Preparation and Characterization of Granular Activated Carbon from *Senna Occidentalis* Seeds

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Abstract

This paper prepared and characterized a low cost carbon from Senna occidentalis seeds (SOS) as an alternate material for environmental remediation. The Senna occidentalis seeds activated carbon (SOSAC) was prepared through chemical activation by impregnating a 50g of SOS into 25% Zinc chloride solution, at a ratio of activator to material (R = 2:1 v/v) for a period of 24 hours, carbonized at temperature (C_T) of 500 ℃ for a duration (Ct) of 2 hours which finally yield a 51.91% product.

The carbon was characterized by employing proximate analyses such as; surface area, total pore sizes, density and spectroscopic analyses such as; Scanning Electron Microscopy (SEM) and Fourier Transformed Infra-red (FT-IR) to understand its chemistry for potential applications to waste management.

The results obtained from proximate analyses include; surface area was $919m^2g^{-1}$, total pore volume was $2.81cm^3g^{-1}$, bulk density was $0.66 g/cm^3$, and moisture content was found to be 3.15%. However, the results of SEM and FT-IR have further revealed that SOSAC was not only good and inexpensive but also promising material (as it has a porous surface covered with adsorption sites), and that can serve as an alternative material to very expensive commercial activated carbons that are in existence.

Keywords: Activated Carbon; Senna occidentalis Seeds; Proximate Analyses; SEM; FT-IR Spectroscopy

Introduction

Several Studies were conducted and concluded on the feasibility and reliability for the production of activated carbon from agricultural wastes ranging from; coconuts shells (Qiao, *et. al*, 2002), oil palm shells (Rengaraj *et. al*, 2002), dates stones (Girgis and El-Hendawy, 2002; Banat *et. al*, 2003; Alhamed, 2002; Alhamed, 2006; Yakubu *et al*., 2008), corncob and maize cob (Tsai *et. al*, 2001; Agunwamba *et al*., 2002a, 2002b), terminalia catappa (Ochonogor *et al*., 2000), apple pulp (Suarez-Garcia *et. al*, 2002), chickpea husks (Hayashi *et. al*, 2002), rice husk (Hayashi *et. al*, 2000; Aloko and Adebayo, 2007), jatropha husk (Namasivayam *et al*., 2007), almond shells and peacan (Bansode, 2002), palm kernel shells and palm tree branches respectively (Ogedengbe *et al*., 1985 and Alhamed, 1995), groundnut shells (Muhammad *et al*., 2015), mango seed shell (Akpen *et al*., 2011) etc.

On the contrary, very little attention is given to Senna occidentalis seeds (SOS) as in exploring its potentials for activated carbon (AC). Hence, this research's main aim is to explore the

possibilities for Senna occidentalis seeds activated carbon (SOSAC), through the objectives which are to; (1) prepare low cost granular activated carbon (GAC) using SOS and, (2) characterize the prepared low cost carbon using proximate analyses and spectroscopic analyses such as SEM and FT-IR.

Activated Carbon

According to Alhamed (2006), "Activated carbon (AC) is an amorphous form of carbon that is specially treated to produce a highly developed internal pore structure and a large surface area, thus, producing reasonably cheap and excellent adsorbent.

AC, also called activated charcoal is a form of carbon that is processed to make it extremely porous and accompanied by a very large surface area for adsorption or chemical reactions.

Methods of Activated Carbon Formation

Physical and chemical activation are the two popular methods for preparation of AC. Physical activation involved carbonization of a raw material at a high temperature then activated by an oxidizing agent such as CO_2 or steam under pressure to increase the porosity and surface area of the carbon. On the other hand, chemical activation is achieved by impregnating the raw material with either mineral acid such as; H_2SO_4, HNO_3, H_3PO_4 or mineral salt such as; $ZnCl_2$, NaOH, followed by carbonization.

