Effect of Sulfonation Treatment Concentration on the Properties of Mulberry Chemi-mechanical Pulp

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Sulfonation chemimechanical pulping (SCMP) of mulberry stalk was studied with different treatment concentrations. The resulting mulberry SCMP pulp contained different content of sulfonic acid groups depending on the concentrations. The brightness, tensile index, and folding resistance of mulberry SCMP pulp increased with the increasing concentration of sulfonation treatment, but the thickness decreased with increasing concentration. There was either a linear or a non-linear relationship between the content of sulfonic groups and the pulp physical properties. The mass average length of mulberry SCMP pulp was 0.66 mm, the fiber width was 16.4 μm, and the content of fine fibers was 20.6%.The mulberry extract and lignin dissolved, and part of soluble lignin may have been deposited on fiber surface in the process of SCMP pretreatment. There were almost no changes to the cellulose crystalline structure.

Keywords: Mulberry SCMP pulp; Sulfonic group; Papermaking performance

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INTRODUCTION

Mulberry is a good raw material for pulping and papermaking. The Jiangxi Provincial Institute of Light Industry compared the chemical composition of mulberry stem, masson pine, and kenaf, showing that mulberry stalk has the highest total cellulose content as well as the lowest lignin content (Cai 1996).

The non-wood fiber raw materials that are most widely used in pulping and papermaking are wheat straw, bagasse, reeds, bamboo, *etc.*, but mulberry stalk is used less frequently. The purchasing price of mulberry stalk raw materials is about 120 to 150 yuan/ton, which is lower than the price of other grass raw materials (Deng and Mo 2008). Mulberry stalk in China is abundant, widely distributed, and produced in an area of about 70 thousand hm^2, ranking first in the world (Li and Yang 2010). Most mulberry stalks are not fully utilized, resulting in environmental pollution. Teng *et al*. (2006) studied the utilization of mulberry in four towns in Sichuan Province, and they found that the highest utilization rate of mulberry stalk was only 35%, the lowest utilization rate was 4%, and the unused mulberry accounted for 72.3% of the total. As a rich and renewable fiber resource, mulberry stalk has the potential for further development for pulping and papermaking.

At present, the chemical pulping technology of mulberry stalk is quite mature and has been in production for many years. However, high yield pulps of mulberry are not commonly manufactured, although they pollute less and are stronger. Hu *et al*. (2009) explored the chemical pretreatment conditions (NaOH and H_2O_2) of preparing mulberry mechanical pulp, focusing on their effectiveness following bleaching. Liu (2012) prepared

mulberry stalk alkaline peroxide mechanical pulping (APMP) and Bio-APMP pulp with better physical properties.

In this research, a pulping method called sulfonated chemimechanical pulp (SCMP) was used to prepare pulping with mulberry stalk. Its principle is to utilize Na_2SO_3 to perform sulfonation reaction with raw material to produce permanent softening, so as to better separate fibers and fibrillate in the refining process. Studies have shown that SCMP pulping has the advantages of reduced pollution, high yields, energy savings, and high pulp strength (Liu 2007). Previous studies have shown that SCMP pulp can be similar to CTMP in terms of optical properties, and its strength is similar to that of the chemical pulp (Pan 1982). Pulp was prepared with mulberry stalk, and the effect of content of sulfonic group on papermaking performance of mulberry SCMP pulp were studied using Fourier transform infrared spectroscopy (FT-IR) and X-ray photoelectron spectroscopy (XPS) to investigate the reaction mechanism of mulberry stalk SCMP pretreatment.

EXPERIMENTAL

Materials

Mulberry stalks were collected (by Guiling Zhao) from a silkworm base in Nanning, Guangxi, China. They were cut into 20 mm to 30 mm long samples and naturally air-dried.

Methods

Preparation of mulberry stalks SCMP

For the sulfonation process, mulberry stalks were immersed in water for 24 h and then placed in an electric heating digester. The sulfonated pulp was washed with clear water after digesting.

SCMP pulping conditions

The amount of NaOH added was 4%. In order to obtain SCMP pulp with different sulfonation treatment concentrations, the amounts of Na_2SO_3 added were: 9%, 12%, 15%, 17%, 18%, and 21%, respectively. Both sets of values were relative to the percentage of dry raw material. The liquor ratio was 1:5. The highest temperature was 130 °C and the heat preservation time was 120 min (Qin 1989; Cai 1996). The conditions are listed in Table 1. The sulfonated mulberry stalks were washed and then prepared at a pulp concentration of about 15% for refining. The millstone was set at intervals of 0.30 mm, 0.15 mm, and 0.15 mm, respectively, and three-stage grinding was carried out on a high-consistency refiner (Jilin Paper Co., Ltd. Paper Machinery Factory, Jilin, China). Finally, the SCMP pulp was washed and had completed a process of moisture equilibration before use.

