

# PROPERTIES OF SINGLE BAMBOO FIBERS ISOLATED BY DIFFERENT CHEMICAL METHODS

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**Abstract.** The objective of this study was to investigate the properties of single Cizhu bamboo fibers isolated by four chemical methods, with and without ultrasonic treatment. Properties tested were static contact angle, lumen size, and mechanical properties (tensile strength, modulus of elasticity [MOE], and elongation) of fibers macerated by four methods: nitric acid and potassium chlorate, sodium hypochlorite (NaClO), hydrogen peroxide and glacial acetic acid ( $H_2O_2 + HAC$ ), and sodium hydroxide. The results showed that the maceration time was different among the four methods. Ultrasonic treatment significantly affected the contact angle of all treatments with the exception of  $H_2O_2 + HAC$ . Lumen sizes treated by NaClO were different but cross-sectional area and cell wall area were similar for all other treatments, with and without ultrasonic treatment. Differences in mechanical properties were found among chemicals whereas elongation was similar for all solutions. Ultrasonic treatment accelerated the maceration rate, thus decreasing treatment time and contact angle of single fibers, but had no effect on cell wall area. Tensile strength and MOE were each affected in one solution by ultrasonic treatment, but those effects were within the range of the other solutions.

**Keywords:** Maceration method, contact angle, maceration time, lumen, mechanical properties.

## INTRODUCTION

Bamboo fibers are becoming the primary feedstock in China for weaving, paper making, and the fiber-based composite industry. Therefore, there is a need to study the properties for these diverse applications, which require different treatments. Each fiber property has its own advantages and deficiencies. For the fiber-based composite industry, adhesion and mechanical properties of the fibers are important (Pezron et al 1995; Aranberri-Askargorta et al 2003). The contact angle is a basic parameter for determining wettability (Aranberri-Askargorta et al 2003), and elongation serves as an indication of mechanical properties such as tensile strength

and modulus of elasticity (MOE). Therefore, studying fibers isolated using various chemical pretreatments can show how maceration methods affect these important properties.

Burgert et al (2002) compared two techniques for spruce fiber isolation, pulling out fiber directly and using a soft chemical treatment. The chemically treated fibers were found to have much lower strength and stiffness compared with mechanically isolated fibers. Burgert et al (2005a, 2005c) further researched structural, chemical, and mechanical characterization of spruce fibers. They discovered that at least 35% of the cell wall material was removed during chemical maceration. Loading of fibers isolated chemically was lower than that of mechanically isolated fibers. However, the surface properties of fibers were not tested. Data obtained by Mott et al

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(2002) found that average earlywood loblolly pine fibers, isolated by hydrogen peroxide and glacial acetic acid mixed at a 1:1 ratio, had tensile strength and MOE of 0.604 GPa and 14.8 GPa, respectively. Cao (2010) researched mechanical properties of moso bamboo (*Phyllostachys heterocycla*) fibers at 2, 4, and 6 yr isolated by hydrogen peroxide and glacial acetic acid with the same concentration as used by Mott et al (2002). They found that the mean values of tensile strength increased with age, tensile MOE increased from 2 to 4 yr, and elongations were unchanged.

Recently, the use of ultrasound, with its low energy consumption, high efficiency, and benign effects on materials, has been increasing (Mo et al 2009). Ultrasonic treatment can lead to chemical and/or physical changes on the surface, which can modify the properties of the fiber (Liu et al 2003). High stirring speeds lead to high reactivity, intense acoustic cavitation, and sonochemical effects (Gong 1999), which can affect the fiber properties during the separation process. Thus, ultrasonic effects, in conjunction with a chemical treatment, need to be quantified for the efficient use of the fiber.

The overall objective of this article was to determine how different maceration methods and ultrasonic treatments affect certain adhesion and mechanical properties of individual bamboo fibers. As a parameter of surface wettability, the contact angle of single bamboo fibers may be affected by the isolation technique, however data are lacking. In addition, the maceration method may have varying influences on the shrinking and swelling of the lumen. Although mechanical properties have been reported in the literature for chemically treated fibers, the effect caused by various isolation methods has not been determined. In this study, we evaluated contact angle, lumen size, and mechanical properties of fibers isolated by four chemical methods: nitric acid and potassium chlorate ( $\text{HNO}_3 + \text{KClO}_3$ ), sodium hypochlorite ( $\text{NaClO}$ ), hydrogen peroxide and glacial acetic acid ( $\text{H}_2\text{O}_2 + \text{HAc}$ ), and sodium hydroxide ( $\text{NaOH}$ ), with or without ultrasonic treatment.

