International Research Journal of Agricultural Science and Technology

Open Access

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Edible Journals

Vol. 1 (2), pp. xxx –xxx
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Full Length Research Paper

Effectiveness of activated carbon from low cost agricultural wastes: physico-chemical and spectroscopic characterization

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Accepted July 12, 2017

Groundnut and Maize is the most widely grown grain crop throughout the West Africa. The shell of the groundnut and cob of the maize were used to prepare activated carbon through chemical activation method using concentrated 75% H_3PO_4/char and under temperature of 60^oC for 24 h. The product of activated carbon was examined for its physico-chemical parameters: such as moisture (9.61±0.21%, 9.85±0.06%) for groundnut shell and maize cob activated carbon respectively, ash (4.85±0.05%, 5.5±0.1%), bulk density (0.46±0.02g/m^3, 0.37±0.01g/m^3), yield (74.15±0.21%, 72.50±0.01%), volatile matter (24.79±0.19%, 22.21±1.0), iodine number (235.36±20.3mg/g, 367.66±30mg/g) and point of zero charge (6.4, 6.8). X-ray Fluorescence study on groundnut shell activated carbon (GSAC) and maize cob activated carbon (MCAC) shows difference concentration of inorganic elements, the major elements in the samples are potassium, calcium, titanium, copper, sodium and magnesium. The Fourier Transform Infra-red spectroscopy results showed a strong and broad absorption peak in both GSAC and MCAC adsorbents which correspond to C=O, O-H, C=C, N-H and C-O stretching. The corresponding Scanning Electron micrographs being obtained at 1000 and 10000 magnifications shows particle grains, ribs like, fibrous and jelly like structure.

Key words: Agricultural wastes, activated carbon, characterization, adsorbents

INTRODUCTION

The agricultural by-products and waste have little economic value, and their disposal not only is costly but may also cause environmental problems. Conversation of these agricultural waste into carbonaceous adsorbents that can be used in food application such as sugar refining or waste water treatments or in pharmaceuticals industry would add value to these agricultural commodities, help reduce the cost of waste disposal, and provide a potentially cheap alternative to existing commercial carbonaceous adsorbents. Activated carbon is prepared and activated by various methods, including thermally and chemically with acids and various salts. Previous study mentioned that activated carbon and the method of activation determine the effectiveness of the carbon as a decolourization of sugar syrup and many other applications (Elnasri et al., 2013).

It has reported that activated carbon is a well known adsorbent that can be used efficiently for a removal of extensive pollutants from air, soil and liquids. Adsorbents are usually porous solids and adsorption occurs mainly on the pore walls inside particles. Among them activated carbon is more efficient adsorbent for elimination of many pollutants (organic, inorganic, and

Table 1. Physico-chemical characterization of Groundnut shell activated carbon (GSAC), and Maize cob activated carbon (MCAC).

S. No.	Properties	GSAC	MCAC
1	Moisture content (%)	9.61±0.21	9.85±0.06
2	Ash content (%)	4.85±0.05	5.5±0.1
3	Bulk density (g/m^3)	0.46±0.02	0.37±0.01
4	%Yield	74.15±0.21	72.50±0.01
5	Volatile matter (%)	24.79±0.19	22.21±1.0
6	pH	6.8±0.01	6.6±0.1
7	Iodine number (mg/g)	235.36±20.3	367.66±30
8	Point of zero charge	6.4	6.8
9	Particle size (mm)	0.25	0.25

biological) of concern in water and waste water treatment.

In the recent years, it has been increasingly used for the prevention of environmental pollution which have been increased the sales of activated carbon for control of air and water pollution. Activated carbon is a broad – spectrum agent that effectively removes toxic and bio-refractive substances such as insecticides, herbicides, chlorinated hydrocarbons, heavy metal ions, and phenols, typically present in many water supplies (Ansari and Mohammed, 2009). Activated carbon in fact is a microcrystalline, non-graphitic form of carbon with porous structure that has been processed to develop its internal porosity. Activated carbon has a high degree of porosity, an extensive surface area, and a high degree of surface reactivity.