Determination of sulfonic groups content

The dry pulp samples of 3 g were soaked twice in 100 mL of 0.1 M HCl solution and stirred with a magnetic mixer for 45 min. They were washed with deionized water (CO_2-free) until the conductivity was stable (conductivity between 1.3 and 1.5 $\mu S \cdot cm^{-1}$) using a conductivity meter (Shanghai Precision Instrument Co., Ltd., Shanghai, China). After filtration, the pulp was dispersed in 450 mL of 0.001 M NaCl. The 0.1 M NaOH standard solution was used to titrate with magnetic stirring by the N_2 protection. The

titration rate was 0.5 mL NaOH solution over 5 min. The titration curve was recorded using a conductivity meter. Finally, the pulp was washed and dried to constant weight. The following formula were calculated using Eq. 1,

$$\text{Sulfonic group} = (C_2V_2\text{-}C_1V_1)/m\times 1000(\text{mmol/Kg pulp sample}) \quad (1)$$

where C_1 is the concentration of the HCl solution (mol/L); V_1 is the volume of the HCl solution added (mL); C_2 is the concentration of NaOH standard solution, (mol/L); V_2 *is* the volume of NaOH standard solution consumed by the first equivalence point (mL); and m is the weight of sample (absolutely dry) (g).

Table 1. Conditions of Sulfonation Used for Mulberry Stalk SCMP

Number	1	2	3	4	5	6
Dosage of Na_2SO_3 (%)	9	12	15	17	18	21
Dosage of NaOH (%)	4	4	4	4	4	4
Maximum temperature (°C)	130	130	130	130	130	130
Preservation time (min)	120	120	120	120	120	120
Content of sulfonic group ($mmol\cdot kg^{-1}$)	60.68	68.28	75.87	83.20	88.10	105.57
Brightness( )	43.01	44.86	46.91	47.39	49.75	50.19
Tensile index ($N\cdot m\cdot g^{-1}$)	24.91	33.44	34.41	37.38	39.92	41.32
Folding strength (times)	1	2	3	4	5	7
Bulk ($cm^3\cdot g^{-1}$)	3.38	2.95	2.87	2.75	2.68	2.62

Measurement of paper attributes

First, the brightness of the pulp was determined by blue light of 457 nm wavelength. The paper quantitative test was measured according to standard GB/T 451.2 (1989). The pulp was also tested in accordance with standard GB/T 453 (1989) for paper tensile strength and tensile strength index data processing and calculation. Finally, the paper folding strength was measured according to standard GB/T 457 (1989).

Analysis of fiber morphology

For the preparation of fiber samples, mulberry stalks (approximately 1 mm × 2 mm × 30 mm) were soaked in a mixture of glacial acetic acid and hydrogen peroxide (1:1) at 60 °C for 30 to 48 h. Then the fiber suspension was at a concentration of 0.05%. For the fiber of mulberry stalk SCMP, the SCMP pulp of 0.1 mg (oven dry) was dispersed to 0.001%. About 30 to 50% of the reserve fiber suspension was placed into a sample cup, and the fiber morphology was evaluated with a fiber mass analyzer (Kajaani Electronics Co., Ltd., Kajaani, Finland).

FT-IR analysis

The model of the instrument is Spectrum BX, produced from PE companies in the United States. The KBr disc technique was used for infrared spectroscopic analysis. The test range was 400 cm^{-1} to 4000 cm^{-1}, and the wavelength coverage was 2.5 μm to 25 μm.

XPS analysis

Mulberry samples were extracted with acetone using a Soxhlet extractor at 60 °C. An Axis Ultra DLDXPS instrument (Kratos, Manchester, UK) was used. The test conditions were CAE scanning mode, the beam spot was 700 μm × 300 μm, and the monochromatic Al Ka source energy was 1486.6 Ev, 10 mA × 12KV.

