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## Adsorption Characteristics of Active Carbons from Pyrolysis of Bagasse, Sorghum and Millet Straws in Ortho Phosphoric Acid

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**Abstract:** Pyrolysis of bagasse, sorghum and millet straws impregnated with ortho phosphoric acid, produced carbon with heterogeneous pore structure and high adsorption capacities. Carbon precursors with particle size of 1180  $\mu\text{m}$  were pyrolysed, to overcome difficulties caused by low density and high ash content. The active carbons obtained from low impregnation ratios (<13.6) were largely microporous. Higher impregnation ratio did not substantially benefit the evolution of micropores in the pyrolysed samples. Fast attainment of sorption equilibrium was skewed towards high initial concentrations of methylene blue. The impregnation ration, controlled by varying the proportion of  $\text{H}_3\text{PO}_4$ , had strong influence on the yield of carbon. Equilibrium yields were highest at 450°C, with impregnation ratio of 13.6. Production of active carbons was completed in 45 min. The adsorptive capacities of the active carbons for large molecular weight compounds such as methylene blue were 502, 662 and 390  $\text{mg g}^{-1}$  for bagasse, sorghum and millet straws, respectively. The iodine numbers which indicate the adsorptive capacities for low molecular weight substances were 626, 667 and 593  $\text{mg g}^{-1}$  for bagasse, sorghum and millet straws, respectively. These iodine numbers are more than 18% higher than the value recommended for low molecular weight compounds by American Water Works Association.

**Key words:** Pyrolysis, activated carbon, micropores, mesopores, impregnation

### INTRODUCTION

The pore structure and adsorption properties of active carbons otherwise known as activated carbons are strongly influenced by physico-chemical nature of precursor materials and the heat treatment profile (Yun *et al.*, 2001; Jagnaribe *et al.*, 2005). Generally, the quality of active carbons is evaluated in terms of their adsorptive characteristics and superficial area, using different analytical methods for liquid and gas phase adsorption (El-Hendawy *et al.*, 2001; Jaguaribe *et al.*, 2005). For the gas phase, characterization may rely on measurement of the adsorption of nitrogen and application of one of the different procedures available to determine superficial area, such as the BET method (Brunauer *et al.*, 1938) or that of Langmuir (Japanese International Standard, 1992). For the liquid phase, the characterization may be achieved with iodine or methylene blue adsorption (Jaguaribe *et al.*, 2005; Omonmheule *et al.*, 2006). Determination of the specific surface of active carbon by the low temperature Nitrogen method and to a limited extent by the electron microscope have the disadvantage of being both complex and tedious in operation and therefore the development of a simpler one was desirable (Ekezie and Onwodi, 2005). Consequently, the investigation into the adsorption of iodine by active carbons (Bhatia *et al.*, 2000; Ikhuoria and Omoumheule, 2006) and subsequent investigation and adoption by ASTM were pursued.

Chemical activation is one of the possible methods for the production of active carbons (Guo *et al.*, 2000; Hayashi *et al.*, 2000, 2002; Baquero *et al.*, 2003; Suarez-Garcia *et al.*, 2004). The carbon precursors are pyrolysed in presence of the chemical activating agent such as

phosphoric acid ( $H_3PO_4$ ), zinc chloride ( $ZnCl_2$ ), potassium hydroxide (KOH) etc. Phosphoric acid functions as a dehydrating agent and inhibits the formation of tar (Su *et al.*, 2003). The use of phosphoric acid is considered to be an environmentally benign technology, because of the ease of its recovery by washing with water (Baquero *et al.*, 2003). Special emphasis on the preparation of active carbons from agricultural by-products has been given, due to the growing interest in low-cost active carbons from renewable safe copious supplies, especially for applications concerning environmental monitoring. Increased use of agricultural residues is an attractive addition to the utilization of wood and other carbonaceous materials for the production of active carbons. It is expected to help combat global warming and climate change when the burning of the agricultural residues on farm lands are minimized. Active carbons from waste agricultural materials such as bagasse, millet and sorghum straws have been considered in this study, as an attractive solution to mitigate the third world environmental protection needs. Previously, woodbark (Darmstadt *et al.*, 2000; Minkova *et al.*, 2001); bagasse (Minkova *et al.*, 2000, 2001; Garcia-Perez *et al.*, 2002; Jaguaribe *et al.*, 2005); maize cobs (Ekanem, 1996), rice straws (Yun *et al.*, 2001, 2002; Oh and Park, 2002), rice husk (Imagawa *et al.*, 2000), coconut shells (Gimba *et al.*, 2001), oil palm shells, walnut shells (Hayashi *et al.*, 2002), coffee bean husks (Baquero *et al.*, 2003); peach stones (Maroto-Valer *et al.*, 2004) and waste tyres (Mui *et al.*, 2004) have been pyrolysed to produce active carbon, but activation by ortho phosphoric acid impregnation of bagasse, millet and sorghum straws have not been well documented.

