Alkaline Peroxide Extrusion Pulping of Cotton Bast and Cotton Stalk

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The chemical compositions of cotton stalk and bast are similar to those of hardwood and superior to those of grass fiber. With respect to the morphological characteristics of their fibers, cotton stalk is similar to hardwood and cotton bast is similar to softwood. The average length of cotton bast fibers is nearly triple that of cotton stalk fibers, and the length-to-width ratio is almost quadruple. Traditionally, cotton stalk and cotton bast are pulped together, which affects the quality and homogeneity of the pulp and complicates bleaching, limiting its use to low-grade paper. In this study, cotton bast and cotton stalk were separated and pulped individually by alkaline peroxide extrusion pulping (APEP). The orthogonal analytical method was used to determine the optimal pulping parameters. Compared to those obtained *via* the kraft pulp (KP) of cotton stalk as a whole, far superior yield and whiteness were obtained in APEP. Further, with APEP, lower amounts of chemicals and less energy were consumed and there was little pollution. The physical performance of APEP was slightly lower than that of KP. With respect to bast alone, the physical performance of APEP was almost as good as that of KP.

Keywords: Cotton stalk; Cotton bast; Pulping; Screw extrusion

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INTRODUCTION

In recent decades, the pulp and papermaking industry has been criticized for its negative influence on the environment. Measures including the increased recycling of paper, more sustainable management of tree plantations, and a shift toward clean pulping technologies have been taken to solve these problems. Agricultural residues have become a raw material for pulp manufacture and may further contribute to the solution of some of these problems due to their abundance and low cost. Such strategies have already been applied to various agricultural crops available in China (Pang *et al.* 2012), Malaysia (Rosli *et al.* 2003), India (Dutt *et al.* 2008), Spain (Jiménez *et al.* 2009; Rodriguez *et al.* 2008a), Portugal (Cordeiro *et al.* 2004), Iran (Hedjazi *et al.* 2008), Tunisia (Gezguez *et al.* 2009), and Canada (Hosseinpour *et al.* 2010).

China is one of the world’s largest consumers of paper and fiber resources and relies greatly on imports. China is a traditionally agricultural country and produces vast amounts of agricultural by-products. Cotton is cultivated for textile production in China, and cotton stalks are abundant. In 2011, considering that each ton of cotton results in 3 tons of stalk, the Chinese cotton stalk output was approximately 22 million tons. Cotton stalk is a concentrated and cheap resource with industrial value as a papermaking material. Thus, the thorough use of cotton stalk is a major issue for both the paper industry and the Chinese national economy. The methods used to pulp cotton stalk are typically chemical, (Gabir and Khristova 1983; Liang *et al*. 2007; Pandey and Shaikh 1987; Wang *et al*. 2005), biological-mechanical (Ardon *et al*. 1996; Hardar *et al*. 1992; Zheng *et al*. 2003), or chemical-mechanical (Cheng and Luo 1994; Liu *et al*. 2005; Tang *et al*. 2005). The pulp is used to produce corrugated base paper, cardboard, and some printing paper.

The chemi-mechanical pulping process involves a mild chemical treatment with a high pulping yield compared to that of the chemical pulping process. Additionally, a lower refining energy is required in the chemi-mechanical pulping process than in mechanical pulping. The recent development of alkaline peroxide mechanical pulp (APMP) and preconditioning refiner chemical (PRC)-APMP for cotton stalk pulp can produce pulps with better whiteness and strength. The outer, dark skin of the cotton stalks adversely effects bleaching and the quality of the pulp. The pulp is often too dusty for use in the manufacture of high-quality paper (Wang *et al*. 2007; Zhu *et al*. 2008). Therefore, a rational, innovative, environmental-friendly pulping method is needed to make the best use of cotton stalk.

Clean pulping with high yield, high quality, low energy consumption, and low pollution has been the focus of numerous scientific works (Caparrós *et al.* 2008; Pang *et al.* 2012; Rodriguez *et al.* 2008b; Sampedro *et al.* 2012). Environmental consciousness is continuously increasing. Developing pulping methods for fibrous agricultural materials, adopting clean pulping technologies, and reducing pollution are effective ways to overcome the current issues of the paper industry and meet environmental protection requirements.