RESULTS AND DISCUSSION

Effect of Sulfonic Groups Content

In the chemical pretreatment stage, Na_2SO_3 reacts with lignin and introduces sulfonic acid groups on the lignin, which increases the hydrophilicity of the lignin. In the sulphonation phase, the main active substance is Na_2SO_3. The degree of sulphonation depends mainly on the percentage of Na_2SO_3 added (Zhan 2011). The chance of collision of lignin with sulfite ions increases with the increase of Na_2SO_3 content, so the sulfonation reaction and the content of sulfonic acid groups also increase (Hu 2009). The different contents of sulfonic groups of mulberry were adjusted and controlled by changing the amount of Na_2SO_3 with other conditions unchanged. The brightness, tensile index, folding resistance, thickness and other properties of mulberry SCMP were also tested. The results are shown in the Table 1.

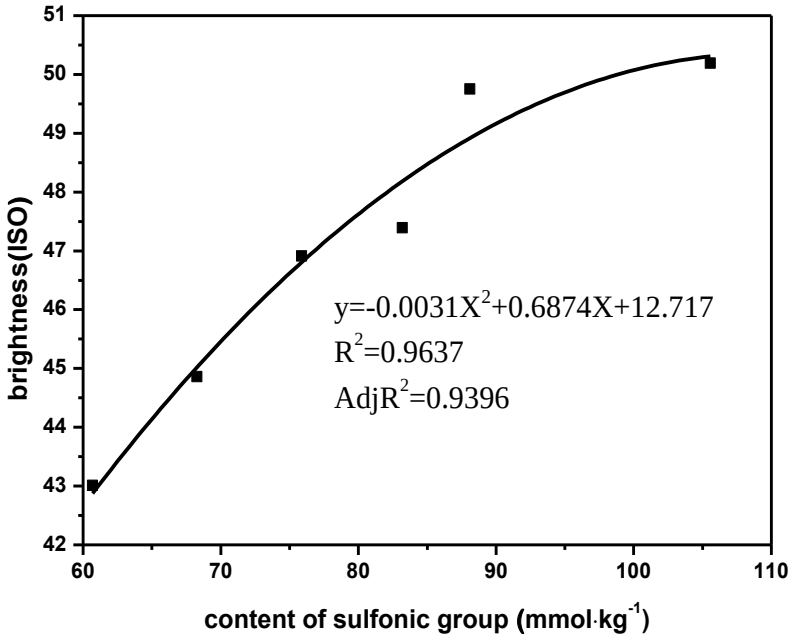


Fig. 1. Effect of sulfonic group on brightness

Effect of Sulfonic Group Content on the Brightness of SCMP Pulp

The paper structure is sensitive to paper optical properties, but the degree of fiber sulfonation changed the structure of paper. Thus, the optical properties of sulfonated thermomechanical pulp (TMP) can be evaluated by the degree of fiber sulfonation; the brightness of pulp increases with increasing degree of sulfonation (McCall *et al*. 1995). This result is consistent with the results shown in Fig. 1. The brightness of mulberry SCMP

was increased to 50.2% ISO with the increased sulfonic group content. The coefficient of determination (R^2 = 0.9637) was greater than the adjusted coefficient (Adj R^2 = 0.9396). If the value is greater than 0.9, it is considered a good fit.

Effect of Sulfonic Group Content on the Tensile Strength Index of SCMP

The sulfonic group, which is hydrophilic, could improve the fiber swelling and fibrillating. This explains the trend of increasing tensile index with sulfonic group content in Fig. 2.

