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Contact information:
[1]University of Bahri, College of Natural Resources and Environmental Studies, Khartoum, Sudan.
[2]Industries Department, College of Applied and Industrial Sciences, University of Bahri Sudan, P.O. Box 1606, Khartoum, Sudan.
[3]Cellulose Chemistry and Technology Research Unit, National Centre of Research, Khartoum, Sudan.

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Abstract

Abelmoschus esculentus okra as whole stalks was examined for its suitability for pulp and paper production. It's, fiber dimensions, morphological and chemical characteristics were reported. The pulping trials with soda- Anthraquinone (AQ,) at different chemical charges. Application of 21% as NaOH with 0.1% AQ gave good results in degree of delignification, mechanical properties. Utilization of okra pulps and blender is recommended due to good pulp properties. Evaluation of general characteristics of okra stalks in terms of fiber dimensions morphological indices, chemical components, Soda-AQ cooking and to study their suitability for paper production. Okra Fiber dimension evaluation done after maceration with a mixture of 30% hydrogen peroxide and acetic acid (1:1) for core and bark parts separately and was carried out under microscope staining with aqueous safranin. The Soda-AQ cooks at different active alkali levels were calculated as NaOH on oven dry raw material. The fibers from okra stalks studied (core and bark) were in the range of hardwood fibers, with short fiber length, especially the core with more or less moderate walls, narrow lumen and fiber width. The fiber width of bark was medium –narrow with medium wall thickness. The ash content was rather high whereas the silica content was comparatively high The hot water extractives from okra stalks was (4.1%), cold water (0.4) ethanol/ cyclohexane (1.1), ethanol extractives (1.2%) and 1% NaOH (27.6%) were rather high. The cellulose (Kurschner-Hoffer) was (48.5%) The lignin content was (15.3%) which was relatively moderate. The use of 0.1% AQ enhanced the delignification in the three trials applied. The screened yield increase with increase of chemical dose applied while the rejects decrease. When 21% NaOH was applied, the screened yield was 32.2% with negligible amount of rejects, however with lower alkali charge 18% the screened yield was decreased to 28% with very low rejects 1.5%. on the other hand rejects were increased to 7% when 15% NaOH was applied with very low screened yield 19%.The pulps produced from okra soda-AQ are suitable for production of printing and writing papers and it is advisable to use them in blending due to good papermaking properties.

Keywords: *Abelmoschus esculentus* stalks, Fiber dimensions, Chemical composition, Soda-AQ cooking, Pulp properties

High lights

- Okra fruits is important vegetable but its stalks is burned in tropical and suptropical areas
- Soda-AQ cooking is suitable methods for pulping the agricultural residues.
- There is seldom literature on pulping of okra stalks and rational utilization is highly needed.
- In large areas in Asia and Africa, these stalks are burned and treated as waste.
- Production of paper pulps in good paper properties with low lignin content (kappa number) and improved viscosity could be expected.

Background

Non woody plants and agricultural residues as flax, cotton, bamboo and cereal straw were used extensively in pulp and paper production. However okra *Abelmoschus esculentus* stalks could be one of promising non woody plants in paper production [1, 2]. The family of okra (*Abelmoschus esculentus*) and cotton (*Gossypium hirsutum*) is Malvaceae, okra is one of the oldest cultivated crops, it was recorded by the Egyptians in 1216 A.D., however there was strong evidence, okra was cultivated

* Correspondence: tarigosmankhider@gmail.com
[2]Industries Department, College of Applied and Industrial Sciences, University of Bahri Sudan, P.O. Box 1606, Khartoum, Sudan
Full list of author information is available at the end of the article

earlier than that in Ethiopia, some other reports considered India as center of origin [3–6].The estimated annual production in the world was about 4.8 million tons; and the share of India (4.528 million tons), Nigeria (0.826 million tons), Sudan (0.249 million tons), Pakistan (0.116 million tons), Cote d'Iviore (0.115 million tons), Egypt (0.100 million tons) and Iraq (0.153 million tons) [7]. Okra is important food supplement in tropical regions and has nutritional and medicinal values, it is used traditionally in treating diseases as diabetes, pyretic syndrome, and spasmodic [7–11].

