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Suitability of Sudanese Cotton Stalks for Alkaline Pulping with Additives

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Abstract: The fibre characteristics and chemical composition of *Gossypium hirsutum*, Sudanese cotton stalks, were assessed for their suitability for papermaking. Soda-anthraquinone (soda-AQ), alkaline sulphite-anthraquinone (AS-AQ) and ASAM (alkaline sulphite-anthraquinone- methanol) cooking was carried out with different alkali charges and pulps with acceptable to good yields and mechanical properties were obtained. ASAM pulping gave the best results in yields, degree of delignification and mechanical pulp properties. AS-AQ pulp cooking with the ratio of 70: 30 as $NaOH_2$: Na_3SO gave better results compared to the ratio of 60: 40.

Key words: *Gossypium hirsutum*; Cotton stalks; Papermaking; Fiber characteristics; Chemical composition

INTRODUCTION

Agricultural residues in Sudan are available in large amounts without rational economical utilization. Research studies reflected the suitability of Sudanese agricultural residues for pulp production [1-4]. Production of cotton stalk, *Gossypium hirsutum*, in Sudan has been bound with high variability attributable to the variation in natural conditions, policy instruments and institutional set-up [5]. In India cotton stalks were used in board industry [6], while in Iran were studied for the production of medium density fiber board [7]. Cotton stalks' pulping was extensively studied with alkaline ethanol which resulted in an increase in pulp yield and reduction in both kappa number and screening rejects with soda and AQ addition [8]. Pulps produced from cotton stalks could be an alternative raw material for pulp and paper industry [9]. It was found that, the Egyptian cotton stalks had lower contents of extractives and lignin and higher cellulose content compared to the American ones [10]. Cotton stalks Kraft-$NaBH_4$ pulps can be used in the production of high quality printing and writing papers [11]. Cotton stalks are suitable for producing mainly writing and printing papers or mixing with conventional wood pulps to produce paper of various uses [12]. The pulp yield is about 40-45 percent and the Kappa number of soda-AQ processed cotton stalk pulp is 30-35 [13]. The cotton fibers can be mixed with other large fibers softwood for the production of high quality paper [14].

The present work aimed to evaluate the characteristics of Sudanese cotton stalks in terms of morphological properties, chemical composition, alkaline pulping and papermaking characteristics and therefore indicate their suitability for pulping purposes.

MATERIALS AND METHODS

Gossypium hirsutum, cotton stalks investigated in this work were from Barakat area in Elgizera scheme (Central Sudan), with clay soil. Cotton stalk were collected, prepared and cleaned according to TAPPI standards 2002 [15]. They were chopped to 3-5 cm and were left for air drying under the sun according to TAPPI standard (T_{257}-cm- 02). Fiber dimensions after maceration with a mixture of 30% hydrogen peroxide and acetic acid (1:1) was determined microscopically at x300 and x400 magnifications after staining with 1% aqueous safranine [16]. The chemical composition of cotton stalks was determined according to TAPPI standard Test methods (T_{204}-cm-97 for solvents extractives, T_{207}-cm99 for water

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solubility, T_{211}-om- 93 for ash, T_{212}-om-98 for 1% NaOH extractives, T_{222}-om-02 for lignin and T223-cm-01 for Pentosans), while Kurchner-Hoffer cellulose was measured according to the literature [17]. Meal was prepared using star mill with standard sieve according to TAPPI standard (T_{11}- wd-79).

Cooking was carried out in the laboratory 7 liters electrically heated digester with forced liquor circulation. The pulping conditions were: cotton stalks to liquor ratio (1: 4), active alkali charge range of 13-17% as Na_2O at the maximum temperature of 165-175°C with 90 min time required to reach the maximum temperature and holding time of 120 min at the maximum temperature, 0.1% dose of AQ was applied and NaOH: NA_2SO_3 ratios were 70:30% and 60: 40% for AS-AQ and ASAM methods. The methanol was added during ASAM cooking on the basis of 15% by volume of white liquor [18].

RESULTS AND DISCUSSION

Fibre Characterization of *Gossypium hirsutum*: The principal factors controlling the strength of paper are fibre density (cell wall thickness), fibre length and fibre strength [19]. The average fibre length of Sudanese cotton stalks was 0.79 mm (Table 1) which is considered short; shorter than fibres of cotton stalks from Iran [14]. The narrow fibre (18.2) µm with lumen diameter of 11.6 µm indicted that fibres would not collapse easily on beating and would give a rather porous and bulky paper sheet compared to ones from Iran, as a result of poor interfibre bonding. The thin walls (3.3 µm) resembling the Iranian cell wall (3.78 µm) expected fibres to be flattening easily and have good surface contact and adhesion, could have the opposite effect, somewhat compensate for the porosity. The flexibility coefficients of both Sudanese and Iranian were more or less good, indicating the number of interfibre bonds positively correlated to the tensile strength, burst factor and double fold endurance. The wall fraction which correlates the wall thickness with the fiber diameter was very good for both types of fibers. The felting power, favorable for bonding strength and tear was 43.4 for Sudanese cotton stalks, indicating an easy beating. The Runkel ratio of 0.57 was too low which makes it suitable for pulping and papermaking materials.