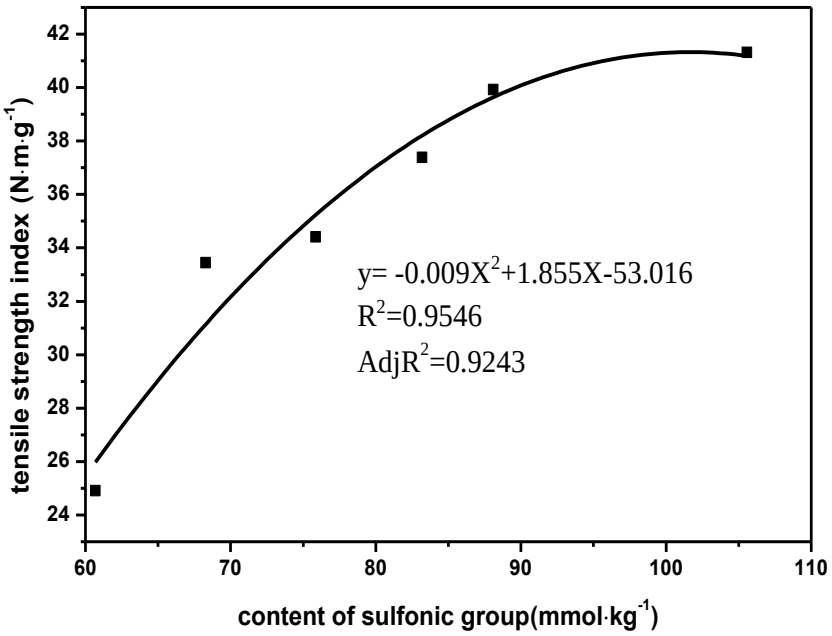


Fig. 2. Effect of content of sulfonic group content on tensile strength index

McCall *et al*. (1995) showed that the content of sulfonic groups in mechanical pulp is linear or has a nearly linear relationship with paper tensile index. As also shown in Fig. 2, the monomial coefficient of *X* was greater than quadratic coefficient in the fitting equation. It could be speculated that the sulfonic groups were approximately linearly correlated with tensile index. Both the coefficient of determination (R^2=0.9546) and the adjusted coefficient (AdjR^2 = 0.9243) were greater than 0.9, indicating that they had a nonlinear relationship.

Effect of Sulfonic Group Content on Folding Strength of SCMP Pulp

The folding strength mainly depends on the length of fiber and is also affected by factors such as fiber flexibility and fiber bonding (Shi and He 2009). The sulfonic acid group could soften the fibers and help to separate fibers and increase the number of long fibers (Zhan 2011), improving the folding strength of SCMP pulp. This explains that the value of folding strength of mulberry SCMP in Fig. 3 increased from 1 to 7, which was similar to that of the mulberry AP-AQ pulping (Gong and Pi 2007). Both R^2 and Adj R^2 of the equation were higher than 0.9. This indicates that the fitting degree of the nonlinear equation was high, and they had linear relationships.

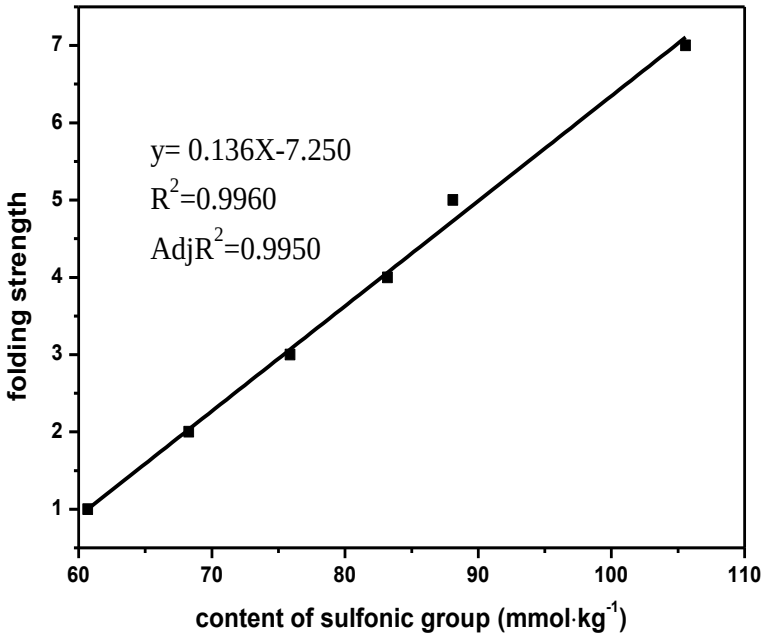


Fig. 3. Effect of content of sulfonic group content on folding strength

Effect of Sulfonic Group Content on Bulk of SCMP Pulp

The long fiber numbers of CMP and CTMP increased with the increasing total ion content. When the total ion content increased to 210 mmol/kg, the tightness of handsheets made by long and short fibers was more than triple that obtained from the default pulp. This was because of the reducing of elastic shear modulus due to the softening of sulfonic acid groups by the treatment with Na_2SO_3 (Heitner and Atack 1983).