Similarly, Zinc Chloride is reported to have widely been used as activating reagent, and found to be the best among other activators studied so far. This is because of its results in high surface areas and high yield (Alhamed, 2006; Santhi and Smitha, 2015).

Description of Senna Plant

Senna occidentalis, also called Coffee Senna, or Coffee weed is a pantropical plant species that is considered as an "edible weed of agriculture" or "famine food" (Mebrahtom *et al.*, 2014). Sena is a smooth annual plant that can grow up to 2 m tall. The seed pods are dark brown, 8–12 cm long, 7–10 mm wide and curve slightly upward. The seeds are dull brown, 4–5 mm long and flattened on both ends. It is found throughout Nigeria (especially in the northern part) along major highways, growing abundantly on waste lands, as shrubs immediately after the rains (Mebrahtom *et al.*, 2014).

Fig. 1: Senna occidentalis Green Plant and Senna occidentalis Seeds

Recently, Mekala et al., (2015) have used batch adsorption technique for the removal of fluoride ion using low cost activated carbon made from stems of Senna occidentalis plant, activated chemically using nitric acid (HNO_3).

Methods
Materials
The Senna occidentalis seeds were obtained from Tudun Wada Area of Birnin Kebbi Local Government, Kebbi State, Nigeria. The analytical equipment used in carrying out this work such as grinding machine, mechanical sieve, hot oven, electric furnace, weighting balance etc. were obtained from Department of Polymer & Textile Engineering, Chemical Engineering, and Geology of Ahmadu Bello University, Zaria, Nigeria. Other chemicals such as $ZnCl_2$ used as activating agent, NaOH, HCl etc. were ensured to be of reagent grade.

Preparation of SOSAC
The seeds of Sena occidentalis plant were obtained, air dried and broken to separate the seeds from the shells, thoroughly cleaned to remove extraneous materials and grounded to smaller particle sizes for chemical activation. The grounded sample material was then sieved using mechanical sieves to get the desired particle sizes of 1.0mm – 0.5mm sieve mesh size.
About 50g portion of the sieved SOS was impregnated with 25% $ZnCl_2$ solution at a ratio of 2:1 v/v for 24 hours, after which it was drained, then put into crucibles and carbonized in a muffle furnace at 500 ^{0}C for 2 hours to produce activated carbon. The resulting carbon was treated with 0.5 HCl solution overnight, followed by successive washings with distilled water decanting the last part into the filter paper until the pH range of 6.5 – 7.0 was reached, dried in an oven at 120 ^{0}C for a period of 4 hours and then, kept in tightly closed plastic bottles and carefully labelled, as similarly reported by (Alhamed, 2006).

Characterization Techniques
Proximate Analyses
Determination of Yield (%) and Weight Loss (%)
The yield of activated carbon is defined as the ratio of the dry weight of the activated carbon product to that of the original raw material as given in the equation below (Okoye, 2010);

$$\%Yield = (W_1/W_0) \times 100 \tag{1}$$

where; W_0 (g) is the initial dry weight of the precursor and W_1 (g) is the dry weight of the carbon after carbonization, washing and drying. The weight loss is calculated according to eq. 2:

$$\%Weight\ loss = [(W_0 - W_1)/W_0] \times 100 \tag{2}$$

Surface Area Measurement
Surface area of the SOSAC was determined according to the method used by Okoye, (2010). In the process, 1.5g of activated carbon was agitated in 100 ml of HCl that had been diluted to pH of 3. Then 30 g of NaCl was added while stirring the suspension. The volume is then made up to 150 ml with de-ionized water which has altered the pH. Then, 0.10N NaOH was used to adjust the pH from 4 to 9 and the volume of NaOH used (V) was finally recorded.
The surface area was calculated using the eq. 3 (Mohammed et al., 2015):

$$S = 32V - 25 \tag{3}$$

where; S is the surface area (m^2/g) and V is the volume of NaOH used (ml).

