Middle		Top	
	Control	Aged	Control	Aged	Control	Aged
Hot-water extractives (%)	7.29±0.46 ^a	6.98±0.54 ^a	6.55±0.61 ^a	4.40±0.43 ^b	6.86±0.39 ^a	6.32±0.32 ^a
Toluene-alcohol extractives (%)	7.26±0.52 ^a	4.29±0.53 ^b	5.72±0.60 ^a	4.75±0.20 ^a	6.65±0.46 ^a	6.10±0.34 ^a
Ash (%)	2.97±0.03 ^a	2.58±0.05 ^a	1.39±0.12 ^a	1.14±0.03 ^a	3.05±0.01 ^a	2.72±0.06 ^a
Alpha-cellulose (%)	42.89±0.07 ^a	40.96±0.12 ^a	40.22±0.52 ^a	39.35±0.19 ^a	43.32±0.03 ^a	42.75±0.14 ^a
Hemicellulose (%)	25.79±1.02 ^a	24.27±1.12 ^a	27.03±0.98 ^a	26.26±1.21 ^a	31.15±1.33 ^a	30.39±1.43 ^a
Klason lignin (%)	25.94±1.86 ^a	31.85±2.09 ^b	23.37±1.26 ^a	24.25±2.03 ^a	24.51±1.26 ^a	29.35±0.98 ^b
1%NaOHsoluble (%)	24.25±0.41 ^a	27.02±0.15 ^b	25.19±0.21 ^a	25.96±1.34 ^a	25.93±0.16 ^a	27.10±0.66 ^a

The values represent the mean ± SD

Values followed by the same letter in the same row are not significantly different at $p < 0.05$

The 1% NaOH solubility in aged specimens was higher than that in controls, and the increasing rate was 11.42% for the base portion. The change in the trend of 1% NaOH was similar to that of Klason lignin. The 1% NaOH solubility of wood contained primarily extraneous components such as acid-soluble lignin, tannins, lipids, low-molecular weight hemicelluloses, and degraded cellulose (Pettersen 1984). Therefore, the results reveal that high-molecular weight components such as hemicellulose and cellulose in bamboo were decomposed into lower-molecular weight entities that can be dissolved by alkali solutions to accelerate the aging process.

Effect of Culm Height on Physical and Mechanical Properties

Physical and mechanical properties

As shown in Table 1, the average values of basic density varied from 0.60 to 0.79 $\text{g}\cdot\text{cm}^{-3}$ and 0.59 to 0.75 $\text{g}\cdot\text{cm}^{-3}$ for the control and aged specimens, respectively. An increasing trend of basic density from the base toward the top was also reported by Razak *et al.* (2010). Significant differences ($p<0.05$) were found in the physical properties with varying culm height. The mean volumetric shrinkage for the base, middle, and top control specimens was 15.78%, 14.31%, and 12.50%, respectively, while for the aged specimens was 14.04%, 13.89%, and 11.26% respectively. Remarkably, a decrease in volumetric shrinkage was observed for both control and aged specimens from the base toward the top, which is consistent with the finding of Kamruzzaman *et al.* (2008). The basic density generally increased from base to top, whereas volumetric shrinkage showed an inverse trend, indicating that there was a negative relationship between volumetric shrinkage and basic density.

To further clarify the variation in physical properties along the culm height, anatomical characteristics including vascular bundle frequency and dimension and fibrous tissue of *Bambusa rigida* bamboo, were measured and are presented in Table 3. The fibrous tissue proportion in cross section increased significantly from the base toward the top, ranging from 39.14 to 57.76%. This trend in fibrous tissue proportion along culm height was in accordance with the findings of Qi *et al.* (2014).

Table 3. Anatomical Characteristics of Control and Aged Specimens of *Bambusa rigida*

Anatomical properties	Base	Middle	Top
Vascular bundle (VB) frequency (VB no./mm ²)	3.27±0.85 ^a	4.85±0.78 ^b	6.79±1.26 ^c
Vascular bundles diameter in radial direction (µm)	586.98±42.83 ^a	538.43±52.24 ^a	441.17±21.94 ^b
Vascular bundles diameter in tangential direction (µm)	310.93±49.29 ^a	259.45±27.11 ^b	239.52±22.57 ^b
Fibrous tissue proportion (%)	39.14±4.46 ^a	49.90±3.37 ^b	57.56±2.94 ^c

The values represent the mean ± SD

Values followed by the same letter in the same row are not significantly different at $p<0.05$

It was reported that fibrous tissues primarily act in a mechanical support role (Razak *et al.* 2010). Thus, it can be concluded that the higher density observed at the top portion of the bamboo is mostly due to the higher fibrous tissue percentage (Table 1).

Similarly, the vascular bundle frequency also showed a significant increase ($p < 0.05$) from the base toward the top (Table 3), this is because of the fact that the top portion had thinner culm wall thickness (Grosser and Liese 1971). This finding is well in agreement with the report of Kelemwork (2009). In the top portion, vascular bundles are massed in a smaller space, which reduces the total air volume within a given area, resulting in a larger basic density (Nordahlia *et al.* 2012). In terms of volumetric shrinkage, the top portion of the specimen showed better dimensional stability (Table 1).