## EXPERIMENTAL PROCEDURE

### Materials and Methods

Material was taken from 1-yr-old Cizhu bamboo (*Neosinocalamus affinis*) grown in Qionglai, Chengdu, Sichuan Province, China, with an initial 8-12% MC. Small bamboo strips (20 mm longitudinally and  $2 \times 2$  mm in cross-section) were selected from the bottom of the trunk. The strips were immersed in the four chemical solutions for fiber separation. Control fibers were isolated mechanically using fine tweezers from 90- $\mu\text{m}$ -thick tangential slices after being softened with about 40 h of hot water treatment.

Four solutions were prepared: 1) 65%  $\text{HNO}_3$  and 5%  $\text{KClO}_3$  mixed at a ratio of 1:1; 2) 20%  $\text{NaClO}$ ; 3) one part  $\text{H}_2\text{O}_2$ , four parts distilled water, and five parts  $\text{HAc}$ ; and 4) 15%  $\text{NaOH}$ . The solutions were heated to  $60^\circ\text{C}$  and the bamboo strips immersed. The fibers were washed in distilled water to neutrality and air-dried to constant weight after treatment. Ultrasonic treatments were made with a KQ5200DE Kunshan Ultrasonic Ltd (Shanghai) apparatus operated at 10 kHz with an output of 100 W.

### Contact Angle

Contact angle testing of distilled water droplets on individual fibers was conducted with a Kruss DSA100 (Hamburg, Germany) at  $25^\circ\text{C}$  and 20% RH. Individual fibers were obtained from the treatments with fine-tipped tweezers and mounted on a slatted platform with double-sided tape. The platform was moved into position using CCD cameras in the x, y, and z directions. The baseline for a sessile drop static contact angle measurement was made at the liquid–solid interface with droplet size held to a constant 10  $\mu\text{L}$ . Contact angle measurements were calculated using the ellipse method at a rate of 72 frames/s in the DSA 3 software (Fig 1). In the ellipse method, the fitted ellipse approximates the drop contour, and deviations from true drop shape are minimal (Amaral et al 2002). This leads to an accurate measurement of the contact angle within

