

Formic Acid Pulping and TCF Bleaching of Cassava Stalks

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ABSTRACT

The formic acid (FA) pulping process is well-known for its advantage to overcome the problems caused by the presence of high silica and fines content of agriculture residues during pulping. In this context a new and unconventional raw material, cassava stalk was pulped using formic acid followed by treatment with peroxyformic acid (PFA) treatment was applied to the formic acid treated pulps at 80 °C for two hours. It was found that the FA/PFA process lowered pulp yield of cassava stalks when FA concentration was increased. The PFA pulping decreased the kappa number of the pulps. After the FA/PFA treatment of cassava stalks, the pulp yield reached 37-42% with kappa number of 21-34 at different formic acid concentrations. FA/PFA pulp from cassava stalks did not show promising result in total chlorine free bleaching and final brightness reached only up to 54%.

Keywords: Pulping, Kappa number, Pulp yield, Formic acid and Papermaking properties.

1. Introduction

Bangladesh has very limited forest area. As the main source for pulping raw material is wood, the local supply is very scarce. Thus, interest on using agricultural wastes as alternative source has been noticed for the last couple of decades in such countries that has abundance of some sort of agriculture residues. Every year Bangladesh produces significant quantity of agricultural wastes [1]. In 2016, the growth in crops production in Bangladesh was reported 2.7863% by World Bank which indicates rise in agricultural wastes during the year [2]. On the other hand, the GDP of Bangladesh has been over 7% for the last 5 years [3]. As the lifestyle has improved over the years, requirement for paper-based consumption products along with other products has also increased. The increased demand for packaging paper, tissue paper along with writing and printing paper has led to the search for alternating cellulosic source for pulp production.

Cassava (*Manihot esculenta*) is fourth most important food staples in tropical countries and it is positioned in number six in the list for providing calories in human food [4]. The cassava plant can grow in very poor soil and harsh climate conditions and the water requirement for its growth is less than 600-1000 mm per year depending on humid conditions [5]. Thus the production is very cheap and requires less effort to grow cassava plant. The plant grows up to 1-4 meters long (Figure 1) [6]. The morphological characteristics of the plant are highly variable depending on high and low land and different type of weather conditions [6]. The cassava stem is developed by alternating nodes and internodes and the morphology of the mature stem is like hardwoods [7]. A mature plant contains many viable sprouts and new plant can be multiplied from the stem cuttings. Thus it is very advantageous and cheap to produce cassava plants. Recently there has been a lot of interest in utilizing different parts of cassava plant for pulping. It was found in a recent study that pulping of cassava bagasse produced 22.4% pulp yield in soda process and 12.3% in organic solvent process

[8]. Another study utilized cassava stalks for pulping in conventional soda-anthraquinone (AQ) process and the yield was only 18.48% [9].

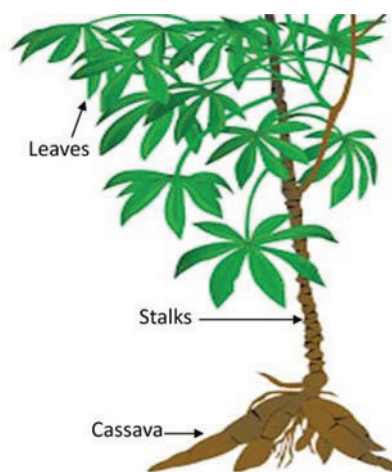


Fig. 1. Illustration of a cassava tree.

Utilization of crops residues for pulp production is environmentally and economically advantageous. Several non-wood fiber based commercially pulping mills are reported to operate in China, India, Latin America, Africa and Middle East [10]. A data in 2005 showed that pulp production from non-wood comprised of 9.27% of world production [11]. Conventional process for pulp production from non-wood crops residues is soda-AQ process [12-15]. In the conventional processes, the main problem is that the silica present in crops residues is dissolved in the cooking liquor which creates difficulty in the recovery process. Also the alkali process utilizes chlorine dioxide as bleaching chemical which is expensive and also harmful for the environment. To make a pulp mill economically viable, a proper chemical recovery system is required. It is also essential in environmental point of view. On the other hand, in case of formic acid (FA) pulping, the silica remains on the fiber and is removed in peroxyformic acid (PFA) delignification stage [9,16-19].