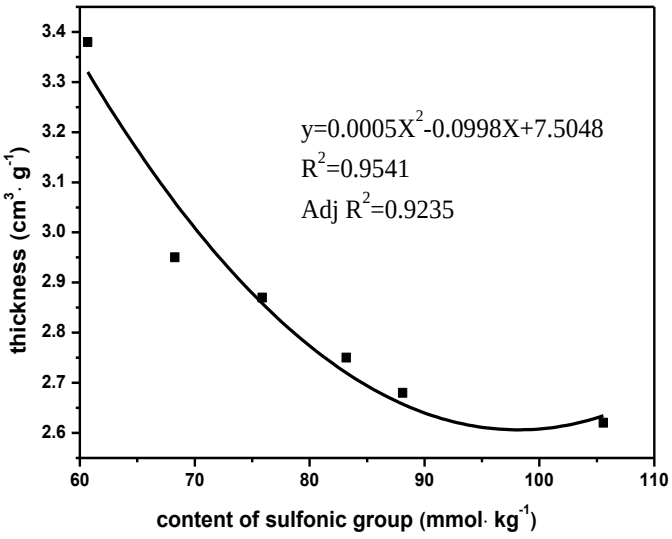


Fig. 4. Effect content of sulfonic group on bulk

As shown in Fig. 4, the bulk of SCMP pulp decreased with the increase of sulfonic group content. In Fig. 4, the nonlinear curve equation were nearly linear, because the quadratic term of X was only 0.0005. The R^2 and the Adj R^2 are higher than 0.9, indicating they fit well.

Table 2. Fiber Morphology of Mulberry Stalk and Mulberry Stalk SCMP

Property		Mulberry stalk	Mulberry stalk SCMP
Fiber length	Number average length (mm)	2.13	0.42
	Mass average length (mm)	2.85	0.66
	Double mass average (mm)	5.62	1.23
Fine fibers	Quantity content (%)	18.32	20.63
	Mass content (%)	3.58	4.25
	Fiber width (μm)	16.66	16.44
	Content of curliness (%)	7.6	8.3

Analysis of Fiber Morphology

The results for SCMP fiber morphology measured by fiber quality analyzer are shown in Table 2 and Figs. 5 and 6.

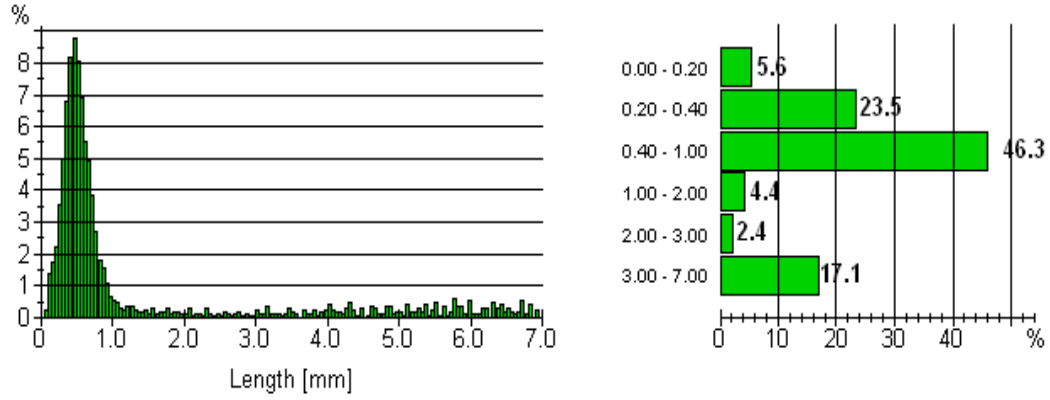


Fig. 5. The fiber length distribution of mulberry stalk

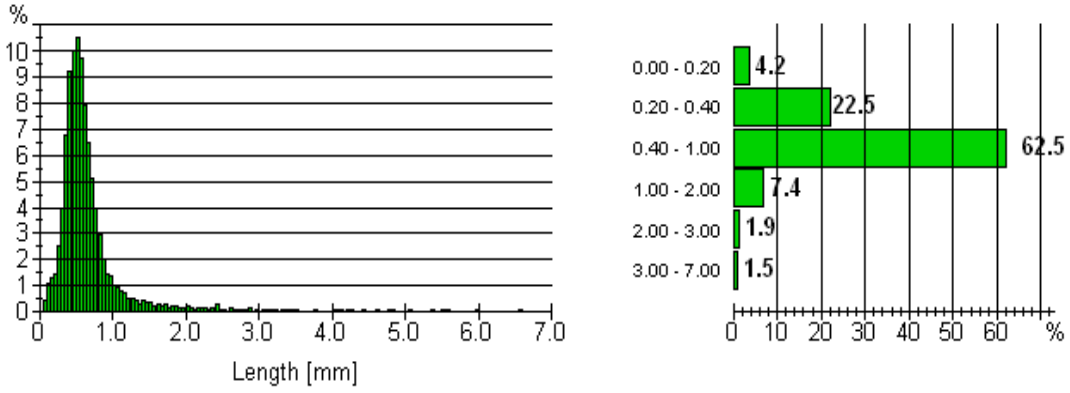


Fig. 6. The fiber length distribution of mulberry stalk SCMP

The average mulberry fiber length was 2.13 mm, and the width was 16.7 μm, which indicated that mulberry was a good raw material for papermaking (Pei 2014). The average mulberry stalk SCMP pulp length was lower than spruce, but higher than kenaf.

