

# CHARACTERIZATION AND EVALUATION OF OKRA FIBRE (*ABELMOSCHUS ESCULENTUS*) AS A PULPING RAW MATERIAL

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ABSTRAC

Increasing concerns for future fibre supplies and potential increases in wood cost have strengthened the pulp and paper industry's interest in alternative pulp fibre sources, i.e., agricultural wastes. Among these, the okra plant (*Abelmoschus esculentus*) is an excellent candidate because of its high crop yield. The okra plant consists of the stick (woody portion) and the fibre (bast). Okra fibre is characterized by high  $\alpha$ -cellulose (56.7%) and low lignin (12.6%) content and the stick by low  $\alpha$ -cellulose (34.3%) and high lignin (25.2%) content. The okra fibre is longer (3.0 mm), while stick fibre is shorter (0.63 mm). Soda-anthraquinone (AQ) and Kraft pulping of okra plants was carried out with varying chemical charges and cooking times. The Kraft process showed better pulp yield and delignification than the soda-AQ process. Kraft pulp also exhibited superior physical properties to those of soda-AQ pulp. Using equivalent bleaching chemicals, the Kraft pulp reached 82.1% brightness, while the soda-AQ pulp reached 76.8% brightness. Both pulps exhibited similar papermaking properties after bleaching.

# **INTRODUCTION**

Paper consumption is continuously increasing in the developing world, even in countries where wood resources are very limited. The acute shortage and high cost of wood-based raw materials is considered to be the most important factor restricting the growth of the Bangladeshi paper industry and its development into a globally competitive industry (1). Conditions are similar in many other regions of the world. Bangladesh is a densely populated, forest-deficient country which is mainly dependent on agriculture. Each year, a huge quantity of agricultural wastes is generated, which may be a potential substitute for forest-based wood. Furthermore, economic utilization of agricultural residues in rational and innovative ways is of prime interest in such countries. Such strategies have already been used to obtain full economic value from various agricultural crops available in different countries (2–7).

In Bangladesh, many studies have been carried out to realize economic value from these agricultural wastes, as well as



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to overcome the shortage of wood resources. Jahan et al. (8,9) produced chemical pulp from dhaincha in the laboratory and showed that a good-quality pulp can be produced from this source for packaging-grade paper. Cotton stalks have also proven to be a good raw material for producing writing- and printing-quality paper (10). Pulps from jute mill wastes have also shown potential as a softwood wood pulp substitute (11).

This research has investigated pulping of the okra plant (*Abelmoschus esculentus*), which is one of the major generators of vegetable wastes in Bangladesh.

Okra is an annual or perennial plant, growing to 2 m tall. It is related to such species as cotton, cocoa, and hibiscus. The leaves are 10–20 cm long and broad, palmately lobed with 5–7 lobes. It is cultivated throughout the tropical and warm temperate regions of the world for its fibrous fruits or pods containing round, white seeds. It is among the most heatand drought-tolerant vegetable species in the world (but severe frost can damage the pods) and will tolerate poor soils with heavy clay and intermittent moisture.

The okra plant consists of a short fibre stalk and a long fibre bast, similarly to the jute plant. The ratio of stalk to bast is 1.5:1. In fine papers, both short and long fibres are needed. In fine papers, shortfibre pulp contributes to good printability. No report on the fibre properties of the okra plant has been found in the literature.

Therefore, in this investigation, okra stalk fibre and bast fibre were characterized to evaluate them as raw materials for pulping. A pulping study was carried out using the soda-anthraquinone (AQ) and Kraft processes with varying cooking times and alkali charges. The pulp produced was bleached by a  $D_0E_pD_1$  bleaching sequence, and finally its papermaking properties were evaluated.

# MATERIALS AND METHODS

# **Materials**

Okra plants were collected from Savar, Dhaka, and were chopped to 2–3 cm in length for subsequent cooking experiments. For chemical analysis, the okra plants were retted for one month, and then the fibres and sticks were separated. Subsequently, the sticks and fibres were separately ground in a Willey mill and screened to 40/60 mesh.

## **Chemical Analysis**

The chemical compositions of the sticks and fibres were determined according to the following TAPPI test methods: extractives (T204 om88), water solubility (T207 cm99), and Klason lignin (T211 om83). Holocellulose samples were prepared by treating extractives-free stick or fibre meal with NaClO<sub>2</sub> solution (12). The pH of the solution was maintained at 4 by adding CH<sub>3</sub>COOH-CH<sub>3</sub>COONa buffer, and the  $\alpha$ -cellulose content was determined by treating holocellulose with 17.5% NaOH (T203 om 93).