In the present study, the effect of phosphoric acid impregnation ratio on the porous texture of the prepared active carbons was investigated with iodine and methylene blue adsorption. The iodine number is a relative indicator of porosity in an activated carbon, although it may not necessarily provide a measure of the carbon's ability to absorb other species. However iodine number may be used as an approximation of surface area and microporosity of active carbons with good precision but the concentration of the standard iodine solution must be maintained at 0.1 N (ASTM D4607-94, 2006). It is also recommended that the standard iodine solution should have an iodide to iodine weight ratio of 1.5 to 1 (ASTM D4607-94, 2006). Sodium thiosulphate was used as a desorbing reagent because it is not adsorbed by active carbon and its strength does not influence the amount of iodine desorbed (ASTM D4607-94, 2006). According to ASTM, the precision of this test method in the determination of iodine number of active carbons ranging from 600 to 1450 is  $\pm 5.6\%$  of the average value measured in milligrams (mg) iodine absorbed per gram of carbon. This range corresponds to two standard deviations (2S) or the 95% confidence limits. The reproducibility of the method is  $\pm 10.2\%$  of the average value of iodine number as measured in mg of iodine absorbed/gram carbon. This range also corresponds to 2S or the 95% confidence limits (ASTM D4607-94, 2006).

The larger size of methylene blue makes it suitable for estimating mesoporosity. The suitability of iodine and dyestuffs adsorption for characterizing active carbon have been implicated in several studies by Bhatia *et al.* (2000), Gimba *et al.* (2001), Ochonogor (2005), Ochonogor and Ejikeme (2005) and Omonmheule *et al.* (2006).

Most important, in adsorption process is a porous solid medium having high adsorptive capacity. A large surface area or large micro-pore volume may be achieved due to the porous structure of the solid. The success of the adsorption process depends on the performance of adsorbents in both equilibria and kinetics (Das *et al.*, 2004). A solid exhibiting favourable adsorption isotherm as well as faster kinetics is supposed to be a good adsorbent. Therefore in order to be a good adsorbent, a solid must have a reasonably larger surface area and relatively larger pore network (Barata-Rodrigues *et al.*, 2003; Das *et al.*, 2004). Transient response of the adsorbent bed to a step-change in the influent concentration is reflective of the adsorbents performance under dynamic conditions (Das *et al.*, 2004). For the applications involving removal of colour, taste, odour and toxicants from aqueous matrices, adsorption capacity of active carbon may be determined by aqueous phase isotherm technique

(ASTM D3860-98, 2003). The amount of constituents removed and the adsorptive capacity is calculated from a Freundlich isotherm (ASTM D3860-98, 2003). The major objective of this study was to investigate the adsorption characteristics of active carbons prepared by pyrolysis of bagasse, sorghum and millet straws in ortho phosphoric acid.