The average poplar CTMP pulp length was 0.68 mm and the width is 19.8 μm (Wang 2011), which was similar to mulberry SCMP pulp, but the width of the mulberry was lower than poplar. In addition, some longer fibers were present in mulberry SCMP pulp. This was due to the friction and shearing during the refining process; when the raw material was divided into individual fibers, the surface morphology and length were also affected (Xiao 2014).

The small size and large specific surface area of the fines affect the properties of papermaking (Li 2006). The content of fines in softwood and hardwood CTMP pulp was from 21.8% to 27.3%. The fines weight contents of spruce and aspen CTMP pulp are 4.65% and 6.07% respectively (Shi 1998). The amount of the mulberry stalk SCMP fines was 20.6%, which was lower than softwood and hardwood CTMP pulp, but the stalk SCMP was 4.25%, which was similar to spruce CTMP pulp.

FT-IR Analysis

The FT-IR test range was 4000 cm^{-1} to 400 cm^{-1}, and the wavelength was 2.5 μm to 25 μm. In this experiment, FT-IR analysis of mulberry and SCMP pulp was carried out to compare the absorption peaks of the chemical bonds and functional groups in the spectrum. The absorption peak of sulfonic group was in the region of 1210 cm^{-1} to 1150 cm^{-1} and 1060 cm^{-1} to 1030 cm^{-1} (Bellamy 1980). The absorption peak was at 1167 cm^{-1} and was deactivated by the stretching vibration of S = O, S-O, ester, *etc.* (Chen 1985). Figure 7 shows the absorption peak of sulfonic group at 1163.1 cm^{-1} and 1055.7 cm^{-1}, indicating the presence of sulfonic acid groups in SCMP pulp. The reaction of sulfonic groups and lignin increased the hydrophilicity of lignin. The absorption peak located at 1113.8 cm^{-1} was deactivated by stretching vibration of hydroxyl groups (OH) in cellulose and hemicellulose.