Stalk fiber of okra is characterized by high lignin, low α-cellulose, and shorter fiber length, while bast fiber is characterized by low lignin, high α-cellulose, and longer fiber length [12] the pulps obtained from okra stalks is good enough [13] with a high carbohydrates content (65.0%), low lignin contents (20.5%) and similar to chemical components of non-woody plants indicating okra stalks as promising source for papermaking utilization, in addition to that okra fibers can be applied as reinforcement in polymer composites [14–16].

The Anthraquinone (AQ) acts as redox catalyst during alkaline pulping, reduce aldehyde end groups of the carbohydrates, forming carboxylic acid, inhibits the alkaline depolymerization results in an increased yield. The soda-AQ cooking suitable for pulping non-woody plants and agricultural residues from economic and environmental points of views [17, 18].

The present study focused in evaluation the characteristics of Sudanese *Abelmoschus esculentus* okra stalks in terms of fiber morphological properties, chemical composition, Soda-AQ pulping and papermaking characteristics and therefore indicate their suitability for paper pulp production.

Methods

Abelmoschus esculentus okra (Fig. 1) stalks were selected randomly according to TAPPI Standards [19] from Marenjan area in Gezira state, central Sudan is characterized with clay soil with low to moderate annual rainfall. The length of stalks was 2. 5-3 m tall and 2-2.5 cm in diameter. The prepared raw materials were packed in plastic bags and transported by bus to National Centre for Research (NCR) in Khartoum state. Leaves and steams were separated manually, and then stalks were chopped into 2-4 cm length. Chips were left for air drying according to TAPPI standard (T 257-cm- 02) [19]. The air dry samples of core and bark of okra were manually separated and chopped to 3–5 cm length. Fiber dimension evaluation done after maceration with a mixture of 30% hydrogen peroxide and acetic acid (1:1) for core and bark parts separately and was carried out microscopically at 300x and 400x magnifications according to (TAPPI-232 cm-01) after staining with aqueous safranin [20, 21]. The raw materials was characterized chemically in accordance with applicable TAPPI standards for different components, namely: preparation for chemical analysis (TAPPI-264-cm-97) sampling and testing for moisture (TAPPI-210 cm-93) lignin (TAPPI-222), alpha cellulose (TAPPI-203 OS-61), hot water soluble (TAPPI-T- 207), Pentosans (TAPPI-223-cm-01) solvent extraction of wood (TAPPI-204), and ash (TAPPI-212) [22].

Fig. 1 *Abelmoschus esculentus* okra plant

According to the suggestions of other authors, the operation conditions used during soda-anthraquinone cooking of okra were selected, as follows: maximum temperature 165C^0, time to maximum temperature 60 min, time at temperature 120 min, anthraquinone concentration 0.1% and liquid/okra stalks ratio was 5. Representative sample of a portion prepared chips for pulping trails was ground in a star mill and the 40X60 mesh fraction was used for chemical analysis according to TAPPI standards and Obolenskaya [23].

All the conditions were constant except for soda concentration as (NaOH, 15-21% on oven dry weight of okra stalks) for optimization of cooking and the concentration of alkali was the most effective variable. After pulping, the cooked material was washed with water at room temperature to remove the residual cooking liquor and was fiberized in a disintegrator at 1200 rpm for 30 min, at room temperature and l0% consistency. The pulp was then beaten in Valley beater according to TAPPI-200-sp-01 freeness of pulp (Canadian standard method TAPPI 227om-99), Kappa number (TAPPI-236 om-99), viscosity (TAPPI-230om – 99), physical testing of pulp sheets (TAPPI-220-sp-01). Conditioning of testing atmosphere (TAPPI-402-sp-98), Burst strength (TAPPI-403om-97), Tensile (TAPP-404-cm-92), tearing resistance (TAPPI-414 om-98), folding endurance (TAPPI-423 cm-98) and density (TAPPI258-om-02) and ISO standards [24].

The chemical charges for all cooks at different active alkali levels were calculated as NaOH on oven dry raw material. All cooking conditions were kept constant (the pulping variables time to reach maximum temperature, time at maximum temperature, maximum temperature, the concentration of AQ on oven dry okra stalks and Liquor to okra stalks ratio, as shown in (Table 1) with chemical charges as NaOH were varied between 15 to 21%. Pulping was carried out in 7 l electrically heated digester with forced liquor circulation.