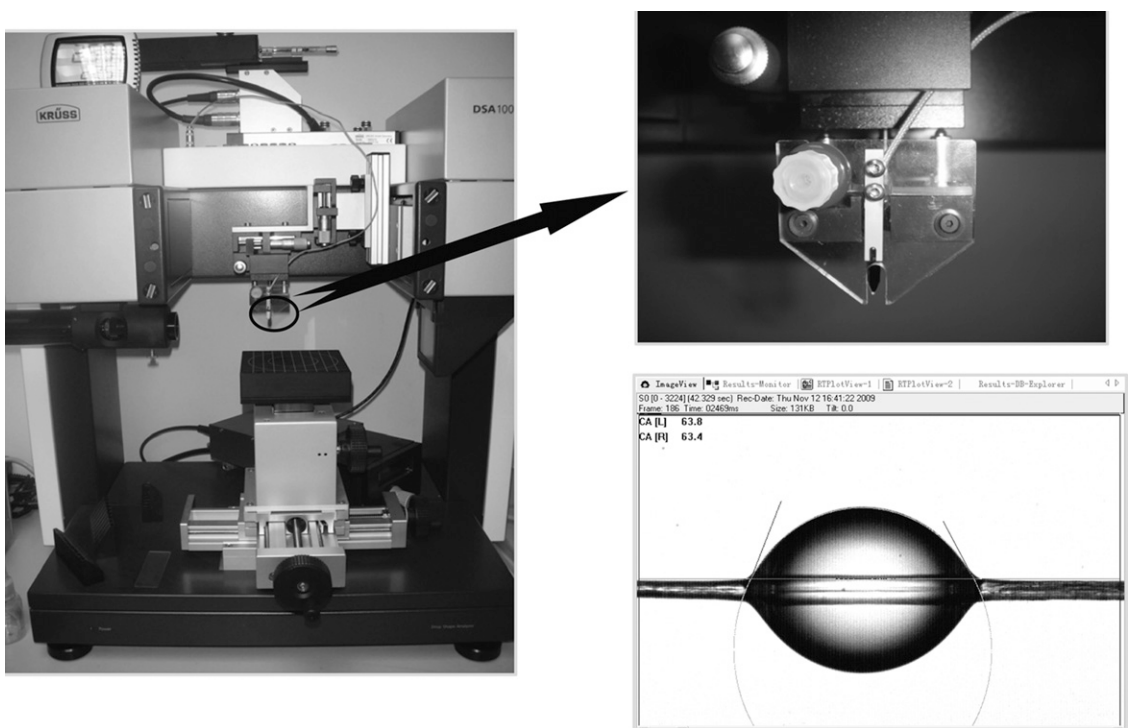


Figure 1. Kruss DSA 100 and contact angle calculated by the ellipse method using DSA 3 software.

fractions of a second and has been applied by various researchers (Amaral et al 2002; Xiangfa and Yuris 2006). Ten samples were investigated for each treatment combination.

### Lumen Size

The fibers were dyed with acridine orange solution for 4 min in a Petri dish. Tissue tack was applied to a glass slide, and the fiber was placed on the slide and allowed to air-dry. One droplet of balsam Canada reagent was placed on the slide, and a cover was carefully placed over the fiber, ensuring that no air was trapped. The area of the fiber cross-sections was determined with a confocal laser scanning microscope (CLSM; Zeiss, LSM 510 Meta, Germany). AxioVision software (Zeiss, Germany) was used to calculate the fiber area. Photographs of the fibers were taken with an emission field scanning electron microscope (ESEM) (FEI Com-

pany, XL30 ESEM FEG, Hillsboro, OR) with 10 replications.

### Mechanical Properties

Tensile testing of single fibers was conducted following the method described by Cao (2010). Briefly, the fibers were first glued across an organic, channeled glass with one droplet of glue on each end. The fibers were then placed in an oven at 60°C for 24 h followed by 22°C for at least 24 h. Tensile testing of single fibers was conducted with an instrument (SF-Microtester I) designed at the International Center of Bamboo and Rattan, Beijing, China (Fig 2). A constant strain rate of 80  $\mu\text{m}/\text{min}$  was set at 25°C and 20% RH. Fibers were removed from the tensile apparatus immediately upon failure and stored for subsequent cross-sectional area measurement with CLSM for tensile modulus and strength calculations. Eight samples were tested for each

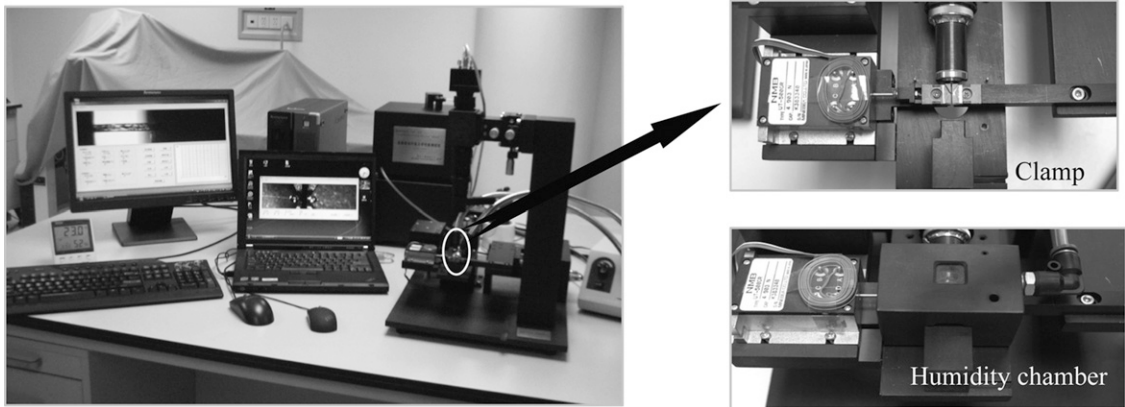


Figure 2. SF-Microtester I designed at the International Center of Bamboo and Rattan.

Table 1. Maceration time for the four solutions and ultrasonic treatments.

