Article Number: 291D744

A Paper presented at the 39th CSN Annual International Conference, Workshop and Exhibition, Rivers State University of Science and Technology, Port Harcourt, Nigeria.
18th – 23rd September 2016

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Full Length Research Paper

Preparation and characterization of activated carbon from *Hura crepitans* Linn seed shells

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Activated carbons were thermally prepared from Hura Crepitan L. seed shells. Zinc chloride (ZnCl2) and phosphoric acid (H3PO4) were separately used as the activating agents. The activated carbons obtained were characterized by determining the percentage yield, moisture content, ash content and percentage fixed carbon. The adsorption of methylene blue by the activated carbon was done using 0.1 to 0.5 g of the activated carbon. The results revealed that the percentage yield and ash content of H3PO4 impregnated activated carbon was higher than ZnCl2 impregnated activated carbon. On the other hand, ZnCl2 impregnated activated carbon had higher moisture content and percentage fixed carbon. It was also revealed that ZnCl2 impregnated activated carbon had greater adsorption capacity than H3PO4 impregnated activated carbon. However, it was found that the higher the adsorbent (activated carbon) dosage, the higher the adsorption capacity.

Key words: Hura Crepitan L., activation, activated carbon, zinc chloride and phosphoric acid.

INTRODUCTION

Activated carbons are carbon of highly micro-porous form with both high internal surface area and porosity, and commercially, the most common adsorbents used for the removal of organic pollutant from air, water and industrial products (Tsai et al., 2001).

Food and pharmaceutical industries uses activated carbon in the removal of compounds that are responsible for undesirable colour, odour and taste in their products. Activated carbon is also used in gas cleaning applications where it is extensively used in air filters at industrial level as well as in air condition equipment (Vigayan et al., 2012).

Commercial activated carbon is produced from bituminous or lignite coal. Because of the environmental problems and increasing high cost associated with petroleum products, researches have reported the production of activated carbon from wood which is environmentally friendly and cost effective. However, because of the environmental problem associated with deforestation, seed shells have been used to replace wood for the production of activated carbon. These products are highly cost effective as most of the seed shells are discarded as waste materials, and the activated carbon produced from them have similar potential in industrial application.

The production of activated carbon from coconut shells,

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palm kernel shells, *Cananium scheqinflurthi* nut shell, corn cob and other seed shells have been reported (Olawale and Ajayi, 2009; Diya et al., 2008; Vijayan et al., 2012).

Hura crepitans L., the sand box tree also known as possum-wood, is an evergreen tree of the spurge family Euphorbiacae. The wood is used for furniture under the name "Hura". The oil extracted from the seed is used in the production of alkyl resin (Ezeh et al., 2012) and metallic soap (Umoren et al., 2013). The seed pods (shells) are not utilized for any industrial purposes; hence they are waste and thereby pollute the environment. These shells are hard and therefore suitable for the production of activated carbon which can be used for the treatment of waste water from textile industries and can also be used in food and pharmaceutical industries to remove pollutants/contaminants.

Therefore, the aim and objectives of this work was to prepare activated carbon from *Hura crepitans L.* shells, characterize and apply it in the removal of methylene blue from contaminated water.

MATERIAL AND METHODS

Sample collection

The seed shells of *H. crepitans L.* also known as sand box were collected from the University of Uyo Town Campus. The samples were cut into chips of workable size.

Activation-carbonization

Activation of the sample was carried out in a muffle furnace using the solution of zinc chloride ($ZnCl_2$) and phosphoric acid (H_3PO_4) separately as the activating agents. 400 g of the sample chips were charred in the furnace at 500°C for 2 h. 30 g of the charred sample was weighed into a beaker containing 300 cm^3 of 1 M solution of each activating chemical. The contents of each beaker was thoroughly mixed and heated until all the activating liquor was absorbed by the sample.

The samples were then transferred into the crucible and dried in the oven at 100°C for 12 h. The crucible was then placed in the furnace and fired at 650°C for 2 h.

The sample was then removed from the furnace and cooled in the desiccator, it was then washed with 0.5 M NaOH followed by distilled water to a neutral pH. The activated carbons obtained were dried in the oven at 100°C to a constant weight.

Characterization of the activated carbon

Percentage yield

The yield of the activated carbon was calculated using:

$$\text{Percentage yield} = \frac{M2}{M1} \times 100\%$$

Where M_1= Weight of the charred sample and M_2 = Weight of the activated carbon

Moisturecontent

Moisture content of the activating carbon was calculated using:

$$\text{Percentage yield} = \frac{M1 - M2}{M1} \times 100\%$$

Where M_1 = Weight of the activated carbon before drying in the oven and M_2 = Weight of the activated carbon after drying in the oven.

Determination of *Ash content and percentage fixed carbon*

Ash content of the activated carbon was determined as follows: 2 g of each activated carbon sample was put in a crucible and fired at 90 °C in a muffle furnace for 3 h. The resultant ash was removed and allowed to cool in the desiccator.

Ash content was calculated using:

$$\% \text{ Ash content} = \frac{W2}{W1} \times 100\%$$

Where W_2 = Weight of the ash and W_1 = Weight of the activated carbon

Percentage fixed carbon was calculated from the ash content in accordance with the method reported by Ogbonnaya (1992) and Duff and Ross (1988).

