

Exploring Sudanese agricultural residues as alternative fibres for pulp and paper manufacturing

H A M Saeed^{1,2*}, Y Liu^{1*} and H Chen¹

¹Key Lab of Pulp and Paper Science and Technology of Ministry of Education (Shandong Province) Qilu University of Technology, Jinan, 250353, Shandong, P.R. China.

²Center of Fibers, Papers and Recycling, Faculty of Textiles, University of Gezira, Box 20, Wad Medani, Sudan.

*E-mail: haroonsaeed75@gmail.com, leoliuyu@163.com

Abstract. In this work, the chemical composition, fibre dimensions and morphology, elemental analysis and paper characterisation of sesame, millet, karkadeh and sorghum stalks were studied. Soda and soda-anthraquinone pulping as sulfur-free processes were applied. It was found that, the cellulose content of karkadeh, sesame, millet and sorghum stalks were 45.80, 42.30, 40.99 and 35.40%, respectively. Klason lignin was found to be highest in sesame 20.85% while it was 19.32, 18.20 and 10.32% in karkadeh, millet and sorghum, respectively. Moreover, karkadeh showed highest pulp yield (46.60% and 43.50%) with viscosity of (655 ml/g and 640 ml/g) and kappa number of (18.00 and 19.40) for soda-AQ and soda pulps, followed by sesame, okra and sorghum, respectively. Overall strength properties of the handsheet made from karkadeh pulp were significantly higher than those made from sesame, millet, and sorghum pulps. Morphological analysis and chemical composition of the studied fibrous materials showed their suitability for producing paper of various grades including writing and printing paper as well as packaging applications.

1. Introduction

An increase in pulp and paperboard consumption, stricter environmental and sustainability regulations, and the increased use of wood materials for furniture production have prompted scientists and researchers to seek additional lignocellulosic material for pulp and papermaking [1]. However, agricultural residues have become one of the most important alternative sources of fibrous resources in the 21st century [2]. However, agricultural residues are not sometimes used eco-friendly as after harvesting they are directly burnt in opened land under not well-controlled conditions, causing destructive environmental impact due to the heavy smog [3-4]. Agricultural residues cellulosic materials can be applied as an effective alternative to constantly decreasing forest resources in different regions especially in the developing countries [5-6]. However, to make better utilization of these cheap and abundant agricultural residues, these stalks could be considered as promising cellulosic raw materials for nano-cellulose pulp and paper making [2, 7].

Sudan is rich in agricultural residues cellulose materials, which can be good candidate cellulosic materials for pulp and paper manufacturing [8]. However, this article investigates the application of Sudanese agricultural residues, namely; sesame, millet, karkadeh and sorghum in pulp and paper making.



Sesame belongs to Pedaliaceae family, it is one of the most common traditional oilseed crops in the world. Sesame crop belongs to the tropical climatic zone; it is largely grown in India, China, Myanmar, and Sudan [9-10]. In Sudan, sesame grown on stored soil moisture with minimal irrigation or rainfall and can produce good yield under high temperature in many states. However, it represents the third important crop after sorghum and millet [11]. Ateş, F. *et al.* [12] investigated the pyrolysis of sesame stalk as biomass, they found that sesame stalk had 48 % cellulose content. This high cellulose content of sesame stalks make it a promising candidate for pulp and paper making.

Millet known as "Dukhun," is the most important cereal crop in Sudan (Kordofan and Darfur States). The average total area annually cultivated is approximately 2.5 million hectares [13] its application in pulp and paper manufacturing can be more beneficial because it is abundant, inexpensive, and can provide economic and environmental benefits [14]. Harinarayana, G., *et al.* [15] investigated millet stalks chemical composition, they found that the millet stalks were rich in cellulose content (39.4 %), hemicellulose (23.9 %), and relatively low in lignin (12.8 %), thus representing a promising feedstock.