Time (min)	Treated				Untreated			
	HNO <sub>3</sub>	NaClO <sub>3</sub>	H <sub>2</sub> O <sub>2</sub>	NaOH	HNO <sub>3</sub>	NaClO <sub>3</sub>	H <sub>2</sub> O <sub>2</sub>	NaOH
1	15	180	2400	1800	34	360	2520	1980
2	18	150	1480	1480	40	510	1520	1520
3	28	270	900	1315	60	390	1255	1500
Avg	20	200	1590	1530	45	420	1765	1670
SD	6.8	62	760	245	14	80	670	270
CV	0.33	0.31	0.47	0.16	0.30	0.19	0.38	0.16

treatment combination. SPSS was used for all data analysis at the 0.05 significance level.

## RESULTS AND DISCUSSION

### Maceration Time

Fiber separation time is summarized in Table 1. The HNO<sub>3</sub> + KClO<sub>3</sub> solution, with and without ultrasonic treatment, had the shortest average maceration times, 15 and 34 min, respectively. Fibers isolated by NaClO with and without ultrasonic treatment took longer to separate, 180 and 360 min, respectively. The other two methods required a minimum of 1 da, with or without ultrasonic treatment. Ultrasonic treatment can shorten maceration time primarily because of the high stirring rate and the intense acoustic cavitation effect, which accelerates molecular velocity in the solution (Weissler 1948, Xie et al 2004). It provides a means of “burning” substances in liquids and enhancing reactions that cannot be achieved by conven-

tional means (Gong 1999). The effect of ultrasonic treatment ranged from a 56% decrease in maceration time for HNO<sub>3</sub> + KClO<sub>3</sub> to a 4% decrease for H<sub>2</sub>O<sub>2</sub> + HAc. The absence of ultrasonic treatment apparently caused the reaction between the fibers and solution to reach equilibrium quicker because of the reduced contact of unreacted chemicals.

### Contact Angle

The contact angles of fibers isolated by the four methods and ultrasonic treatments are shown in Fig 3. The mean contact angle of those isolated by HNO<sub>3</sub> + KClO<sub>3</sub> were the lowest, whereas the fibers isolated by H<sub>2</sub>O<sub>2</sub> + HAc had the largest mean angle. Overall, the contact angle of fibers fluctuated only 4°, and there were no significant differences in contact angles for ultrasonic treatments of all solutions. Comparisons between fibers with and without ultrasonic treatment found that ultrasound did decrease the mean

contact angle of fibers in all solutions but had a significant effect only for those treated with NaClO (0.04 significance level) (Table 2). One possible explanation is that the mechanical shaking, thermal activity, and acoustic cavitation caused the surface of the fibers to be much rougher, resulting in a larger fiber surface area (Liu et al 2003). Generally, the greater surface area contributes to a lower contact angle and thus a larger surface free energy, which greatly contributes to an increase of adhesion by improving wettability (Silva and Al-Qureshi 1999). The fact that H<sub>2</sub>O<sub>2</sub> + HAc and NaOH solutions had longer residence times in solution could have offset the effect of ultrasound. However, the maceration time for HNO<sub>3</sub> + KClO<sub>3</sub> may have been too short to change the surface of fibers (Table 1).

The contact angle of fibers macerated chemically was lower than that of fibers isolated

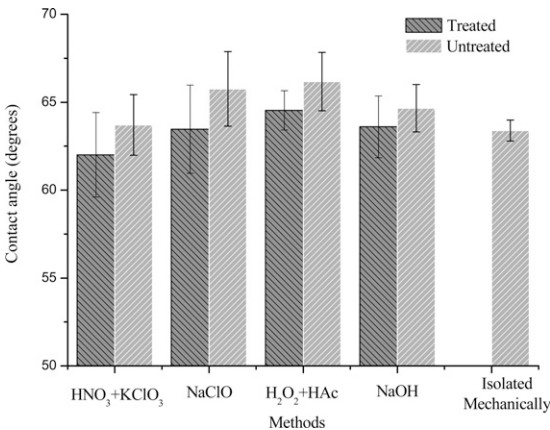


Figure 3. Contact angle of single fibers macerated in different solutions and conditions.

mechanically, possibly because predominantly plane fracture surfaces were produced when the fiber was peeled with fine tweezers (Burgert et al 2005a) (Fig 4). These fractures made the surfaces of fibers rougher compared with those macerated chemically.