## MATERIALS AND METHODS

### Pyrolysis and Activation

Dry straws of sorghum and millet were collected after harvest, from Rigasa farm center (non industrial area) in Kaduna state (Northern Nigeria), in March 2006. Sugarcane were obtained from the same area and processed to bagasse. The straws and bagasse were air-dried for 3 months on plastic mesh, to ensure adequate drying for easy milling and avoid the loss of carbon residue due to oven-drying. The straws were cut into pieces of approximately 3 cm to obtain the samples for milling. The bagasse and chopped straw samples were milled with Christy and Morns miller at the National Animal Production Research Institute (NAPRI), Zaria. The milled bagasse and straw samples were sieved into 1180 particle size with Endecotts laboratory test sieve on Omron No. 17748 (manual timer) sieve shaker. In porcelain crucibles, 0.5 g of the 1180  $\mu\text{m}$  grains of bagasse, sorghum or millet straws was impregnated with  $\text{H}_3\text{PO}_4$ , by adding drop wise, (while stirring the solid to facilitate homogeneous absorption of the acid) the amount of the aqueous acid (2.5  $\text{cm}^3$ ) necessary to produce swelling until incipient wetness (Baquero *et al.*, 2003). Different concentrations of the  $\text{H}_3\text{PO}_4$  corresponding to 2.5, 3.0, 3.5, 4.0, 4.5, 5.0, 5.5, 6.0, 6.5 and 7.0  $\text{cm}^3$   $\text{H}_3\text{PO}_4$  in aqueous solutions were used to vary the content of impregnation agent. After impregnation, the samples were dried for 1 h at 110°C in an oven. The dried samples were transferred to digital thermo control, Gallenkamp electric furnace and heated at the various pyrolysis temperatures (100-450°C) for the various heating periods (residence time) (16-280 min). The solid pyrolysis residues were cooled at room temperature and later transferred to desiccators containing silica gel as desiccant. The carbonized samples were washed sequentially, several times with hot deionised water and finally with cold deionised water until the solutions were neutral to litmus paper. The washed samples were dried at 110°C to prepare the active carbons.

### Iodine Adsorption

American standard test method (ASTM D4607-94, 2006) for determination of iodine number of activated carbon was used to assess the porosity of the active carbons prepared. The test is based on a three-point adsorption isotherm. The amount of iodine adsorbed ( $\text{mg g}^{-1}$  carbon) at a residual iodine concentration of 0.02 N was reported as the iodine number. Granular forms of the active carbons were used throughout.

The carbon dosages used were estimated as follows:

$$M = [A - (DF)(C)(126.93)(50)]/E \quad (\text{ASTM D4607-94, 2006})$$

Where:

M = Carbon (g)

A = ( $N_2$ ) (12693.0)

DF = Dilution factor

C = Residual iodine

E = Estimated iodine number of the carbon

$N_2$  = Iodine (N)

Three carbon dosages (2.2594, 1.9801 and 1.7009 g) were calculated using three standard values of C (0.01, 0.02 and 0.03, respectively) and estimated iodine number of 500 mg g<sup>-1</sup> (minimum value recommended for active carbon to be used in removing compounds of low molecular weight (Jaguaribe *et al.*, 2005)). Each weighed sample of carbon was transferred to a clean, dry 250 cm<sup>3</sup> Erlenmeyer flask equipped with a ground glass stopper. Ten cubic centimeter of 5 wt.% HCl solution was added to each flask containing carbon. Each flask was stoppered and swirled gently until the carbon was completely wetted. The stoppers were loosened to vent the flasks and they were heated to bring the contents to boil for 30 sec. The flasks were removed and cooled to room temperature (29°C). One hundred cubic centimeter of 0.1 N iodine solution was pipetted into each flask. The addition of iodine solution to the three flasks was staggered to minimize delay in handling. The flasks were immediately stoppered and shaken vigorously for 30 sec. Each mixture was quickly filtered by gravity through one sheet of folded filter paper (Whatman No. 2v). Clean beakers were used to collect the filtrates after discarding the first 30 cm<sup>3</sup> portions of the filtrates. Each filtrate was swirled and 50 cm<sup>3</sup> of it pipetted into a clean 250 cm<sup>3</sup> Erlenmeyer flask. Each filtrate was titrated with 0.1 N sodium thiosulphate solution until the solution turned yellow. Two cubic centimeter of freshly prepared starch indicator solution was added and the titration continued with sodium thiosulphate until one drop produced a colourless solution. The volume of sodium thiosulphate used was noted.