Maize (*Zea Mays*) and groundnut (*Arachis Hypogaea)* is the most widely grown grain crop throughout the West Africa and Americas. However, the cob and shells are unutilized, thereby becoming environmental nuisance. It is found that abundant functional group, such as hydroxyl, amino and carbonyl groups which contain lone pairs of electrons needed for adsorption of cationic pollutants in solution (Babarinde et al., 2012). Present research work was aimed to produce activated carbon from groundnut shell and maize cob using H_3PO_4 activating agent, and to characterize the produced activated carbon physico-chemically and microscopically in order to know the effectiveness of the adsorbents.

MATERIALS AND METHODS

The maize cob and groundnut shell was obtained from Ipata market in Ilorin metropolis, Kwara state of Nigeria. The samples were then dried at laboratory temperature for 7 days, sample was later dried in an oven at 105^{O}C for 12 h (Fan et al., 2003 and Itodo et al., 2009), after which it was crushed using laboratory mortar and pestle, followed by sieving using 0.25 mm sieve size. Five grams of the sieved samples was weighed into 10 different pre-weighed crucibles and it was introduced into the furnace at the temperature of 500^{o}C for 20minutes, the pyrolysed sample was cooled and ground into a granulated size and then activated (Viboon et al*.,* 2008). Activation was made by using H_3PO_4. The char sample was mixed with 75% concentrated orthophosphoric acid in a beaker with the weight ratio of H_3PO_4/ char to be 0.1. The slurry was evaporated at 60^{o}C for 24 h, and a dried mixture consisting of activated char and H_3PO_4 were obtained (Lillo- Rodenas et al., 2003). This was followed by washing to neutral pH of 7 (Viboon et al., 2008). The sample was then poured in a beaker containing 250ml of 0.1M HCl for purification and stirred for 1hour. Finally the sample was washed to a pH 6-7 (Strivastava, 1989). The resulted activated carbon was kept in tightly closed container for application (Viboon et al*.,* 2008).

RESULTS AND DISCUSSION

physico-chemical characterization of the adsorbents

The physical characteristics of GSAC and MCAC are shown in Table 1. Moisture content is the amount of water retained by the sample and suggests extensive porosity in the structure of adsorbents. Although, it has been observed that if the moisture content of adsorbent is high its adsorptive capacity will be reduced and it would necessitate utilizing an additional weight of the carbon during treatment process (Sugunadevi et al*.,* 2002). The moisture content of the adsorbents are 9.61±0.21 and 9.85±0.06 respectively, which is approximately the same as reported by Mane et al., 2005, (9.7%) and higher than the value reported by Salman et al., 2011, (4.5%), Pattabhi et al*.,* 2008 (2.5%).

The ash content shows the amount of inorganic constituents associated with carbon. The percentage ash contents for GSAC and MCAC adsorbents were obtained as 4.85±0.05% and 5.5±0.1% respectively, these are lower than the value reported by Salman et al., (2011), Raffiea et al., (2012), (10.7%), and close to the reported research work by Mane et al*.,* 2005, (5.6%). The ash content of adsorbents increased with decreased percentage of volatile matter which indicates that ash is non-volatile as reported by Gan et al., (2004).

Bulk density indicates the fibre content of the precursor. It is an important parameter when carbon is removed by filtration, because it determines the quantity of carbons that is contained in a filter of a given solid capacity and how much treated liquid is retained by the filter cake. Carbons with adequate density also help to