Table 1: Fiber dimensions and morphological indices for *Gossypium hirsutum* from central Sudan and Iran

Fiber properties	Sudan	Iran
Fiber dimensions		
Average fiber length (mm)	0.79	0.926
Average fiber Diameter (µm)	18.2	23.88
Average lumen diameter (µm)	11.6	16.1
Average cell wall thickness (µm)	3.3	3.78
Morphological indices		
Flexibility coefficient (%)	63.7	67.42
Wall fraction (%)	18.1	15.8*
Felting power	43.4	38.78
Runkel ratio	0.57	0.47

*Calculated

Chemical Characterization of *Gossypium hirsutum*: The chemical composition of Sudanese *Gossypium hirsutum,* cotton stalks (Table 2), in comparison to the Egyptian and American cotton stalks [10] was generally in the range of tropical hardwoods and non woody plants.

Table 2: Chemical Composition of *Gossypium hirsutum* from central Sudan compared to stalks from other locations

Chemical composition	Sudanese Cotton stalks	Egyptian Cotton stalks	American Cotton stalks
Ash content (%)	2.4	1.84	1.12
Silica content (%)	0.8	0.49	N/A
Cold water solubility (%)	12.3	N/A	N/A
Hot water solubility (%)	15.7	10.77	14.23
Alcohol solubility (%)	6.3	N/A	N/A
Alcohol cyclohexane (%)	4.4	2.93*	3.03*
1% Sodium hydroxide (%)	34.1	39.6	40.64
Holocellulose (%)	74.6	N/A	N/A
Pentosan (%)	22.1	17.45	N/A
Cellulose, Kushner -Hoffer (%)	46.9	48.83**	42.22**
Acid insoluble lignin %	18.4	22.5	26.94
Cellulose / lignin ratio	2.4	2.17	1.57

N/A -Not Available

*alcohol benzene

** Alpha cellulose

Table 3: *Gossypium hirsutum* pulping conditions and unbleached pulp evaluation

Pulping		Pulp yield, %			Pulp evaluation.		
Proces	active alkali as Na O$_2$, %	Total	Screened	Rejects	Kappa No	Viscosity	Brightness
Soda							
CS1	17	41.6	39.5	2.1	27.2	833	16.5
CS3	15	43.5	36.2	7.3	31.6	801	12.3
Soda-AQ							
CS2	15	44.8	42.4	2.4	24.1	845	20.6
CS	4	17.0	42.4	41.2	0.2	22.2	85019.4
AS-AQ							
CS5 $_{(70:30)}$	13	45.6	44.5	0.1	25.7	909	33.5
CS6 $_{(60:40)}$	14	46.7	43.3	3.4	23.5	880	38.7
ASAM							
CS7	13	47.1	46.9	0.2	19.6	916	35.0

Other pulping conditions were constant
- Cotton stalks to liquor ratio was (1:4)
- Time to reach the maximum temperature was 90 min
- Holding time at the maximum temperature 120 min
- Maximum temperature of 165°C for soda and soda-AQ cooks
- Maximum temperature of 175°C for AS-AQ and ASAM cooks

The ash and silica contents were higher than those of Egyptian and American cotton stalks, but in normal range for pulpwood especially silica. This implies normal alkali consumption and fewer problems at waste liquor recovery [20]. The cold water (12.3%), hot water (15.7%) and organic solvents (4.4-6.3%) were much higher compared to the corresponding Egyptian and American cotton stalks, but they were rather higher than usual for commercial pulpwood and tropical species. This is due to the presence of high percentage of phenolic compounds and soluble polysaccharides. While, 1% NaOH extractives were 34.1% less than that of Egyptian and American cotton stalks but higher than normal pulpwood materials indicating an open anatomical structure of cotton stalks with an easy access of cooking liquor and degradation of cell wall materials by weak alkali. This implies that pulping chemical charges should be low-moderate. However, the high extractives with organic solvents are totally undesirable for pulping (pitch problems).

The cellulose Kurscher-Hoffer of Sudanese cotton stalks (Table 2), higher than alpha cellulose of American cotton stalks but lower than alpha cellulose of Egyptian one indicate acceptable to good yields. However, the presence of higher amount of Pentosans compared to the Egyptian cotton stalks would improve the solutions permeability during the pulping and accelerate the beating. The lower acid insoluble lignin meant low-moderate cooking chemicals. This is supported further by higher cellulose/ lignin ratio compared to the Egyptian and American cotton stalks.