Iodine absorbed (X/M) per gram of carbon (mg g<sup>-1</sup>) was calculated as follows:

$$X/M = [A - (DF) (B) (S)]/M \quad (\text{ASTM D4607-94, 2006})$$

- N<sub>2</sub> = Iodine (N) = 0.1
- S = Sodium thiosulphate (cm<sup>3</sup>)
- M = Carbon used (g)
- DF = Dilution factor = (100+10)/50 = 2.2
- B = (N<sub>1</sub>) (126.93)
- N<sub>1</sub> = Sodium thiosulphate (N) = 0.1

Concentration of residual filtrate (C) expressed in normal (N) was calculated as follows:

$$C = (N_1 \cdot S)/F$$

- N<sub>1</sub> = Sodium thiosulphate (N) = 0.1
- S = Sodium thiosulphate (cm<sup>3</sup>)
- F = Filtrate (cm<sup>3</sup>) = 50 cm<sup>3</sup>

### **Methylene Blue Adsorption**

0.1 g each of the active carbons from bagasse, sorghum and millet straws (obtained using 13.6 acid impregnation ratio) was dispensed into 250 cm<sup>3</sup> Erlenmeyer flasks. Fifty cubic centimeter of different solutions of methylene blue containing 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 4.5 and 5.0 mg of methylene blue, respectively, were added and stoppered. In each case, the flask was shaken on Griffin flask shaker at room temperature of 29°C, for various times of 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55 and 60 min, respectively. The mixture in each flask was filtered by suction through a sintered glass crucible and the different filtrates were collected and analysed using Cole 7506 UV-VIS spectrophotometer. Standard solutions of methylene blue were used for calibration. To determine the adsorptive capacity of the active carbons produced, 1.0, 2.5, 5.0, 7.5, 10.0, 15.0, 25.0, 35.0, 45.0 and 50.0 mg of active carbons were used for the adsorption of methylene blue from solutions containing 5 mg methylene blue in 500 cm<sup>3</sup> (ASTM D3860-98, 2003).

## RESULTS AND DISCUSSION

### Starting Materials

From the proximate studies reported by Lori *et al.* (2007) the effects of particle size on weight loss characteristics, rates of dehydration and volatile matter of the carbon precursors were used to adjust particle size of 1180  $\mu\text{m}$  as appropriate for the carbonization of the bagasse, sorghum and millet straws. High contents of volatile matter ( $64.52 \pm 1.18$ - $66.65 \pm 3.07$ ), fixed carbon ( $21.93 \pm 2.74$ - $24.16 \pm 1.94$ ) and low ash ( $2.27 \pm 0.15$ - $3.80 \pm 0.17$ ) are favourable properties of the precursors for the production of active carbon (Lori *et al.*, 2007).

### Pyrolysis and Activation

Carbon precursors with particle size of 1180  $\mu\text{m}$  were used, in order to overcome difficulties caused by low density and high ash content. The impregnation ration, which was controlled by varying the proportion of  $\text{H}_3\text{PO}_4$  used for activation, had a strong influence on the yield of active carbon. The yields of active carbons indicate that the temperature domain in which carbonization occurs depends on the interaction between the furnace residence condition and chemical activation. Equilibrium yields of the active carbons were highest at 450°C. At 450°C productions of active carbons from bagasse, sorghum and millet straws were completed in 45 min. The percentage yields of active carbons from bagasse, sorghum and millet straws at 450°C, were 53.6, 30.6 and 29.6% with residence times of 28, 36 and 32 min, respectively. The yields were obtained with optimum impregnation ratio of 13.6. Attainment of steady equilibrium during pyrolysis was the criteria for selecting the best temperature for the production of active carbons from the precursors. Consequently, only carbons produced at 450°C were studied, as there were no steady equilibria attained at 100-350°C.

### Adsorption Characteristics

Pyrolysis of bagasse, sorghum and millet straws impregnated with ortho phosphoric acid produced carbon with well developed pore structure and high adsorption capacities. The porous texture of the active carbons was characterised by aqueous phase adsorption of iodine and methylene blue. From Fig. 1, it was deduced that the active carbons obtained from low impregnation ratios are

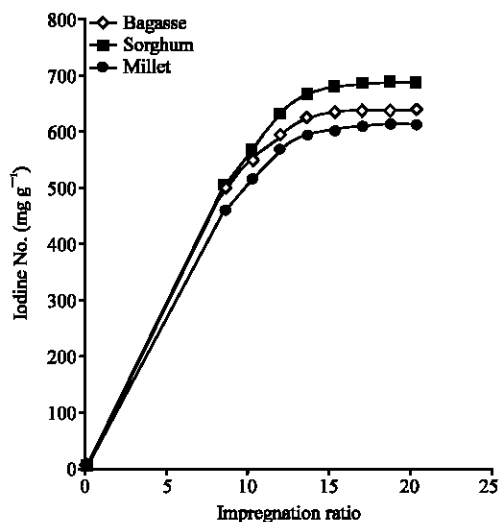


Fig. 1: Effect of phosphoric acid impregnation ratio on evolution of micropores in active carbons from bagasse, sorghum and millet straws