**Lumen Size**

Lumens of single fibers isolated with different treatments (Table 3) were dissimilar as determined by images from the CLSM (Fig 5). Lumens of fibers macerated by HNO<sub>3</sub> + KClO<sub>3</sub> and NaClO varied widely, possibly because the high rates of separation influence the fiber diversely. However, lumens isolated by H<sub>2</sub>O<sub>2</sub> + HAc and NaOH exhibited less variation, possibly because of the longer residence time.

The average cross-sectional area of fibers isolated mechanically was 264 μm<sup>2</sup> and the average lumen area was 126 μm<sup>2</sup>, leaving an average cell wall area (cross-sectional area minus lumen area) of 138 μm<sup>2</sup>. The fibers were compared with those isolated chemically with H<sub>2</sub>O<sub>2</sub> + HAc and NaOH. Cross-sectional area decreased by 72% after treatment with H<sub>2</sub>O<sub>2</sub> + HAc, and the lumen was collapsed. NaOH decreased the cross-sectional area by 29%, the lumen by 72%, and the cell wall area by 61%. NaOH probably decomposed lignin and hemicelluloses to a large extent, whereas the H<sub>2</sub>O<sub>2</sub> + HAc treatment removed lignin extensively in addition to partially hydrolyzing some hemicelluloses (Burgert et al 2005b; Xu and Tang 2006). These treatments should greatly influence the mechanical properties of macerated fibers because the fiber area is required for calculating tensile strength and MOE.

Table 2. Data analysis of the contact angle by SASS at a significance level of 0.05.

	Equal variances	F	Significance	t	df	Significance (two-tailed)
HNO <sub>3</sub>	Assumed	0.766	0.393	-1.818	18	0.086
	Not assumed			-1.818	16.329	0.087
NaClO	Assumed	0.672	0.423	-2.209	18	0.04
	Not assumed			-2.209	17.495	0.041
H <sub>2</sub> O <sub>2</sub>	Assumed	0.253	0.621	-2.587	18	0.19
	Not assumed			-2.587	15.749	0.2
NaOH	Assumed	1.37	0.257	-1.509	18	0.149
	Not assumed			-1.509	16.869	0.15

