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Production, characterization and activity test of activated carbon from Moringa seed husks for dyes removal

Eyitayo A. Afolabi, U.G, Akpan, I. G, Babalola and C.J Ibeh

Department of Chemical Engineering, Federal University of Technology, Minna.

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ABSTRACT

In this study, activated carbon was prepared from moringa seed husk by chemical activation method using of potassium hydroxide (KOH) impregnation. The activated carbon produced was characterized and tested for the removal of two different dyes from wastewater. The Activity tests were carried out for five different masses of activated carbon and three contact times in order to investigate the effect of mass of activated carbon and contact time respectively. The experimental results showed that an increase in the mass of the activated carbon produced leads to a higher percentage removal of dye from wastewater.

Introduction

Activated carbon is a form of carbon produced from carbonaceous materials such as nutshells, coconut husk, wood, lignite, coal and petroleum pitch to create millions of tiny pores between the carbon atoms. This increase in the surface area of the substance from about 500 to 1500m2/g, or 300-2,000 square meters per gram makes activated carbon a suitable material for adsorption process. Activated carbon possesses some capacity to remove heavy metals, organic compounds, bacterial and viral pathogens, pesticides and algal toxins from drinking water. In medicine, activated charcoal is used to treat poisonings, reduce intestinal gas (flatulence), lower cholesterol levels, prevent hangover, and treat bile flow problems (cholestasis) during pregnancy [1]. The impure molecules removed are held within the carbon's internal pore structure by electrostatic attraction or chemisorption.

In industries such as textiles, rubber, paper, plastic, leather, cosmetic, coloured organic effluents are produced and when discharged directly or indirectly into water bodies even in small quantity cause environmental damage to surface water [2]. Dyes generally are synthetic in nature with complex aromatic molecular structures and this makes them to be chemically stable and difficult to biodegrade. As in [3], water polluted with dye was observed to have caused allergic dermatitis, skin irritation and sometimes found to be carcinogenic and mutagenic. The global water source is increasingly contaminated with series of pollutants and the poor masses are turning to low cost household water treatment such as boiling and the usage of relatively cheap chemicals known as coagulants. Unfortunately, the cost associated with manufacturing or more often importation of water treatment chemicals tend to push the cost of processed water to an unaffordable level to ordinary citizen in Nigeria. Therefore, there is an urgent need for the provision of safe water at affordable cost.

Recently, the uses of activated carbon produced from agricultural wastes to replace conventional coagulants such as aluminium salts in both domestic and industrial scale water treatment are considered as an effective and efficient method of removing undesirable chemicals from contaminated water [4]. Adsorption technique for waste water treatment has become more popular in recent years due to its simplicity and the availability of a wide range of adsorbents. For example, as in [3] adsorption was observed to be an effective and efficient process for removing non-biodegradable pollutants (including dye) from waste water. Many researchers have demonstrated the potential in some agro-forestry wastes such as macadamia (macadamia integrifolia), nutshells, Jatropha carcas cakes, maize (Zea mays) cobs, baobab (adansonia digitata) husks, marula (Scerocaryabirrea) stones and rice (oryza sativa) husk, as raw materials for producing activated carbon [5 6 7; 8].

The production of activated carbon from agricultural by products has potential for positive economic and environmental impacts. Firstly, it converts unwanted, lower value agricultural waste to useful, high value adsorbents. Secondly, activated carbon are increasingly used in water treatment plant to remove organic chemicals and metals of environmental and (or) economic concern. Thirdly, it reduces the importation of activated carbon, thereby increasing the economic base of the country [9; 10]. The objectives of this study are three folds; firstly, to prepare activated carbon from moringa oleifera seed husks using chemical activation. Secondly, to investigate the physical characteristics of the prepared activated carbon. And lastly, to use the produced activated carbon to remove methylene dye and local dye from simulated wastewater.

Experimental Procedures

Materials

The reagents and equipments used to produce activated carbon from moringa seed husk are presented in Tables 1 and 2 respectively.

Table 1: List of Reagent used

S/ No	Reagent	
1	Distilled water	
2	Potassium Hydroxide	

Sample Collection and Pre-treatment

The Moringa Oleifera seed husk was collected from local processors at Maitumbi Market in Minna, Nigeria. The sample was thoroughly washed with distilled water to remove all dirt and sun-dried for seven days.