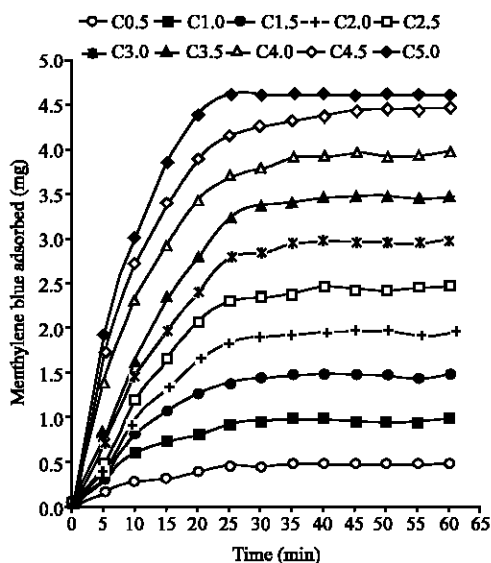


Fig. 2: Effect of contact time on the adsorption of different initial concentrations of methylene blue [Cx (mg/50 cm<sup>3</sup>)] on active carbons from sorghum straws

microporous as indicated by the steady increase in iodine number below impregnation ratio of 13.6. Higher impregnation ratio did not substantially benefit the evolution of micropores in the carbonised samples as shown in Fig. 1. Except active carbons from millet straws with iodine number of 593, carbons from bagasse and sorghum straw gave iodine number within the reported values (600-647) for carbons from pelletised straws and bagasse, that were activated physically during carbonisation (Minkova *et al.*, 2001; Zanzi, 2001). The results from the present study may be considered superior to those reported by Minkova *et al.* (2001) and Zanzi (2001) with regard to energy demand of the two processes.

The adsorption of different concentrations of methylene blue on active carbons from bagasse, sorghum and millet straws appeared to depend heavily on the contact time and initial adsorbate concentration (Fig. 2-4). Similar effect of adsorption contact time and initial adsorbate concentration was earlier reported for indigo carmine removal by activated carbons from *Terminalia catappa* and *Cinnarium schweinfurthi* nut shell (Ochonogor, 2005). Fast attainment of sorption equilibrium was skewed towards high initial concentrations of methylene blue. With active carbons from sorghum straws, initial methylene blue concentrations of 1.5- 4.5 mg in 50 cm<sup>3</sup> aqueous solutions produced adsorption that tends to equilibria after 30 min. The Fastest equilibrium was attained in 25 min with the optimum initial adsorbate load of 0.1 mg cm<sup>-3</sup> methylene blue solution (5 mg/50 cm<sup>3</sup> solution). However, adsorption was nearly all time linear below initial methylene blue concentration of 1.5 mg/50 cm<sup>3</sup> solution. The adsorption profile of carbons from bagasse and millet straw were similar to the sorption profile obtained for carbons from sorghum straw with the various initial concentrations, except the variability in their adsorption capacities (Fig. 2-4). The maximum amount of methylene blue adsorbed by active carbons from the different precursors in 5 mg/50 cm<sup>3</sup> solutions were in the order, sorghum straw (4.65 mg) > bagasse (4.545 mg) > millet straw (4.435 mg) for sorption equilibrium time of 25 min. High impregnation ratio that resulted in increased amount of impregnated phosphoric acid, may have developed pores in the carbon, with wide range of sizes that may be predominantly mesoporous as indicated by the high percentage of methylene blue adsorbed (Fig. 5). This may have resulted from the increased amount of the intercalated phosphoric acid and various polyphosphates,