### Pulping

Pulping of okra plants was carried out by the soda-anthraquinone (AQ) and Kraft processes in an electrically heated oil bath containing four bombs of 1.5-L capacity. The bombs were rotated at 1 rpm. In the soda-AQ process, the alkali charge was varied from 14%, 16%, and 18%, to 20% on o.d. raw materials at cooking times of 1, 2, and 3 h. In the Kraft process, the cooking time was fixed at 2 h. For evaluation of papermaking properties, okra plant pulps of almost identical kappa number were cooked in a 20-L capacity digester. The following parameters were kept constant:

-Anthraquinone charge: 0.1 % on o.d. raw material
-Fibre-to-liquor ratio: 1:5 g/mL.
-Temperature: 170°C.
-Sulphidity: 30% (for Kraft cooking).

After digestion, the pulp was washed until free from residual chemicals and screened in a flat vibratory screener (Yasuda, Japan). The pulp yield and screened rejects were determined gravimetrically as percentages of oven-dry raw materials.

### **Evaluation of Pulps**

Okra pulps were beaten in a Valley beater for different times. Handsheets of approximately 60g/m<sup>2</sup> were made in a Rapid Kothen sheet-making machine. The sheets were tested for tensile, burst, and tear strength according to TAPPI Standard Test Methods.

#### Bleaching

The pulp obtained from okra plants in the Kraft and soda-AQ processes was bleached by a  $D_0E_pD_1$  bleaching sequence. The ClO<sub>2</sub> charge was 2%, and the temperature was 70°C for 60 min in the  $D_0$  stage. The pH was adjusted to 2.5 by adding dilute  $H_2SO_4$ . In the alkaline extraction stage, the temperature was 70°C for 60 min, and the NaOH and  $H_2O_2$ charges were 2% and 0.5% respectively. In the final  $D_1$  stage, the ClO<sub>2</sub> charge was 1%, and the pH was adjusted to 4 by adding dilute  $H_2SO_4$ . The sheets of bleached pulp were tested for density, tensile, burst, and tear strength according to TAPPI Standard Test Methods.

# **RESULTS AND DISCUSSION**

## **Chemical Characteristics**

Table 1 shows the chemical characteristics of retted okra bast fibre and stalk fibre. The holocellulose and *a*-cellulose content are higher and the lignin content is lower in the bast fibre than in the okra sticks. Similar results have been observed for jute sticks and jute fibre (13). The holocellulose content in bast fibre is higher than in wood (14), but holocellulose in the stick fibres is at similar levels to wood and higher than in other non-wood fibres (15). The  $\alpha$ -cellulose content is related to higher pulp yield. The  $\alpha$ -cellulose content in bast fibre was 56.7%, which is higher than in wood and other non-woods (14), but lower than in jute bast fibre (11). Okra stalk fibre contains much lower  $\alpha$ -cellulose (34.4%) than bast fibre, even less than straw (16). The pentosan content of the bast fibre was 17.8%, while in the stalk fibre, it was 16.9%. These values are lower than in non-woods and hardwood (15), but close to the values for softwood (14).

Low lignin content is always desirable

TABLE 1Chemical and morphological characteristics of stick and bast fibre of okra plants.						
	Stick	Bast Fibre				
Holocellulose (%)	70.6	85.9				
α-cellulose (%)	34.3	56.7				
Lignin (%)	25.2	12.6				
Pentosan (%)	16.9	17.8				
1% alkali solubility (%)	29.2	17.3				
Extractives (acetone) (%)	2.92	1.5				
Ash (%)	0.6	2.0				
Fibre length, mm	0.63	3.0				
Fibre width, µm	15.4	13.4				

for chemical pulping. It is evident from Table 1 that the lignin content in the bast fibre is distinctly lower than in the okra stalk fibre. The lignin content of bast fibre is similar to that of jute fibre (11), while the lignin content of okra sticks is similar to that of common hardwoods (8) and other non-wood fibres (10). The okra stalk fibre lignin content of 25.2% is almost identical to that found in mature poplar chips (25.2%) (17).

The acetone extracts content of bast fibre is lower than in agricultural residues. Some substances, including resins, wax, fat, and ethanol–benzene extracts, can precipitate upon pulping and leave stains in the resulting paper sheets. The low acetone extracts content of 1.5 wt% suggests that the proportion of these compounds in the raw material is low. On the other hand, the acetone extracts content of okra sticks is higher than that of bast fibre. The extractives in the fibre are almost similar to those in jute, kenaf, and hemp bast fibre (18).

In the bast fibre, 1% alkali solubility is only 17.3%, while in the stick fibre, it is 29.2%. Based on their moderate content of 1% NaOH solubles, okra plants can be expected to provide a medium pulp yield. This solubles content is lower than that of agricultural residues (19) and also lower than those of pine and eucalyptus.