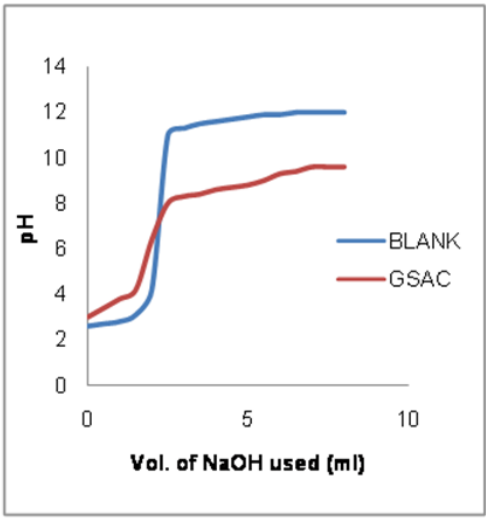


Figure1: PZC curve for GSAC adsorbent

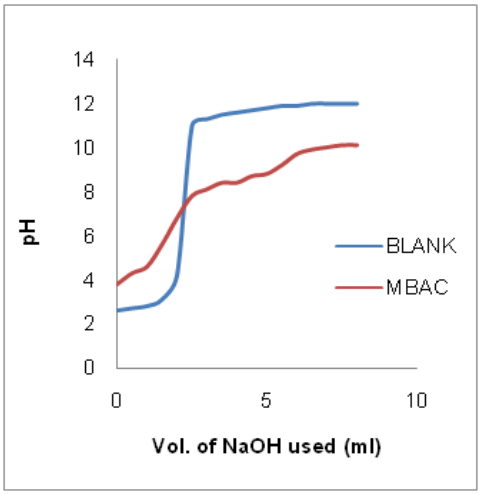


Figure 2: PZC curve for MCAC adsorbent

improve the filtration rate by forming an even cake on the filter surface. The bulk density of the GSAC and MCAC adsorbents are 0.46±0.02 and 0.37±0.01 g/m^3 respectively, which is lower than the value reported by Itodo et al., 2011, (0.53g/m^3) by raw mango seed back, (Onundi)* et al., 2011, (0.864g/m^3), however, the values are moderate compare with literature. Generally, an adsorbent with bulk density need not be regenerated continuously because it can hold more adsorbate per unit weight (Yang et al., 2003).

The percentage yield obtained for GSAC and MCAC adsorbents are 74.15±0.21% and 72.50±0.01% respectively. The adsorbents have high percentage yield compared to the reported percentage yield of 40% and 51% by Faust and Aly, (1983); Sahu et al., (2010) (46.08%).

Volatile matter is due to the presence of organic compounds present in the raw material. It was evident that at longer carbonization period, more volatiles are released from the char, thereby resulting to a higher burn off and a corresponding lower yield (Martinez et al., 2006). The volatile matter obtained for the GSAC and MCAC adsorbents are 24.79±0.19% and 22.21±1.0% respectively, which is higher than the value reported by Raffiea et al., (2012) (20.9%).

The pH of adsorbent is the degree of acidity or basicity of that adsorbent and this depends on number of factors which include preparation methodology, inorganic matter content, chemically active oxygen groups on its surface as well as the kind of treatment to which the adsorbent was subjected to Elnasri et al., (2013). The pH of GSAC and MCAC adsorbents are 6.8±0.01 and 6.6±0.1 respectively. These values were lower than reported by El-nasri et al., (2013), (7.8±0.01), and higher than those reported by Idris et al., (2012) (6.32 ± 0.12, 6.37 ± 0.1, and 6.41 ± 0.11).

The Iodine number is the most fundamental parameter used to characterize activated carbon performance (Raffiea et al., 2012). It is measure of activity level, the higher the number, the higher the degree of activation and the development of the microporous structure. It is often reported as mg/g. Iodine number may also be used as an approximation of surface area between 600mg/g and 1100mg/g (Aziz et al., 2009; Baccar et al., 2009). The iodine number of GSAC and MCAC adsorbents were 235.36±20.3mg/g and 367.66±30mg/g respectively, the results showed that these are lower compared to 798 (Raffiea et al., 2012). The adsorbents have higher iodine number and higher degree of micropore, and it is an indication of better performance as adsorbents (Raffiea et al., 2012).

The Point of Zero Charge (PZC) is a phenomenon of adsorption, and it describes the condition when the electrical charge density on a surface is zero (Lyklema 1995; and Kirby 2010). The PZC is located at the point of interception of the curves when traced to the pH axis. The PZC is (usually) the pH value at which a solid submerged in an electrolyte exhibit zero net electrical charge on the surface. The PZC of GSAC and MCAC adsorbents as shown in figure 1 and 2 below, are 6.4, and 6.8 respectively which are lower to the reported Elnasri et al., (2013) (7.3).

spectroscopic characterization of adsorbents

X-ray Fluorescence.