Tele:

E-mail addresses: elizamos2001@yahoo.com

Table 2: List of Apparatus and Equipments

S/No	Equipment/Apparatus used	Specification
1	Electric muffle furnace	Gallenkamp, England
2	Digital weighing balance	Adventurer, England
3	Mortal and Pestle	Wooden
4	pH meter	Genway 3510
5	Electric oven	Gallenkamp, England
6	Measuring cylinder	Glassware
7	Tray dryer	Steel
8	Beaker	Glassware
9	Crucible	Ceramics
10	Desiccator	Pyrex, England
11	Water bath electric shaker	Labtech
12	Ultra violet spectrophotometer	Model 210 VGP
13	Conical flask	Pyrex
14	Filter paper	Whatman

Carbonization Procedure

1.232 kg of the dried raw material was weighed into a crucible and was pyrolized in an electric muffle furnace at 450 ^{0}C for 25 min after which the carbonized moringa husk was allowed to cool to room temperature. The yield on pyrolysis was determined from the weight before (W_{bc}) and after carbonization (W_{ac}). The percentage yield is given in Equation 1.

$$\% \text{ yield} = \frac{W_{ac}}{W_{bc}} \times 100 \quad (1)$$

Activation/ Impregnation Procedure

The carbonized sample was divided into three parts of 100 g each and impregnated into 500 ml of the prepared standard solution and stirred properly to form a paste. Three different standard KOH solutions of molar concentration of 0.25 M, 0.5 M and 1.0 M were used as the activating agent for each carbonized sample. Thereafter, it was covered and allowed to soak for 24 h so as to allow the char to become activated. The resulting mixture was filtered and the residue thoroughly washed with distilled water until it has a pH close to 7 after which it was oven dried at 60 ^{o}C for 24 h to remove moisture. The impregnated ratio is given in Equation 2.

$$\text{Impregnation Ratio} = \frac{Mass\ of\ he\ char\ (Carbon)}{Activating\ agent\ (KOH) in\ ml} \quad (2)$$

Characterization of the Produced Activated Carbon

Moisture Content

3 g of the carbonized sample was placed in a clean, dry and pre weighted petri-dish and dried in an oven at 105 ^{o}C for 90 min. The sample was then cooled in a dessicator for 30 min and the weight of the sample before and after heating was determined. The percentage moisture was determined using Equation 3;

$$\%\text{Moisture} = \frac{Weight\ of\ sample\ before\ drying - weight\ of\ sample\ after\ dying}{Weight\ of\ sample\ before\ drying} \times 100 \quad (3)$$

Ash content

2 g of moringa husk was weighed in a dry, pre-weighed crucible and placed in the furnace and was heated up to 550 ^{o}C for 1 h. The sample was removed and allowed to cool in a dessicator before it was weighed. The percentage ash content is obtained using Equation 4;

$$\% \text{ Ash content} = \frac{Weight\ of\ ash}{Weight\ of\ dry\ sample} \times 100 \quad (4)$$

Volatile Matter Content

5 g of activated carbon was weighed in a clean pre-weighed closed crucible and placed in the furnace at a temperature of 450 ^{o}C for 10 min. The sample was retrieved and left to cool in a desiccator. The weight of the sample before and after heating was used to determine the amount of volatile matter present in the sample as shown in equation 5;

$$\%\text{ Volatile content} = \frac{Weight\ of\ sample\ before\ drying - weight\ of\ sample\ after\ dying}{Weight\ of\ sample\ before\ drying} \times 100 \quad (5)$$

Fixed Carbon Content

The fixed carbon content was determined using Equation 6.

$$\text{Fixed Carbon Content} = \{100 - (\text{Ash content} + \text{Volatile matter content} + \text{Moisture content})\}\ \% \quad (6)$$

Burn- off

10 g of moringa husk was weighed in a clean pre-weighed crucible and placed in a furnace at 450 ^{o}C for 20 min. The mass of the sample was measured after carbonization to obtain the weight loss on carbonization. The percentage burn off is given by Equation 7;

$$\%\text{ Burn off} = \frac{Weight\ of\ husk\ before\ carboniztion - weight\ of\ sample\ after\ carboniztion}{Weight\ of\ sample\ before\ drying} \times 100 \quad (7)$$

pH Measurement

A pH electrode was calibrated with pH of 4 and immersed into the washed samples and the pH values were read from the meter.