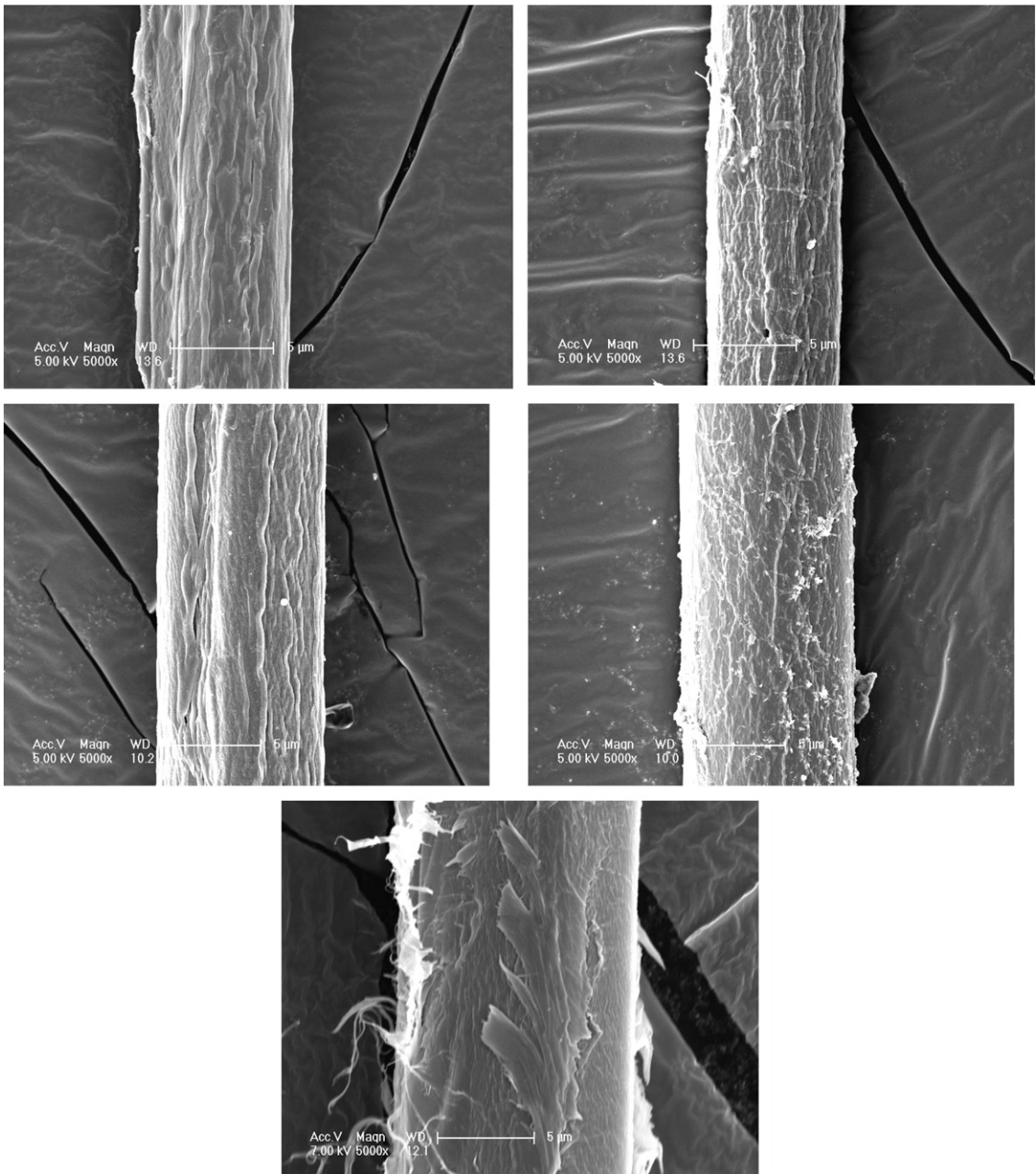


Figure 4. Emission field scanning electron microscope images of fibers macerated in nitric acid and potassium chlorate ( $\text{HNO}_3 + \text{KClO}_3$ ), sodium hypochlorite ( $\text{NaClO}$ ), hydrogen peroxide and glacial acetic acid ( $\text{H}_2\text{O}_2 + \text{HAc}$ ), and sodium hydroxide ( $\text{NaOH}$ ) solutions and isolated mechanically.

Chemical treatment removes a large portion of the lignin fraction and partially degrades some hemicelluloses, leading to shrinkage of fiber cell walls during drying (Burgert et al 2005b). Fur-

thermore, the external surface has been found to be influenced much more compared with the lumen surface area (Parameswaran and Liese 1976). Because the external surface of the fiber

shrank quicker, this caused the lumen area to decrease.

ESEM images of fibers isolated chemically and mechanically (Fig 5 show that mechanically isolated fibers were smoother and larger, whereas

the fibers isolated chemically shrank transversely and had wrinkled surfaces. Na<sup>+</sup> ions transported water molecules into fibers, which increased the intervals between among molecular chains. This breaks or weakens hydrogen bonding, causing the fiber to swell transversely (Zhao et al 2009). The swelling compensates for a fraction of the shrinkage so that the area of the lumen in the fibers isolated by NaOH decreased less than that in fibers isolated by H<sub>2</sub>O<sub>2</sub> + HAc. ESEM images of fibers in Fig 4 illustrate that the wrinkles on the surface of fibers macerated by H<sub>2</sub>O<sub>2</sub> + HAc were more pronounced than those on fibers isolated by NaOH, which caused greater swelling (Zhao et al 2009).

Table 3. Area of lumens in the center of single fiber cross-section and area of single fiber cross-section.

Methods	Area	Average area (μm) <sup>2</sup>	Standard deviation	
HNO <sub>3</sub> + KClO <sub>3</sub>	Treated	CS	72.1	34.3
		Lumen	18.3	24.2
	Untreated	CS	79.9	31.4
		Lumen	25.5	25.0
NaClO	Treated	CS	87.2	40.0
		Lumen	16.6	25.9
	Untreated	CS	119	45.9
		Lumen	46.1	26.0
H <sub>2</sub> O <sub>2</sub> + HAc	Treated	CS	66.0	13.6
		Lumen	0	0
	Untreated	CS	72.5	20.1
		Lumen	0	0
NaOH	Treated	CS	83.0	6.89
		Lumen	35.3	11.0
	Untreated	CS	89.8	14.7
		Lumen	35.7	13.3

CS, cross-sectional area.

