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Characterization of Sugarcane Bagasse and Moringa Oleifera as Potential Adsorbent for the Treatment of Wastewater containing Heavy Metals

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ABSTRACT: Activated carbon from sugarcane bagasse and moringa oleifera were prepared and their textural properties were evaluated using Fourier transform infra-red (FT-IR) and scanning electron microscope (SEM). The chemical composition of sugarcane bagasse and moringa oleifera was determine with the aid of X-ray fluorescence (XRF) while the surface area, micropore volume and total pore volume were estimated with the aid of structural characterization (SCAC) software using the iodine value and methylene blue number as the input parameters. The calculated iodine value and methylene blue value of sugarcane bagasse was 24.56mg/g and 2.27g/100g while for moringa oleifera, it was 29.48mg/g and 3.02g/100g. Based on these values, the surface area and total pore volume of sugarcane bagasse was estimated as 234m^2/g and 0.14cm^3/g, for moringa oleifera it was 277.4m^2/g and 0.21cm^3/g. Results of the textural analysis revealed the presence of O-H stretching (which is the site for adsorption) in both the sugarcane bagasse and moringa oleifera. In addition, significant morphological differences were observed between the sugarcane bagasse and moringa oleifera with moringa oleifera exhibiting better irregular and porous surface characteristics of different lamellae sizes.

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Wastewater treatment is an essential process of any region, without which waterborne pathogens can spread resulting in outbreak of diseases. The discharge of untreated wastewater is responsible for the degradation of receiving water bodies and the impacts of such degradation can result in the spread of various waterborne diseases, decreased levels of dissolved oxygen, and decreased water quality (Ilaboya, 2017). Heavy metals, also known as trace metals, are one of the most persistent pollutants in wastewater. The discharge of high amounts of heavy metals into water bodies leads to several environmental and health impacts (DWI, 2014). The assimilation of relatively small amounts of these heavy metals over a long period of time in the human body can lead to chronic toxicity coupled with numerous health challenges such as skin irritation, lung tumor including severe damage to the nervous systems and circulatory system (DWI, 2014). The presence of these heavy metals in the environment is of great concern to scientists and engineers because of their toxic nature and other adverse effects posed by the discharge of untreated effluents containing heavy metals on receiving water bodies (Sekar *et al*, 2004). Physico-chemical processes that have been employed in the treatment of wastewater containing heavy metals prior to being discharged into water bodies include chemical and electrolytic precipitation, reverse osmosis, electrodialysis and ion exchange method (Gupta *et al*, 2003). Adsorption offers good prospects over other treatment techniques owing to its flexibility and ease of operation (Ahmad *et al*, 2006). Adsorption is a process which involves the movement of molecules of the fluid phase (liquid or gas) to the surface of the solid. In adsorption, liquid or gas molecules diffuse from the bulk solution to the surface of the solid forming an adsorbed phase of distinct characteristics (Abdullah *et al*, 2001; Babu *et al*., 2012). With the selection of a proper adsorbent, the adsorption process has been identified as a promising technique for the removal of certain types of contaminants including heavy metals (Weng *et al.,* 2007). Until recently, commercial activated carbon is the most used adsorbent for wastewater treatment owing to its high surface area. The difficulties of preparing commercial activated carbon and the associated problem of reuse call for research into local materials. To act as good adsorbent for wastewater treatment, the material must possess good textural properties and contain polar functional groups such as N-H bending of amides and O-H stretching of the hydroxyl group. High surface area and good cation exchange capacity are also very important (Weng *et al.,* 2007, Ilaboya, 2017, Ilaboya and Izinyon, 2019). The focus of this study is to

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evaluate the characteristics of sugarcane bagasse and moringa oleifera as potential adsorbent for water and wastewater treatment.

MATERIALS AND METHODS

Preparation of Adsorbent from Sugarcane Bagasse and Moringa Oleifera: Moringa oleifera leaves was collected and dried in hot air oven at 50-70°C for 2 hours, pulverized and screen sieved to obtain geometric sizes of 150mm (Mariadas *et al.,* 2012). The sugarcane bagasse was obtained from fresh sugarcane after extracting the juice. The bagasse was then chopped manually and washed with distilled water to remove any impurities and extra sugar contents. Thereafter, it was oven-dried at 50-70°C for 2 to 3 hours, pulverized and screen sieved to obtain geometric sizes of 150mm. Carbonization was done using the method recommended by Ekpete and Horsfall (2011) with slight modification as follows. 500g weight of the pulverized samples was placed in a muffle furnace which allows limited supply of air at a temperature of 250^0C for 1 to 2hours. The carbonized samples were activated using the method recommended by Mansfield (1998) with slight modification as follows: 25g of the charred samples was soaked in 250 ml of 5.5M $ZnCl_2$ solution and mixed thoroughly to form a paste. The paste was transferred into an evaporating dish placed in an oven and heated at 150ºC for 30 minutes. The samples were then allowed to cool and washed with distilled water to remove any residual salt, oven dried at 105^0C for one hour, grind using mortar and pestle and sifted with 106µm Standard Tyler Sieve before use.