Samples with undesired pH were re washed until a pH between 7.1 and 8.0 was obtained.

Pore Volume

10 g of activated carbon was soaked in 100 ml of distilled water and boiled for 20 minutes to displace the air contained in the pores. The sample was filtered, left to dry and the final weight obtained using equation 8:

$$\text{Pore Volume} = \frac{Final\ weight\ of\ activated\ carbon - intial\ weight\ of\ activated\ carbon}{Density\ of\ water} \quad (8)$$

Bulk Density

10 ml beaker was weighed and tightly packed with activated carbon. The weight of the beaker with the sample was measured and the bulk density determined using equation 9:

$$\text{Bulk density} = \frac{Mass\ of\ activated\ carbon}{Volume\ occupied\ (10\ ml)} \quad (9)$$

Porosity

The porosity of the sample is given by equation 10;

$$\text{Porosity} = \text{Bulk density} \times \text{pore volume} \quad (10)$$

Treatment of the Wastewater

Preparation of Aqueous Solution

Methylene blue and local dye are the two types of dye used in the preparation of wastewater used in this study upon which the activity test of the activated carbon was established. A simulated wastewater containing 0.0005 M concentration of methylene blue dye was prepared in the laboratory. The initial concentration of the dye present in the wastewater was determined using a 210 VGP UV-Spectrophotometer.

Adsorption Experiments

The adsorption experiments were carried out at room temperature. The effect of the mass of the adsorbent was investigated with 1 g, 2 g, 3 g, 4 g, and 5 g of the sample activated with 0.25 M KOH. The required weight of activated carbon was placed in 100 ml of the dye solution in a conical flask. These conical flasks were shaken with a water bath electric shaker at agitation speed of 200 rpm.

The mixture was filtered with Whatman 125 mm filter paper. The concentration of the filtrate before and after adsorption was obtained by Spectrophotometer measurement at a wavelength of 670 nm.

The effect of contact time in the adsorption process was also investigated with 5 g of sample activated with 0.25 M KOH. 100 ml of methylene blue solution was added to each of the flasks and the process time was varied from 15 to 45 min. The same procedure was repeated for sample activated with 0.5 M and 1 M of KOH, and for the wastewater polluted by local dye.

Separation Technique

The adsorbent was separated from the solution by filtration using a Whatman 125 mm filter paper. 20 ml of the filtrate was subjected to UV- Spectrophotometer analysis while the remaining discarded.

Analysis of Data

Data were analysed by calculating the percentage removal of dye and the amount adsorbed using equation 11;

Percentage Removal =

$$\frac{\textit{Initial concentration of the sample} - \textit{Final concentration of the sample}}{\textit{Initial concentration of the sample}} \qquad (11)$$

Results and Discussion

The results of the proximate analysis and carbonization of the moringa husks at 450 ^{o}C are presented in Tables 3 and 4 respectively. Table 5 presents the characterization of the activated carbon produced from moringa seed husk with different concentration of KOH. The effect of mass of activated carbon produced from moringa seed husk and contact time on the adsorption of methylene blue and local dyes are presented in Figures 1 to 3.

Table: 3 Proximate Analysis of Moringa Seed Husk

Parameter	Composition (%)
Ash content	3.8
Moisture content	4.5
Volatile matter content	10.0
Fixed carbon content	81.7

