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Utilization of *Coula Eludies* Nut Shells in the Preparation of Activated Carbon

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ABSTRACT

Activated carbons were prepared by carbonization and activation of *Coula eludies* seed shells. This was done using $ZnCl_2$ as the activating chemical and the activation was done using sample to activating chemical ratios of 1: 10 and 1:20 (g/v). The activated carbon 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 100mg to 500mg of the activated carbon. The results revealed that the yield, moisture content, ash content and percentage fixed carbon of 1:10 impregnated activated carbon were 41.94%, 11.20%, 7.00% and 92.00% respectively while that of the 1:20 impregnated activated carbon were 38.76%, 18.56%, 5.50% and 96.50% respectively. These results show that the yield and the ash content of 1:10 impregnated activated carbon is higher than that of 1:20 impregnated activated carbon. While 1:20 impregnated activated carbon has higher moisture content and percentage fixed carbon. The adsorption study also revealed that 1:20 impregnated activated carbon had greater adsorption capacity. However adsorption efficient was increased with increase in adsorbent dosage.

Introduction

Activated carbons are carbon obtained as a product of carbonization and activation of carbonaceous materials. This process gives carbon with large surface area and high porous structure. These properties provide carbon with the ability to absorb gases, odour, dissolved and dispersed substances from solution (Odebumi and Okeola, 2001). These important characteristics of activated carbon are influenced by the raw material, activation method and processing conditions (olawale and Ajayi, 2009).

Activated carbon is usually prepared from the carbonaceous material either by physical or chemical activation methods. Physical activation involves carbonization of carbonaceous materials followed by the activation of the resulting char at a temperature between 1073 and 1373K in the presence of suitable oxidizing gases such as carbon (iv) oxide or steam. In chemical activation method, both the carbonization and activation processes are carried out simultaneously. The raw material is first mixed with the activating chemical in the proper ratio. The resultant paste is dried and then carbonized between 673 and 873K in the absence of air (Tsai et al., 1997). Chemical activation offers several advantages over physical activation in that it is carried out in a single step combining carbonization and activation, it is also perform at the lower temperature therefore saving the energy and the resultant activated carbon obtained has a better porous structure (Tsai et al., 1997). These methods have been employed in the preparation of activated carbon from carbonaceous materials like used tyre, maize cob, coconut shell guinea corn stem, orange peels (Tsai et al., 1997). *Cananium scheqinflurthi* nut shell (Olawale and Ajaji, 2007), Palm kernel shells (Adewumi And Ogedengbe, 2005), Fluted pumpkin (*Telfairia occidentalis* Hook.F) Seed Shell (verla,et al., 2012).

Coula edulies commonly known as African walnut or Gabon nut tree due to its edible seed is from the family Olacaceae. It is referred to as Ekom in Efik/Ibibio, Udo in Igbo, Ivianlegde in Edo and Asala in Yoruba , Nigeria (Ajibesin and Adebayo-Tayo 2008). *Coula edulis* is a tree in the genus *Coula*, native to tropical western Africafrom Sierra Leone to Angola. It is plentiful in the Democratic Republic of Congo, Nigeria and Sierra Leone. It is an evergreen tree growing to a height of 25-38 m (Orwa *et al*., 2009). The plant flowers between January and May (Alan, 1999). Its timber and nuts are used extensively but the nuts shells are use only as fire wood (Orwa *et al*., 2009).

The nut shell is extremely hard, hence it can be utilize in the production of activated carbon which will compare favorably with the other activated carbon prepared from biomass.

Material and methods

Sample collection and preparation

Sample collection and preparation The raw materials used in this experiment was collected from Ikot Obio Inyang in Etinan L.G.A., Akwa Ibom state. The samples were prepared at the Department of Chemistry, Akwa Ibom state University.

Carbonization and Chemical Activation

Activation of the samples was carried out in a muffle furnace using Zinc chloride ($ZnCl_2$) solution at different impregnation ratios. 25g of the sample was put in a beaker containing 250cm^3 of 1.0M solution of $ZnCl_2$ (ratio 1:10). The content of the beaker was thoroughly mixed and heated until it forms a paste. The paste was then transferred to crucibles.

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The crucible was placed in the oven and fired at 500°C for two hours. The crucible was then removed and cooled in the desiccators .The sample was washed with distilled water and then dried in the oven at 100°c to a constant weight. The final product was kept in an air-tight vail ready for use (Odebunmi and Okeola, 2001).