Determination of Ash Content (%)
The ash content of SOSAC was determined in accordance with the standard method ASTM E1755-01. In the process, a sample of the carbon was weighed, pre-dried at 150 °C, and then burnt in a muffle furnace at 650 °C for 4 hours in the presence of air. The ash content (%) was then calculated from the eq. 4 (ASTM, 2003):

$$Ash\ Content\ (\%) = W_{ash}/W_{ads} \times 100 \qquad (4)$$

where; W_{ash} is the weight of dry ash (g) and W_{ads} is the weight of dry adsorbent (g).
The above method was repeated severally until constant ash content was obtained.

Determination of Bulk Density
The bulk density of each adsorbent will be determined by using the standard procedure already employed by other researchers. A known weight of each sample, after being dried at 105 °C, was parked into a 10 ml capacity graduated cylinder. The bottom of the cylinder was tapped gently on the laboratory bench top several times until there was no further diminution of the sample level. The bulk density was calculated using the eq. 5 (Okoye, 2010):

$$Bulk\ Density\ (g/ml) = W_{mat}/V_{mat} \qquad (5)$$

where; W_{mat} is the weight of dry material (g) and V_{mat} is the volume of dry material (ml).

Determination of Total Pore Volume
The total pore volume of SOSAC is determined by boiling its weighed 2g sample and immersed in water. After the air in the samples had been displaced, the samples were superficially dried and weighed. The increase in weight divided by the density of water gave the pore volume (Mohammed *et al*., 2015).

Determination of Moisture Content (%)
Moisture content was determined using the procedure used by Roshan, (2015), where a crucible with its lid was taken and weighed. 1gram of the SOSAC was taken in the crucible with lid and weighed. It was kept in hot air oven at 110 °C for one and half hours. It was taken out and kept in the desiccator. Then the weight was measured using eq. 6:

$$M = 100 \times \frac{(B-F)}{(B-G)} \qquad (6)$$

where; M = moisture content, G = mass of crucible with lid, B = mass of crucible with lid plus sample, F = mass of crucible with lid plus dried sample.

Determination of Volatile Matter (%)
A crucible with lid was taken and weighed, then add 1 gram of sample in the crucible with lid and weighed. It was kept in the muffle furnace at a temperature of 910 °C for 7 minutes. Then it was taken out and kept in the desiccator for half an hour to cool down. The weight of the sample in crucible with lid was taken. Volatile matter (%) was then calculated using eq. 7 (Roshan, 2015):

$$V = 100 \times 100\ (B\text{-}F) - M \times (B\text{-}G)/(B\text{-}G) \times (100 - M) \qquad (7)$$

Determination of pH
The pH of SOSAC was determined using the standard test ASTM D 3838-80 (ASTM, 1999).

Determination of Fixed Carbon (Carbon Content, %)
Fixed Carbon of SOSAC was determined using the procedure employed by Roshan, (2015). The relation for obtaining the carbon content is given in the eq. 8:

Fixed Carbon = 100 – [%Moisture Content + %Volatile Matter + %Ash Content]   (8)

Spectroscopic Analyses
Fourier Transform Infrared (FT-IR) Spectroscopy
FT-IR Spectroscopy is most useful for identifying chemicals that are either organic or inorganic. It can be applied to the analysis of solids, liquids, and gasses. FTIR is perhaps the most powerful tool for identifying types of chemical bonds (functional groups). The wavelength of light absorbed is characteristic of the chemical bond as can be seen in the annotated spectrum. By interpreting the infrared absorption spectrum, the chemical bonds in a molecule can be determined. FTIR spectra of pure compounds are generally so unique that they are like a molecular "fingerprint". While organic compounds have very rich, detailed spectra, inorganic compounds are usually much simpler. For most common materials, the spectrum of an unknown can be identified by comparison to a library of known compounds. We have several infrared spectral libraries including online computer libraries (Mohammed *et al.*, 2015).