Functional group determination: Fourier Transform Infra-Red (FT-IR) spectra of the adsorbents (sugarcane bagasse and moringa oleifera) was obtained by using an FTIR spectrophotometer (Model: FTIR 2000, Shimadzu Kyoto, Japan) to determine the presence of functional groups which could influence the adsorption capacity of the adsorbent. About 150mg KBr disks containing approximately 2% of sugarcane bagasse and moringa oleifera samples were prepared prior to recording the FTIR spectra in the range of 400-4000cm^{-1} with a resolution of 4.0cm^{-1} according to (Dawodu *et al.,* 2012).

Analysis of Microstructures: The microstructural arrangements of sugarcane bagasse and moringa oleifera was analyzed using scanning electron microscopy (APEX 3020 PSEM 2) to give adequate information about their morphology and topological presentations. Such presentations provide possible explanations for the solid behaviour according to (Omisanya *et al.,* 2012).

Chemical Composition: The chemical composition of the adsorbents; (sugarcane bagasse and moringa oleifera) was studied using X-Ray Fluorescence (APEX 3022). Chemical digestion of the solid adsorbent was done in accordance with the procedure given by (Omokaye, 1999) as follows: (1:1 v/v) mixture of 0.25M solution of hydrochloric acid and Nitric acid was prepared. A mixture of (1:10 w/w) of the solid adsorbents (sugarcane bagasse and moringa oleifera) to acid solution was obtained and stirred for 30 minutes. The solution was filtered and the filtrate was used for the analysis.

Iodine Number Determination: Iodine number was determined in accordance with the procedure given by (ASTM, D4607-94) as follows: 10 ml of 5% HCl was added to 1g of adsorbent. The mixture was allowed to boil for 30 seconds before being cooled to room temperature. 100ml of 0.1N iodine solution was added to the mixture; the content was shaken vigorously and filtered. 25 ml of the filtrate was thereafter titrated against 0.1N sodium thiosulphate using starch as indicator. The iodine number was defined as the quantity of iodine adsorbed in (ml).

Methylene blue absorption value: Methylene blue absorption number was determined as follows: 2g dried sample of the adsorbent was transferred into a conical flask containing 100 mL distilled water and stirred properly. The mixture was titrated against a standard methylene blue solution. The methylene blue solution was prepared by dissolving 1g of dry methylene blue (with molecular weight of 319.9g/mol) in 250 mL of distilled water. The end point of titration of the methylene blue adsorption was determined by using the halo method given by (Kahr and Madsen, 1995; Yukselen and Kaya, 2008). Following this method, Methylene blue solution was added drop by drop from a burette to the stirred solid adsorbent suspension in the conical flask and drops of the suspension was placed with a glass rod on a filter paper to examine the spread. At the end point, a light blue halo colour was seen at the center point of the circle which remained visible for over a minute. The methylene blue adsorption value (MBA) (Expressed in g/100g of sample) was calculated from the equation given by (Salwa *et al.,* 2011).

$$MBA = \frac{(\frac{A}{B} x\ C)}{(\frac{D}{100})} \qquad (1)$$

Where; A is the weight of dry methylene blue, B is the volume of methylene blue solution (1 litre), C is the volume of MB added to the sample until the end point was reached, D is the weight of the dry powdered adsorbent sample (2g)

Surface area, micropore volume and total pore volume estimation: Surface area, micropore volume and total pore volume of sugarcane bagasse and moringa oleifera were estimated from the iodine and Methylene blue adsorption number. Iodine and Methylene blue adsorption number were determined using methods as described in section 2.2.4 and 2.2.5 respectively. Data obtained were analyzed using particulate characterization software (SCAC) to compute the surface area, micropore and total pore volume respectively (Cleiton *et al,* 2011). The surface area of adsorbents is usually measured using the Brunauer-Emmett-Teller (BET) method, which employs the nitrogen adsorption at different pressures at the temperature of liquid nitrogen (77K). Additional information about the structure of microporous materials can also be obtained by the adsorption characteristics of different adsorbate such as methylene blue and iodine. These characteristics confer such molecules the potential for using them as probes in the study of the physical structure of microporous materials (Cleiton *et al.,* 2011).