Karkadeh belongs to Malvaceae family, is commonly known as Roselle, hibiscus, Jamaica sorrel or red sorrel (English) and in Arabic, karkadeh [16]. It is an ideal crop for developing countries as it is relatively easy to grow, can be grown as part of multi-cropping systems and it has multi usages. It can grow annually and attain a height in range of 2 to 2.5 m [17-18]. In Sudan, karkadeh is grown in many different areas, it is one of the cash crops cultivated by traditional farmers in Kordofan and Darfur states under rain-fed conditions, where large quantities are produced both for local consumption and for export [19]. Khristova, P. and M. Tissot [20] studied the chemical composition of the karkadeh, the results shown that it constituted of 45.56 % cellulose, lignin 20.11% and 2% ash. Sorghum is an important crop in Sudan, and the country ranked eighth in the world in sorghum grain production (3.5 million tonnes) in 2014 [21]. Jiménez *et al.* [22] studied the application of sorghum stalks in pulp and papermaking and determined they had better properties than other agricultural residues, such as olive tree fellyings, wheat straw, sunflower stalks, vine shoots, and cotton plant stalks. Gençer *et al.* [23] identified conditions for producing pulp and paper from sorghum. Indeed, the length of sorghum stalks is sufficient to make them a viable alternative for pulp and paper production.

Agricultural residues materials are beneficial in pulp and paper industry in terms of environmental and socio-economic aspects. Due to this reason it was thought that these stalks could be utilised as an alternative resources for pulp and paper making. In his study, sesame, millet, karkadeh and sorghum stalks were investigated on their fibres dimensions and morphological properties, chemical composition, soda and soda-anthraquinone pulping and pulp and paper properties.

2. Experimental

2.1 Materials

Sesame, millet, karkadeh and sorghum stalks were collected from a farm in north Kordofan State (latitude 15° 40' N and 3° 32' E) Sudan in February 2016. The stalks were air dried for weeks then fragmented separately into small chip sized 25 x 25 x 2.5 mm using band and dick saw. All the chemicals were purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China) and were used without further purification.

2.2 Chemical composition

The chips were grinded in Wiley mill to produce sawdust on 40/60 mesh, which was analyzed for chemical composition. The amount of extractive in hot and cold water, 1% sodium hydroxide and benzene-ethanol mixture (20/80 v/v) were determined using the TAPPI standard methods; TAPPI T207 cm-08, TAPPI T212 om-07 and TAPPI T204 cm-07, respectively [24]. The mean chemical components; ash and klason lignin, were determined according to TAPPI T 211 om-02 [25] and

TAPPI T211-om-93, while holocellulose was determined by Wise's chloride method [26]. Moreover, acid-soluble lignin was determined based on the method of UV-vis spectro-metric at wavelength 205 nm TAPPI um 250 [27].

2.3 *Pulping*

The pulping of these agricultural residues was carried out using soda and soda-anthraquinone process. However, pulping were performed in electrically heated laboratory rotary a 5-L stationary stainless steel digester consisting of 4 vessels each with a capacity of 1 L (NAC Autoclave Co. Ltd., Japan) according to . A total of 100 g of each material were cooked in 25 % a freshly prepared caustic soda. The amount of anthraquinone was kept at 0.1% (w/w), with respect to the oven dried (o.d.) initial solid material for soda-AQ pulping. Liquor-to-material ratio of 5:1, time to maximum temperature of 60 min, maximum temperature of 160 °C, cooking time at maximum temperature of 120 min. At the end of pulping, the pulp was pressed to remove the liquor then washed with running water to neutral pH, then, disintegrated in a pulp mixer and screened on a 0.15 mm slotted plate. The pulp yield was determined by gravimetric method as dry matter obtained on the basis of oven dried raw material.

2.4 *Pulp Properties*

Kappa number, Canadian Standard Freeness (CSF) and viscosity, were determined according to the following methods TAPPI T236 om-99, TAPPI T227 om-99 and TAPPI T230 om-89, respectively [28]. Totally chlorine-free bleaching (ECF) was carried out for the screened pulp in polyethylene bags in a water bath at 10% pulp concentration using (D₀ED₁) bleaching sequence (D: chloride 3% ClO₂, Ep: alkaline-peroxide 1% NaOH) at 80 °C for 2 h for each stage [29].

2.5 *Paper making and testing*

The screened pulp was beaten in a Hollander beater for 5000 revolutions and 35 °SR freeness according to TAPPI T248 sp-00. Standard handsheets with basis weight of 60 g/m² were made using a laboratory hand-sheet former (PTI laboratory Equipment, Vorchdorf, Austria) according to the TAPPIT 205 sp-95 standard method.

2.6 *Paper Properties*

The handsheets properties were tested based on the following standards: apparent density TAPPI T 411 om-89, tear index TAPPI T 414 om-98, tensile strength TAPPI T 494 om-01, burst index TAPPI T 403 om-97, opacity TAPPI T1214 sp-98 and brightness TAPPIT1216 sp-98.