Scanning Electron Microscope (SEM)
SEM is a type of electron microscope that images a sample by scanning it with a high-energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition, and other properties such as electrical conductivity. The types of signals produced by a SEM include secondary electrons, back-scattered electrons (BSE), characteristic X-rays, light (cathode luminescence), specimen current and transmitted electrons. Secondary electron detectors are common in all SEMs, but it is rare that a single machine would have detectors for all possible signals. The signals result from interactions of the electron beam with atoms at or near the surface of the sample. SEM can produce very high-resolution images of a sample surface, revealing details less than 1 nm in size. Due to the very narrow electron beam, SEM micrographs have a large depth of field yielding a characteristic three dimensional appearance useful for understanding the surface structure of a sample (Subhashree, 2011).

Results
Proximate Analyses Results
Table 1: Proximate Analyses Result for SOSAC Produced at 500 ℃, 120 min. using ZnCl₂ Activating Agent.

Parameters	Results
% Yield	51. 91
Weight Loss (%)	48.09
Surface Area (m^2/g)	919
Total Pore Volume (cm^3/g)	2.81
Bulk Density (g/cm^3)	0.66
pH	6.4
Moisture Content (%)	3.15

Ash Content (%)	7.83
Volatile Matter (%)	13.20
Fixed Carbon (%)	75.82

The physico-chemical characteristics of SOSAC were examined and presented in the Table 1. The surface area was found to be considerably higher (919 m^2/g) hence, indicating high porosity and it may be obtained as result of the influence of the activating agent ($ZnCl_2$) activity (Alhamed, 2002; Alhamed, 2006). The typical activated carbon has value ranges from 600 – 1500 m^2/g.

However, the low ash content value (7.83%) and volatile matter (13.20%) indicated that the SOSAC has low inorganic content and high fixed carbon (75.82%). Similarly, an activated carbon is said to be good, if it has low ash content (which directly relates to minerals composition) of carbon. This is because the ash content can reduce the efficiency of reactivation.

Furthermore, the moisture content (3.15 %) found in this study is said to be good. This is because high moisture content tends to dilute adsorbent and increases its weight during adsorption process – which reduces the efficiency of activation carbon in terms of contact with the adsorbate. Hence, the low moisture content of SOSAC signifies that the carbon structure has developed adequate porosity through the activation process.

Other parameters like; pH 6.4 of SOSAC is in line with the acceptable values of 6–8 as reported by Okiemmen *et al*., (2008); Bulk density is better when compared with values found by other researchers (Okoli *et al*., 2015). It is important to mention here, that the importance of bulk density of activated carbon to the wastewater treatment study is in design of filtration columns. It determines the minimum amount of adsorbent that can be used to achieve maximum amount of adsorbate per small filtration column (i.e. it determines the retention capacity of adsorbent).
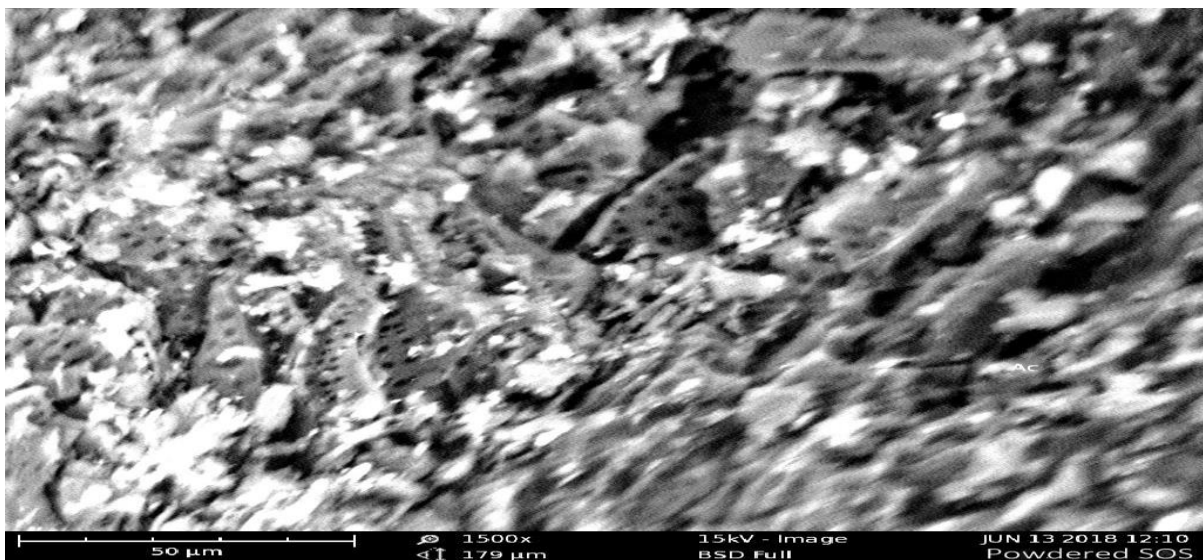
Scanning Electron Microscopy