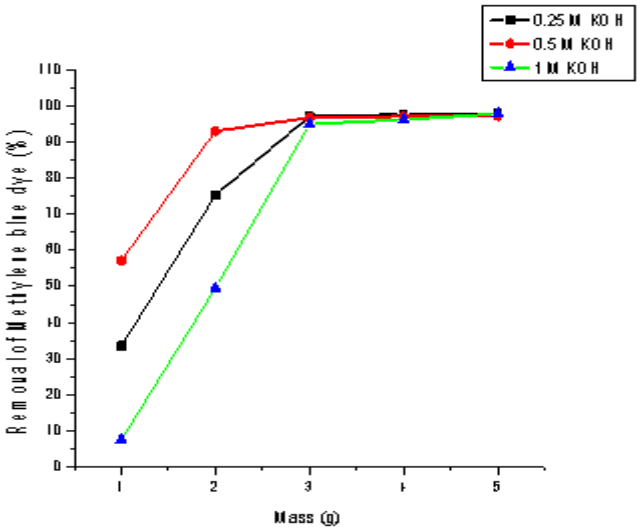

Figure 1: Effects of the mass of activated carbon on the percentage removal of methylene blue dye

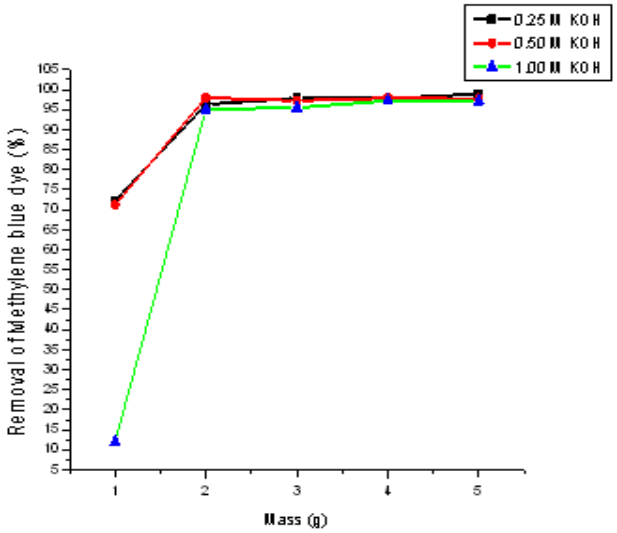

Figure 2: Effects of mass of the activated carbon on the percentage removal of local dye

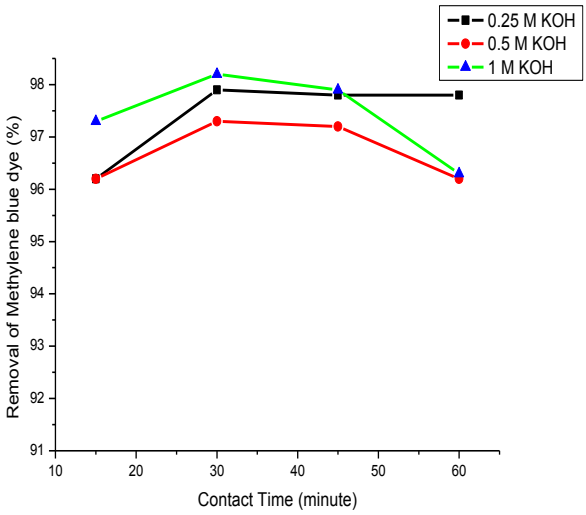

Figure 3: Effects of contact time on the percentage removal of methylene blue dye

Discussion of Results

Activated carbon from moringa seed husk was produced, characterized and activity tested using water polluted with dye. As shown in Table 3, the proximate analysis indicates that moisture content; volatile content, ash content and fixed carbon content were 4.5 %, 10 %, 3.8 % and 81.7 % respectively. The high carbon content and low ash content as shown in Table 3 are the most desirable properties that make moringa seed husk a suitable precursor for the preparation of high grade activated carbon. This is in the agreement with the results obtained in works of [11; 12; 13]. Table 4 shows the percentage yield of moringa seed husk after carbonization to be 24.19%. This percentage is considered relatively fair as moringa seed husk is light and a drastic loss in the weight of the husk is expected after carbonization. The characterization of activated carbon produced from moringa seed husks as shown in Table 5 revealed that an increase in the concentration of activating agent leads to an increase in porosity and surface area of the activated carbon. This is in agreement with the work of [13; 14], who observed that an increased in porosity and surface area is directly proportional to the adsorption property of activated carbon.

Figure 1 shows that an increase in the mass of the activated carbon produced from moringa seed husk resulted in an increase in its percentage adsorption capacity.