RESULTS AND DISCUSSION

Result of the chemical analysis of moringa oleifera and sugarcane bagasse conducted using X-Ray Fluorescence is presented in Tables 1

Table 1: XRF result of Moringa Oleifera and Sugarcane Bagasse

Moringa Oleifera			Sugarcane Bagasse		
Element Symbol	Element Name	Weight Conc.	Element Symbol	Element Name	Weight Conc.
C	Carbon	59.34	K	Potassium	36.96
K	Potassium	8.19	Si	Silicon	28.82
O	Oxygen	7.86	Ca	Calcium	7.69
Ca	Calcium	6.17	Fe	Iron	7.30
Si	Silicon	3.34	Al	Aluminium	6.86
S	Sulphur	2.72	P	Phosphorus	6.04
Al	Aluminium	1.67	S	Sulfur	2.23
P	Phosphorus	1.61	Mg	Magnesium	2.22
Mg	Magnesium	1.16	Ti	Titanium	0.89
N	Nitrogen	0.96	Na	Sodium	0.53
Fe	Iron	0.57	Mn	Manganese	0.45
Na	Sodium	0.33			

It was observed from the result of Table 1 that carbon is the dominant element present in Moringa followed by potassium, oxygen, calcium and silicon with weight concentration of 59.34mg/l, 8.19mg/l, 7.86mg/l, 6.17mg/l and 3.34mg/l respectively. For sugarcane bagasse, the dominant element was observed to be potassium, silicon, calcium, iron, aluminium and phosphorous with weight concentration of 36.96mg/l, 28.82mg/l, 7.69mg/l, 7.30mg/l, 6.86mg/l and 6.04mg/l. The adsorption properties of activated carbon and other clay minerals depend mainly on the presence of microporous structure and the reactivity of the various functional groups present on the surface. To examine the microstructural arrangement of sugarcane bagasse and moringa oleifera scanning electron micrograph (SEM) was employed. Result of the scanning electron micrograph of sugarcane bagasse and moringa oleifera is presented in Figures 1 and 2 respectively. Significant morphological differences were observed between the sugarcane bagasse and moringa oleifera samples with moringa oleifera exhibiting better irregular and porous surface characteristics of different lamellae sizes as shown in the result of Figures 2.

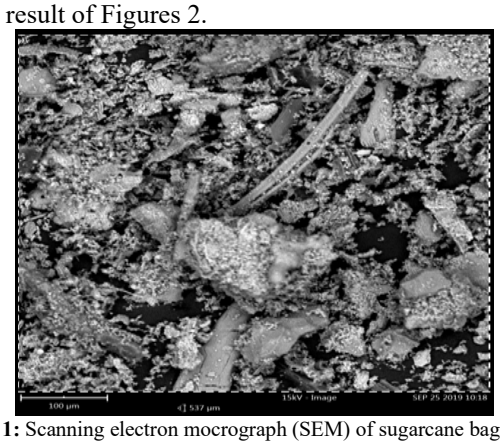

Fig 1: Scanning electron mocrograph (SEM) of sugarcane bagasse

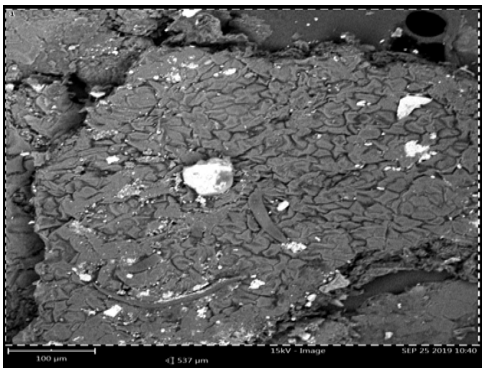

Fig 2: Scanning electron mocrograph (SEM) of moringa oleifera

Insight into the nature of functional groups that make up the surface of adsorbent would create a better picture on the adsorption potentials of the material. To identify the functional groups, present on the surface of moringa oleifera and sugarcane bagasse, Fourier Transform Infrared spectra were obtained and presented in Figures 3 and 4. To identify the functional group based on the FTIR spectra, a summary of absorption assigned bands from the works of previous researchers were adopted to analyze and evaluate the spectrum of sugarcane bagasse and moringa oleifera. Result of the analysis is presented in Tables 2 and 3. It was observed from the result of Table 2 that; O-H vibration and O-H stretching mode of hydroxyl groups (N-H stretch) where the dominant functional groups present in sugarcane bagasse and are responsible for the sorption potential of sugarcane bagasse. From the

result of Table 3, it was observed that O-H stretching mode of hydroxyl groups N-H stretch is the dominant functional group present in moringa oleifera and it is the functional group responsible for metal ion adsorption onto moringa oleifera.