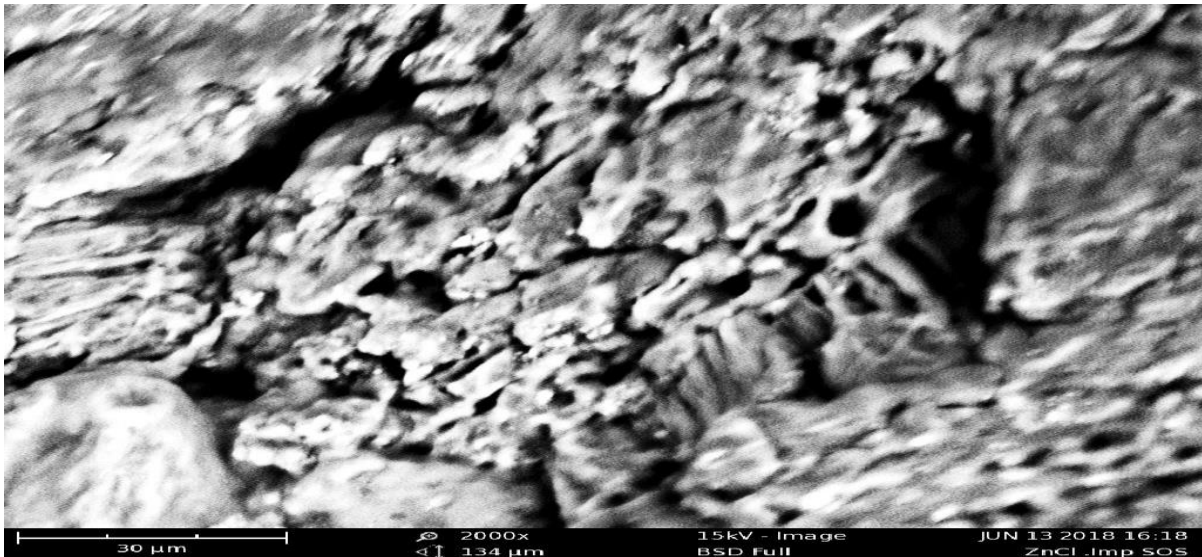**Fig. 2:** Scanning Electron Micrograph for Raw Grinded SOS at 1500X Magnification