Adsorption study

The adsorption potentials of the activated carbon sample obtained from different activating agents were determined according to the method reported by Odebunmi and Okelola (2001). In this method, batch sorption experiments were carried out at room temperature. 0.1 to 0.5 g of the activated carbon was added into a beaker containing 100 ml of 250 mg/l methylene blue solution. The mixture was thoroughly mixed and shaken for 1 husing a mechanical shaker. After shaking, the suspensions were filtered and remaining concentration was determined spectrophotometrically at ë$_{nm}$ of 25 0nm using UV/V spectrophotometer. The amount of methylene blue adsorbed from each solution was calculated as shown below.

$$\text{Percentage methylene blue adsorbed} = \frac{Co - C}{Co} \times 100\%$$

Where, C_O (mg/L) = Initial concentration of methylene blue and c (mg/L) = post-adsorption concentration of methylene blue.

RESULTS AND DISCUSSION

The percentage yield, ash content, moisture content and percentage fixed carbon of the activated carbon are presented in Table 1. These parameters are factors which could influence the adsorption capacity of the activated carbon (Okeola et al., 2012).

Result revealed that the percentage yield of H3PO4

Table 1. Physical parameters of the activated carbon.

Activating Agent	H_3PO_4	$ZnCl_2$
Yield (%)	44.90	31.94
Moisture content (%)	5	17
Ash content (%)	28	16.5
Percentage fixed carbon (%)	72	83.8

Table 2. Adsorption of methylene blue by H_3PO_4 activated Carbon.

AC dosage (mg)	Post adsorption concentration of MB (mg/l)	Adsorption efficiency (%)
100	201.67	19.33
200	197.54	20.98
300	180.64	27.74
400	167.36	33.05
500	140.58	43.77

Table 3. Adsorption of methylene blue by **$ZnCl_2$** activated carbon.

AC dosage (mg)	Post Adsorption concentration of MB (mg/l)	Adsorption efficiency (%)
100	195.84	21.66
200	186.29	25.48
300	178.64	28.54
400	145.06	41.98
500	122.77	50.89

activated carbon was 44.90% and that of $ZnCl_2$ activated carbon was 31.94%. The decrease in the yield of $ZnCl_2$ activated carbon may be due to the reactions between $ZnCl_2$ and the sample.

The ash content and percentage fixed carbon compare favourable with those for the commercial activated carbon. Also, the ash content of H_3PO_4 activated carbon compared favourable with that of H_3PO_4, $ZnCl_2$ and KOH activated carbon from maize cob (Odebunmi and Okeola, 2001) and NaCl activated carbon of *Jatropha curcas* seed coats (Okeola et al., 2012). The ash content obtained for ZnCl2 activated carbon compared favourably with the value obtained for coconut shell (Odebunmi and Okeola, 2001). However, $ZnCl_2$ impregnated activated carbon had greater percentage fixed carbon than H_3PO_4 impregnated activated. There is a correlation between percentage fixed carbon and adsorption capacity. Sample with higher percentage fixed carbon are expected to have high adsorption capacities (Ogbonnaya, 1992). The moisture content of $ZnCl_2$ activated carbon was higher than that of H_3PO_4 activated carbon, implying that $ZnCl_2$ activated carbon has greater pore size for absorption than H_3PO_4 activated carbon.

Adsorption studies

Methylene blue was chosen in this study because of its recognized usefulness in characterizing adsorptive material (Itodo et al., 2010). The size of methylene blue molecule is much larger and its adsorption is restricted mainly to the mesopores adsorbent. However, a small portion is also found in larger micropores (Cleiton and Guerreiru, 2011).

The results of the methylene blue adsorption by the activated carbons are presented in Tables 2 and 3. The results revealed that the quantity of the adsorbent has greater effect on the adsorption of methylene blue from aqueous solution. The result shows that the adsorption efficiency increased with increase in mass of the adsorbent. The increase in adsorbent mass increases the contact surface area of the adsorbent particles which means that it become more probable for solute molecules to be adsorbed on the adsorption sites and

thereby increasing adsorption efficiency (Okeola et al., 2012).

Effect of activating agents

With all the adsorbent dosages, $ZnCl_2$ impregnated activated carbon sample has greater adsorption efficiency than the one prepared with H_3PO_4. This is due to the fact that samples with high percentage fixed carbon always have high adsorption capacities (Ogbonnaya, 1992). This result also suggests that $ZnCl_2$ impregnated activated carbon sample have well developed mesoporosity favorably for adsorption of larger molecules compared to H_3PO_4 impregnated activated carbon sample. However, the percentage adsorptions of methylene blue by these activated carbons are low, this may be due to the fact that the size of the methylene blue molecule is much larger and its adsorption is restricted mainly to the mesopores activated carbon (Cleiton and Guerreiru, 2011).

Conclusion

H. Crepitan L. seed shells were successfully converted into activated carbon using H_3PO_4 and $ZnCl_2$ as the activating agents. The results of the physical properties of the activated carbon compared favourably with the commercial activated carbon and with other activated carbons prepared from seed shells and cob. The adsorption of methylene blue by $ZnCl_2$ impregnated activated carbon was higher than H_3PO_4 impregnated activated carbon. However, H_3PO_4 impregnated activated carbon is recommended for used in food and pharmaceutical industrials since it is a non-toxic activating chemical as it does not contain heavy metal while $ZnCl_2$ impregnated activated carbon can be apply for the treatment of waste water from textile and allied industries.

CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

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