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## オクラ茎からの製紙

ドウルドウラオ マリセル ナルシソ\*、渡邊 拓\*\*、釜谷保志\*\*\*、鈴木恭治\*\*\*\*

### Papermaking Potential of Okra Stalks\*

Maricel DULDULAO\*, Hiromu WATANABE \*\*, Yasushi KAMAYA \*\*\* and Kyoji SUZUKI \*\*\*\*

The papermaking potential of stalks, core, and bast parts of okra (*Abelmoschus esculentus* Moench syn. *Hibiscus esculentus* L.), an agricultural waste, was evaluated by determining their chemical composition and converting the raw material into pulp utilizing soda-AQ and kraft pulping processes. Chemical analysis showed that the okra has alphacellulose and lignin content comparable to commonly used nonwood and wood resources. Okra stalk and core fiber lengths were comparable to hardwoods. Fiber distribution showed a high presence of pulp fines. Although total pulp yield was low, and kappa numbers were high, handsheets from unbeaten pulp were found to be adequate in terms of paper strength. Further refining increased strength properties.

Keywords :Okra stalk, Papermaking, Kraft pulping, Soda-AQ pulping

### 1. Introduction

Although wood is still the major source of raw material for pulp and paper, studies on the potential of nonwood cellulose resources are still ongoing to meet the ever-increasing demand for pulp and paper products and to help alleviate the destruction and depletion of natural forests. In the 1960's, when the USDA launched a comprehensive study to identify new fiber sources, researchers identified representatives of

Graminae (bamboo and sorghum), Leguminosae (*Crotalaria* and *Sesbania*), and Malvaceae (kenaf and okra) as the most promising fiber sources among more than 1200 screened fiber crops samples<sup>1) 2)</sup>. Of these, kenaf received the most concentrated attention by the pulp and paper industry<sup>3) 4)</sup>. This study focuses on the other member of the Malvaceae family, Okra (*Abelmoschus esculentus* Moench syn. *Hibiscus esculentus* L.), which was also considered as a promising fiber source.

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\* Maricel DULDULAO (Student, The United Graduate School of Agricultural Science, Gifu University):

Tel: (81)9091765114 Email: maryand12@yahoo.com

\*\* Associ. Prof. (Dr.) Hiromu WATANABE: Tel: (81)54-238-4589, Fax: (81)54-237-3028, Email: ahwatanabe@agr.shizuoka.ac.jp

\*\*\* Prof. (Dr.) Yasushi KAMAYA: Tel: (81)54-238-4857, Email: ahykama@agr.shizuoka.ac.jp

\*\*\*\* Prof. (Dr.) Kyoji SUZUKI: Tel: (81)54-238-4859, Fax: (81)54-237-3031, Email: afksuzu@agr.shizuoka.ac.jp

All from Shizuoka University Faculty of Agriculture, 422-8529 Shizuoka City, Ohya 863

Similar to kenaf, okra is a perennial woody-stemmed herbaceous dicotyledon, but, unlike kenaf, it is mainly grown for its fruits for food and for other medicinal uses. However, as early as 1869, okra stalks were used on a commercial scale in American papermaking, and in 1870, the Mobile Register newspaper in Mobile, Alabama, USA, was printed on paper made from okra although its use was discontinued<sup>5)</sup>. To this day, handsheets made from okra, using the traditional mould and deckle, are quite common, but few formal investigations have examined its potential in pulp and papermaking. Furthermore, according to the National Research Council of the National Academies<sup>6)</sup>, after harvesting the okra fruits, the remaining biomass, which include the leaves, stalks and roots, which are considered to be wastes, can amount to about 27 tons per hectare. Most of this biomass is either, burned, thrown or utilized as compost. With okra being cultivated in countries with tropical and warm temperate regions, there is, therefore, a considerable amount of agricultural waste whose potential is untapped. Thus, we aimed to determine the potential of okra stalks in papermaking using soda-AQ and kraft pulping conditions.

## **2. Materials and Methods**

After harvesting the fruits, whole okra stalks (excluding the root part) were cut and collected from a local field. The stalks were then cleaned and sun dried. On the average, stalks of okra are composed of about 33% bast and 67% core. For the chemical composition analysis, whole stalks, core and bast parts were milled and passed through a standard 40-mesh screen. The

stalks were cut into 2-cm long chips for pulping experiments. The core and bast parts of the stalks were also separated, cut, and used for pulping. The powdered samples were further dried in a 105 °C oven for 24 hours prior to chemical characterization. The chemical composition of whole okra stalks, core, and bast parts, particularly the amount of alcohol (ethanol) extractives, lignin, holocellulose, and alphacellulose were determined using TAPPI T204, Klason, Wise method, and sodium hydroxide extraction, respectively.