2.7 *The elemental analysis*

The elemental analysis (Carbon (C), oxygen (O), Aluminium (Al), Nitrogen (N), Silicon (Si), Magnesium (Mg), and Calcium (Ca)) were observed under a scanning electron microscope (SEM) (OCTANE 9.88/1114658 AMETEK®, Mahwah, USA). Before testing, paper samples were gold coated in a Sputtergerät SCD 005 sputter coater (England). A sputter current of 60 mA, sputter time 90 s.

2.8 *The fibre morphological characteristics*

For the measurements of fibre's dimensions, samples were macerated in a mixture of 30% hydrogen peroxide and acetic acid (1:1). Fibre length, width and kinked and curled indices were analyzed with an FQA device (LDA02128, OpTest Equipment Inc., Ontario, Canada), model LDA-02 according to TAPPI T271 om-07. Lumen diameter and wall thickness were measured by Leica DMLB (Leica Microsystems GmbH, Wetzlar, Germany) connected to a video camera Leica DFC490 (Leica Microsystems GmbH, Wetzlar, Germany) at 1000× magnifications.

3. **Results and discussion**

3.1 Chemical composition of residues material

Table 1 shows the percentages of chemical contents and some solubility values in sesame, millet, karkadeh and sorghum stalks. Karkadeh shown the highest cellulose content 45.80 % with low lignin 19.32 % , while cellulose and lignin contents are (42.30 and 20.85 %), (40.99 and 18.20 %) and (35.4 and 10.35 %) of sesame, millet and sorghum stalks respectively. Compared to other non-woods, karkadeh millet, and sesame shown same cellulose content as date palm 45.04 % and wheat straw 47.14 % [30], pineapple leaves 41.15 % [31] and higher than cotton and corn stalks [32]. In pulp and paper, a high cellulose content is preferable because it leads to produce high pulp yield as well as the quality paper. However, less lignin content is desirable in the pulp and paper because less chemicals and energy are required during the pulping process. Furthermore, low lignin will also produce paper with good optical properties and therefore requires less bleaching [33-34]. These raw fibrous materials content less lignin than rapeseed straw, rice straw and corn straw [35].

Table 1: Chemical composition of sesame, millet, karkadeh and sorghum stalks

Chemical Composition (%)	Sesame	Millet	Karkadeh	Sorghum
Cellulose	42.30±2.2	40.99±2.5	45.80±2.5	35.40±1.8
Hemicellulose	22.10±2.1	20.90±2.0	22.82±2.4	19.41±1.3
Klason lignin	20.85±1.3	18.20±1.8	19.32±1.4	10.27±0.8
Acid Insoluble Lignin	0.05±0.01	0.06±0.01	0.06±0.01	0.05±0.01
Ash	4.50±1.0	5.96±0.5	3.20±1.1	5.31±0.6
Hot water extractives	10.70±1.2	10.20±1.4	8.60±1.3	13.20±1.2
Cold water extractives	5.80±1.1	4.50±0.7	4.60±1.0	11.60±1.1
Alcohol-benzene	5.90±1.1	4.60±1.1	3.40±1.2	5.20±0.7
1% NaOH	20.30±2.5	21.30±2.0	19.55±2.3	16.20±1.6

Hemicellulose content of karkadeh (22.82 %) is the highest compared to sesame (22.10 %), millet (20.90 %) and sorghum (19.41 %) as shown in Table 1. High hemicellulose content is granulated for pulp and paper making because it enhance the mechanical properties of the paper produced particularly in tensile and burst index [36]. However, paper produced from karkadeh are expected to have high tensile strength and burst index. The highest ash was observed in millet stalk 5.90%, while karkadeh shown the lowest 3.20 %. However, the lower ash content is desirable, because, it expected to decrease chemicals required during pulping and bleaching.

Solubility in water is the measure of the extractive compositions of the lignocellulosic material. However, the high extractives values were indicating high presence of tannins, alkaloids and starch [37]. However, sorghum shown the highest amount of extractives in hot and cold water (24.80 %) compared to that (16.50, 14.70 and 13.20 %) for sesame, millet and karkadeh, respectively. These values are very high compared with that found in hardwood (2–8 %) [38] but similar to that of some non-wood materials such as sunflower and corn stalks [5], and less than that of rapeseed straw reported by 18 % [35]. Moreover, the solubility in different solvents indicated the extractive contents, which were not cell wall components [33].