The ash content of okra bast fibre is higher than that of stalk fibre (2.3% vs. 0.6%). High ash content can cause problems in chemical recovery. Fortunately,



Fig. 1 - Photograph of okra-plant pulp.

both stalk fibre and bast fibre have lower ash content than rice straw, switchgrass, or alfalfa (16,17). Stalk fibre ash content is very similar to that of mature aspen (0.5%) (17).

# **Morphological Characteristics**

Fibre length is recognized as an important parameter for pulp and paper properties. Fibre length influences paper strength, particularly tear and paper machine runnability (20,21). Table 1 shows the lengths of stalk and bast fibre of the okra plant. The average length of bast fibre is definitely greater (3.0 mm) than that of stick fibre (0.63 mm). Stalk fibre is similar to the shorter range of tropical hardwoods (0.7-1.5 mm), which are considered as short fibre (22). Stalk fibres, with a length of 0.63 mm, are close to, but slightly shorter than, the switchgrass fibre length of 0.88-1.0 mm (23). Bast fibres are even longer than jute fibres (11).

The microscopic structure of okra pulp fibres is shown in Fig. 1. It can be seen that most of the anatomical elements of the okra plant are long, narrow, thinand thick-walled fibres. Because this pulp is derived from the okra plant, which includes both stick and bast fibres, a variety of characteristic cell types is evident in the micrograph, including short fibres and vessels from the stick.

## Pulping

Several cooks were carried out to

TABLE 2	Effect of chemic	cal charge and coo	king time on p	oulping of okra plar	nts.	
Process	Alkali charge (%)	Time at 170°C (h)	Screened	Pulp yield (%) Rejects	Total	Kappa number
Soda-AQ	14	1	1.7	59.2	64.3	-
	16	1	17.0	29.0	46.0	37.3
	18	1	29.6	9.8	39.4	33.6
	20	1	30.4	7.1	37.5	29.5
	14	2	4.5	55.5	60.0	-
	16	2	18.0	27.4	44.4	37.2
	18	2	30.6	7.8	38.4	32.8
	20	2	32.2	5.5	37.7	28.2
	14	3	19.8	34.7	54.5	41.2
	16	3	31.4	11.1	42.5	35.2
	18	3	32.5	5.5	38.0	32.3
	20	3	32.6	4.3	36.9	27.4
Kroft	14	2	39.0	6.4	45.4	40.8
	16	2	38.8	3.4	42.2	30.4
Nat	18	2	38.9	1.8	40.7	25.2
	20	2	37.6	0.8	38.4	22.2

determine suitable soda-AQ and Kraft pulping conditions for the okra plant. The okra plant consists of bast fibres and sticks. The data presented in Table 2 show that increasing cooking time and alkali charge led to decreased total pulp yield, as expected. With increasing cooking time and alkali charge, screened yield is increased, and rejects are decreased. The okra plant is not cooked thoroughly, even under drastic conditions. At 20% alkali charge and 3 h cooking, the okra plant produces a screened pulp yield of 32.6% with kappa number 27.4, while screen rejects are 4.3%. The maximum screened yield is obtained under this condition. Therefore, it can be concluded that the soda-AQ process is not suitable for okraplant cooking.

Kraft cooking of okra plants was carried out at varying active alkali levels. Total pulp yield decreased from 45.4% to 38.4% and kappa number from 40.8 to 22.2 with an increase in active alkali from 14% to 20%, but still 0.8% rejects remained. The total pulp yield obtained with varying alkali charges in 3 h of cooking in the soda-AQ process was compared with the pulp yield obtained from the Kraft process in 2 h of cooking. It can be clearly seen in Fig. 2 that the pulp yield in the Kraft process is markedly better than that in the soda-AQ process within the kappa range of 20-35. At kappa number 27, the Kraft process produces 5% higher pulp yield than the



soda-AQ process. Usually, the soda-AQ process is suitable for non-wood pulping because of the relatively low average molecular weight of lignin (9,24). However, in this study, better pulping selectivity for the okra plant was observed in the Kraft process. This may be explained by the morphological properties and chemistry of the okra plant. Saka et al. (25) found that the addition of AQ and sodium sulphide resulted in a transition from a slow initial to a rapid bulk delignification, particularly in the middle lamella, and in an enhanced bulk delignification in the secondary wall. However, sodium sulphide addition significantly improved bulk delignification in the middle lamella.