2.2 Characterization of the Activated Carbon.

Percentage Yield

The yield of the activated carbon was calculated using;

$$\text{Percentage yield} = \frac{M_2}{M_1} \times 100\%$$

Where M_1 = Weight of the sample

M_2 = Weight of the activated carbon

Moisture content

Moisture content of the activating carbon was calculating using

$$\text{Percentage moisture content} = \frac{M_1 - M_2}{M_1} \times 100\%$$

Were M_1 = Weight of the activated carbon before drying in the oven

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: 2g of each activated carbon were put in a crucible and fired at 900°C in a muffle furnace for 3 hour. The resultant ash was removed and allowed to cool in the desiccators the weighed using electronic weighing balance.

Ash content was calculated using:

$$\%\ \text{Ash content} = \frac{W_2}{W_1} \times 100\%$$

Where W_2 = Weight of the ash

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

A batch adsorption test was carried using methylene blue (MB) a cationic basic organic dye. The absorbance MB was measured using a spectronic 20 spectrophotometer at 250nm wave length. Adsorption measurement for each carbon sample was made under equilibrium condition over a period of time of one hour (Mkayula and Matumbo, 1994; Odebunmi and Okeola, 2001).

The adsorbent-adsorbate equilibrium point were generated by contacting different quantities of activated carbon samples (0.1 – 0.5g) with 100ml of MB stock solution containing 250mg/l. the mixture was shaken using a mechanical shaker for one hour, followed by filtration using whatmann filter paper(No1). The post-adsorption concentration of MB was determined spectrophotometrically.

$$\text{Percentage methylene blue adsorbed} = \frac{(C_o - C)}{C_o} \times 100$$

Where:

C_o (mg/L) = Initial concentration of methylene blue

C (mg/L)= Post-adsorption concentration of methylene blue

Result and Discussion

The results of the physical parameters of the active carbon prepared using different impregnation ratios are presented in table 1.

The percentage yield of the activated carbon obtained was 41.94% and 38.76% for 1:10 and 1:20 impregnated activated carbon. These results revealed that the higher impregnation ratios, the lower the percentage yields. This may be due to the reactions that occur between the raw material and activating chemical.

The results of percentage ash and percentage fixed carbon as presented in table 1, show that 1:10 impregnated activated carbon has a ash content of 7% and percentage fixed carbon of 93%, while 1:20 10 impregnated activated carbon has a ash content of 5.5% and percentage fixed carbon of 95.5%. it is expected that 1:20 impregnated activated carbon which has greater percentage fixed carbon should have greater adsorption efficiency (Ogbonnaya, 1992).

Table 1. Physical parameters of the activated carbon

Activating Agent	1:10	1:20
Yield (%)	41.94	38.76
Moisture content (%)	11.20	18.56
Ash content (%)	7.00	5.50
Percentage fixed carbon (%)	93. 00	96.50

Table 2. adsorption of M.B by the 1:10 activated carbon

AC dosage (mg)		Post adsorption concentration (mg/l)	Adsorption Efficiency (%)
100	48.25	80.70	
200		46.44	81.42
300		46.19	81.52
400		44.48	82.21
500		44.6	82.40

Table 3. adsorption of M.B by the 1:20 activated carbon

AC dosage (mg)	Post adsorption Concentration (mg/l)	Adsorption Efficiency (%)
100	35.55	85.78
200	35.04	86.00
300	32.52	87.00
400	29.92	88.03
500	23.67	90.57

Adsorption Study:

The adsorption study was done based on the Methylene blue removal for each activated carbon sample. The results of the methylene blue adsorption by the activated carbons are presented in Table 2 and 3. The adsorption was seen to be directly related with the percentage fixed carbon. Sample with greater percentage fixed carbon exhibit higher adsorption efficiency. The adsorption was also directly related to the impregnation ratios. Samples with higher impregnation ratio, has greater adsorption capacity. The results also revealed that the quantity of the adsorbent has greater effect on the removal 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 will be more probable for solute molecules to be adsorbed on the adsorption site and thus adsorption efficiency is increased (Okeola *et al.*, 2012).

Conclusion

The use of *Coula eludies* seed shells as raw materials for the preparation of activated carbon has been examined and the results showed that activated carbon made from this sample can be use as effective adsorbent for the removal of methylene blue from aqueous solution.

Results also revealed that the adsorption of methylene increases with increase in percentage fixed carbon, impregnation ratio and adsorbent dosages.

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