In this study, a new pulping method, alkaline peroxide extrusion pulping (APEP), is introduced. Alkaline peroxide extrusion pulping is similar to APMP and uses a screw extruder as the refining machine with sodium hydroxide and peroxide as its primary pulping chemicals. The objective of this study was to determine the chemical compositions of cotton stalk and cotton bast and their APEP properties. The cotton stem was separated into three fractions (cotton stalk, cotton bast, and outer dark skin) by a special machine. The materials were pretreated with various dosages of sodium hydroxide and peroxide. Then, the treated materials were refined by a screw extruder, and the pulp's properties were systematically evaluated and compared to those of KP, as reported in other literature. This investigation shows the potential of using cotton stalk for the production of APEP. This method combats the disadvantages of traditional pulping technology by achieving higher yield and better performance at lower chemical dosages and energy consumption.

EXPERIMENTAL

Materials

Cotton stalk was obtained from a farm in China. Leaves and other unwanted material were removed from the stem. The cotton stem was divided into cotton stalk, cotton bast, and outer dark skin fractions. Cotton bast accounts for about 25 to 29% of whole cotton stem. A debarker and shredder were then used. The cotton stalk was cut into sticks ranging in length from 10 to 30 mm and in width from 2 to 10 mm, air-dried, and stored at room temperature. The cotton bast was threadlike and fluffy after the outer, dark skin had been removed. The raw materials were analysed for hot water solubles (HWS), 1% soda solubles (1SS), ethanol–benzene extractables (EBE), ash, cellulose, lignin, hemicellulose (HEM), and pectin in accordance with the applicable TAPPI standards T-207cm-08, T-212 om-07, T204 cm-07, T211 om-93, T-203 0S-61, and T-222 om-06, respectively (TAPPI Standards, 1997).

Pulping Procedure

The fibrous material was immersed in water and steamed for 5 to 10 min in a steaming chamber. In the chemical pretreatment stage, liquids including NaOH, H_2O_2, $MgSO_4$, Na_2SiO_3, and EDTA were added to reactor with the fibrous material and the mixture was heated to a fixed temperature for a specified time. The liquor-to-fiber ratio was 6:1. The blended materials were added to an extruder for pulping and refining.

Extrusion Pulping Principle

Extrusion pulping involves the use of a co-rotating twin screw extruder as the main pulping device. Figure 1 shows the basic operating principle of the extruder.

The pulping extruder consisted of an “8”-shaped barrel with two co-rotating, intermeshing screws inside. Pretreated fiber was fed into the barrel by transport screws. The main screw element used in the extrusion pulping processes was the reversed screw element (RSE) (see Fig. 2a).

There was a thread on the RSE whose pitch was opposite to the transport screws. This results in accumulation and compression of the fibers in the space between the transport screws and the RSEs. The high compressive and shear forces cause defibration, fibrillation, and shortening of fibers. Excess water pressed from the fiber mass is extracted through barrel filters placed upstream of the RSE. The pressure drop created in passing the RSE heats the pulp and allows for rapid impregnation of liquids. Cooking liquids are supplied through an injection port downstream of the RSE. Machined slots, through which the fibers could eventually pass forward, are regularly distributed throughout the threads of the RSE. The combination of a transport screw, RSE, filter, and injection port could be repeated along the barrel. A combination of a subsequent transport screw, RSE, and transport screw is shown in Fig. 2b. The cutaway view of the screw extruder used to extrude the cotton bast pulp is shown in Fig. 2c.