# **Physical Properties**

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The physical properties of okra-plant pulp obtained from the soda-AQ and Kraft processes are shown in Table 3. The sheet density, tensile index, and burst index are increased, and the tear index is decreased with increasing °SR value. The tensile index and burst index are a reflection of fibre-to-fibre bonding, and the tear index also depends strongly on fibre length. In the soda-AQ process, the tensile index is increased from 40 N.m/g to 76 N.m/g with increasing °SR value from 23 to 50; further increases in °SR decrease the tensile index slightly. Figure 3 compares the tensile-tear properties of soda-AQ and Kraft pulps. Kraft pulp shows superior physical properties compared to soda-AQ pulp. From the papermaker's point of view, the tear-tensile relationship is one of the most important properties of pulp. Figure 3 shows a clear superiority in the tensile-tear relationship of Kraft pulp. At a tensile index of 76N.m/g, the tear index of Kraft pulp was 8% higher than that of soda-AQ pulp. The tensile-tear strength was even better than for jute-plant pulp (14). The properties are similar to or better than those of hardwood pulp (26).

The tear index of okra plant pulp is much higher than those of switchgrass and alfalfa fibre (17). This higher tear index is contributed by the longer okra bast fibre. Khristova et al. (27) also observed a much higher tear index for kenaf bast fibre than for core fibre.

#### **Bleaching**

Soda-AQ pulp with kappa number 27 and Kraft pulp with kappa number 25 were bleached by a  $D_0 E_p D_1$  bleaching sequence. At the same ClO2 consumption, Kraft pulp showed better bleachability than soda-AQ pulp. Using equivalent bleaching chemicals, the Kraft pulp reached 82.1% ISO brightness, while the soda-AQ pulp reached 76.8% ISO brightness. This may be attributed to the higher initial kappa number of the unbleached soda-AQ pulp. Bleachability tends to decrease as the kappa number increases (28). As the brownstock kappa number increases, higher doses of bleaching chemicals are required to remove the remaining lignin entering the bleach plant (29). Low kappa number represents low lignin content and easy bleachability of pulps (30). A lower PhOH concentration in residual lignin was suspected to lead to lower bleachability (28).

Bleached pulps were beaten to almost similar beating degree (°SR) and their papermaking properties determined as shown in Table 4. Both pulps exhibited almost similar papermaking properties. The papermaking properties of bleached okra-plant pulp are similar to those of the mixed hardwood pulp that is used in Kharnaphuli Paper Mills, Bangladesh. It may be concluded that the okra plant will produce bleached pulp similar to the existing mixed hardwood pulp.

#### CONCLUSIONS

The okra plant (*Abelmoschus esculentus*) consists of stalk fibre and bast fibre. Stalk fibre is characterized by high lignin, low  $\alpha$ -cellulose, and shorter fibre length, while bast fibre is characterized by low lignin, high  $\alpha$ -cellulose, and longer fibre length. The Kraft process is suitable for okra plant pulping. Screened pulp yield with an acceptable kappa number was obtained



Fig. 2 - Effect of pulping processes on delignification in okra plant pulping.



Fig. 3 - Effect of pulping processes on tensile and tear strength in pulping of okra plants.

TABLE 3Papermaking properties of unbleached pulp obtained from the Kraft and soda-AQ processes.							
Process	°SR	Density	Tensile index	Tear index	x Burst index		
Soda-AQ	23	0.422	39.6	11.7	2.3		
	50	0.539	75.7	9.2	5.1		
	67	0.569	71.4	7.6	4.3		
Kraft	22	0.534	51.3	11.2	3.5		
	53	0.622	80.7	9.4	4.3		
	68	0.671	76.3	8.4	5.3		

<b>TABLE 4</b> $D_{\mu}E_{\mu}D_{\gamma}$ bleaching of okra-plant pulp.							
Process	Brightness (%)	Drainage resistance (°SR)	Viscosity (mPa.s)	Density (g/cc)	Tensile index (N.m/g)	Tear index (mN.m²/g)	Burst index (kPa.m²/g)
Soda-AQ	76.8	54	14.8	0.564	60.7	8.9	5.0
Kraft	82.1	54	16.1	0.556	58.2	8.6	4.4

only at 20% alkali charge for 3 h of cooking time in the soda-AQ process, while the Kraft process required 18% active alkali for 2 h of cooking time to reach a similar degree of delignification. The Kraft process produced better pulp yield than the soda-AQ process at the same kappa number. Kraft pulp showed better tensile index, tear index, and burst index than the soda-AQ pulp. The Kraft pulp also showed better bleachability than that of soda-AQ pulp in a D<sub>2</sub>E<sub>2</sub>D<sub>1</sub> bleaching sequence. The Kraft pulp reached 82.1% ISO brightness, while the soda-AQ pulp only reached 76.8% ISO brightness. The okra plant (Abelmoschus esculentus) can be an effective alternative to traditional tropical hardwoods in Bangladesh.

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