The solubility predicts the mass loss of the carbohydrates and other alkali soluble matters in raw fibrous materials [4]. On the other hand, high solubility suggests high mass loss during the pulping process and low pulp yield [39]. The high extractives contents of okra led to decrease in pulp yield, thus, increasing in chemicals needed in cooking and bleaching [5].

3.2 Pulp characterization

Table 2: Properties of pulp from sesame, millet, karkadeh and sorghum stalks

Material	Pulp yield (%)		Kappa number		Viscosity (ml/g)		CSF (ml)	
	Soda	So-AQ	Soda	So-AQ	Soda	So-AQ	Soda	So-AQ
Sesame	40.60	43.30	20.31	18.17	620	635	410	380
Millet	40.10	42.04	14.50	12.43	650	665	560	555
Karkadeh	43.50	46.60	19.40	18.00	640	655	425	405
Sorghum	33.00	33.15	16.30	15.12	670	744	440	430

The properties of the pulps obtained from the sesame, millet, karkadeh and sorghum stalks are listed in Table 2. The results reveal that there were some differences in pulps properties of the investigated agricultural residues produced by the two pulping methods. However, pulp yield for the studied materials was in the range of (43.50 to 33.00 %) when cooked with soda process while the yield was increased by 3 % with the use of 0.1 % AQ similar results were reported previously [40]. Among the studied materials, karkadeh shown the highest pulp yield regardless the pulping methods, this is attributed to the high cellulose content in karkadeh stalks. However, a higher pulp yield with soda-AQ pulps, compared to the soda method, was due to higher hemicellulose retention [41]. Moreover, the combination of AQ to soda may accelerates the delignification rate and protect cellulose degradation, which was illustrated by the quantity of pulp yield [42]. Similar results have been observed by several authors [43]. On the other hand, sesame pulp shown the highest kappa numbers of 20.13 and 18.17 for soda and soda-AQ pulps, respectively, while millet shown the lowest kappa numbers 14.50 and 12.34 for soda and soda-AQ pulps, respectively. It is noted that pulp yield improved and kappa number reduced when soda method was modified by AQ, this result could be attributed to the acceleration of delignification rate with the addition of AQ [43-44]. Moreover, low kappa number also led to less consumption of bleaching chemicals, which directly led to decreasing the effluent load [33]. It was reported that the viscosity decreased with decreasing yield percentages for both types of pulping [45]. However, among the studied materials, sorghum shown highest viscosity (670 and 744 ml/g) while sesame shown the lowest (635 and 620 ml/g) for soda-AQ and soda pulp, respectively.

Table 3: Fibres properties of sesame, millet, karkadeh and sorghum stalks

Material	Sesame	Millet	Karkadeh	Sorghum
Fibre length (mm)	0.80±0.2	0.41±0.3	0.92±0.3	0.52±0.3
Fibre diameter (µm)	16.30±1.2	27.10±1.5	14.00±1.5	26.80±1.4
Lumen diameter (µm)	14.20±1.3	13.31±1.4	15.20±1.4	14.10±1.2
Cell wall thickness (µm)	6.80±0.3	7.40±0.4	6.20±0.2	6.10±0.3

As shown in Table 3, the results reveal that these materials contained short fibre. However, karkadeh shown the highest fibre length 0.92 mm [46] compared to that of 0.80 mm of sesame, 0.65mm of sorghum and 0.41mm of millet. These results are similar to pea [47], *C. orientalis* and *C. tataria* [48]. However, based on the results of the fibre length, karkadeh produced paper is expected to show best tea strength. On the other hand, higher fibre length improve tensile properties of paper but causes distortion of paper [49]. The average fibre diameter of karkadeh 14.00 µm is relatively smaller than that of sesame 16.30 µm, 26.80 sorghum and 27.10 µm millet and. A small diameter of fibre indicates that less fibre flexibility. Lumen width of the fibre has remarkable influence on the beating of the pulp. The wider the lumen width, better responses to the beating of the pulp can be observed because of the permeation of liquids into free spaces in and between the fibres. The thickness of the fibres cell wall play important role on most paper properties, with thick-walled fibres forming bulky sheets with less quality of tensile and burst indices and folding endurance but with a high tearing

index [33]. It was reported that increase in fibre length and decrease in cell wall thickness had noticeable effects on the physical properties of paper [48]. However, millet stalks shown highest cell wall thickness 7.40 μm compared to sesame 6.40 μm and followed by karkadeh 6.20 μm and sorghum 6.10 μm . These are comparatively similar to kenaf 6.4 μm [42] and sunflower stalk 5.9 μm [7].