Pulping of *Gossypium hirsutum*: The cooking conditions of cotton stalks were as follows, maximum temperature was in the range of 165-175°C, the time required to reach the maximum temperature was 120 min and the holding time at the maximum temperature was 90 min and these conditions were constants for all cooking trials. On the other hand, the alkali charges were varied from 13-17% as Na_2O with constant doses of anthraquinone of 0.1% was applied (Table 3). The black liquor analysis (Fig. 1) indicated the highest pH for soda cooking (CS1 and CS3), the ASAM cooking had the lowest pH implying that this process consumed most of chemicals, the same patterns were observed for residual active alkali and total solids.

In soda pulping of cotton stalks, alkali charge of 15-17% (Na_2O were applied as reference cooks) resulted in 41.6-43.5% total yield, 36.2-.39.5% screened yield, 27.2-31.6 kappa number, 12.3-16.5% ISO brightness and 801-833 ml/g viscosity. The addition of AQ with 0.1% dose to soda cooking improved the delignification rate (kappa number 22.2-24.1) with higher total yield 42.4-44.8% and screened yield 41.2-42.4% compared to the reference soda cooks. This could be attributed to the preservation of polysaccharides by the anthraquinone. Furthermore, it improved the strength properties (Figs. 2-6).

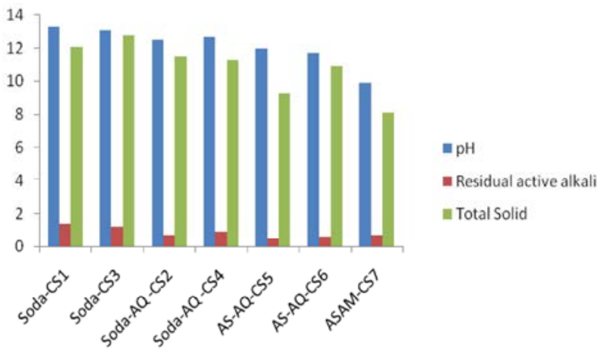

Fig. 1: Black liquor analysis for soda, soda-AQ, AS-AQ and ASAM cooking

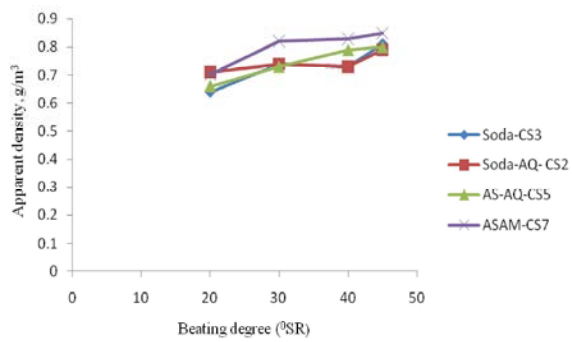

Fig. 2: Apparent density vs. beating degree (°SR) of *Gossypium hirsutum* unbleached pulps

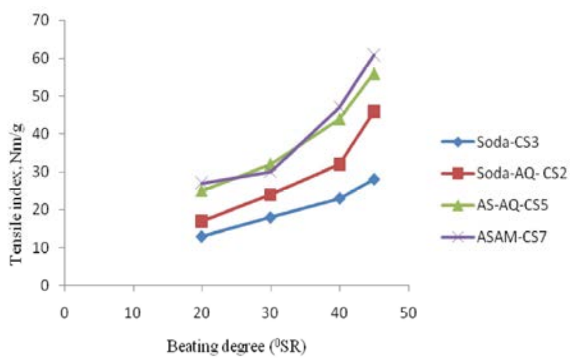

Fig. 3: Tensile index vs. beating degree (°SR) of *Gossypium hirsutum* unbleached pulps

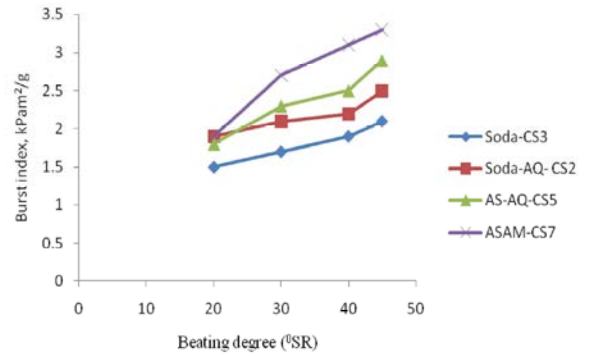

Fig. 4: Burst index vs. beating degree (°SR) of *Gossypium hirsutum* unbleached pulps