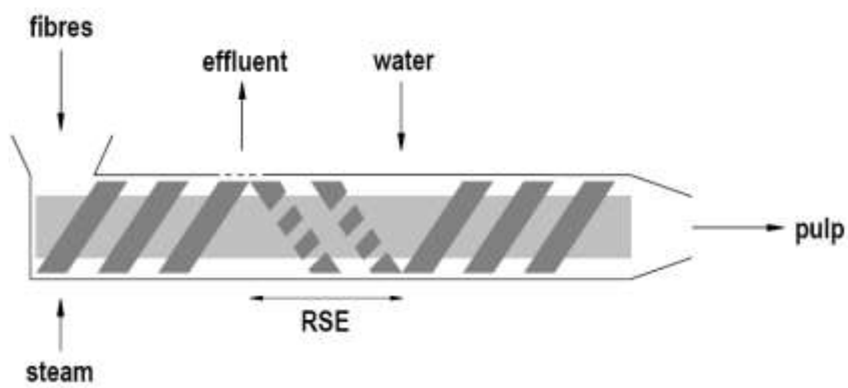

Fig. 1. Side view of the Bivis extruder

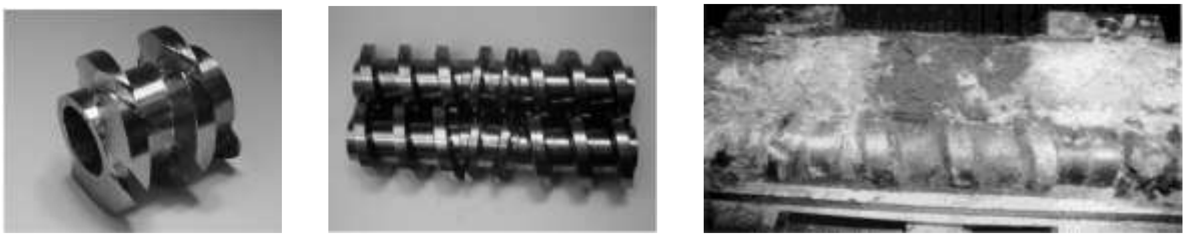

Fig. 2. Reversed screw element and intermeshing transport screws

Paper Sheet Formation and Characterization

In each trial, a set of paper sheets (80 g/m^2) were made from each pulp sample according to TAPPI T 205 and kept overnight in a conditioning room in accordance with TAPPI T 402. The paper sheets obtained from the pulp were tested for brightness (TAPPI T 452 om-08, Brightness of pulp, paper, and paperboard), yield (TAPPI T 257 cm-12, Sampling and Preparing Wood for Analysis), kappa number (TAPPI standards T203 cm-99), tensile index (TAPPI T 494 om-13, Tensile properties of paper and paperboard), tear index (TAPPI T 414 om-12, Internal tearing resistance of paper), and burst index (TAPPI T 403 om-10, Bursting strength of paper).

RESULTS AND DISCUSSION

Characterization of Raw Materials

Cotton stem is composed of various fractions (skin, xylem, and pith), and the skin is composed of the outer skin and the bast. In this work, the outer skin and pith were removed, and the cotton stalk and bast were prepared individually. The proportion of the cotton bast is about 25% of the whole cotton stalk. Table 1 shows the results of the chemical analyses of the cotton stalk and bast. The morphological characteristics of each are listed in Table 2. Some conventional short-fiber materials are described in Tables 1 and 2 for comparison.

Table 1. Chemical Analyses of Cotton Stalk and Cotton Bast Compared to those of Some Common Fibrous Materials

Material	HWS (%)	1SS (%)	EBE (%)	Ash (%)	HEM (%)	Cellulose (%)	Lignin (%)	Pectin (%)
Cotton stalk	3.8	20.34	1.66	2.17	18.34	41.43	21.11	3.35
Cotton bast	10.21	37.82	3.07	5.32	16.42	35.83	15.38	3.01
Aspen	2.46	15.61	1.94	0.32	22.61	42.24	17.1	1.76
Eucalyptus	3.3	12.67	1.98	0.29	27.7	46.85	18.06	--
Wheat straw	11.53	42.59	4.37	5.97	24.04	39.72	18.12	--

HWS = Hot water-solubles; 1SS = 1% soda-solubles; EBE = Ethanol-benzene-extractables; HEM = Hemicellulose.

Table 2. Morphological Characteristics of Cotton Bast and Cotton Stalk Fibers Compared to those of with Some Common Fibrous Materials

Material	Fiber Length (mm)	Average Length (mm)	Fiber Width (μm)	Average Width (μm)	Length/Width Ratio
Cotton bast	1.44 to 3.50	2.26	15.7 to 22.9	20.6	113
Cotton stalk	0.63 to 0.98	0.83	21.6 to 34.3	27.7	30
Aspen	0.65 to 1.14	0.86	14.7 to 23.5	17.4	50
Eucalyptus	0.55 to 0.79	0.68	13.2 to 18.3	16.8	43
Wheat straw	1.03 to 1.60	1.32	9.3 to 15.7	12.9	102
Spruce	1.84 to 4.05	3.06	39.2 to 68.6	51.9	59

As shown in Table 1, the contents of the three main components (cellulose, hemicellulose, and lignin) of cotton stalk and bast were similar those of hardwood, while the contents of solubles and ash were higher than in hardwood and lower than in wheat straw. The pectin content of cotton stalk and bast was the highest of any of the listed materials. Thus, cotton stalk and bast are both good materials for pulping and are superior to grass fiber.

Table 2 reveals a large difference between the morphological characteristics of cotton stalk and bast fibers. The fiber morphological characteristics of cotton stalk were similar to those of hardwood fibers, whereas those of cotton bast were similar to those of softwood fibers. The results were consistent with the reports of other literature (Jiménez *et al.* 2007; Li 2007). The average length of cotton bast was nearly triple that of cotton stalk, and the length-to-width ratio was almost quadruple. Based on the differences in both morphology and chemical composition, different parts of the cotton stalk must be separated and processed separately to be used efficiently.