When untreated fibers were compared with those treated by ultrasound, it was found that ultrasound decreased fiber cross-sectional area slightly but not significantly. The lumen area of the fibers was not significantly impacted, with the exception of NaClO, possibly because of the ultrasonic acceleration of the separation rate, thus affecting the fiber to a greater degree in the chemical solution (Weissler 1948). Overall, cell wall area was not significantly affected by

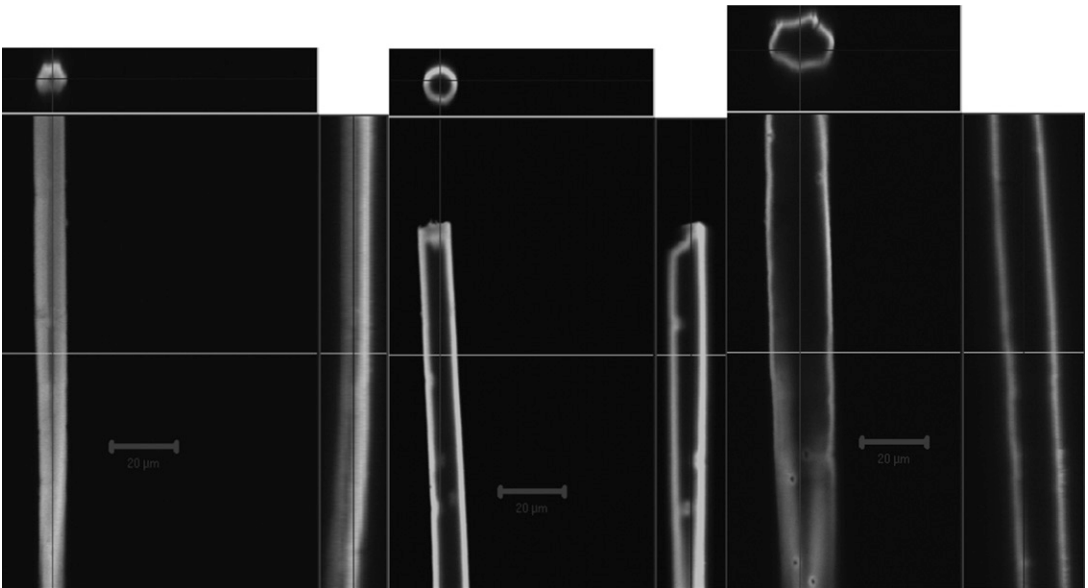


Figure 5. Images of fibers macerated in hydrogen peroxide and glacial acetic acid (H<sub>2</sub>O<sub>2</sub> + HAc) and sodium hydroxide (NaOH)solutions, and isolated mechanically from a confocal laser scanning microscope.

ultrasonic treatment. Thus, using ultrasound can shorten maceration time and save energy without detrimental effects to the fiber size (Gong 1999).

### Mechanical Properties

Tables 4 and 5 show that the tensile strength of single fibers isolated by H<sub>2</sub>O<sub>2</sub> + HAc and treated ultrasonically was significantly lower than that of fibers without treatment. Ultrasound did not have an effect for any other solution. The MOE of fibers macerated in NaClO significantly

decreased with ultrasonic treatment but was within the range of fibers isolated by HNO<sub>3</sub> + KClO<sub>3</sub> and H<sub>2</sub>O<sub>2</sub> + HAc. Otherwise, no differences were detected for MOE. Ultrasonic treatment caused a more uniform elongation of fibers in NaClO and NaOH, whereas untreated fibers produced variable elongations when they were tested in tension. Because the elongation of fibers isolated and treated with ultrasound was similar, fiber elasticity should not be affected. This was possibly caused by the ultrasound keeping the solutions uniform during separation (Gong 1999). Ultrasonic treatment causes the

Table 4. Mechanical properties of individual fibers isolated with different treatments.