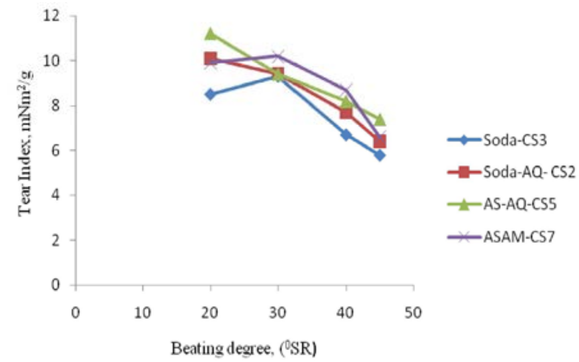

Fig. 5: Tear index vs. beating degree (°SR) of *Gossypium hirsutum* unbleached pulps

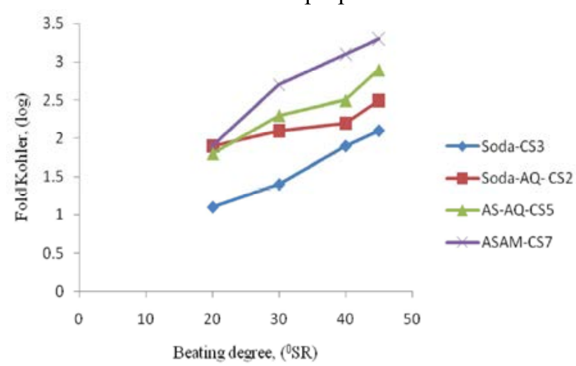

Fig. 6: Fold Kohler vs. beating degree (°SR) of *Gossypium hirsutum* unbleached pulps

When 70:30 (NaOH: Na_2SO_3) ratio used, compared to the 60:40 ratio during alkaline sulphite anthraquinone (Table 3), an improvement in the viscosity, reduction in total yield and rejects, increase in screened yield and lower brightness attributed to the higher share of sodium hydroxide was observed intensifying the delignification process. The overall results of AS-AQ cook reflect higher yield, viscosity, same kappa numberand excellent initial brightness in contrast to the soda-AQ cooks. The ASAM (CS7) at the same active alkali with addition of 15% v/v methanol had lower kappa number, better viscosity, brighter pulp sheets, higher total yield, screened yield and more or less same rejects amounts compared to the AS-AQ (CS5) cook. However, this indicates the suitability of cotton stalks cooking by ASAM method.

Comparison of the strength properties of cotton stalks unbleached pulps (Figs. 2-6) indicated, in general, the superior strength properties of the ASAM (CS7). The high bonding strength (tensile and burst indices) was mainly based on the good bonding ability of the fibers

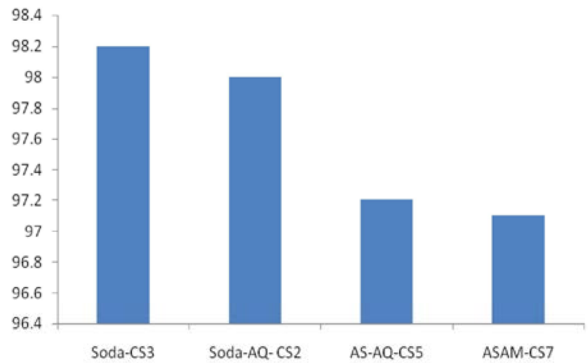

Fig. 7: Opacity (%), of *Gossypium hirsutum* unbleached pulps at beating degree of 45

resulted from the high carbohydrate content of ASAM pulps due to the high stability of xylan and cellulose in the outer cell wall layers. However, the AS-AQ (CS5) showed the same pattern. The soda pulps (CS3) reflected lower tensile burst and tear indices (Figs. 3 - 5). Although the soda pulps (CS3) had similar apparent density (Fig. 2), it reflected inferior tear resistance (Fig. 5) compared to soda-AQ pulps (CS2). Fig. 6 showed the highest folding Kohler for ASAM pulps (CS7). The most porous sheet was soda (CS3), while the ASAM (CS7) was the most compact sheet (Fig. 7).

CONCLUSION

The main objective of this work was to establish the suitability of *Gossypium hirsutum,* Sudanese cotton stalks as a potential source of pulping. By present work on the fibre properties, chemical composition and pulping optimization process, it could be concluded that: (i) the cotton stalks have short, narrow fibres but could collapse easily during beating due to the good morphological indices (ii) regardless of high extractive content mainly 1% NaOH, a good yield of pulping was obtained with acceptable to good Kappa numbers (iii) during AS-AQ pulping, the 70:30 $NaOH:Na_2SO_3$ ratio gave better pulping results compared to the 60: 40 ratio (iv) the ASAM cooking reflected the best pulp properties and yield within the applied methods in the study and (v) pulps from Sudanese cotton stalks could be blend with wood fibres to produce high quality paper.

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