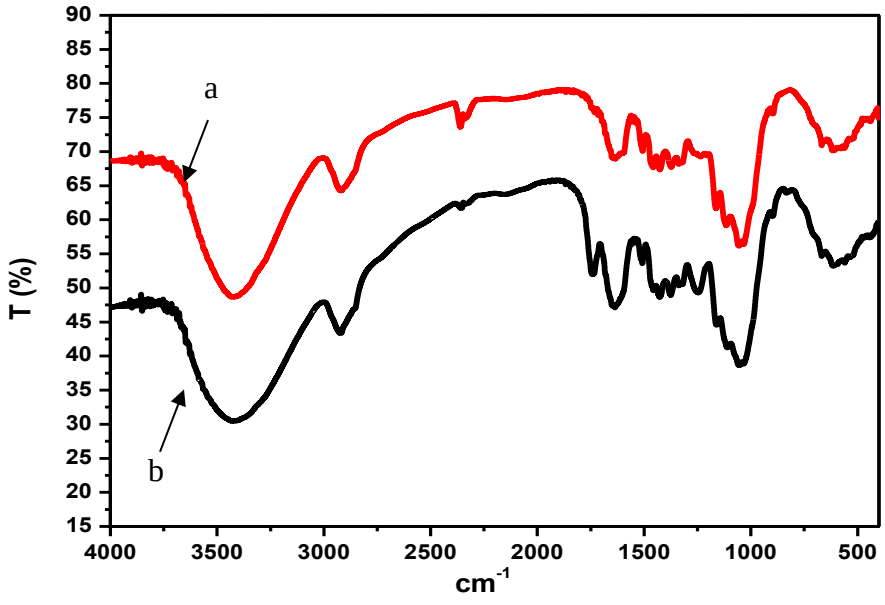
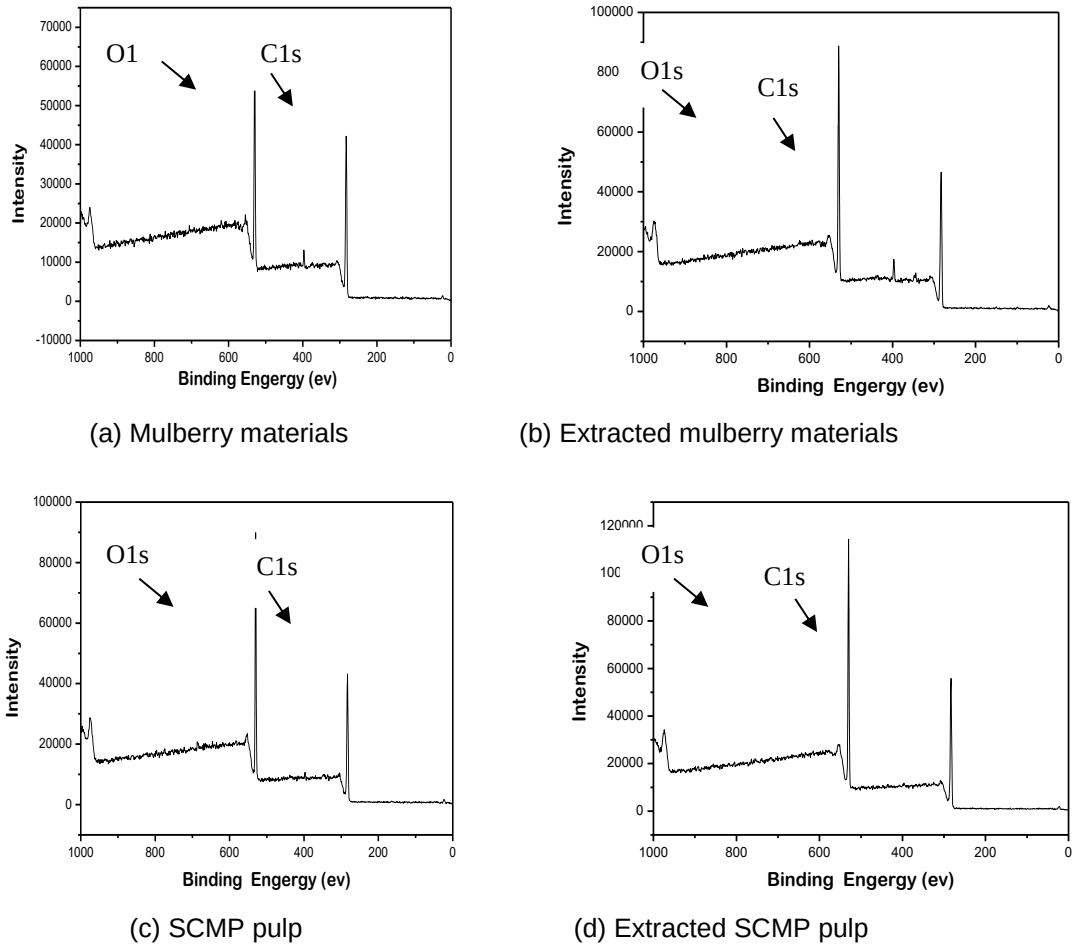


Fig. 7. FT-IR spectra of (a) mulberry stalk and (b) mulberry stalk SCMP



(a) Mulberry materials (b) Extracted mulberry materials

(c) SCMP pulp (d) Extracted SCMP pulp

Fig. 8. XPS Low-resolution spectrum of mulberry stalk

XPS Analysis

XPS spectrum of fiber surface

The main chemical components of mulberry stalk and mulberry stalk SCMP pulp are cellulose, hemicellulose, and lignin. Their fiber surface contains certain extracts, which mainly contain C, H, and O. The SCMP pulp may contain some S, but the XPS cannot check the content of H. As shown in the XPS low-resolution spectrum (Fig. 8), mainly the C_{1s} peak and O_{1s} peak were observed, and their binding energies were at 285 eV and 532 eV, respectively. The SCMP pulp was low in S content, which was difficult to detect in low-resolution XPS spectra.

Table 3. O/C Ratio of Mulberry Stalk and SCMP Fiber Surface

Samples	O (%)	C (%)	S (%)	N (%)	O/C
Mulberry stalk	32.84	64.41	0.39	2.36	0.51
Mulberry after extraction	39.18	56.66	0.19	3.97	0.69
SCMP pulp	38.72	57.75	0.78	2.76	0.67
SCMP pulp after extraction	39.58	56.62	1.16	2.63	0.70

Analysis of O / C ratio on fiber surface

A higher carbon content indicates that the fiber surface contained lignin and extractives. The O/C ratio can be used to explain the content of lignin and extractive on the fiber surface because the O/C ratio varies inversely relative to their content (Wang *et al.* 2009). The higher the O/C ratio, the higher content of carbohydrate was on the fiber surface.