Another advantage of organic acid pulping is that solvent recovery is very easy and complete recovery of the solvent used is possible [20]. As the cooling solvent is not dumped in the environment, the process is also very environment friendly. In case of using formic acid as organic solvent, solvent recovery as well as the separation of other valuable products such as lignin and hemicellulose are also very convenient [20-21].

In previous studies, FA/PFA pulping generated pulp yield of 43.4% and 44.4% from rice straw and bagasse respectively [18,21]. Our previous study utilized FA/PFA pulping process to produce 38.7-54.0% pulp yield from fourteen different non-wood agricultural residues of Bangladesh [22]. When compared with conventional pulping process, it was found in other studies that in case of non-wood pulping, acid pulping gives higher pulp yield [23-24]. Our previous study of cassava stalks pulping in conventional alkaline process produced very low pulp yield (18.84% with kappa number 30.3) [9].

Thus, the aim of the work was to perform pulping of cassava stalks in FA/PFA process at different acid concentrations and to bleach the produced pulp in total chlorine free bleaching (TCF) process. The results of cassava stalks pulping were compared with eggplant and okra stalks pulped under similar conditions in FA/PFA process.

2. Materials and Methods

2.1. Raw materials and chemicals

The cassava stalks (*M. esculenta*) was collected from Hobiganj district and eggplant stalks (*S. melongena*) and okra stalks (*A. esculentus*) were collected from Mymensing. The raw materials were cut into 2-3 cm size. They were ground using a Wiley mill into smaller particles that were able to pass through a 40-60 mesh size and these were utilized for chemical characterization. All the chemicals used were reagent grade and were bought from Merck, Germany.

2.2. Morphological properties

A solution consisting of 1:1 HNO₃ and KClO₃ was prepared and the samples were steeped in it. Slides containing small quantity from these samples were prepared and fiber properties were determined using an Euromex-Oxion image analyzer. About hundreds of individual fibers were measured using Image Focus Alpha software and an average of the measurements was utilized [9, 22-23].

2.3. Chemical analysis

Tappi test methods were followed to measure solvent extractives (T204 om88), 1% alkali solubility (T212 om98), water solubility (T207 cm99), Klason lignin (T211 om83) and ash content (T211 os76). For the measurement of holocellulose content, chopped samples were treated with NaClO₂ solution. CH₃COOH-CH₃COONa buffer was used as buffer and a NaOH solution (17.5%) was used to determine α -cellulose content [9, 22-23].

2.4. Formic acid (FA) /Peroxyformic acid (PFA) treatment

The cassava, eggplant and okra stalks samples were heated at 100 C for 4 hrs using reflux system. The FA

concentration was varied 70, 80 and 90 % (v/v) maintaining the liquor ratio at 10.

After the cooking, the pulp was filtered and washed and was treated with PFA at 80°C for 2 hrs. The PFA was prepared by adding 4% H₂O₂ with 90 % formic acid (on o.d. FA pulp). After the PFA delignification stage, the pulp was filtered and washed. 80% FA was used as washing liquor. Finally, the pulps were washed with hot water and pulp yield and kappa number (T236 om-99) of the pulp were determined. All experiments were performed three times and an average of the data was taken. The used formic acid was distilled to be used further and the extracts containing hemicellulose and lignin were preserved for separate experimentations [22-23].

2.5. Bleaching of FA/PFA pulp

Unbleached pulp (50g) of the samples were bleached by alkaline peroxide treatment. The pH was maintained at 11 using NaOH and consistency of the pulp was 10%. The bleaching operation was performed at 80°C for 1 hr by varying hydrogen peroxide concentration to 2, 4 and 6% on o.d. pulp [22-23].

2.6. Evaluation of FA/PFA pulp

The resulting pulps were tested for kappa number, viscosity (T230 om-99) and brightness (T452 om-92). The tests were performed three times and an average of the readings was taken. Finally, hand sheets were prepared after blending individual bleached pulps in a waring blender for 10 min and tested for tensile (T494 om-96), tear (T414 om-98) and burst (T403 om-97) [22-23].