XRF analysis of GSAC and MBAC adsorbents were carried out with XRF instrument (AMP TEK-100CR Model), at Centre for Energy Research and Development Obafemi Awolowo University, Ile –Ife. Osun State. Nigeria.

The XRF data of GSAC and MCAC adsorbents indicates various elemental concentrations. From the Table 2, K, Ca, Ti, Cu, Na and Mg constitute higher concentration of inorganic elements in the adsorbents while compared to Cr, Mn and Fe concentrations. This showed that GSAC and MCAC cannot be used as adsorbent in the removal of metals such as K, Cr, Mn and Fe. It will serve better purpose for the removal of metals such as Mn, Co, Pb, Cd and As. In catalytic processes, those used in organic synthesis required transition metals which include Fe, Ti and Zn etc. The prepared GSAC and MCAC adsorbents contain

Table 2. X-ray fluorescence (XRF) analysis result for GSAC and MCAC adsorbents

Elements	GSAC (conc)	MCAC (conc)
K	19.89±1.2	21.201±3.1
Ca	9.639±2.64	11.02±1.18
Ti	1.965±0.17	0.232±0.1
Cr	0.011±0.02	0.016±4.2
Mn	0.39±0.10	0.887±0.22
Fe	0.432±0.01	1.423±0.21
Cu	1.12±1	3.711±0.8
Na	1.13±2	1.927±12.1
Mg	5.61±2	6.311±8