Table 4: Results for the Carbonization of Moringa seed husk at 450 ^{o}C

Carbonization time (min.)	Mass before carbonization (g)	Mass after carbonization (g)	% Yield	%Burn off	Bulk density (g/m^3)
25.0	1232	298	24.19	69.67	0.44

Table 5: Characterization of Activated carbon produced from moringa seed husks activated with different concentrations of KOH

Parameter	Activated carbon activated with 0.25M of KOH	Activated carbon activated with 0.5M of KOH	Activated carbon activated with 1M of KOH
Percentage yield (%)	24.19	24.19	24.19
Ash content (%)	3.8	4.0	3.9
Volatile matter content (%)	10.00	10.40	9.80
Moisture content (%)	4.50	4.40	4.50
Fixed carbon (%)	81.70	81.20	81.80
pH	7.46	7.38	7.44
Burn off (%)	69.67	69.0	71.10
Pore volume (cm^3/g)	5.29	6.63	6.44
Bulk density (g/cm^3)	0.73	0.70	0.71
Porosity	3.86	4.64	4.57

For example, the percentage removal of methylene blue dye increases from 33.5 to 97.8 % with an increase in the mass of activated carbon from 1 g/100 ml to 5 g/100 ml at 45 min for 0.25 M KOH. In the same manner, an increase in the mass of activated carbon from 1 g/100 ml to 5 g/100 ml at 45 min for 0.5 M and 1 M KOH leads to an increased in the percentage removal of methylene blue dye from 57.2 % to 97.2 % and 7.5 % to 97.9 % respectively.

This is expected as an increased in availability of surface active sites is associated with increased in the mass of the activated carbon. The same trend is observed in Figure 2 where an increased in mass of the adsorbent activated by 0.25 M, 0.5 M and 1 M KOH increases the percentage removal of local dye from polluted water.

As shown in Figure 1, maximum removal of 97.85%, 97.2% and 97.9% were obtained at 5g/100 ml of activated carbon activated with 0.25M, 0.5 M and 1M KOH respectively. Although, the maximum percentage removal of dye was at 5 g/100 ml activated carbon, the optimum mass of adsorbent activated with different concentrations of KOH is 3 g. The reason for optimizing the operating conditions was to maximize profit and minimize cost of production. In the same manner, the optimum mass of adsorbent activated by 0.25M, 0.5M and 1M KOH as observed in Figure 2 is 2 g/100 ml for the different concentration of KOH.

Figure 3 shows the variation of the percentage removal of dye with contact time of the sample with the activating agent. The percentage removal of dye rapidly increased with contact time from 15 to 30 min for the sample activated with 0.25 M KOH. Thereafter, at 30 min it becomes steady and reached equilibrium for the remaining period of time. The same trend is observed for the first 45 min with the sample activated with 0.5 M and 1 M KOH. However, after 45 min the percentage removal of dye begins to rapidly decrease. This means, the percentage removal of dye was higher at the initial stage due to availability of more surface area that positively influences the adsorption process. However, as contact time increased the percentage removal decreases. This is due to the active sites that becomes saturated and could no longer adsorb the dye released back into the solution and desorption occurs [15; 16]. In general, Figure 3 showed that the optimum contact time for samples activated with 0.25 M, 0.5 M and 1M KOH is 30, 30 and 45 min respectively. That is, at these contact times highest percentage removals of dye is observed.

Conclusions

The following conclusion can be drawn from this work;

1. The activated carbon produced from moringa seed husks was found to possess high surface area, pore volume and adsorptive properties. Therefore, moringa seed husks which are an agricultural waste can be used as a suitable precursor for the preparation of high grade activated carbon.
2. An increase in the mass of the activated carbon produced from moringa seed husk employed for the removal of dye from wastewater resulted in an increase in its percentage adsorption capacity. 3 g/100 ml of activated carbon was observed as the optimum mass of adsorbent activated by 0.25 M, 0.5 M and 1 M KOH for the removal of methylene dye from waste water. However, for the removal of local dye from wastewater 2 g/100 ml is the optimum mass of the adsorbent.
3. The percentage removal of dye is higher at the initial stage due to availability of more surface area and as contact time increases the percentage removal decreases. The optimum contact time for samples activated with 0.25 M, 0.5 M and 1M KOH is observed to be 30, 30 and 45 min respectively.
4. Contact time is observed to have more effect on the removal of dye from wastewater than mass of the activated carbon.
5. Finally, activated carbon prepared from moringa seed husks could be employed as a low cost alternative for the removal of dye from wastewater.