3. Results and discussion

3.1. Chemical characteristics

The chemical properties of cassava stalks along with two other woody non-wood plants samples were performed and the data are shown in Table 1. The results were almost similar to our previous results [9]. Cold water and hot water solubilities of cassava stalks were higher than the other two samples. The higher cold and hot water solubilities suggested the existence of high amount of inorganic matter, tannins, gums, sugars etc. Additionally, starch is also extracted in hot water. The higher water solubility has unfavourable outcome on pulp yield as some carbohydrates are removed [24]. 1% alkali solubility of cassava stalks was higher than that of eggplant and okra stalks. One percent alkali extracted lower molecular carbohydrates. The water solubilities and 1% alkali extractives in cassava stalks were higher than eggplant and okra stalks. Higher solubility in 1% percent alkali, cold and hot water may affect pulp yield and cooking chemical consumption [25-26]. Lower extractives content is also desirable for better pulp yield. Presence of extractives can adversely affect the pulp bleaching and paper machine runnability. Acetone extract in cassava stalks was 1.12%, which was close to acetone soluble in *Setaria-Italika* [24] and lower than lemon grass [26].

The holocellulose content is directly linked to pulp yield and physical properties of paper [27]. The higher α -cellulose

contents in lignocellulosic are better for higher pulp yield [28]. The cellulose content is also correlated with physical strength properties of paper [29]. Table 1 also shows that the α -cellulose contents in cassava stalks was 30.4%, which was lower than other two non-wood plant stalks. The α -cellulose contents in hardwood are found to be higher than these values [30-31]. Pentosan is the leading hemicellulose in agricultural residues. The pentosan content in cassava stalks was 14.8%. High amount of hemicellulose has positive impact on the separation of fibers during beating process which is common for large scale pulping for paper production. Thus presence of hemicellulose helps in the bonding of paper sheets [32]. It was found in our experiments that the pentosan content in cassava stalks was lower than a commonly used industrial non-wood, bamboo [22].

Higher lignin content in lignocellulosic is undesirable for papermaking and is extracted from raw materials by using the cooking chemicals. High amount of lignin in a sample requires higher chemical and energy consumption during pulping. Thus low lignin containing raw materials are acceptable for pulping as they require less chemicals and lower temperatures. The lignin content in cassava stalks was 20.8%, which was lower than eggplant stalks (28.5%) and higher than the lignin content in okra stalks (18.6%).

The ash content in cassava stalks was 2.40%, which was higher than eggplant and okra stalks (Table 1). As compare to rice, wheat and kaun straws, the ash content in cassava stalks was low [22].

Table 1. Chemical and morphological characteristics of crops residues

	Cassava stalks	Eggplant stalks	Okra stalks
Hot Water Solubility (%)	33.88	17.13	22.4
Cold Water Solubility (%)	28.21	12.5	13.8
1% NaOH Solubility (%)	39.85	25.3	37.29
Extractives in acetone (%)	1.12	0.32	1.5
Ash Content (%)	2.40	1.62	0.76
Holocellulose (%)	50.5	63.8	56.9
Alpha Cellulose (%)	30.4	35.2	29.8
Klason lignin (%)	20.8	28.5	18.6
Acid soluble lignin (%)	3.62	2.06	3.55
Total lignin (%)	24.02	30.56	22.15
Pentosan (%)	14.9	14.6	15.3
Fiber length, L (mm)	0.65	0.58	1.14
Fiber width, D (μ m)	25.5	13.2	21.0

3.2. Morphological properties

Macerated samples from cassava, eggplant and okra stalks were studied on optical microscope (Fig. 2). The fiber lengths obtained in this study from cassava stalks was 0.65 mm, which was shorter than okra stalks (1.14 mm) and slightly longer than eggplant stalks (0.58 mm) (Table 3). Fiber length determines papermaking properties especially tear strength [32]. The fiber width of cassava stalks was 25.5 μ m, which was wider than the other two fibers (Table 1). The shorter and wider fiber produces inferior slenderness ratio, indicating lower tearing resistance. The reason for this is that shorter and wider fibers are unable to make bonding due to lack of surface proximity between them [33].