The ratio of O/C on the surface of mulberry materials and SCMP pulp increased after acetone extraction in Table 3, which indicated that most of the mulberry extractives were removed by chemical pretreatment. The O/C ratio of SCMP pulp after extraction was very close to that of the SCMP pulp. It is believed that there was very little dissolution of lignin during the preparation of SCMP pulping treatment. Furthermore, the O/C ratio of SCMP pulp after extraction was 0.70, which was higher than the CTMP pulp after acetone extraction without washing, which is 0.624 (Koljonen *et al*. 2005).

As shown in Table 3, the content of S in SCMP pulp before and after extraction increased by 50.0% and 83.6%, respectively, relative to the mulberry materials. This indicated that the SCMP had introduced S in the form of sulfonic groups during the SCMP pulping treatment. Low levels of sulfonic groups were detected on the surface of CTMP pulp, S_2p = 0.20 to 0.30% (Koljonen *et al*. 2005). The sulfonate lignin had strong hydrophilicity, it was easy to remove from the fiber surface.

Analysis of lignin and extract on fiber surface

The benchmark of fiber surface's lignin and extract was the percentage of C1 instead of the O/C ratio. This is because the chemical change of O/C during chemical treatment ratio is more sensitive (Johansson 2002). The content of lignin and extract on fiber surface were estimated from Eqs. 2 and 3 (Koljonen *et al*. 2005),

$$\Phi \text{ lignin } = \frac{\text{C1}extracted\,pulp - \alpha}{49} \times 100\% \quad (2)$$

$$\Phi\, extractives = \frac{C1pulp - C1extracted\ pulp}{C1extractives - C1extracted\ \ pulp} \quad (3)$$

where *C1pulp* is the C1 percentage of the unextracted pulp; *C1extracted pulp* is the C1 percentage of the extracted pulp; 49 is the C1 percentage of the milled wood lignin; *C1extractives* is the C1 percentage of the extractives (usually 94%); and α is the measurement of C1 in pure fiber (usually 2%).

Table 4. Content of Lignin and Extractives of Mulberry Stalk and Mulberry Stalk SCMP Fiber Surface

Sample	Lignin Content (%)	Extractives Content (%)
Mulberry stalk	58.47	16.64
Mulberry stalk SCMP pulp	41.12	4.13

As shown in Table 4, the content of lignin and extractives in SCMP pulp was lower than in mulberry stalk. Part of the lignin was dissolved during chemical pre-treatment. Some extractives decomposed at an elevated temperature in an alkali environment. Li (2010) studied *Bambusa chungii* SCMP pulp by X-ray photoelectron spectroscopy (XPS) and found that the content of surface lignin was 57.4% and the content of surface

extractives were 7.6%. Koljonen *et al*. (2005) found that the surface extractives of spruce's PGW (pressure groundwood) and CTMP pulp was 13 to 17%, and it was reduced to 10 to 15% after cleaning with water. The content of surface lignin was 40 to 45% (Koljonen *et al*. 2005).

CONCLUSIONS

1. The brightness, tensile index, and folding resistance of mulberry SCMP pulp increased with the increasing concentration of sulfonation treatment, as indicated by the content of sulfonic groups. The thickness decreased with the content of sulfonic groups. Fitting equations of sulfonic groups and pulp physical properties (whiteness, tensile index, folding strength, and bulk) were obtained. The coefficient of determination for the fitting equation was greater than 0.9.
2. The average length of mulberry stalk fiber was 2.85 mm and the width was 16.7 μm. The weight average length of SCMP pulp was 0.66 mm, the fiber width was 16.4 μm, and the content of fine fibers was 20.6%. It was shown that mulberry stalk SCMP pulp has good qualities for papermaking.
3. The FTIR and XPS analyses of showed that mulberry extract and lignin dissolve, and part of soluble lignin might be deposited on the fiber surface in the process of SCMP pretreatment. At the same time, a sulfonic group was added to lignin with a sulfonated reaction. The results of XRD indicated that there were almost no changes to the cellulose crystalline structure during SCMP pretreatment.

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