References:

1. Olafadehan, O. A., Jinadu, O.W., Salami, L. and Popola, O. T. (2012). Treatment of brewery wastewater effluent using activated carbon prepared from coconut shell. International Journal of Applied Sciences and Technology, 2(1), 165-178.
2. Crini, G and Badot, P.M (2008), Application of chitosan, a natural aminopolysacchride, for dye removal from aqueous solutions by adsorption process using batch studies: A review of recent literature, Progress Polymer Science, 33, 399-447.
3. Aksu, Z and Tezer, S (2005), Bio sorption of reactive dye on the green alga Chlorella Vulgaris, Process Biochemistry, 40, 1347-1361.
4. Pradhan, Subhashree (2011), Production and characterization of Activated Carbon produced from a suitable Industrial sludge, B. Tech thesis, Department of Chemical Engineering, National Institute of Technology, Rourkela, India.
5. Ahmedna, M., Marshall, W.E. and Rao, R. M. (2000). Production of granular activated carbons from select agricultural

by-products and evaluation of their physical, chemical and adsorption properties. Bioresour. Technol. 71, 113–123.
6. Girgis, B.S; Yunis, S.S and Soliman S (2000), Characterization of activated carbon from pecan hulls in relation to conditions of preparation, Matter. 57, pp 16-172.
7. Hapazari, I; Ntuli, V. And Parwira, W (2011) valuation of single –step steam pyrolysis- activated carbons from Lesotho Agro-Forestry Residues, Journal of Science, 13(4), pp100-121.
8. Martinez,M.L., Moiraghi,L., Agnese, M and Guzman, C (2003) Making and some properties of activated carbon produced from Agricultural industrial residues from Argentina, The Journal of the Argentine Chemical Society Vol. 91, No 4/6 103-108.
9. Abia A.A; Horsfall. M and Didi, O (2003), The use of chemically modified cassava waste for the removal of Cd^{2+}, Cu^{2+} and Zn^{2+} ions from aqueous solution, Journal of Bioresourse Technology, 90, 345-348.
10. Misihairabgwi, J; Ntuli, V; Kasiyamburu, A; Zinyowere,S; Ncube, I and Chipofya, V (2007), Application of locally produced activated carbons in water treatment, Deparment of Biochemistry, University of Zimbabwe, Zimbabwe. Retrieved from http:www.bscw.ihe.nl/pub/bscw.cgi/d2607370/misihairabgwi on 19th August 2012.
11. Mc Connachie, G.L; Warhurst, A.M and Pollrd, S.J (1996), Activated carbon from Moringa husks and pods. In reaching the unreached: Challenges for the 21st Century, Proceeding of the twenty second WEDC Conference, New Delhi, India, pp. 279-282.
12. Lua, A and Guo, J (2001), Textural and Chemical properties of adsorbent prepared from Palm shell by Phosphoric Acid activation , Material of Chemistry Physic 80: 114-119.
13. Bhatnagar, A. and Minocha, A.K. (2006), Convectional and non convectional adsorbents for the removal of pollutants from water, Journal of Chemical Technology 13(5), pp 203-208.
14. Amin, N.K. (2008), Removal of reactive dye from aqueous solutions by adsorption onto activated carbons prepared from sugarcane bagasse pith. In the European Desalination Society nd center for Research and Technology Hellas, Proceedings of a conference held at Sani Resort, Halkidiki, Greece, April -25, 2007, pp 152-161.
15. Amri, N.B (2008), Preparation of Activated Carbons from Waste Tyres Char impregnated with Potassium Hydroxide and Carbon dioxide Gasification. Published M. Sc. Thesis, Universiti Sains Malaysia pp 5-17
16. Bibek, D (2010), Competitive Adsorption of dyes (Congo red, methylene blue and Malachite green) on Activated Carbon. Published B. Tech. Thesis, Department of Chemical Engineering, National Institute of Technology, Rourkela Orissa, India, pp 19-21.