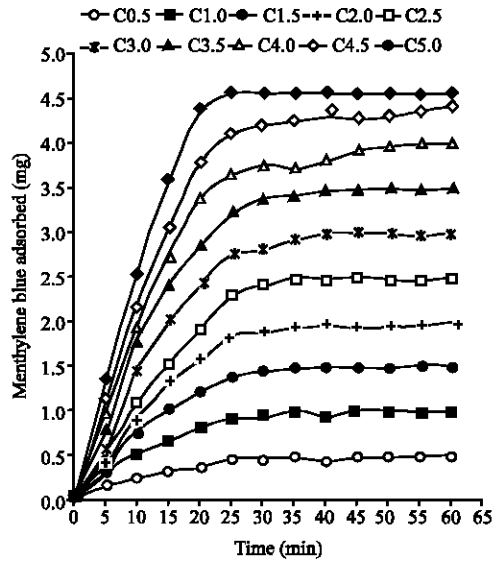


Fig. 3: Effect of contact time on the adsorption of different initial concentrations of methylene blue [Cx (mg/50 cm<sup>3</sup>)] on active carbons from bagasse

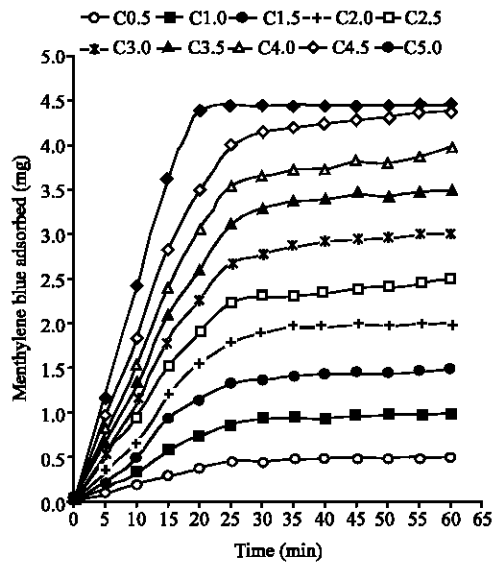


Fig. 4: Effect of contact time on the adsorption of different initial concentrations of methylene blue [Cx (mg/50 cm<sup>3</sup>)] on active carbons from millet straw

which may produce larger pore volume and size when washed away from the carbons. Similar effects of the phosphoric acid impregnation on porosity development have been reported for other cellulosic precursors (Baquero *et al.*, 2003). The evolution of wide micropores and mesopores for methylene blue adsorption on carbons from bagasse, sorghum and millet straws follow the sequence, sorghum (93%) > bagasse (90.9%) > millet straw (88.7 %) (Fig. 5). General decrease in evolution of the pores with impregnation ratios greater than 13.6 was also showed in Fig. 5.



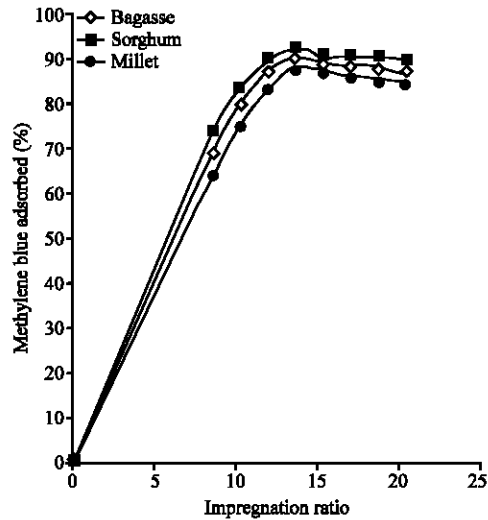


Fig. 5: Effect of phosphoric acid impregnation ratio on evolution of wide micropores and mesopores in active carbons from bagasse, sorghum and millet straws

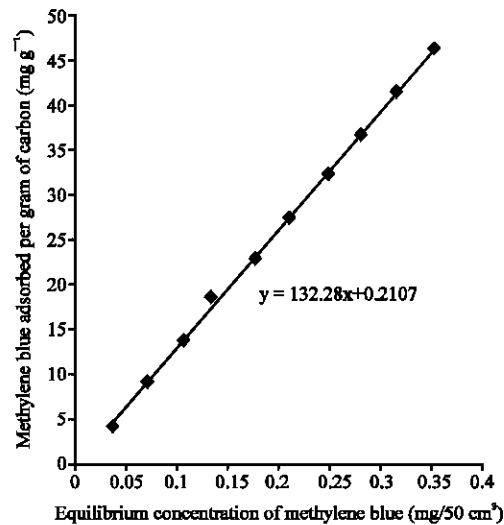


Fig. 6: Adsorption isotherm of methylene blue on active carbons from sorghum straw