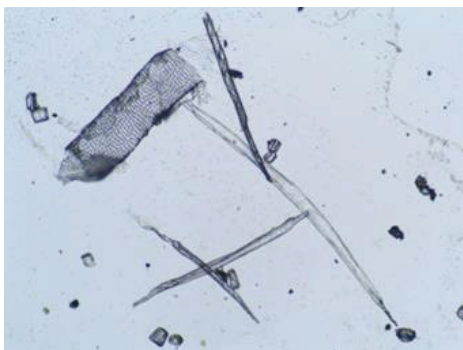


Fig. 2. Microscopic image of macerated fiber of cassava stalks (4X magnification)

3.3. FA/PFA pulping

FA pulping of cassava stalks was carried out with varying FA charge at boiling temperature for 4 hr followed by PFA treatment for 2 h at 80 °C, and pulp yields and kappa numbers of the produced pulps are shown in Table 2. For comparison, FA/PFA pulping was carried out for eggplant and okra stalks under similar conditions. Cassava stalks produced pulp yield of 37.1% with kappa number of 20.8 at 90% FA charge, while the pulp yield for eggplant stalks and okra stalks were 45.1% (kappa number 21.6) and 40.9% (kappa number 21.4) respectively at 90% FA charge (Table 2). The pulp yield of cassava stalks in

Table 2. Formic acid and Peroxyformic acid (FA/PFA) pulping of cassava plant and comparison with other non-wood plants

Raw material	Formic acid (%) v/v	Pulp yield (%)	Kappa number
Cassava stalks	70	41.8	34.1
	80	39.7	29.2
	90	37.1	20.8
Eggplant stalks	70	51.4	37.2
	80	50.5	33.8
	90	45.1	21.6
Okra stalks	70	43.2	35.0
	80	41.3	29.3
	90	40.9	21.4

FA/PFA process was higher than previously reported yield in soda-AQ process (18.48% with kappa number 30.3) [9]. Fig. 3 shows the total pulp yield and kappa number relationship of cassava, eggplant and okra stalks in FA/PFA processes at different FA concentrations. From Table 2 it can be observed that with increase in FA concentration, kappa value and pulp yield of all the samples decreased. The data in Table 2 are represented graphically in Figure 3.

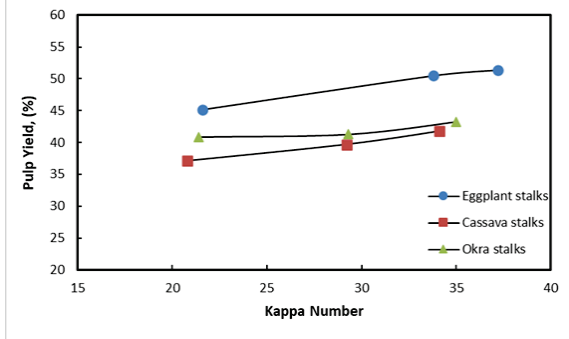


Fig. 3. Pulp yield kappa relationship of eggplant cassava and okra plants pulping

3.4. Bleaching

The bleaching of FA/PFA treated cassava, eggplant and okra stalks pulp was performed by chlorine free alkaline peroxide. Eggplant and okra stalks pulp responded well as compared to cassava stalks pulp. The brightness of cassava stalks pulp reached to 54.1% at 4% H_2O_2 charge, which was lower than that of the other two non-wood plant pulps (72-76%) (Fig. 4). The viscosity of the pulps decreased when peroxide charge was increased from 4 to 6% and the brightness also did not improve as was expected (Fig. 5). The viscosity decreased as some of the carbohydrate degraded with the increase of peroxide charge [34].

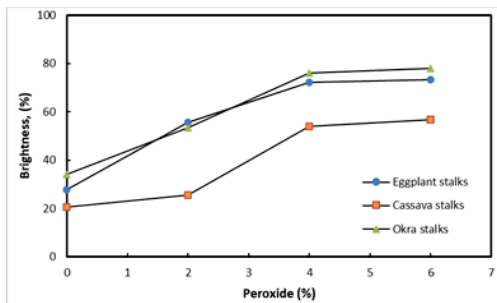


Fig. 4. Effect of peroxide charge on the brightness of different non-wood pulps.