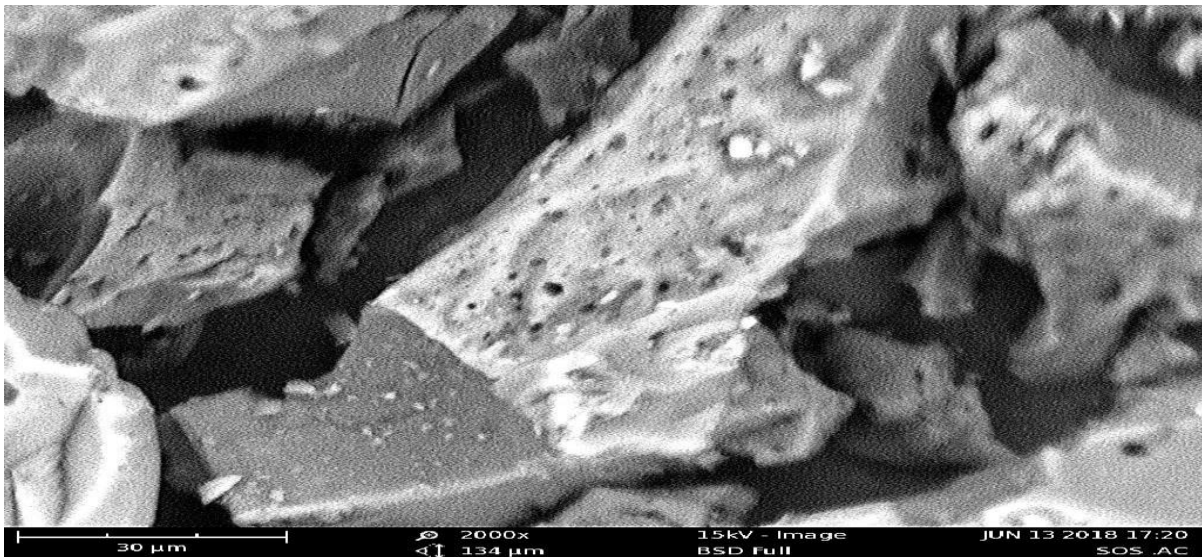**Fig. 3:** Scanning Electron Micrograph for $ZnCl_2$ Impregnated SOS at 2000X Magnification

Fig. 4: Scanning Electron Micrograph for SOSAC at 2000X Magnification

Scanning electron microscopy for raw grinded SOS, $ZnCL_2$ impregnated SOS and SOSAC was carried out in order to better understand the morphology of the starting material and end product, as well as bringing out the picture of the surface chemistry (or transformation) involved in the activation and production processes of SOSAC. The SEM result of SOS at 1500X and 2000X magnifications are shown in figures 3 and 4.

Fig. 2 shows SEM image of raw grinded SOS which appeared to be somewhat as a crystalline solid material with numerous dark spots that are highly suspected to be clogged but potential pores for adsorption. Fig. 3 shows a sign of material modification and improvement due to chemical activity of $ZnCl_2$ activating agent on the raw material. Fig. 4 finally shows an image of a successfully modified or/and new material called SOSAC formed after going through several production processes. The activated carbon clearly appeared to have developed several holes (pores) distributed over its surface – which are believe to be adequate and ready for adsorption.