Results

The fibers from the two okra stalks studied (core and bark) were in the range of hardwood fibers, with short fiber length, especially the core (0.66 mm) as shown in (Table 2), with more or less moderate walls, narrow lumen and fiber width. The fiber width of bark was medium –narrow and in the hardwoods range (10-35 μm) the bark fiber has wall thickness (6.2 μm) could be classified as medium thick (Fig. 2).

The ash content of *Abelmoschus esculentus* okra stalks was rather high (6.3%) as indicated in (Table 3), but typical for tropical non-woody plants. The silica content was comparatively high as usual for such non-woody raw material. However the silica content was rather high (1.6%) so there is some problems during cooking. The hot water extractives from okra stalks was (4.1%), cold water (0.4) ethanol/ cyclohexane (1.1), ethanol extractives (1.2%) and 1% NaOH (27.6%) were rather high due to the presence of many soluble polysaccharides and phenolic compounds.

On the other hand they are an indication of easy access and degradation of the cell wall materials by weak alkali. The cellulose (Kurschner-Hoffer) was (48.5%) which meant good pulp yields at suitable alkali utilization. As Obolenskaya, [21] mentioned that the kurschner-Hoffer values are usually 4-7% higher than alfa-cellulose. The lignin content was (15.3%) which was relatively moderate. This should result in moderate cooking chemical charges and a short cooking cycle. Cellulose to lignin ratio was higher than 2 (3.2) and predicted normal pulping with alkaline methods [25]. The presence of water-soluble hemicelluloses in pulp fibers increases their swelling tendency and water absorption during beating as indicated by relatively high pentosans content (19.3%).

Table 1 *Abelmoschus esculentus* okra stalks*:* Pulping conditions

Cooking conditions	Soda-AQ1	Soda-AQ2	Soda-AQ3
Active alkali charge as NaOH %	15	18	21
Anthraquinone, %	0.1	0.1	0.1
Liquor to okra stalks ratio,	5	5	5
Maximum temperature, ^{0}C	165	165	165
Time to maximum temperature, min	60	60	60
Time at maximum temperature, min	120	120	120

Table 2 *Abelmoschus esculentus* okra stalks: fiber dimensions and morphological indices. Fibers after cooking conditions presented in Table 1

Fiber dimensions	Measured value of core	±SD	Measured value of bark	±SD
Fiber length, mm	0.66	0.09	0.92	0.08
Fiber width, μm	17.6	0.94	21.7	0.89
Lumen width, μm	6.4	0.95	9.3	0.97
Wall thickness, μm	5.6	0.41	6.2	0.22
Morphological indices				
Runkel index	1.8		1.3	
Wall fraction	63.6		57.1	
Flexibility coefficient, %	36.4		42.9	
Rigidity coefficient, %	31.8		28.8	
Felting power (slenderness)	37.5		42.4	

Anthraquinone (AQ) is a powerful redox-catalyst in alkaline pulping especially when non-woody raw material is cooked. The results of soda-AQ cooking of okra with 15-21% NaOH showed in Table 4. Use of 0.1% Anthraquinone (AQ) enhanced the delignification in the three trials. The screened yield and rejects were indicated in (Fig. 3), thus screened yield increase with increase of chemical dose applied with the rejects decrease, during cooking with alkali charge 21% as NaOH it seemed no or negligible amount of reject whereas during 15% NaoH trial about 7% rejects with lower degree of delignification (kappa number 29.6). Good viscosity, kappa number when high chemical charge applied (21%). However at lowest alkali dose 15% NaOH, highest kappa number (29.6), and lowest viscosity 1010 ml g^{-} 1. The brightness for the three cooks were more or less the same. According to Gierer [26, 27] the intermediate formation of quinonemethide is preceded by the cleavage of β-ether aryl-ether bonds.

The strength properties of three trails pulps (Table 4) indicated in general the slight difference in strength