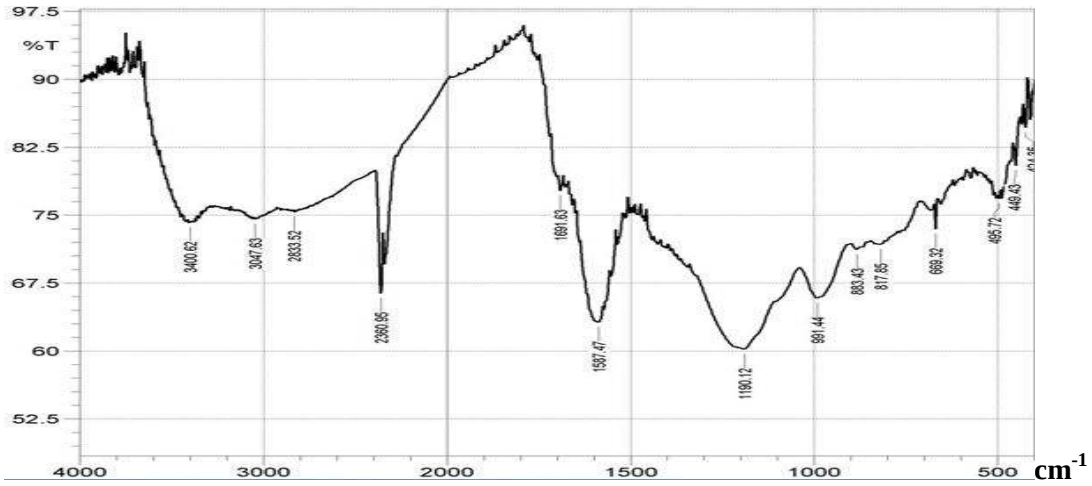


Figure 3 : FTIR spectrum of GSAC adsorbent

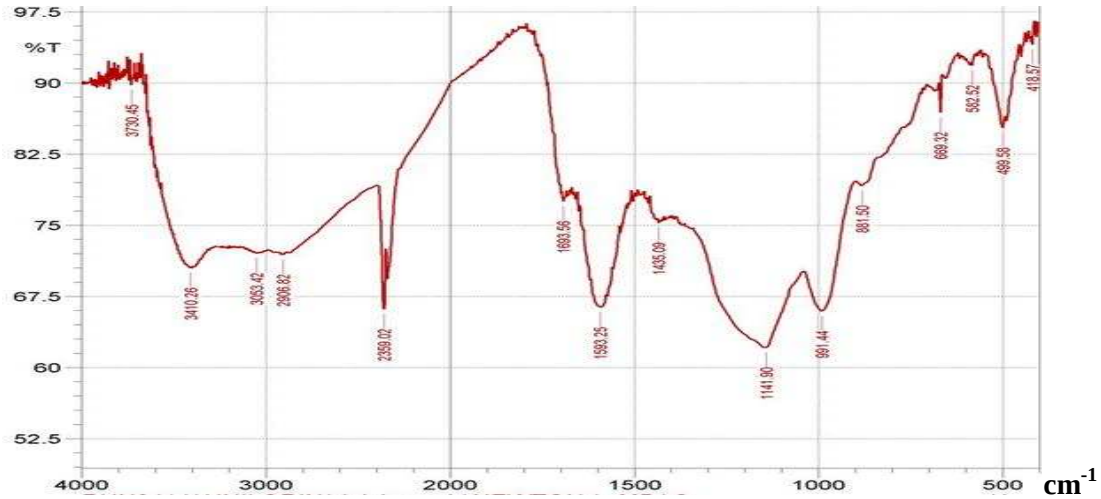


Figure 4 : FTIR spectrum of MCAC adsorbent

concentration of transition metals which may be useful in catalytic processes and during adsorption (Mishra et al., 2009).

Fourier Transform Infra-Red Spectroscopy

The FTIR of the GSAC and MCAC respectively were done with instrumental model Shimadzu 8400S at Redeemer University, Sango, Ogun State, Nigeria. The spectrums of each sample were shown in Figure. 3 and Figure 4. The FTIR analysis was carried out in order to identify the different functional groups present in GSAC and MCAC adsorbents which were responsible for adsorption process. The peaks appearing in the FTIR

