

Preparation and Characterization of Activated Carbon from *Hura Crepitans Linn* Seed Shells

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ABSTRACT

Activated carbons were thermally prepared from *Hura Crepitans L.* seed shells. Zinc chloride ($ZnCl_2$) and Phosphoric acid (H_3PO_4) 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 0.5g of the activated carbon. The results revealed that the percentage yield and ash content of H_3PO_4 impregnated activated carbon was higher than $ZnCl_2$ impregnated activated carbon. On the other hand $ZnCl_2$ impregnated activated carbon had higher moisture content and percentage fixed carbon. It was also revealed that $ZnCl_2$ impregnated activated carbon had greater adsorption capacity than H_3PO_4 impregnated activated carbon. However it was found that the higher the adsorbent (activated carbon) dosage, the higher the adsorption capacity.

Keywords: *Hura Crepitans L.*, activation, activated carbon, Zinc chloride and Phosphoric acid

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I. 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, research have been reported on 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, palm kernel shells, *Canarium scheginqflurthi* nut shell, corn cob and other seed shells have been reported (Olawale and Ajayi, 2007; Diya *et al.*, 2008; Chang *et al.*, 2001 and Vijayan *et al.*, 2012).

Hura crepitans L., the sand box tree also known as possum-wood, is an evergreen tree of the spurge family Euphorbiaceae. 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 is to prepare activated carbon from *Hura crepitans L.* shells, characterize and apply it in the removal of methylene blue from contaminated water.

II. MATERIAL AND METHODS

2.1 Sample Collection

The seed shells of *Hura 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.

2.2 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. 400g of the sample chips were charred in the furnace at $500^\circ C$ for 2 hours. 30g of the charred sample was weighed into a beaker containing $300cm^3$ of 1M 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 $100^\circ C$ for 12 hours. The crucible was then placed in the furnace and fired at $650^\circ C$ for 2 hours.

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

2.3 Characterization of the Activated Carbon.

• *Percentage Yield*

The yield of the activated carbon was calculated using;

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

$$\begin{aligned} \text{Where } M_1 &= \text{Weight of the charred sample} \\ M_2 &= \text{Weight of the activated carbon} \end{aligned}$$

• *Moisture content*

Moisture content of the activating carbon was calculated using:

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

Where 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 sample was put in a crucible and fired at $900^\circ C$ in a muffle furnace for 3 hour. The resultant ash was removed and allowed to cool in the desiccator.

Ash content was calculated using:

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

$$\begin{aligned} \text{Where } W_2 &= \text{Weight of the ash} \\ W_1 &= \text{Weight of the activated carbon} \end{aligned}$$

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

2.4 Adsorption Study

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

$$\text{Percentage methylene blue adsorbed} = \frac{(C_0 - C)}{C_0} \times 100$$

Where:

C_0 (mg/L) = Initial concentration of methylene blue

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

III. 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.*, 2011).

Result revealed that the percentage yield of H_3PO_4 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 compare favourable with that of H_3PO_4 , $ZnCl_2$ and KOH activated carbon from maize cob (Odebunni and Okeola, 2001) and NaCl activated carbon of *Jatropha curcas* seed coats (Okeola *et al.*, 2011). The ash content obtained for $ZnCl_2$ activated carbon compare favourably with the value obtained Coconut shell (Odebunmi and Okeola, 2001). However, $ZnCl_2$ impregnated activated carbon has 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.

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

3.1 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 *et al.*, 2011).

The results of the methylene blue adsorption by the activated carbons are presented in Table 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).

3.2 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 *et al.*, 2011).