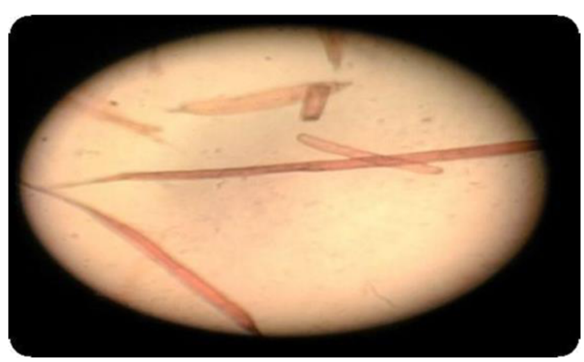

Fig. 2 *Abelmoschus esculentus* okra stalks, fiber structure under microscope

Table 3 The Chemical components of *Abelmoschus esculentus* (okra) whole stalks

Chemical Composition, %	Whole stalks
Ash	6.3
Total Silica	1.6
Solubility in	
Hot water	4.1
Cold water	0.4
Alcohol (Ethanol)	1.2
Ethanol: cyclohexane(1:2)	1.1
1% NaOH	27.6
Kurschner- Hoffer cellulose	48.5
Alfa-cellulose	43.7
Pentosans	19.3
Lignin	15.3
Total Extractives	9.3
Cellulose to lignin ratio	3.2

properties with relatively higher of 21% soda-AQ pulp properties.

The highest viscosity was for 21% soda-AQ which reflected positively for all strength properties especially at high degree of freeness except for tear resistance. Due to adverse effect of the large amount of fines produced, all soda-AQ had low initial brightness since alkali treatment induces darkening of the pulp as shown in (Fig. 4).

Discussion

The lumen width of the bark (9.3 μm) indicated that these fibers should collapse easily upon beating, resulting in improved interfibre bonding in the pulp and producing compact and low porosity sheet. The core had lower fiber dimensions and inferior morphological indices, indicating the bark as better pulpable material. This clearly showed by Runkel index and flexibility coefficient. Both two types of fibers could be classified in third group of Istas Classification [28], characteristic for fibers with fairly thick walls and rather narrow lumen. Although there is still controversy in accepting morphological data in predicting the properties of the pulp, the multi-regression technique seems undoubtedly valuable in showing which morphological characters are important in the pulp properties.

The amount of ash and silica content showed that ash composed of a high proportion of inorganic components other than silica. This should increase the alkali consumption and may cause some problems at waste liquor recovery [29]. The high silica content causes difficulties in the regeneration of chemical pulping, but might be beneficial in total chlorine free (TCF) bleaching where sometimes silicates are added for stabilization of cellulose. A high hot water soluble content also indicated a higher accessibility of the cell wall components to pulping liquor. Therefore, pre-extraction of the raw material before cooking might help to decrease the chemical consumption and obtain useful substances.

Table 4 Pulping results and strength properties of hand-sheets obtained from *Abelmoschus esculentus* okra stalks

Pulping Process		15% Soda-AQ	18% Soda-AQ	21% Soda-AQ
Pulping results				
Kappa number		29.6	27.8	22.2
ISO brightness, %		19.6	20.4	22.3
Viscosity, ml g^{-1}		1010	1045	1065
Strength properties				
Initial pulp freeness,	SR			
Apparent density, g cm^{-3}	25	0.52	0.61	0.61
	50	0.62	0.65	0.67
Breaking length, Km	25	7.5	8.1	8.1
	50	8.1	8.3	8.5
Tensile index, N m g^{-1}	25	40.1	43.2	45.3
	50	60.2	61.4	63.1
Tear index,, m N m^2 g^{-1}	25	7.8	7.4	7.2
	50	5.6	5.6	5.7
Burst index, K Pa m^2 g^{-1}	25	2.2	2.1	2.3
	50	3.6	3.9	3.7
Folding endurance, $log_{10}n$	25	06	0.9	0.9
	50	1	1	1.1

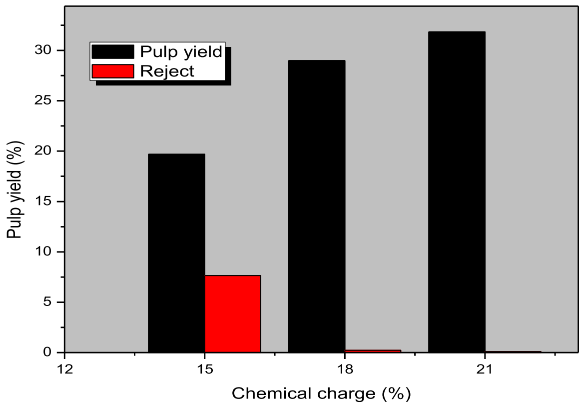

Fig. 3 Screened yield and rejects of *Abelmoschus esculentus* okra stalks versus chemical charge% as NaOH

It is well known that tensile strength, burst and double folds increase with increase in freeness level, this clearly reflected during application of freeness 25 and 50. The high tensile strength, which mainly based on the good bonding ability of the fibers, results from high pentosans content due to the high stability of xylan and cellulose in the outer cell wall layers. The high hemicellulose content also improved the beating [25, 29].