The SS and CS of both control and aged specimens significantly increased with culm heights (Table 1). The SS for the control specimens was 11.50 to 13.07 MPa, and that for aged specimens was 8.71 to 11.89 MPa. The CS of the control and aged specimens increased from 69.10 to 87.87 MPa and 58.17 to 85.32 MPa, respectively. The results in the variation in SS and CS along the culms height were similar to the findings of Xie *et al.* (2012). Accordingly, higher SS and CS at top portion were attributed to the higher density. In this present study, a significant positive correlation ($p < 0.05$) were found among SS, CS, vascular bundle frequency, and vascular bundle diameter (dimension in radial and tangential direction of culm thickness), which indicates that smaller and denser vascular bundles contributed to higher SS and CS.

Bending properties for both control and aged specimens, including MOR and MOE, increased from the base to the top. The increasing trend of MOE from base to top does not agree with the finding of Kelemwork (2009), who reported that the highest MOE was found in the bottom. In this experiment, MOR values of the control specimens were in the range of 170.34 to 214.32 MPa, while values for the aged specimens ranged from 131.23 to 205.55 MPa (Table 1).

The MOE values of the control specimens ranged from 15.50 to 19.57 GPa. The vascular bundle frequency increased with increasing basic density. In addition, basic density and vascular bundle density were positively correlated with MOR and MOE for both control and aged specimens.

Surface color

The color parameter and color parameter changes in the ΔC^* , ΔH^* , and ΔE^* values of both control and aged specimens are shown in Table 4. After the aging procedure, the bamboo surface became light brown, as indicated by the significant decrease ($p < 0.05$) in the value of L^* ($\Delta L^* = 9.1$). The lightness (L^*) of wood is commonly affected by hydrothermal treatment (Bourgois *et al.* 1991). Both the control and aged specimens in the middle portion were much lighter compared to these in the base and top portions.

The higher values of a^* for the aged specimens demonstrate that bamboo became more red compared to the control specimens ($\Delta a^* = 0.8$). From the base to the top, a^* values for both controls and aged specimens decreased with increasing culm height. The b^* values decreased significantly from 37.9 to 19.0 ($\Delta b^* = 18.9$) after the aging treatment. The b^* values increased slightly from the base toward the top portions. Meanwhile, a huge ($p < 0.05$) chroma difference ($\Delta C^* = -18.7$) and total color difference ($\Delta E^* = 21.0$), as well as a slight hue difference ($\Delta H^* = 2.7$) were observed. Furthermore, the largest hue difference was found in the specimens at the top portion.

Table 4. Effect of Accelerated Aging on Color Parameters of *Bambusa rigida*

Specimens		CIE LAB			ΔC^*	ΔH^*	ΔE^*
		L^*	a^*	b^*			
Control	Base	36.77±3.57 ^a	3.26±2.95 ^a	36.16±3.55 ^a	—	—	—
	Middle	36.87±4.66 ^a	2.05±3.67 ^b	37.92±6.26 ^a	—	—	—
	Top	35.11±3.61 ^a	1.24±3.87 ^c	39.49±5.31 ^a	—	—	—
	Means	36.25 ^x	2.19 ^x	37.86 ^x	—	—	—
Aged	Base	27.12±5.52 ^a	4.04±5.08 ^a	16.26±3.66 ^a	19.56±7.11 ^a	3.78±2.25 ^a	22.14±7.14 ^a
	Middle	30.35±5.66 ^a	3.18±3.18 ^a	20.54±2.95 ^b	17.19±2.79 ^a	2.79±1.58 ^b	18.60±3.79 ^b
	Top	24.14±5.82 ^b	1.81±5.26 ^b	20.20±1.27 ^b	19.23±5.53 ^a	1.63±0.26 ^c	22.20±3.08 ^a
	Means	27.20 ^y	3.01 ^y	19.00 ^y	-18.66	2.74	20.98

Color parameters: L^* , value on white/black axis; a^* , value on red/green axis; b^* , value on blue/yellow axis; ΔC^* , chroma difference; ΔH^* , hue difference; ΔE^* , color difference

The values represent the mean \pm SD

Values followed by the same letter in the same column are not significantly different at $p \leq 0.05$

CONCLUSIONS

1. A significant difference ($p < 0.05$) was found in the density and volume shrinkage between the control and aged specimens. The aged specimens had lower mechanical properties than the controls; however, no significant difference in mechanical properties was observed.
2. The lower chemical component content in the aged specimens caused a decrease in the physical and mechanical properties for the aged specimens. The significant difference ($p < 0.05$) in surface color before and after aging test may also be associated with the difference in chemical components between the control and aged samples.
3. The physical and mechanical properties for both the control and aged specimens increased from culm base to top, which may correspond to the variation in anatomical characteristics among different portions of the culms.

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