Methods		Tensile strength (GPa) (CV)	MOE (GPa) (CV)	Elongation (%) (CV)
HNO <sub>3</sub> + KClO <sub>3</sub>	Treated	1.32 (0.32)	34.4 (0.20)	2.33 (0.54)
	Untreated	1.32 (0.32)	30.9 (0.22)	1.84 (0.22)
NaClO	Treated	1.16 (0.19)	30.5 (0.18)	2.29 (0.22)
	Untreated	1.30 (0.23)	42.0 (0.32)	1.96 (0.30)
H <sub>2</sub> O <sub>2</sub> + HAc	Treated	1.34 (0.18)	30.2 (0.22)	2.41 (0.21)
	Untreated	1.78 (0.15)	26.8 (0.06)	2.89 (0.16)
NaOH	Treated	1.45 (0.36)	19.6 (0.36)	2.49 (0.17)
	Untreated	1.52 (0.30)	18.3 (0.18)	2.80 (0.31)

Table 5. Data analysis of mechanical properties by SASS at a significance level of 0.05 for ultrasonic treatment.

		Equal variances	F	Significance	t	df	Significance (two-tailed)
TS	HNO <sub>3</sub>	Assumed	0.146	0.708	0.006	14	0.996
		Not assumed			0.006	13.996	0.996
	NaClO	Assumed	2.143	0.165	-1.026	14	0.322
		Not assumed			-1.026	12.958	0.324
	H <sub>2</sub> O <sub>2</sub>	Assumed	1.318	0.27	-3.365	14	0.005
		Not assumed			-3.365	13.732	0.005
NaOH	Assumed	0.358	0.559	-0.266	14	0.794	
	Not assumed			-0.266	13.749	0.794	
MOE	HNO <sub>3</sub>	Assumed	0.026	0.874	1.017	14	0.327
		Not assumed			1.017	13.999	0.327
	NaClO	Assumed	10.322	0.006	-2.208	14	0.044
		Not assumed			-2.208	9.187	0.054
	H <sub>2</sub> O <sub>2</sub>	Assumed	2.296	0.152	1.194	14	0.252
		Not assumed			1.194	12.13	0.255
NaOH	Assumed	1.998	0.179	-0.204	14	0.842	
	Not assumed			-0.204	11.132	0.842	
E	HNO <sub>3</sub>	Assumed	6.182	0.026	1.04	14	0.316
		Not assumed			1.04	8.499	0.327
	NaClO	Assumed	0.052	0.823	1.185	14	0.256
		Not assumed			1.185	13.692	0.256
	H <sub>2</sub> O <sub>2</sub>	Assumed	7.058	0.019	-2.462	14	0.027
		Not assumed			-2.462	8.675	0.037
NaOH	Assumed	4.278	0.058	-0.825	14	0.423	
	Not assumed			-0.825	9.9	0.429	



chemical solutions to remain reactive, decreasing maceration time and the likelihood of damaging fibers (Weissler 1948). Tensile strength and MOE of bamboo fibers isolated by  $H_2O_2$  + HAc compared favorably with past work on isolated moso bamboo fibers (Cao 2010).

For mechanically isolated fibers, tensile strength (0.932 GPa) was lower than that for fibers macerated chemically but elongation was 4.3% greater. The transverse shrinkage of fibers macerated chemically and the plane fractures on fibers isolated mechanically were possibly the major reasons. Groom et al (2002) found the average tensile strength and MOE of loblolly pine fibers, in equal proportion of earlywood and latewood, to be 0.824 and 17.3 GPa, respectively, with the same maceration solution ( $H_2O_2$  + HAc) as this study. Different species may be responsible for the different results. The average tensile strength of spruce fibers isolated mechanically obtained by Burgert et al (2005c) was similar to the results of this study, but his MOE was lower because of the different species.

### CONCLUSIONS

Several fiber properties were determined for bamboo fibers isolated by four chemical methods and subjected to ultrasonic treatment. The fibers isolated by  $HNO_3$  +  $KClO_3$  required the shortest maceration time and had the lowest contact angles. Fibers isolated by  $H_2O_2$  + HAc required the longest residence time and had the largest contact angles. Ultrasonic treatment shortened maceration time for all chemical solutions. Tensile strength was decreased in  $H_2O_2$  + HAc, but all solutions were similar after ultrasonic treatment. Lumen area was affected by NaClO treatment, but overall, cell wall area was similar for all chemical treatments. MOE was not significantly different among solutions after ultrasonic treatment. The four methods produced similar fiber elongations after ultrasonic treatment. These results suggest that ultrasound decreases the maceration time and does not have an overall deleterious effect on contact angle and mechanical properties of bamboo fibers.

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