3.3 Elemental analysis

Table 4: Elemental analysis of sesame, millet, karkadeh and sorghum stalks

Material	C (%)	O (%)	N (%)	Mg (%)	Al (%)	Si (%)	Ca (%)
Sesame	52.60	41.20	4.20	0.34	0.54	2.80	0.45
Millet	45.21	20.90	2.09	0.38	0.17	0.48	0.50
Karkadeh	50.80	39.30	2.40	0.24	0.07	3.05	0.55
Sorghum	43.54	51.14	2.02	0.53	0.23	0.57	0.75

The SEM-EDS elemental analysis is shown in Table 4. However, the results reveal that the predominant elements in paper obtained from the studied materials were C and O and lesser amounts of mineral elements such as N, Mg, Al, Si and Ca were also discovered. Among the studied material sesame stalks shown highest C and O content (52.60 and 41.20%), respectively [50] compared to (50.8 and 39.30%) of karkadeh, (43.54 and 51.14%) of sorghum and (45.21 and 20.90%) of millet. However, similar results were observed in wheat straw [51], corn hull [52] and rice husk and bagasse [53]. However, the mineral substances appear in these agricultural residues do not present any counter indication for chemical pulping.

3.4 Paper Properties

Table 5: Physical properties of papers from of sesame, millet, karkadeh and sorghum stalk.

Properties	Sesame		Karkadeh		Sorghum		Millet	
	Soda	So-AQ	Soda	So-AQ	Soda	So-AQ	Soda	So-AQ
A. D (g/cm^3)	0.50	0.54	0.55	0.58	0.51	0.53	0.40	0.41
T. I (N m/g)	45.90	46.80	51.70	52.30	47.10	50.70	32.15	33.95
Te. I (mNm^2/g)	3.80	4.10	4.60	5.50	2.40	2.61	2.28	2.31
B. I (KPa m^2/g)	3.40	3.10	3.50	3.90	2.15	2.55	1.90	1.96
Opacity (%)	85.40	86.50	86.60	87.90	81.40	82.60	88.00	88.50
B (%)	65.90	67.25	75.50	77.64	67.10	71.15	65.10	66.30

AD: Apparent Density, T I: Tensile Index; Te.I: Tearing Index; B.I: Bursting Index; B: Brightness.

The paper properties tested were those pertinent for assessment the quality of liner and fluting papers [54]. Among the studied materials papers produced from karkadeh pulp shown the highest tensile strength (52.30 and 51.70N m/g), tearing indices (13.50 and 12.60 mNm^2/g) and bursting indices (3.90 and 3.50 KPa m^2/g) for soda-AQ and soda pulp, respectively Table 5. Similar result were reported by [55-56]. This result may be due to the higher fibre flexibility, higher bonding ability, higher slenderness ratio [7] and lower Runkel ratio [43] of the karkadeh pulp fibres. On the other hand, millet hand sheet shown the lowest tensile strength (33.95 and 32.15 N m/g), tearing indices (2.31 and 2.28 mNm^2/g) and bursting indices (1.96 and 1.90 KPa m^2/g) for soda-AQ and soda pulp,

respectively. Comparing the paper properties obtained from soda and soda-AQ pulps, the results reveal that the mechanical properties of the paper samples were influenced by addition of AQ as well as the delignification degree through the reaction with quinone methide intermediates of lignin [42, 43]. Furthermore, the additional removing of lignin and extractives and formation of new hydrogen bonds between molecules of cellulose and hemicelluloses may also led to increasing in paper physical properties [5]. Moreover, the soda pulp of the studied materials shown a lower brightness than that soda-AQ.

Hand sheet produced from karkadeh fibres shown highest apparent density (0.58 and 0.55 %) compared to that (0.54 and 0.50%) of sesame followed by (0.53 and 0.51 %) of sorghum and (0.41 and 0.40 %) millet for soda-AQ and soda pulp, respectively. It was reported that the apparent density of the hand sheet increased with the increased of short fibres content in the pulp that led to increases the fibre packing efficiency . The apparent density of soda -AQ pulps were generally higher than soda pulps, this is could be due to highly refined flexible fibres obtained by the soda- AQ pulping which increase the fibre packing efficiency [57].

The best bleachability and highest brightness (77.64 and 75.50 %) was achieved by karkadeh for soda-AQ and soda pulps, respectively, while it was (71.15 and 67.10 %), (67.00 and 65.50 %) and (66.30 and 65.10 %) of soda-AQ and soda pulps of sorghum, sesame and millet respectively. However, brightness represent the kappa number, which in turn to evaluates the level of delignification. Opacity is an important aspect of paper appearance [58]. Millet showed the highest opacity of (88.50 and 88.00 %) followed by karkadeh (87.90 and 86.60 %) for soda-AQ and soda pulps, respectively, while it was (86.50 and 85.40 %) and (85.60 and 84.40 %) of soda-AQ and soda pulps of sesame and sorghum respectively. This could be attributed to the extraction of lignin during pulping which led to replace hemicellulose on the fibre surfaces, therefore, hemicellulose may enhances fibre-to-fibre bonding, this will led to less voids between the fibres then reduces the light scattering at fibre-air and pigment-air interfaces [58].

3.5 *Surface Morphology Analysis*

Scanning electron microscopy (SEM) analysis of the paper produced from sesame, millet, karkadeh and sorghum fibres were magnified at 1000X are shown in Figure1 (A, B, C &D). However, the strength of the fibre can be understood based on the order and formation of the fibre pattern [59]. From Figure 1 (B), karkadeh handsheet fibres are parallel, aligned and closed to each other rather than that of millet Figure 1 (A), sesame Figure 1 (C) and sorghum Figure 1 (D). This is attributed to the higher fibre content as well as long fibres in karkadeh compared to the other species. Moreover, in Figure 1 (B) as can be seen that the structure of the karkadeh fibres are closely compacted and formed many more bundles of fibre matrix and fibres are quite homogeneous and resembled more than that of millet, sorghum and sesame handsheets.

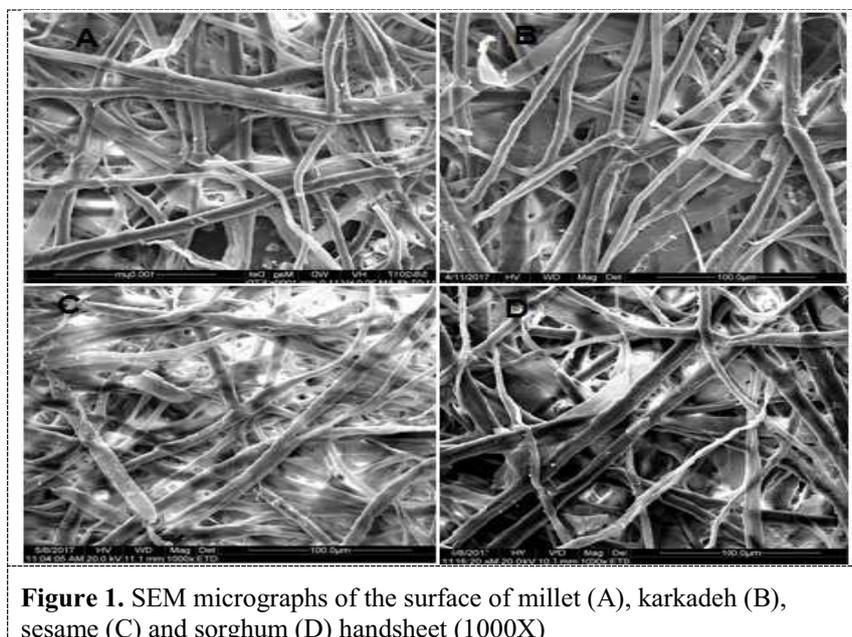


Figure 1. SEM micrographs of the surface of millet (A), karkadeh (B), sesame (C) and sorghum (D) handsheet (1000X)

4. Conclusion

Agricultural residues could be considered an interesting and promising candidate as cellulosic fibre source for pulp and paper industry since they are abundant, inexpensive and renewable. Moreover, the high cellulose contents (45.80, 42.30, 40.99 and 35.40 %) and low lignin contents (19.32, 20.85, 18.20 and 10.35 %) of karkadeh, sesame, millet and sorghum stalks, respectively, led to high-quality pulp and paper with good physical properties. The use of soda-AQ pulping process for these agricultural residues helped delignification, increased pulp yield and enhanced the paper mechanical properties. However, the results showed that a lower kappa number, higher screened pulp yield, and better paper properties were obtained for the pulp produced via soda-AQ pulping than the soda process. As paper is usually a mixture of short fibres and long fibres, the morphological analysis shows that the studied fibres can be as promising short fibres for various paper grades.

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