The adsorption isotherm for iodine on active carbons has been reported to follow Langmuirian isotherm (Bhatia *et al.*, 2000). Figure 6, 7 and 8 show the methylene blue, Freundlich adsorption isotherms at room temperature (29°C), on active carbons from bagasse, sorghum and millet straws impregnated with phosphoric acid at 450°C with null soaking time. The amounts of the adsorbed methylene blue were calculated from the experimental data using material balance (ASTM D3860-98, 2003). The amount of methylene blue (x) adsorbed per unit weight of carbon (m) was expressed as the difference between the amount of methylene blue before carbon treatment and amount of methylene blue after carbon treatment, per unit weight of carbon. The isotherms were extrapolated and the equations of the lines were used to calculate the x/m that corresponds to the initial amount of

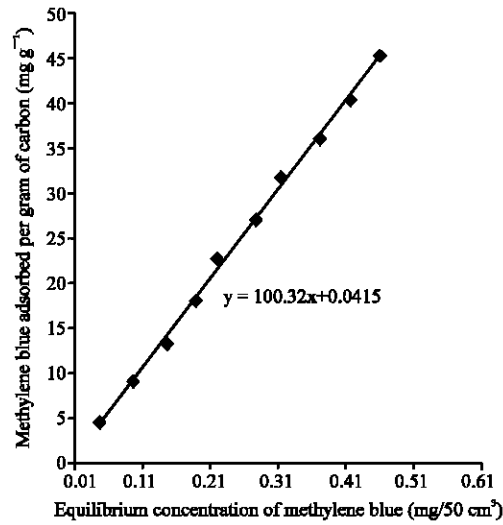


Fig. 7: Adsorption isotherm of methylene blue on active carbons from bagasse

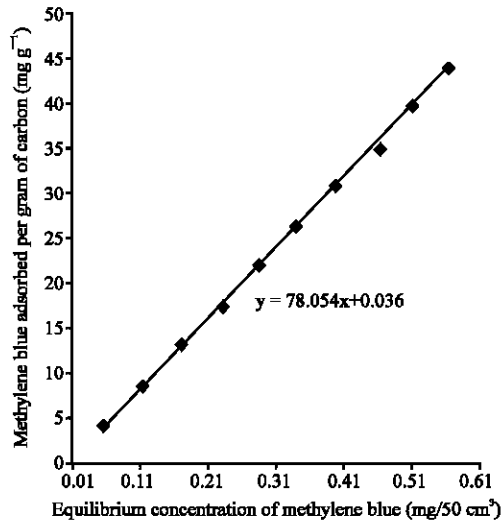


Fig. 8: Adsorption isotherm of methylene blue on active carbons from millet straw

methylene blue ( $C_0$ ) before carbon treatment. This value termed  $[x/m]_{C_0}$  represents the amount of methylene blue adsorbed when the carbon is in equilibrium with the influent concentration. The  $[x/m]_{C_0}$  is the ultimate capacity of the active carbon for the methylene blue (ASTM D3860-98, 2003). From Fig. 6-8, the adsorptive capacities of the active carbons from bagasse, sorghum and millet straws, for large molecular weight compounds such as methylene blue, were 502, 662 and 390  $\text{mg g}^{-1}$ , respectively. The iodine numbers which indicate the adsorptive capacities for low molecular weight substances were 626, 667 and 593  $\text{mg g}^{-1}$  for bagasse, sorghum and millet straws, respectively. These iodine numbers are more than 18% higher than the value recommended for low molecular weight compounds by American Water Works Association.

## CONCLUSIONS

The adsorptive capacities of the granular active carbons from bagasse, sorghum and millet straws, for large molecular weight compounds such as methylene blue, were 502, 662 and 390 mg g<sup>-1</sup>, respectively. The iodine numbers which indicate the adsorptive capacities for low molecular weight substances were 626, 667 and 593 mg g<sup>-1</sup> for bagasse, sorghum and millet straws, respectively. These iodine numbers are more than 18% higher than the value recommended for low molecular weight compounds by American Water works Association. The active carbons from the various cellulosic precursors had similar capacities to adsorb iodine and methylene blue. They are thus, recommended for removal of waste dye from textile effluents and trace metals from wastewater.

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