Fourier Transformed Infrared Spectroscopy

Shift in bands and changes in wavelength numbers and absorbance between the untreated and treated samples which were discovered by the help of Fourier Transformed Infrared (FT-IR) Spectroscopy has indicated that chemical transformation would have taken place during chemical treatment.
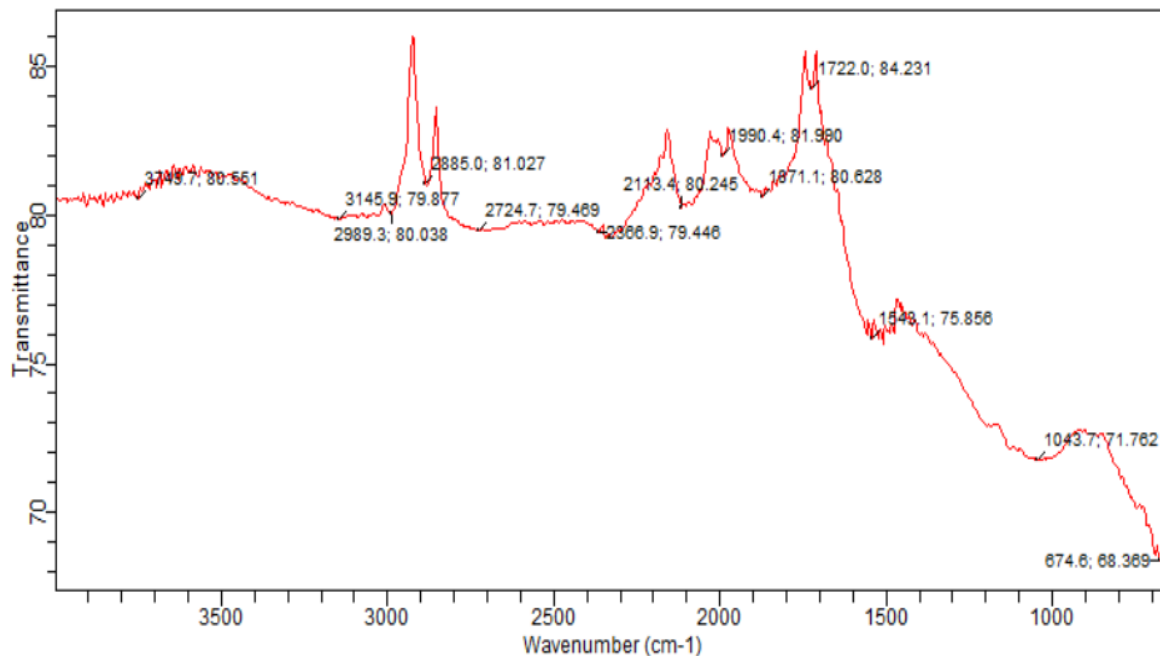

Fig. 5: FT-IR Spectrum for low cost SOSAC

The surface functional groups of SOSAC was analysed before it put to test (later) using FT-IR spectroscopy, it's spectra were recorded between absorption ranges of 650 to 4000 cm^{-1} and presented in the figure 5. The carbon showed characteristic bands at 3749.7 cm^{-1} due to O–H stretching, 3145.9 cm^{-1} represents O–H stretch of vibrations of carboxylic acids, peaks at 2989.3 cm^{-1} and 2885.0 cm^{-1} represent H–C–H stretch of alkanes, peaks at 2366.9 cm^{-1} and 2724.7 cm^{-1} represents hydrogen-bonded O–H stretch of carboxylic acid, peak at 2113.4 cm^{-1} represents –C≡C– stretch of monosubstituted alkynes. Other peaks observed includes; 1990.4 cm^{-1} and 1871.1 cm^{-1} represents C=O stretching of transition metal carbonyls, peak at 1772.0 cm^{-1} represents C=O stretching of ester, carboxylic acid, aldehyde or ketone, peak at 1543.1 represents cm^{-1} –C=C– aromatic ring stretching while, 1043.7 cm^{-1} and 674.6 cm^{-1} represents C–O stretching of alcohol, carboxylic acid, ester, ether and C–H aromatic C–H out-of-plane bending respectively (Figure 5).

Meanwhile, the adsorption capacity of adsorbent depends upon the porosity as well as the chemical reactivity of the functional groups at the adsorbent surface (Okoli et al., 2015). Hence, looking at the kind of functional groups SOSAC has, it might said to be a good adsorbent.

Conclusions

The techniques employed to characterize SOSAC including proximate analyses; FT-IR Spectroscopy and Scanning Electron Microscopy were all adequately appreciated in explaining its physico-chemical properties, functional groups and structural morphology respectively. Proximate analyses revealed that SOSAC has good surface area, high fixed carbon which means low ash content and low volatile matter; low moisture content etc.; FT-IR revealed that it has considerable number of functional groups capable of providing bonding sites while; SEM

revealed that several cavities were successfully developed on the external surface of the chemically activated carbon of SOS. Hence, SOSAC might serve as an alternative to the commercial activated carbons that are currently available but not affordable.

Moreover, there is the need for further investigations to be carried out on the production optimization of SOSAC. It is also recommended that SOSAC should be used to treat different wastewaters especially for the removal of organic molecules and heavy metals ions.

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