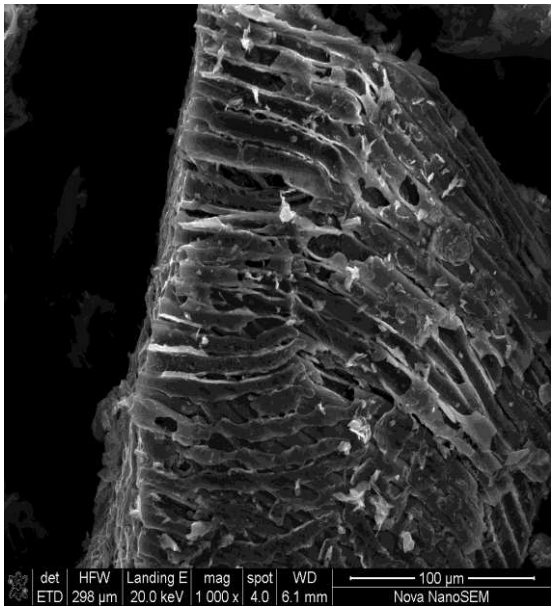


Figure 5: SEM Micrograph of GSAC at X1K

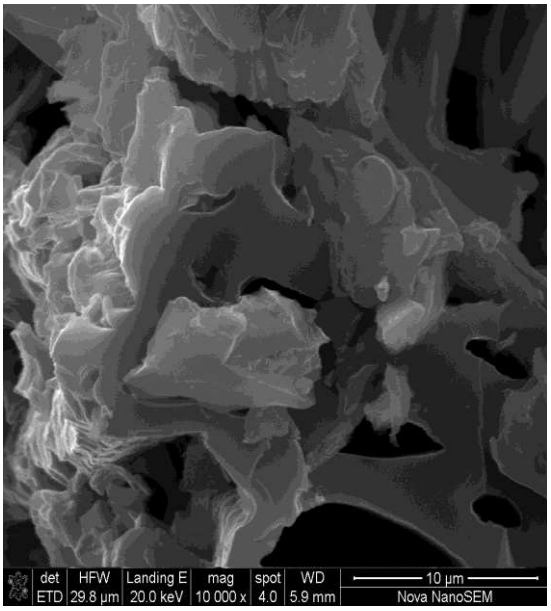


Figure 6: SEM Micrograph of GSAC at X10K

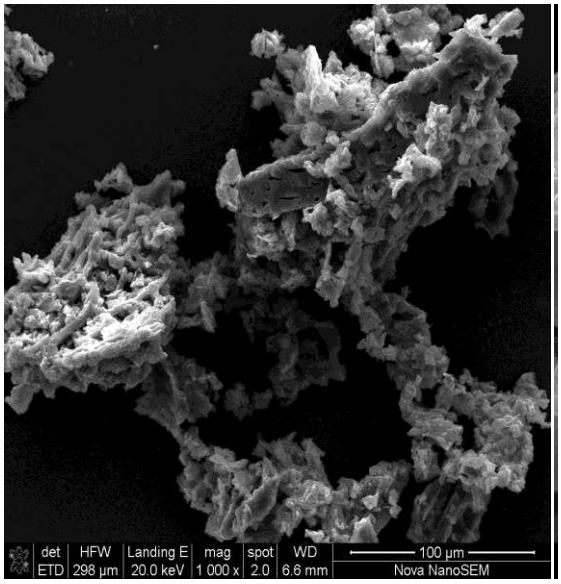


Figure 7: SEM Micrograph of MCAC at X1K

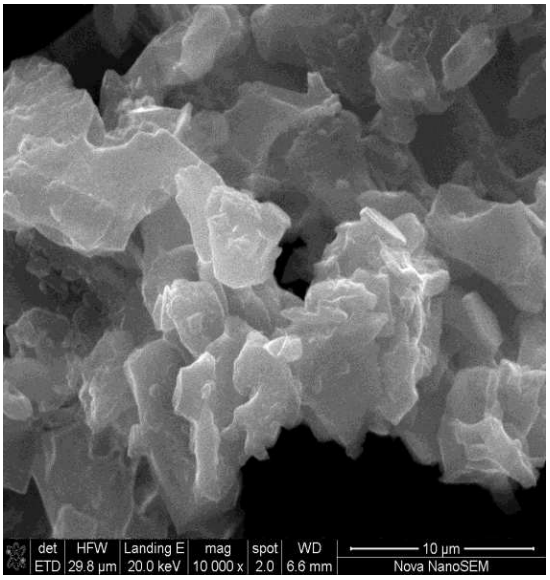


Figure 8: SEM Micrograph of MCAC at X10K

spectra were assigned to various functional groups according to their respective wave number (Das and Mondal, 2011).

The GSAC and MCAC adsorbents exhibit functional groups, including O-H and N-H stretching in the range of 3417-3400 cm^{-1}, the reduction of hydrogen bonding showed that the phosphoric acid used as activating agent act as a dehydrating agent, reacting with raw materials as soon as both substances are mixed (Jagtoyen and Derbyshire 1998; Suarez-Garcia et al., 2002). The stretching absorption band in the range of 1735-1691 cm^{-1} observed for the adsorbents is associated with the carbonyl C=O. The peaks at approximately 1580-1638 cm^{-1} in the samples corresponded to the C=C stretching attributed to the presence of lignin aromatic bond (Florida et al., 2009). After activation, a significant reduction was observed for a series of complex bands in the range of 1200-1000 cm^{-1} which corresponded to the C-O stretching in carboxylic acids, alcohol and esters (Deng et al., 2010). Both GSAC and MCAC adsorbents are observed in the range of 700-600 cm^{-1} indicating C-C stretching.

Scanning Electron Microscopy

The surface morphology of GSAC and MCAC before adsorption was analyzed by scanning electron microscopy (SEM), the corresponding SEM micrographs being obtained at 1000 and 10000 magnifications are shown in Figure 5-8. The micrographs revealed rough

surface area of the carbon which generally believes to be a good requirement for the adsorptive properties. It is generally observed that pore structure development is influenced by many factors, such as inorganic impurities and the initial structure of the carbon precursor (Deng et al., 2010).

The surfaces showed particle grains, ribs like, fibrous and jelly like structure. The presence of small pores on the image from SEM (Figure.5 and 7) showed that the adsorbents developed an elementary pore network.

CONCLUSION

Preparation of activated carbon from agricultural wastes was done successfully. The various properties studied in the present research work indicated that applicability of groundnut shell and maize cob waste converted to value added product-AC; it has extensive applications and effective low cost adsorbent for the removal of contaminants from various industrial waste water/effluents.

ACKNOWLEDGEMENTS

The author is grateful to the entire laboratory Technologist, Department of Industrial chemistry, University of Ilorin, Ilorin, Kwara state, Nigeria, for their support during the research period.

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