Table 3. Fiber quality analysis of cassava, eggplant and okra stalks

Sample	Fines (%)	Length Weighted (mm)	Mean Curl Index	Mean Kink Index (mm^{-1})	Mean Width (μm)	Degree of External Fibrillation (%)	Coarseness (mg/m)
Cassava stalks (FA/PFA)	68.7	0.377	0.035	0.902	25.3	0.67	0.109
Cassava stalks (SAQ)	53.2	0.436	0.040	0.938	26.9	1.0	0.034
Eggplant stalks (FA/PFA)	31.5	0.329	0.062	1.29	14.8	1.06	0.188
Eggplant stalks (SAQ)	18.2	0.449	0.067	1.40	16.4	1.71	0.058
Okra stalks (FA/PFA)	52.1	0.834	0.078	1.57	18.5	0.83	0.209
Okra stalks (SAQ)	31.6	1.236	0.101	1.75	21	1.40	0.143

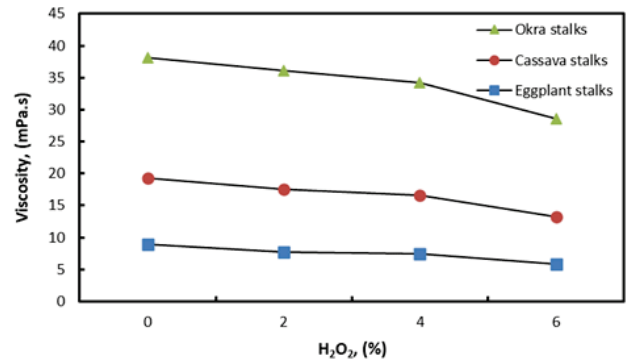


Fig. 5. Effect of peroxide charge on the viscosity of different non-wood pulps.

3.5. Evaluation of pulp fiber quality

After blending the pulps, the pulp fiber quality was analyzed and compared with soda AQ pulps [9]. It was found that FA/PFA pulp produced more fines than soda-AQ pulps (Table 3). The reason might be the brittleness of acid pulps. Fiber length and fines have significant impact on paper sheet formation. Also reduced length of the fibers directly affect papermaking properties, while optical and structural properties might not be hampered [35]. It was found that the coarseness of cassava stalks pulp in FA/PFA process was much lower than soda-AQ process (3.5 vs 10.9 mg/100 m) [9]. The low value of coarseness of FA/PFA cassava stalks pulp indicated good paper sheet formation which produced paper with better physical properties [36]. FA/PFA cassava stalks pulp had lower curl and kinks index than soda-AQ pulp. Curl and kinks form bulk and porosity in paper sheets. These are deformations in fiber which cause less fiber-to-fiber bond affecting physical properties of paper sheets.

3.6. Papermaking properties

Hand sheets were prepared from bleached pulps to find out the physical properties of paper. Tensile index of cassava stalks pulp was 26.3 N.m/g with tear index of 2.8 mN.m²/g, which was lower than other non-wood plant pulps (Table 4). Papermaking properties of cassava stalks pulp did not reach to desired level, which can be explained by generated fines in organic acid treatment and the fines were poorly bonded with fibers (Table 3) [19].

Table 4. Physical properties of bleached pulp

Raw materials	Tensile index (N.m/g)	Tear index (mN.m ² /g)	Burst index (kPa.m ² /g)	Elongation (%)	TEA (J/m ²)
Cassava stalks	26.3±6.7	2.80±0.6	0.19±0.2	1.4±0.1	6.3±3.0
Eggplant stalks	35.7±2.3	4.5±0.9	2.1±0.3	2.3±0.3	46.0±4.1
Okra stalks	46.8±1.8	3.2±0.8	1.9±0.2	1.5±0.3	25.2±2.3

4. Conclusion

Cassava stalks was evaluated as a raw material for pulping and compared with eggplant and okra stalks. Cassava stalks had lower holocellulose and α -cellulose than other common non-wood. The FA/PFA pulping process produced higher pulp yield than conventional soda process. The formed paper sheets in unrefined state were inferior in tensile and tear index to the other two non-wood samples due to the generation of high amount of fines during acid pulping. Thus, according to the finding of this study, cassava stalks pulps produced by FA/PFA process can be utilized in production of corrugated packaging papers/boards where bleaching is not required.

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