Whole stalks (S), core (C) and bast (B) parts of okra were cooked under the following conditions: Soda-AQ process utilized a soda:anthraquinone ratio of 15%:0.1%(SAQ-1) and 18%:0.2%.(SAQ-2); for kraft process – active alkali concentration used were 16% (K-1) and 18%(K-2) as Na<sub>2</sub>O, with 25% sulphidity. To facilitate labeling, SSAQ-1 would stand for stalks cooked in 15%:0.1% soda:anthraquinone ratio, and so on. The cooking liquor-to-material ratio was 6:1, and the cooking temperature of 170 °C was reached in 90 minutes and maintained for another 90 minutes.

The resulting pulps were screened and unbeaten pulps were made into handsheets for the determination of their mechanical and optical properties. TAPPI test standards for folding endurance (T511), tensile strength (T494), bursting index (T404) and tearing resistance (T414) were used. Optical properties such as ISO brightness and opacity were measured. Pulp properties such as kappa number (TAPPI T236 cm-85), fiber length and width (HiRes Fiber Quality Analyzer), yield, and Canadian Standard of Freeness (CSF) were determined. Selected pulp samples (stalk and core pulps) were also

beaten using a TAPPI Standard valley beater at 1.2% pulp consistency for 15 minutes and 30 minutes, to determine the effect of beating. Handsheet properties of the beaten pulps were further compared.

### 3. Results and Discussion

#### 3.1 Chemical Composition of Okra Stalk and Pulp

Table 1 shows the chemical composition of uncooked okra stalks, core, and bast parts. The analysis showed that the holocellulose and lignin contents of whole stalk, core and bast samples compose about 83-93%, with the alphacellulose content of the okra core and bast comparable to that of both softwood and hardwood whereas the stalk was slightly lower. Compared to the listed stalk fibers<sup>7)</sup>, alphacellulose and lignin contents fall within the same ranges, whereas okra bast alphacellulose content is significantly higher than the bast fiber sources which include jute, kenaf

and textile flax. Low lignin content is always advantageous in chemical pulping. As shown in Table 1, the lignin contents of okra stalk and core are lower than that of wood and comparable to common non-wood sources. Furthermore, okra bast lignin content was considerably lower. As expected from a nonwood resource, ash and hemicellulose contents were higher than those of wood but were also within the same ranges as those of nonwoods. These data served as the primary indication of the potential for okra stalk, core and bast in papermaking. This set of data would also serve as a reference for future pulping considerations for okra stalk considering the high amount of ash it contains which could also contribute to problems in chemical recovery and paper quality. For the purpose of establishing baseline data standard chemical pulping conditions, particularly soda-AQ and kraft, were only applied in this study.

Table 1 Chemical composition of okra stalk, core and bast.

	Okra*			Softwood <sup>7)</sup>	Hardwood <sup>7)</sup>	Kenaf core <sup>7)</sup>	Kenaf bast <sup>7)</sup>	<i>Arundo</i> <i>donax</i> <sup>7)</sup>	Cereal straw <sup>7)</sup>
	Stalk	Core	Bast						
EtOH Extractive **(% )	3.0	2.0	1.0	-	-	-	-	-	-
Holocellulose***(% )	62.0	74.0	78.0	-	-	-	-	-	-
Alphacellulose***(% )	35.0	39.0	49.0	40-45	38-49	34	31-39	29-33	28-37
Hemicellulose***(% )	27.0	35.0	29.0	7-14	19-26	19.3	21-23	28-32	23-38
Lignin***(% )	21.0	19.0	9.0	26-34	23-30	17.5	15-18	21	12-21
Ash(% )	11.0	8.0	13.0	1	1	2.5	2-5	4-6	5-20

\* This study

\*\* based on OD weight

\*\*\* based on OD weight of extractive-free material

### 3.2 Pulp Properties

Okra stalk, core and bast were first cooked under standard soda-AQ pulping and kraft conditions utilizing 15%NaOH: 0.1% anthraquinone ratio and an active alkali concentration of 16% as Na<sub>2</sub>O-25% sulphidity, respectively. The resulting pulps showed low screened yield and high amounts of reject (Table 2). Whole stalks used in pulping were not depithed and this may have contributed to low yields and affected CSF<sup>8)</sup>. The presence of pith is known to have an influence on the amount of ash as well as in chemical consumption during pulping. Kappa numbers were high in all pulp samples, and CSF values ranged from 65 mL to 450 mL. Increasing the soda:anthraquinone concentration ratio to 18%:0.2%, and active alkali concentration to 18%, improved the screened yield and considerably reduced the amount of rejects, although total pulp yield slightly decreased. Holocellulose and alphacellulose contents improved and CSF increased with higher chemical charge except for core pulps. Kappa numbers, on the other hand, were greatly lowered, with kraft-cooked pulps showing lower kappa numbers than soda-AQ-cooked pulps. This suggests that the delignification process was further improved with increased chemical charge. Although a kappa number of at least 20 is desirable for evaluating handsheet properties of wood pulps, it should be noted that the pulping treatments were done based on the resulting pulp yields. Since screened yield was below 40%, the amount of rejects have been greatly decreased and although the kappa number was still higher than 20, the resulting and available pulped material was prepared and examined for its handsheet properties without

further continuing to bleaching process.

The fiber length weighted length distributions of different okra pulps are shown in Figure 1. The graphic profiles indicate the high presence of fines in all pulp types. Again, the inclusion of pith in pulping may have contributed to the amount of fines in the stalk and core pulps however, even the bast pulp exhibited similar profiles thus aside from the parenchyma cells, small fiber fragments, epidermal cells as well as vessel elements may account for the high amount of fines. Average dimensions of okra fibers are further shown in Table 3. Okra fiber lengths fall within the range of hardwood fiber lengths and widths.

### 3.3 Handsheet Properties

Unbeaten okra pulps were made into handsheets, and their optical and strength properties were tested using standard TAPPI tests (Table 4). ISO brightness of the handsheets was generally improved with an increase in chemical charge with the kraft-cooked core handsheets showing the highest brightness value and handsheets from bast pulps showing the least ISO brightness. In terms of tear index, burst index, tensile index and folding endurance, the handsheets showed relatively high values for unbeaten pulps. Comparing the strength values of the unbeaten okra pulps to recently published data on wood pulp, particularly that of willow and eucalyptus<sup>10)</sup>, cooked under similar conditions as in this research, okra pulps showed higher values in terms of tear index, tensile index, burst index and folding. However, the yield, kappa numbers and ISO brightness values of both willow and eucalyptus were more superior to okra.



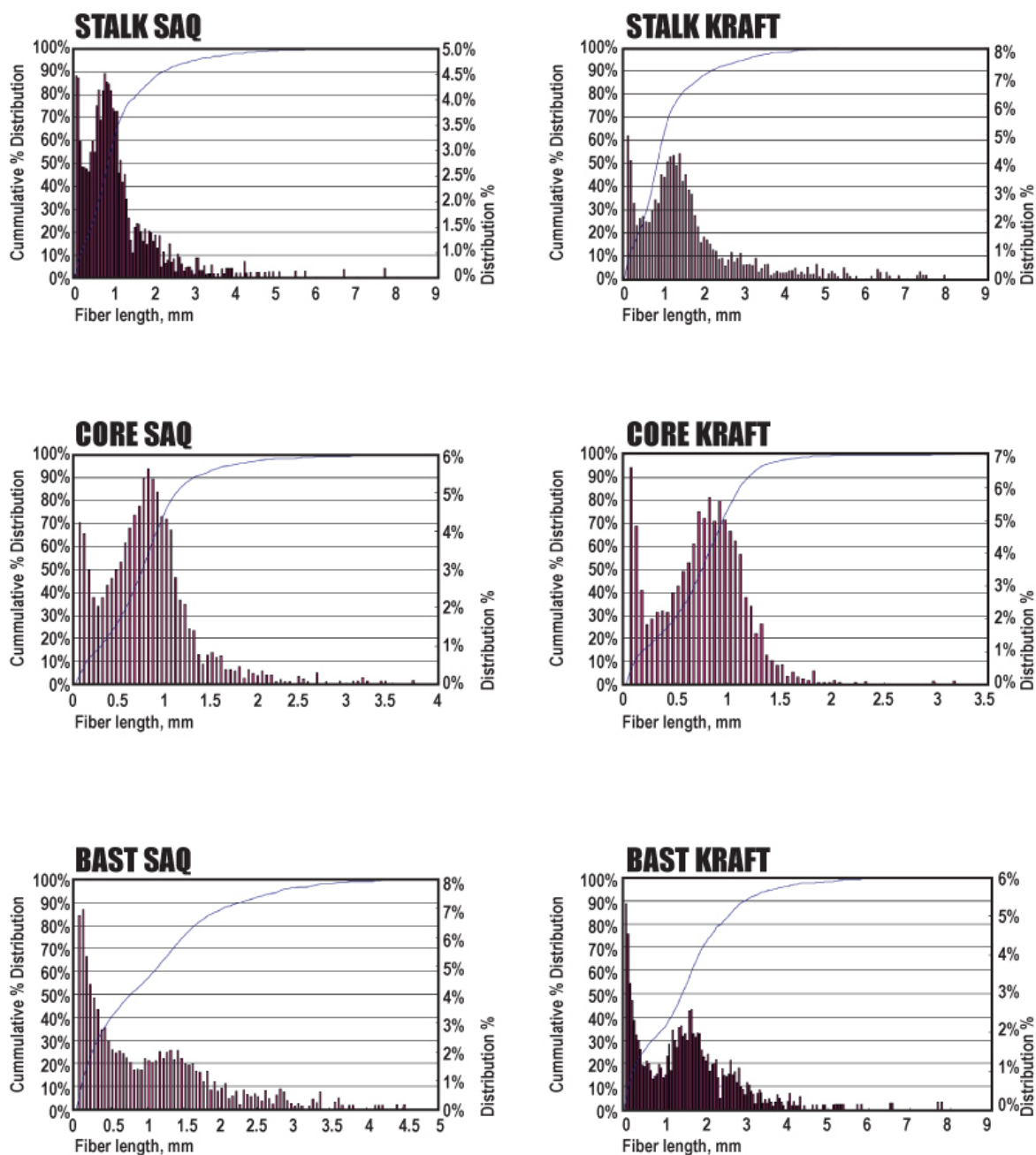


Figure 1 % length weighted length distribution of okra fibers.

Table 3. Fiber dimensions of okra pulps.

	Okra Pulps*		
	Stalk	Core	Bast
Length (mm)	1.06 (1.11)	0.75 (0.81)	1.55 (1.03)
Width (um)	22.3 ( 23.8)	23.8 ( 24.6)	20.9 (21.5)

\* This study – average values for Kraft (SK-1, CK-1 and BK-1 in Table2))  
(Soda-AQ (SSAQ-1, CSAQ-1 and BSAQ-1 in Table2)) pulps.

Table 4 Optical and strength properties of unbeaten okra pulps.

	ISO Brightness (%)	Tear Index (mN.m <sup>2</sup> /g)	Burst Index (kPa.m <sup>2</sup> /g)	Tensile Index (Nm/g)	Folding endurance (no. of times)
<b>SSAQ-1</b>	15.7	7.39	1.07	49.73	83
<b>SSAQ-2</b>	14.3	5.23	1.46	54.74	96
<b>SK-1</b>	14.1	5.79	0.76	42.82	34
<b>SK-2</b>	16.4	5.23	2.34	66.22	141
<b>CSAQ-1</b>	16.2	7.36	0.92	47.31	76
<b>CSAQ-2</b>	15.5	5.04	1.23	57.79	71
<b>CK-1</b>	16.3	6.05	0.92	43.16	42
<b>CK-2</b>	20.2	5.30	1.29	57.08	114
<b>BSAQ-1</b>	11.9	5.70	1.06	50.79	91
<b>BSAQ-2</b>	13.9	5.69	0.80	48.29	162
<b>BK-1</b>	12.3	11.52	1.94	50.79	140
<b>BK-2</b>	13.35	5.97	1.11	55.59	79

Figure 2 shows the relationships of strength properties of okra handsheets. Black-shaded and gray-shaded symbols represent samples cooked at a lower chemical charge and at a higher chemical charge, respectively. Figure 2A, shows the relationship

between tear and tensile indices. Although tear index slightly decreased with higher chemical charge in all pulp cooks, the tensile index increased, except for bast pulp cooked in soda-AQ. Figures 2B and 2C show the relationship

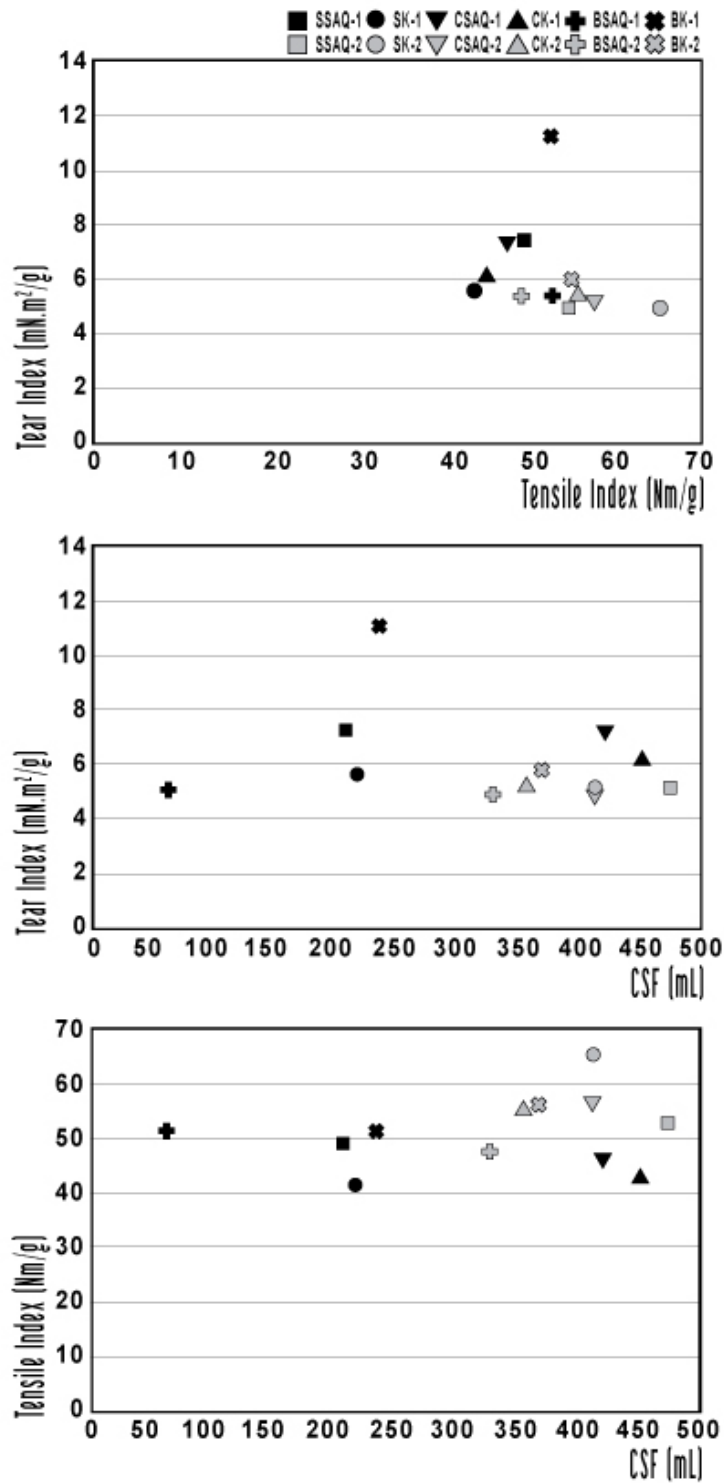


Figure 2 Relationships of mechanical properties of unbeaten okra handsheets.



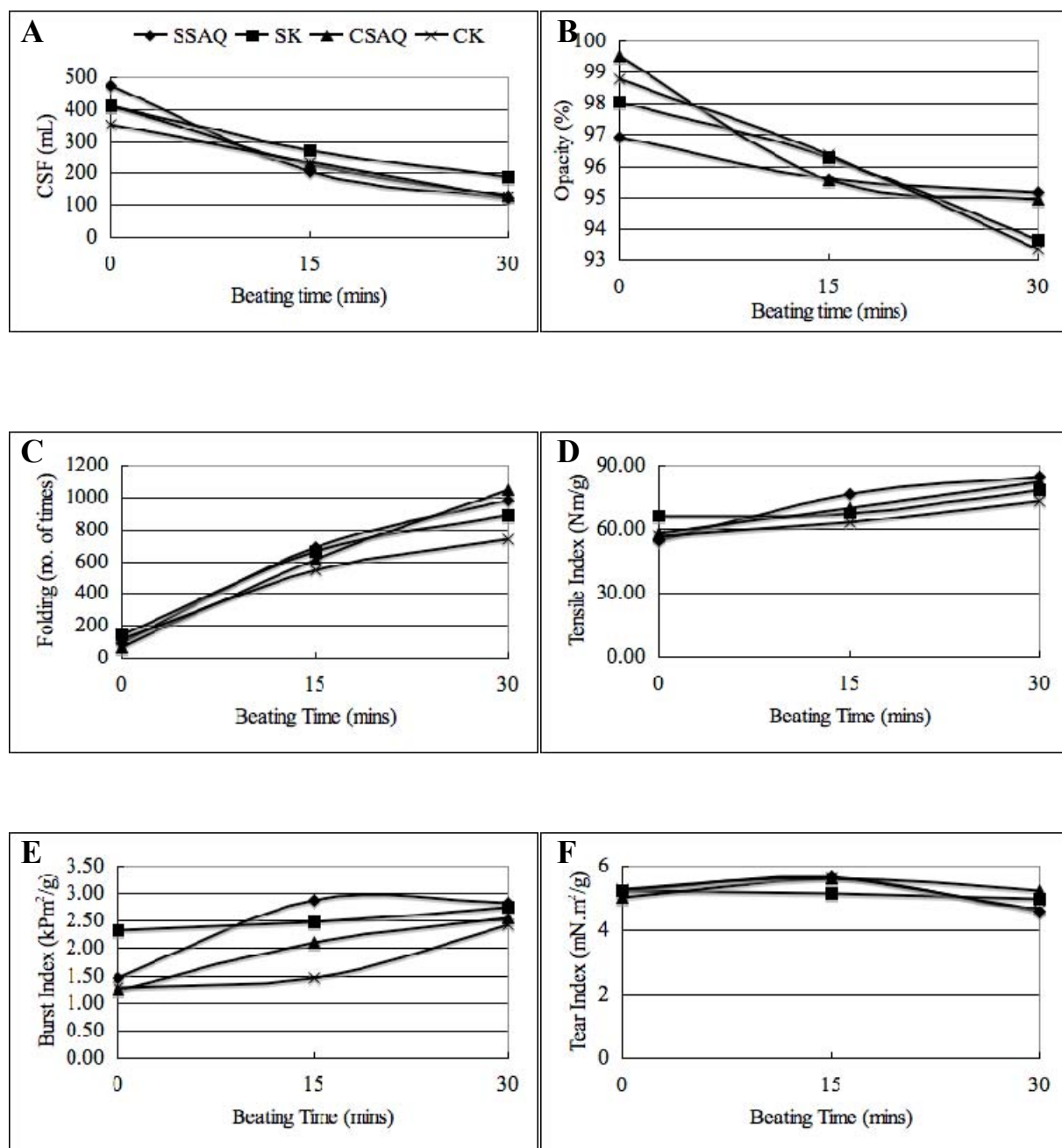


Figure 3 Effect of beating on CSF (A), opacity (B), folding endurance (C), tensile (D), burst (E) and tear (F) indices.

between CSF and tear and tensile indices, respectively. It can be observed that, generally, the strength properties were more or less maintained even with increased CSF values.

### **3.4 Effect of Beating**

The effect of beating was evaluated on whole stalk and core pulps cooked with a higher chemical charge (SSAQ-2;SK-2;CSAQ-2 and CK-2). Figure 3 shows the effect of beating on optical and strength properties of handsheets (0 min. representing unbeaten state). As expected, CSF (Figure 3A) and opacity (Figure 3B) decreased with longer beating time. Refining increases fiber-to-fiber bonding resulting to a denser sheet and lower opacity <sup>9</sup>. Strength properties such as folding endurance (Figure 3C), tensile (Figure 3D) and burst (Figure 3E), increased with further beating.

## **4. Conclusions**

In chemical composition analysis, the alphacellulose content of okra stalk was comparable to the listed common nonwoods. Although, ash was found to be high, it is still within the range of the nonwood resources. These primarily indicated the potential of okra stalk in papermaking.

Chemical pulping of the whole stalk, core and bast using both soda-AQ and kraft processes yielded pulps of fiber dimensions comparable to hardwoods. Total pulp yield was relatively low and kappa numbers were high but were greatly reduced with pulping utilizing higher chemical charge. Resulting handsheets exhibited high strength properties except tear index for unbeaten pulps. Refining of the pulp resulted to even

higher strength properties and lower freeness and opacity.

So far, results of this research suggest the suitability of okra stalk, core and bast in papermaking. In light of this, the pulps produced may not be of adequate quality as a sole fiber source but may have potential as reinforcing pulp taking into account its availability, fiber characteristics and strength properties. To fully establish its potential, further studies on optimization of pulping conditions and bleachability are underway.

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