Orthogonal Experimental Design and Analysis of Cotton Stalk Pulping

The cotton stalk APEP was studied using an orthogonal experimental design L9(3^4). Four major factors (hydroxide dosage, hydrogen peroxide dosage, temperature, and pretreatment time) were investigated. Table 3 shows the values of the operational variables and the characterizations of the resulting pulp. The notable differences are shown in Table 4.

Table 3. Operational Variable Values and the Characterization of the Resulting Cotton Stalk Pulp

No.	NaOH (%)	H_2O_2 (%)	Temperature (°C)	Time (min)	Yield (%)	Kappa Number	Tensile Index ($N·m^2/g$)	Tear Index ($mN·m^2/g$)
1	4	0	70	60	92.1	35.5	21.3	10.52
2	4	1	80	90	85.3	30.1	29.4	9.44
3	4	2	90	120	79.4	27.8	35.2	8.56
4	6	0	90	90	78.5	23.8	54.5	8.30
5	6	1	70	120	85.2	26.7	38.6	9.34
6	6	2	80	60	81.5	24.7	49.3	8.74
7	8	0	80	120	80.7	17.9	53.2	8.52
8	8	1	90	60	76.1	18.1	56.7	7.52
9	8	2	70	90	83.3	19.4	47.3	8.90

Table 4. Results of Range Analysis of Cotton Stalk Pulp

Item	Yield (%)				Kappa Number			
	A	B	C	D	A	B	C	D
I/3	85.6	83.8	86.9	83.2	31.1	25.7	27.2	26.1
II/3	81.7	82.2	82.5	82.4	25.0	25.0	24.2	24.5
III/3	80.0	81.4	78.0	81.8	18.5	24.0	23.2	24.1
R	5.57	2.37	8.87	1.47	12.61	1.72	3.92	2.02
Item	Tensile Index ($N·m^2/g$)				Tear Index ($mN·m^2/g$)			
	A	B	C	D	A	B	C	D
I/3	28.6	43.0	35.7	42.4	5.5	9.1	8.6	8.9
II/3	47.5	41.6	44.0	43.7	4.8	8.8	8.9	8.9
III/3	52.4	43.9	48.8	42.3	4.3	8.7	8.1	8.8
R	23.77	0.93	13.07	0.10	1.19	0.38	1.46	0.12

As shown in Tables 3 and 4, the most significant factor affecting both the yield and the Kappa number was temperature, followed by the NaOH charge. Both yield and Kappa number decreased rapidly with increasing temperature and NaOH charge. The significant factors affecting the tensile index were the NaOH charge and the temperature. The optimal NaOH charge was 8%. The more NaOH used, the higher the value of the tensile index; however, the yield decreased. Considering yield, Kappa number, and tensile index, an 8% NaOH charge was deemed the optimal condition. The tensile index increased rapidly with temperature throughout the range tested. The optimal temperature tested was 90 °C. The time and amount of H_2O_2 used were minor factors. The performance was best when the time was 90 min and the amount of H_2O_2 was 2%. Thus, the optimal scheme for cotton stalk pulping was $A_3B_3C_3D_2$.

Orthogonal Experimental Design and Analysis of Cotton Bast Pulping

Cotton bast APEP was also studied using an orthogonal experimental design $L9(3^4)$. Table 5 shows the values of the operational variables and the characterization of the resulting pulp. The notable differences are shown in Table 6.

Table 5. Values of the Operational Variables and the Characterization of the Resulting Cotton Bast Pulp

No.	NaOH (%)	H_2O_2 (%)	Temperature (°C)	Time (min)	Yield (%)	Kappa Number	Tensile Index ($N·m^2/g$)	Tear Index ($mN·m^2/g$)
1	4	0	70	80	92	37.48	39.2	17.04
2	4	1	80	100	85	32.09	47.04	14.88
3	4	2	90	120	79	29.78	52.92	13.12
4	6	0	90	100	78	25.84	71.54	12.6
5	6	1	70	120	85	28.67	56.84	14.68
6	6	2	80	80	81	26.72	67.62	13.48
7	8	0	80	120	80	19.85	75.46	13.04
8	8	1	90	80	76	20.13	73.5	11.04
9	8	2	70	100	83	21.42	63.7	13.8

