

Production and Characterization of Activated Carbon (AC) From Groundnut Shell and Its Application in Water Treatment

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Abstract

Activated carbon was prepared from groundnut shell which has been considered as agricultural waste, through chemical activation using Phosphoric acid (H_3PO_4) and subsequently applied for the removal of excess iron and manganese in raw water using a Batch adsorption experiment. Scanning Electron Microscopy (SEM) technique was employed to observe the surface morphology of the groundnut shell-derived activated carbon. Fourier Transform Infrared Spectroscopy (FT-IR) was carried out in order to identify the functional groups present on the activated carbon. Groundnut shell activated carbon was evaluated as an adsorbent for the removal of Iron (Fe) and Manganese (Mn) from Alau Dam raw water solution. The Alau Dam water has high amount of iron and manganese. The pH of the produced activated carbon was found to be 6.8, volatile matter 11%, carbon content 68%, moisture content 15% and the surface area $975m^2/g$. The maximum sorption efficiency of Fe and Mn was found to be 90% and 84% respectively. Sorption was most efficient at adsorbent dose of 3g and at contact time of 50-60 minutes. The data was tested by Freundlich and Langmuir isotherm equations and it was found that the Langmuir isotherm is fitted better to the experimental data than Freundlich model.

Keywords: Activated Carbon, Adsorbent, Alau Dam, Iron, Manganese

1.0 Introduction

Iron and manganese are common metallic elements that occurs together naturally especially in deeper wells with little or no oxygen present. Natural sources of iron and manganese may include weathering of iron and manganese bearing minerals like amphibole, iron sulphide and iron rich minerals. In areas where ground water flows through an organic rich soil, iron and manganese will also dissolve in the ground water. