Sudanese Agro-residue as a Novel Furnish for Pulp and Paper Manufacturing

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Sudan has rich sources of lignocellulose materials from agricultural waste that have potential to be used as a papermaking furnish following adequate chemical compositions, elemental analysis, fibre dimensions, and morphology of millet stalks and date palm leaves. Paper sheet properties from the various pulps made were investigated, and it was found that there was no difference in the polysaccharide (cellulose and hemicelluloses) content between millet stalks and date palm leaves, although millet stalks had a high lignin content of 18.20% relative to date palm leaves' content of 15.34%. Moreover, millet stalks showed a high pulp yield (42.04%) with a viscosity of 665 mL/g compared to that (34.43%, 551 mL/g) and (38.50% and 534 mL/g) of date palm leaves and the blend, respectively. Papers produced from date palm leaves and millet stalk blends showed better physical properties compared to that of pure millet stalks and date palm leaves. The Scanning Electron Microscopy (SEM) analysis showed that fibres in the blend were more closely packed than that of the pure millet stalks and date palm leaves fibers. Based on their physical and chemical composition properties, millet stalks and date palm leaves have a high potential as a furnish for pulp and papermaking.

Key words: Sudanese agro-residue; Millet stalks; Date palm leaves; Cellulose; Pulp and paper

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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 (Danielewicz and Surma-Slusarska 2011; Xing *et al.* 2016). Globally, non-wood cellulosic materials are a major part of raw material inventories for pulping and paper. Sudan is rich in non-wood cellulose materials, such as bagasse, cotton linters, sorghum, sunflower, millet and sesame stalks, and date palm rachis and leaves, which can be used in pulp and paper (Elzaki *et al.* 2012).

The use of agricultural residues in pulp and papermaking has many benefits for farmers and the environment, such as reducing the need for waste disposal, which currently increases farming costs and sustainability by reducing environmental pollution, fires, and pests (Hammett *et al.* 2001; Ashori 2006). Compared with other classical pulping processes, soda pulping is the most economical, efficient, and simple for non-woody feedstock. Soda-anthraquinone pulping has been applied to various agricultural residue non-wood materials, such as bagasse (Samariha and Khakifirooz 2011; Sánchez *et al.* 2016), banana (Rosal *et al.* 2012), and cotton stalks (Khider *et al.* 2012), but no work on Sudanese millet stalks and date palm leaves has been reported to date.

Pearl millet (*Pennisetum glaucum*) 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 ha. However, millet stalks are used as feed for animals and mostly used as building material or fuel (Abuelgasim 2011). Their application in pulp and paper manufacturing can be more beneficial because they are abundant, inexpensive, and can provide economic and environmental benefits (Elzaki *et al.* 2012; Dulermo *et al.* 2016). Harinarayana *et al.* (2005) 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.

Date palm (*Phoenix dactylifera*) is common in Northern Sudan along the Nile (El Amin 1990). Khristova *et al.* (2005) investigated alkaline pulping with additives of date palm rachis and leaves from Sudan. Date palm rachis gave the best yields and displayed the best mechanical properties. The leaves were best pulped with soda at a low yield with very good strength properties. Nevertheless, date palm is a promising material for the paper industry. Khiari *et al.* (2010) studied the Tunisian date palm rachis as an alternative source of fibres for papermaking applications. It was found that the physical properties of the prepared handsheets were like those displayed by other papers made of common lignocellulosic fibres. Moreover, the pulps displayed good drainability together with excellent mechanical properties. No similar previous study was conducted, and only a few studies have evaluated the potential use of Sudanese date palm leaves for pulp and paper manufacturing (Khristova *et al.* 2005).

The authors herein study millet stalks and date palm leaves to create a novel furnish for pulp and paper manufacture with resources available in Sudan.

EXPERIMENTAL

Materials

Millet stalks (*Pennisetum glaucum*) were collected from a farmer in north Kordofan state, Sudan, January 2016. They were harvested from a tropical and subtropical area of low soil fertility and limited moisture where rainfall ranges from 200 mm to more than 1000 mm. The date palm leaves (*Phoenix dactylifera*) were collected from 7 to 9-year-old trees from Khartoum state, Sudan, January 2016. The air-dried samples were cut 3 to 5 cm in size and a part of each individual sample was ground in a star mill. The 40-mesh to 60-mesh fraction was analyzed according to TAPPI standards. Hydrogen peroxide (H_2O_2), sodium hydroxide (NaOH), and anthraquinone were purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China).

Methods

Chemical composition

Holocellulose was determined by Wise's chloride method (Wise and Jahn 1952), and cellulose (Rowell 2005), as well as lignin and ash, were determined per TAPPI T222 om-06 (1996) and TAPPI T211 om-93(1993), respectively.

Pulping processing and testing

The pulping process of millet stalks, date palm leave, and their blend (50% of each raw material) was performed in a 10-L capacity rotating autoclave according to Khiari *et al.*(2010) with a total alkali solution with 20% (w/w with respect to oven dried (o.d). material) expressed in NaOH, in the presence of an anthraquinone concentration of 0.1% (w/w with respect to o.d. material) at a constant temperature for 150 min. The liquor to solid ratio was 5:1 and the cooking temperature was 160 °C. After cooking, the pulp was washed, disintegrated in a laboratory disintegrator, and then screened on a 0.15-mm laboratory slot screen. The pulp yield was calculated and the kappa number and freeness of pulp were determined according to TAPPI T236 om-13 (2013) and TAPPI T227 om-99 (1999), respectively, while the pulp viscosity was determined according to TAPPI T230 om-08 (2008). Pulp bleaching was performed in two stages for 2 h at a 10% pulp concentration by hydrogen peroxide (H_2O_2) at 4% in sodium hydroxide (NaOH) at 80 °C.

Papers making and testing

Sets of hand-sheets paper (60 g/m^2) were made from the pulp of the samples using a laboratory hand-sheet former (PTI laboratory Equipment, Vorchdorf, Austria) according to the TAPPIT 205 sp-95 (1995) standard method. All of the hand-sheets tests were based on the following standards: thickness, TAPPI T411 om-97 (1997); bulk, TAPPI T500 cm-98 (1998); tensile resistance, TAPPI T494 om-96 (1996); burst resistance, TAPPI T403 om-97 (1997); tear resistance, TAPPI T496 sp-99 (1999); opacity TAPPI T1214 sp-98 (1998); and brightness TAPPI T1216 sp-98 (1998). The reported results represent the average values of 5 tested hand-sheets.

Papers surface morphology

The fiber morphological properties were observed under a scanning electron microscope (SEM) (OCTANE 9.88/1114658 AMETEK®, Mahwah, USA). Images were taken under several magnifications to observe the content, arrangement, and compactness of the fibers. Furthermore, C, O, N, Si, Ca, Mg, and Al were also identified by the same machine. A suspension of materials pulp was used for detailed anatomical features including fibre length, fibre width, curl index, and kink index, by using a Fiber Quality analyzer (LDA02128, OpTest Equipment Inc., Ontario, Canada).

RESULTS AND DISCUSSION

Chemical Composition

Table 1 shows the chemical properties of millet stalks and date palm leaves. The holocellulose content of millet stalks and date palm leaves was 61.89% and 61.63%, respectively. Their cellulose content was 40.99% and 39.00%, respectively, which is suitable for pulp and paper manufacturing (close to or above 40%) (Samariha and Khakifirooz 2011). However, millet stalks had a similar content of 39.4%, and date palm leaves have 39.37% as observed by Harinarayana *et al.* (2005) and Nasser *et al.* (2016), respectively. Compared to other non-woods, millet stalks and date palm leaves were higher than that of sorghum 35.87% (Cardoso *et al.* 2013), corncob residues 38.8% (Liu *et al.* 2010), rice husk 36%, and cotton stalk 38% (Singh and Chouhan 2014).

Hemicellulose content from millet stalks and date palm leaves ranged from 20% to 22%. However, higher hemicellulose values resulted in higher paper strength (especially tensile, burst, and fold). The pulp yield may have a negative effect during pulping because hemicellulose is the cell wall polymer component with the highest water sorption (Syed *et al.* 2016). The Klason lignin of millet stalks and date palm leaves was 18.20% and 15.34%, respectively, which was lower than that of bagasse at 20.35% (Samariha and Khakifirooz 2011), rice husk at 23%, cotton stalk at 20.88%, bagasse at 19%, and sesame at 23.3% (Singh and Chouhan 2014). Lignocellulose material with low lignin content needs a lower temperature and high pulping chemical charges, as well as bleaching to achieve a satisfactory kappa number. When more lignin can be removed from pulp, the paper that will be made is of higher quality compared to paper made from other lignocellulosic materials. The ash content of date palm leaves was low, 2.06%, while the ash content for millet stalks was high, 5.96%. The high ash contents, however, were unfavorable for pulp and papermaking, because they increase alkali consumption, incur recovery problems for cooking chemicals (evaporation, combustion, and lime mud reburning), and result in operational problems for further pulping (Agnihotri *et al.* 2010). Moreover, millet stalks showed relatively high hot and cold water extractives 13.00% and 5.20% respectively, compared to that 12.12% and 3.02% of date palm leaves. This is evidence of solubility of carbohydrates as well as lignin and dyes. The solubility in 1% NaOH was 15.30% and 12.20% of millet stalks and date palm leaves reflected the degradation of the cell wall material by weak alkali in pulping and bleaching process.

Table 1. Chemical Composition of Millet Stalks and Date Palm Leaves

Chemical Composition (%)	Millet Stalks	Date Palm Leaves
Holocellulose	61.89	61.63
Cellulose	40.99	39.00
Hemi Cellulose	20.90	22.63
Klason Lignin	18.20	15.34
Acid Insoluble Lignin	0.06	0.04
Ash	5.96	2.06
Soluble in hot water	13.00	12.12
Soluble in cold water	5.20	3.02
Soluble in 1% NaOH	15.30	12.20

Elemental Analysis

Low mineral content in the lignocellulose materials is desirable in pulp and paper production (Plazonić *et al.* 2016). In Table 2, the millet stalks had elemental proportions of carbon (C) 45.21%, oxygen (O) 20.90%, sodium (Na) 2.09%, calcium (Ca) 0.50%, silica (Si) 0.48%, magnesium (Mg) 0.38%, and aluminium (Al) 0.17%, respectively. The composition of date palm leaves was oxygen (O) 52.98%, carbon (C) 43.29%, sodium (Na) 1.86%, silicon (Si) 0.37%, aluminium (Al) 0.30%, and magnesium (Mg) 0.26. The millet stalks and date palm leaves contained relatively little silica, which should not therefore induce issues in the chemical recovery. Moreover, elemental analyses showed that the minerals components, which represent the inorganic constituents, were relatively low. However, this lower silica content is expected to reduce the amounts of alkali required during pulping and bleaching processing.

Table 2. Elemental Analysis

Material	C (%)	O (%)	Na (%)	Mg (%)	Al (%)	Si (%)	Ca (%)
Millet Stalks	45.21	20.90	2.09	0.38	0.17	0.48	0.50
Date Palm Leaves	43.29	52.98	1.86	0.26	0.30	0.37	0.68

Pulp Yield, Kappa Number, and CSF

In Table 3, the pulp yield of millet stalks, date palm leaves, and their 50% blend was 42.04%, 34.43%, and 38.50% with kappa numbers of 12.43%, 13.50%, and 11.96%, respectively. Considering the pulping yields and kappa numbers, it could be recommended that the simple soda process be chosen for the pulping of millet stalks/date palm leaves blend. Moreover, the blend of millet stalks and the date palm leaves produced better quality pulp as indicated by a lower kappa number. However, the combination of high yield with good papermaking properties suggested that date palm leaves could be used in blends with other cellulosic material, or pulped together with the rachis to give bleachable pulp grades (Khristova *et al.* 2005). Millet stalks, date palm leaves, and their blend showed viscosities of 665 mL/g, 551 mL/g, and 534 mL/g, respectively (Fig.1). Moreover, date palm leaves obtained a higher freeness value at CSF 720 mL compared to that of the millet stalks and the blend, 555 mL and 600 mL, respectively.

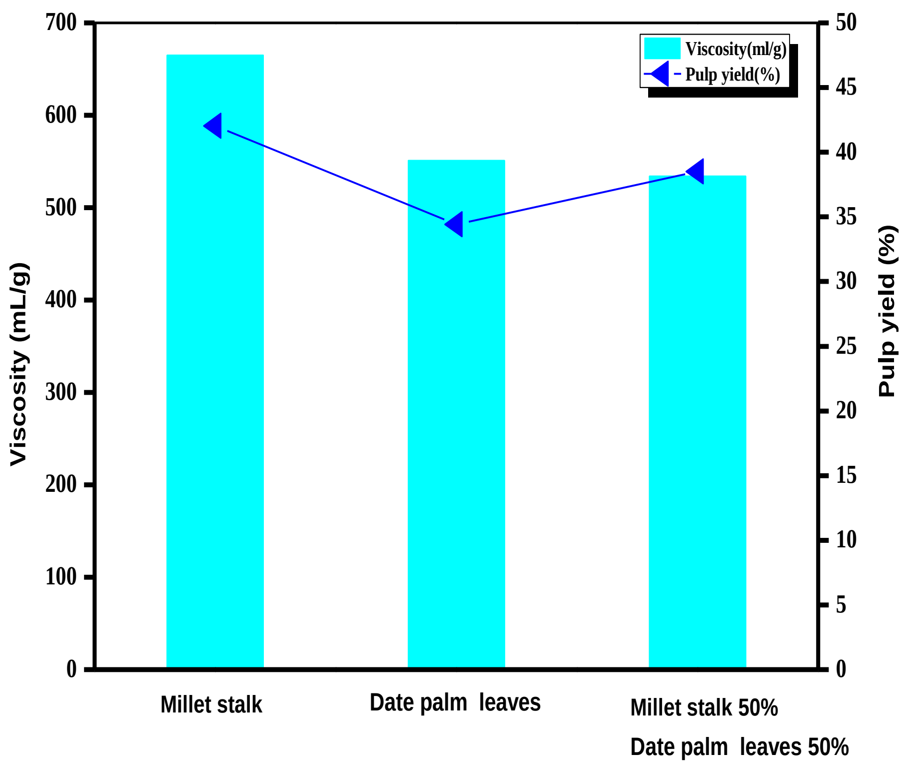

Fig. 1. Pulp yields and viscosities of millet stalks, date palm leaves, and their 50% blend

Table 3. Pulp Properties

Material	Pulp Yield (%)	Pulp Viscosity(mL/g)	Kappa Number	CSF (mL)
Millet Stalk	42.04	665	12.43	555
Date Palm Leaves	34.43	551	13.50	720
Millet Stalk (50%)+ Date Palm Leaves (50%)	38.50	534	11.96	600

Fibre Properties

The fibre length is considered an important parameter for pulp and paper properties because it has a significant impact on the paper mechanical properties (Jahan *et al.* 2010). As shown in Table 3, the average fibre length of millet stalks and date palm leaves was 0.41 mm and 0.51 mm, respectively. These results were in the range of those associated with common agriculture plants such as date palm rachis (Khiari *et al.* 2010). Moreover, these materials showed fibre thicknesses similar for millet stalks and date palm leaves, 27.10 mm and 28.80 mm, respectively. Fibre curl strongly affects the tensile strength and bonding ability of fibres within a network (Robertsén and Joutsimo 2005). However, millet stalks showed higher fibre curl and kink indices (0.12 mm and 1.67 mm) compared to that of date palm leaves (0.09 mm and 1.33 mm). Fibre curl affected the tensile index such that a sheet formed from such fibres would have a low tensile index, but may have a high tear strength. This has been explained by the uneven distribution of stress along the length of a curled fibre in a fracture zone (Robertsén and Joutsimo 2005).

Table 3. Fibre Properties

Material	Millet Stalk	Date Palm Leaves
Average Fibre Length (mm)	0.41	0.51
Average Fibre Diameter (µm)	27.10	28.80
Mean Kink Index (mm)	1.67	1.33
Mean Curl Index (mm)	0.12	0.09

Paper Properties

The physical strength properties of handsheets from millet stalks, date palm leaves, and their 50% blend are shown in Table 4 and Fig. 2. The paper made from the 50% blend of millet stalks and date palm leaves had better physical properties, such as tensile **s**trength 45.23 N m/g, tearing **i**ndex 11.04 mNm^2/g, bursting **i**ndex 2.34 KPa m^2/g, and a folding **e**ndurance of 16 ($log_{10}\ n$). For date palm leaves and millet stalks, the values were for tensile **s**trength (39.55 N m/g, 33.95 N m/g), tearing index (6.24 mNm^2/g, 2.31 mNm^2/g), bursting **i**ndex (1.64 KPa m^2/g, 1.96 KPa m^2/g), and folding **e**ndurance (11, 5 ($log_{10}\ n$)), respectively. However, this data showed that millet stalks and date palm leaves were promising for papermaking applications. Date palm leaves showed the best bleachability and reached the highest brightness of 70.50%, while it was 66.05% and 63.60% of millet stalks and 50% blend, respectively. Moreover, the highest opacity was 88.90% and was achieved by the 50% blend, while it was 88.50% and 86.00% of millet stalks and date palm leaves, respectively. However, opacity is very important for printing and writing papers, while for tracing paper, lampshades, and some packing papers, brightness is considered very important (Tajik *et al.* 2016).

Table 4. Paper Properties

Material	Millet Stalk	Date Palm Leaves	Millet Stalk (50%)+ Date Palm Leaves (50%)
Thickness (µm)	150.50	180.00	150.50
Tensile Strength (N m/g)	33.95	39.55	45.23
Tearing Index (mNm^2/g)	2.31	6.24	11.04
Brightness (%)	66.05	70.50	63.60
Bursting Index (KPa m^2/g)	1.96	1.64	2.34
Folding Endurance (log_{10}n)	5	11	16
Opacity (%)	88.50	86.00	88.90
Density (cm^3/g)	2.50	3.00	2.50

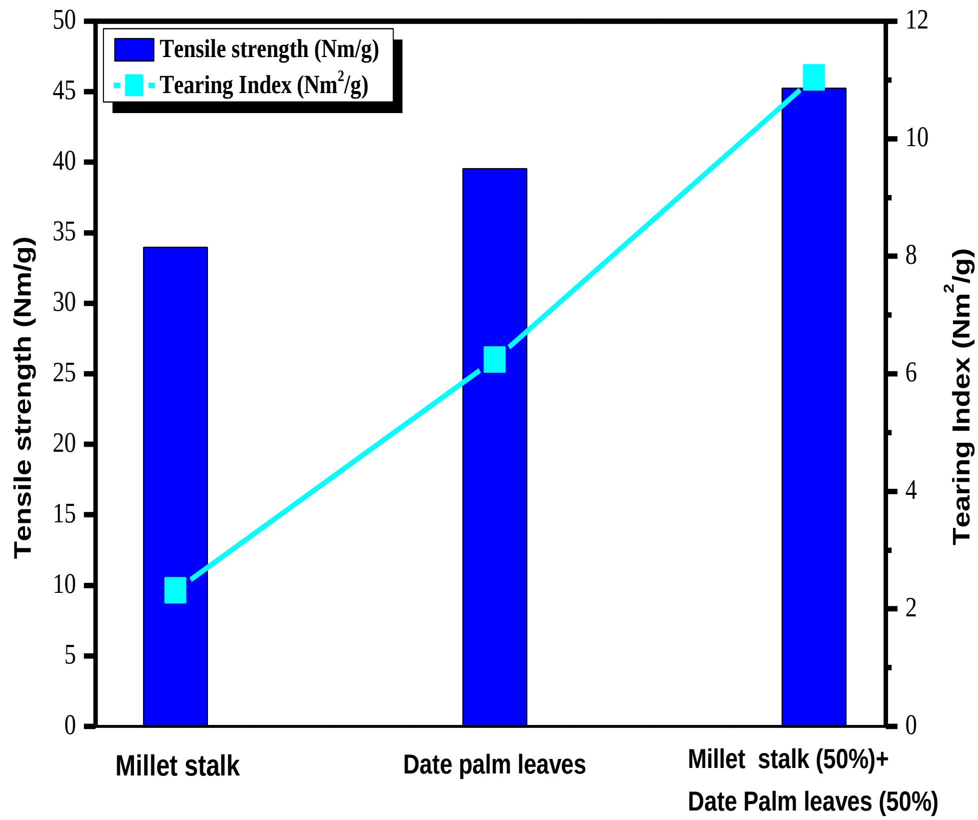

Fig. 2. Tensile strength and tearing index of the materials

Paper Morphology

The strength of the fiber matrix could be extrapolated based on its packing of the fibers. In Fig. 3, the millet stalks, date palm leaves, and 50% blend handsheets were magnified at 100 µm. (Fig. 3(A)) showed that the millet stalk fibers were more closely compacted compared to the date palm leaves (Fig. 3(B)). SEM images showed that the produced papers from the millet stalks were quite homogeneous and compact compared to that produced from date palm leaves; however, this will lead to smooth surface and good structure on produced paper. Alternatively, the blend handsheet in Fig. 3(C) showed a more closely packed arrangement than the millet stalks and date palm leaves fibers. Inevitably, the highly dense arrangement and compact packing imparted better mechanical properties and quality (Daud *et al.* 2013).

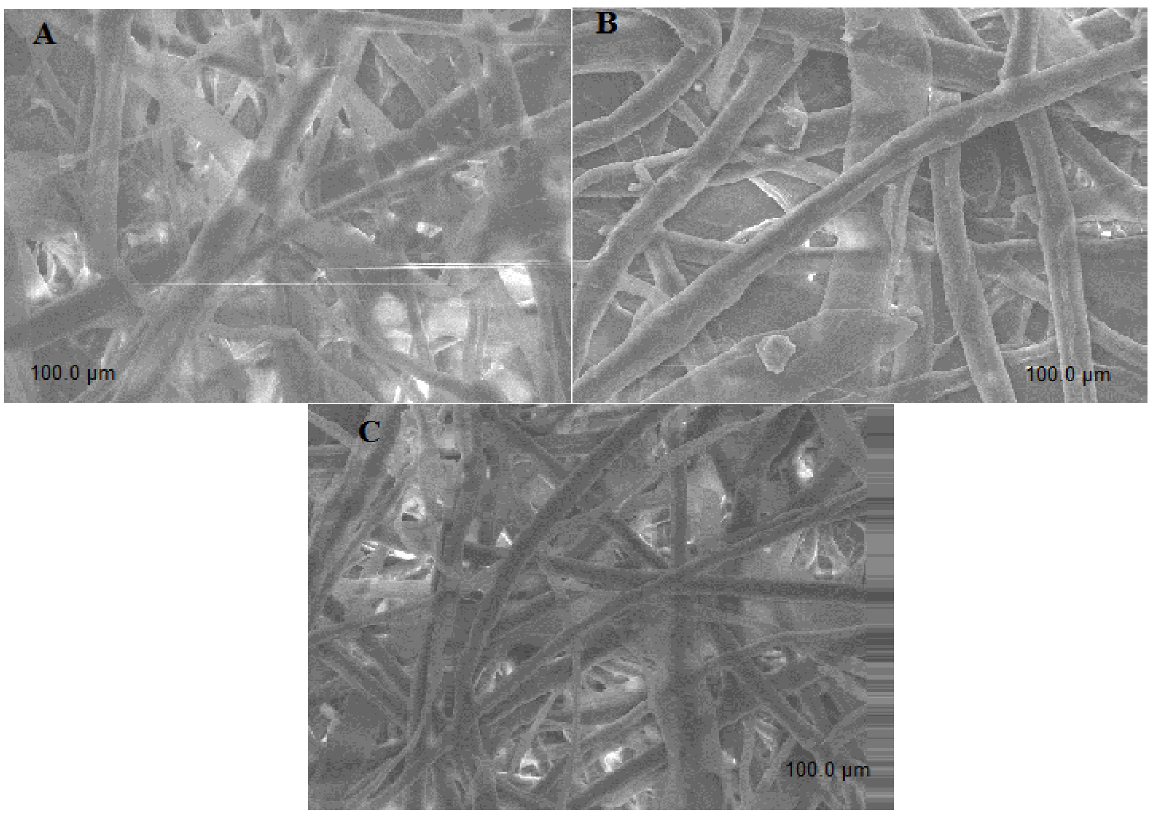

Fig. 3. Scanning electron microscope images of millet stalks (A), date palm leaves (B), and 50% blend (C)

CONCLUSIONS

1. The relatively high cellulose contents (40.99% and 39.00%) and low lignin contents (18.20% and 15.34%) of millet stalks and date palm leaves, respectively, led to high-quality pulp and paper.
2. The millet stalks and date palm leaves blend showed high mechanical properties (tensile strength, tear index, and fold test) similar to wood materials when compared to pure millet stalks and date palm leaves.

3. The SEM analysis showed a condensed arrangement of fibres that led to a stronger structure in the date palm leaves and the blend than in the millet stalks.
4. The papers produced from millet stalks, date palm leaves and 50% blend, could be used for writing and printing paper as well as in packaging applications.

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