Table 2: Functional groups present in Sugarcane Bagasse

S/ No	Wave Number (cm^{-1})	Bond Source	Sugarcane Bagasse
1	3695 – 3655	O-H vibration	3763.00
2	3450.63	O-H stretching mode of hydroxyl groups N-H stretch	3422.00
3	2367.18	stretch of alkyne	2360.45
4	1653.92	N-H bending of amides, C = O stretch, carbonyl	1644.08
5	1566.65	Quinonic and carboxylate groups, N-H bending, C = O stretch	1522.13
6	1427.31	CH_2 and CH_2 bend, pyrones and aromatic group	1431.00
7	1350 – 1250	Organic phosphate, (P = O stretch)	1379.20
8	1110 – 1080	Organic siloxane or silicone, Si-O-C stretch	1158.29
9	1095 – 1075	Organic siloxane or silicone, Si-O-Si stretching	1039.00

Table 3: Functional groups present in Moringa Oleifera

S/ No	Wave Number (cm^{-1})	Bond Source	Moringa Oleifera
1	3450.63	O-H stretching mode of hydroxyl groups N-H stretch	3434.00
2	2367.18	stretch of alkyne	2361.22
3	1653.92	N-H bending of amides, C = O stretch, carbonyl	1641.71
4	1427.31	CH_2 and CH_2 bend, pyrones and aromatic group	1421.00
5	1095 – 1075	Organic siloxane or silicone, Si-O-Si stretching	1094.12

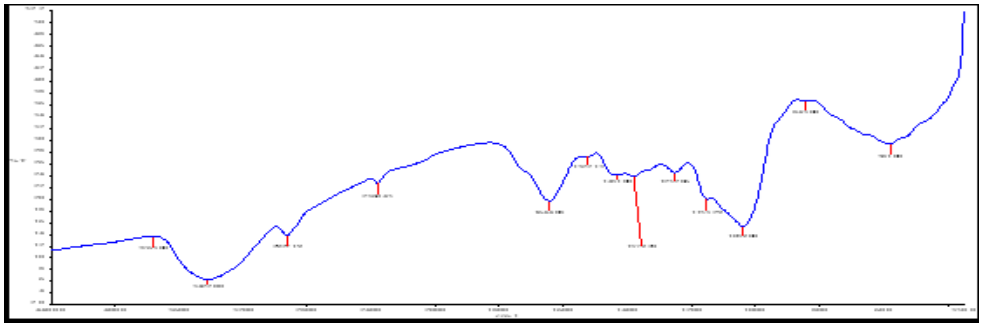

Fig 3: FTIR spectra of sugarcane bagasse

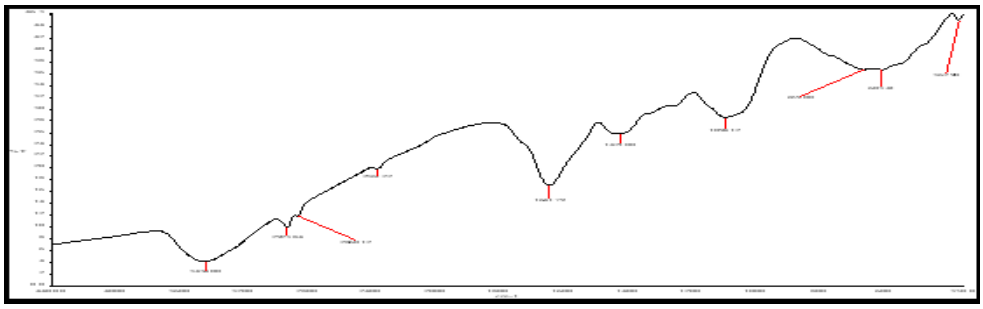

Fig 4: FTIR spectra of moringa oleifera

*Conclusion***:** The textural characteristics and chemical composition of moringa oleifera and sugarcane bagasse as cost effective, locally/readily available, environmentally friendly alternative adsorbent for wastewater treatment has been successfully investigated. It was concluded from this study that moringa oleifera and sugarcane bagasse possess good adsorbent properties which can be harnessed for the treatment of domestic and industrial wastewater.

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