Table 2: Adsorption of Methylene Blue by H_3PO_4 activated Carbon

AC dosage (mg)	Post Adsorption Conc. 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 2: Adsorption of Methylene Blue by ZnCl₂ activated Carbon

AC dosage (mg)	Post Adsorption Conc. 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

IV. CONCLUSION

Hura Crepitans L. seed shells were successfully converted into activated carbon using H₃PO₄ and ZnCl₂ 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 ZnCl₂ impregnated activated carbon was higher than H₃PO₄ impregnated activated carbon. However H₃PO₄ impregnated activated carbon is recommended for used in food and pharmaceutical industries since it is non toxic activating chemical as it does not contain heavy metal while ZnCl₂ impregnated activated carbon can be apply for the treatment of waste water from textile and allied industries.

REFERENCES

- [1]. Ajayi, O.A., and Olawale, A. S. (2009). A comparative study of thermal and chemical activation of *Canarium Schweinfurthii* Nutshell. *J. Appl. sci. Res.*, 5(12): 2148-2152.
- [2]. Cleiton Nunes A., Guerreiro Mario C., (2011). Estimation of surface area and pore volume of activated carbons by methylene blue and iodine numbers, *Quim. Nova*, 34(3) 472-476.
- [3]. Diya udden, B. H., mohammed, I. A. Ahmed, A.S. and Jibril, B. Y. (2008). Production of activated carbon from corncobs and its utilization in crude oil spillage clean up. *Agricultural Engineering International: The CIGR E-Journal*. Vol. x: 1-9.
- [4]. Duff, O.G. and Ross, S.M. (1988). Adsorption on activated charcoal. *J of chem. Educ.* 65(3) 815
- [5]. Ezeh, I.E., Umoren, S.A., Essien, E.E. and Udoh. A.P. (2012). Studies on the utilization of *Hura Crepitans L.* seed oil in the preparation of alkyl resins, industrial crops and products 36:94-99,
- [6]. Itodo, A. U., Abdulrahman, F. W., Hassan. L. G., Maigandi, S. A. and Itodo, H. U. (2010). Application of Methylene blue and iodine Adsorption in the measurement of specific area by four acid and salt treated activated carbons. *New York Science Journal*, 3(5): 25-33.
- [7]. Joshi, S., Adhikari, M., Pokharel, B. P. and Pradhanga, R. R. (2013). Effect of Activating agents on the Activated carbons prepared from Lapsi seed stone. *Research Journal of Chemical Sciences*, 3(5): 19-24.
- [8]. Odeunmi, E. O. and Okeola, O. F. (2001). Preparation and characterization of activated carbon from waste material. *J. Chem. Soc. Nigeria*, 26(2): 149-155.
- [9]. Ogbonnaya, O. (1992). Preparation and comparative characterization of activated carbon from Nigerian sub-bituminous coal, palm kernel and cow bone. *J. Chem. Soc. Nigerian*, 17,11.
- [10]. Okeola, O. F., Odeunmi, E. O. and Ameen, O. M. (2012). Comparison of Sorption capacity and surface area of Activated carbon prepared from *Jatropha curcas* fruit pericarp and seed coat. *Bull. Chem. Soc. Ethiop*, 26(2) 171-180.
- [11]. Olawale, A. S. and Ajayi, O. A. (2009). Thermal activation of *Canarium Schweinfurthii* nutshell. *Australian Journal of Basic and Applied Sciences*, 3(4): 3801-3807.
- [12]. Tsai, W., Chang, C., Wang, Y., Chang C., Chien, F., Sun, H.(2001). Preparation of activated carbons from corn cob catalyzed by potassium salt and subsequent gasification with CO₂. *Bioresource technology* 78(2) ; 203-208
- [13]. Umoren. S.A., umoren, I. U. Johnson A.S.(2013). Metal soaps of *Hura crepitans* seed oil: potential stabilizer for unplasticised polyvinyl chloride against thermal degradation. *Journal of chemical Acta*, 2:92-94
- [14]. Vijayan, S. N., Makesh Kumar, M. and Sridhar, K. (2012). Physical and chemical Analysis of Activated carbon prepared from coconut shell charcoal and usage-A case study. *International Journal of Advanced Scientific Research and Technology*, 2(3): 168-175.