Table 6. Results of Range Analysis of Cotton Bast Pulp

Item	Yield (%)				Kappa Number			
	A	B	C	D	A	B	C	D
I/3	85.3	83.3	86.7	83.3	33.12	27.72	29.19	28.11
II/3	81.7	82	82.3	82	27.08	26.96	26.22	26.45
III/3	79.7	81.3	77.7	81.4	20.47	25.97	25.25	26.1
R	5.6	2.0	9.0	1.9	12.55	1.75	3.94	2.01
Item	Tear Index (mN·m^2/g)				Tensile Index (N·m^2/g)			
	A	B	C	D	A	B	C	D
I/3	15.02	14.22	15.18	13.86	36.55	58.80	53.21	60.07
II/3	13.6	13.54	13.8	13.76	55.57	59.09	60.17	60.76
III/3	12.6	13.48	12.26	13.6	57.82	61.45	65.95	58.51
R	2.42	0.74	2.92	0.26	21.27	2.65	12.74	2.25

Table 6 shows that the most significant factors affecting the yield and Kappa number were the temperature and NaOH charge. Both yield and Kappa number decreased rapidly with increasing temperature and NaOH charge. The time and the H_2O_2 charge were minor factors. The performance of the pulping was best when the time was 100 min and H_2O_2 charge was 2%. The significant factors affecting breaking length were the NaOH charge and the temperature. The NaOH charge was 8%. The more NaOH used, the greater the decrease in yield. With a 6% NaOH charge, the tensile index was not as high as that achieved with an 8% NaOH charge, but the pulping performance was similar otherwise. Considering yield, Kappa number, and tear index, a 6% NaOH charge was determined to be optimal. The tensile index rapidly increased with temperature throughout the range of temperatures tested, but considering all factors, the optimal temperature was 80 °C. Thus, the optimal scheme for cotton bast pulping was $A_2B_3C_2D_2$.

Verifying Experiment and Comparison with KP

To validate the optimum pulping conditions determined by the orthogonal experiments, a verifying experiment was carried out. Cotton stalk and bast were pretreated under the determined optimal conditions. The data obtained were compared with those of kraft pulp (KP). The KP operating conditions for cotton bast were a 25% NaOH charge, 15% sulfidity, a temperature of 160 °C, a 2-h heat preservation time, and a 5:1 liquid-to-solid ratio. The KP operating conditions for the whole cotton stalk were an 18% NaOH charge, 20% sulfidity, a temperature of 165 °C, a 150-min heat preservation time, and a 5:1 liquid-to-solid ratio (Liang *et al.* 2007). The performance of pulp samples were compared in terms of their main physical indexes. The results are shown in Table 7.

Table 7. Values of Validating Experiment Compared with KP Method

Sample	Pulping Method	Yield (%)	Whiteness (%)	Tensile Index (N·m^2/g)	Tear Index (mN·m^2/g)	Burst Index (kPa·m^2/g)
Whole cotton stalk	KP	43.5	19	62.3	11.1	5.13
Cotton stalk	APEP	79.3	52	53.21	9.2	3.3
Cotton bast	KP	54.63	22	70.3	15.26	4.17
	APEP	82.21	48	75.3	13.53	4.65

As shown in Table 7, pulp from the APEP process was produced with a much higher yield and whiteness, with lesser chemical dosages, and lower energy consumption, than KP. For cotton stalk, the physical performance of APEP was slightly lower than that of the KP of whole cotton stalk, because the cotton stalk fibers were short. With regard to cotton bast, the physical performance of the APEP was nearly as good as that of the KP of the whole cotton stalk and cotton bast, except in the case of the tear index. Cotton bast and cotton stalk separated and individually pulped *via* the APEP method were superior to whole cotton stalk pulped by the traditional method.

CONCLUSIONS

1. The optimal cotton stalk pulping scheme was an 8% NaOH charge, a 2% H_2O_2 charge, a temperature of 90 °C, and a duration of 90 min. That for cotton bast pulping was a 6% NaOH charge, a 2% H_2O_2 charge, a temperature of 80 °C, and a duration of 100 min. Because cotton bast was threadlike and fluffy, it could absorb chemicals more easily than cotton stalk, requiring lower dosages of chemicals and reacting at lower temperatures.
2. Compared to the kraft process (KP) and pulping of whole cotton stalks, a great improvement in the yield and whiteness with lesser chemical dosages, energy consumption, and pollution were achieved with alkaline peroxide extrusion pulping (APEP). The physical performance of cotton stalk APEP was slightly lower than that of whole cotton stalk KP because the fibers of the cotton stalk were short. With respect to cotton bast, the physical performance of APEP was almost as good as that of KP pulp.
3. Extrusion pulping is a technically and economically viable process for the chemi-mechanical pulping of non-wood fibers. It provided a much higher yield (on the order of 80%) with much lower chemical usage. Moreover, extrusion pulping also allowed cutting of the fibers to a desired length, such that the resulting pulps could be more easily handled by bulk papermaking systems.

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