In paper production the primary role of hemicelluloses is to imbibe water and thus to contribute to fiber swelling. This leads to internal lubrication of the fiber and improves its flexibility and ease of beating. The swelling pressure contributes to loosening of the structure and fibrillation. The hemicelluloses being amorphous and adhesive in nature tend to hornify as the fiber shrinks and dries. Thus hemicelluloses serve as a matrix binding substance between fibers in a pulp. Burst and tear strengths are highly correlated to a high proportion of hemicelluloses [30, 31].

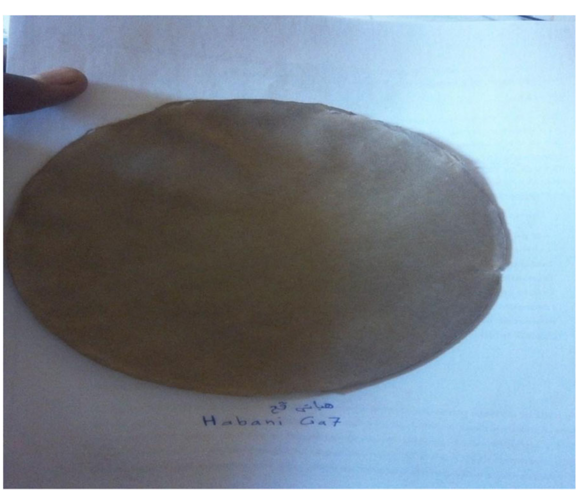

Fig. 4 Prepared hand sheet from pulp of *Abelmoschus esculentus* okra stalks for physical properties evaluation

The dominating source of darkening, however is definitely the condensed and degraded lignin. Nevertheless, too high alkali concentrations must be avoided especially at the end of the cooking procedure. Otherwise, overproportional degradation and dissolution of hemicelluloses and cellulose might take place, resulting in reduced yield and viscosity. The combination of low yield but good papermaking properties suggested that okra pulps could be used in blends with the short-fibered hardwood pulps.

Conclusion

The okra stalks has multiple purposes and could be promising raw material for pulp and paper production as agricultural waste. The use of soda-AQ pulping accelerated the delignification and beating rate, increased yield and viscosity and gave pulp with superior properties and highest alkali charge. The screened yield of 32.2% was achieved with negligible amount of rejects, at bleachable Kappa number 22.2 and viscosity 1065 ml g^{-1} during application of high alkali charge 21% as NaOH. Decreasing this alkali charge to 18% gave screened yield 28% with very low rejects 1.5% kappa number 27.8 and viscosity 1045 ml $g^{-1.}$ The rejects were increased to 7% during application of 15% NaOH with high Kappa number 29.6 and viscosity 1010 ml g^{-1}. The fiber dimensions and morphological indices represented okra stalks as in range of tropical non woody plants. The chemical composition of okra gave indication of suitability of soda-AQ cooking for these agricultural residues. The pulps produced are suitable for production of printing and writing papers. Due to relatively low screened yield and good papermaking properties, it is advisable to blend these pulps with other high yield pulps.

Additional file

Additional file 1: Physical properties of Abelmoschus esculentus (Okra plant). (DOCX 795 kb)

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Availability of data and materials
We have already included most of data in the manuscript, the lab and data, some data not included in the manuscript attached as Additional file 1.

Authors' contributions
SHO and SKS were carried out, supervised the chemical analysis, soda-AQ pulping and revised the draft manuscript. OTE did the fibers dimensions and

their analysis, review the draft manuscript, SDM supervised and carried out the evaluation of paper properties and revised the draft manuscript. TOK wrote the draft manuscript, designed the study and supervised the work. All authors read and approved the final manuscript.

Ethics approval
Not applicable.

Consent for publication
Not applicable.

Competing interests
The authors declare that they have no competing interests.

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Author details
[1]University of Bahri, College of Natural Resources and Environmental Studies, Khartoum, Sudan. [2]Industries Department, College of Applied and Industrial Sciences, University of Bahri Sudan, P.O. Box 1606, Khartoum, Sudan. [3]Cellulose Chemistry and Technology Research Unit, National Centre of Research, Khartoum, Sudan.

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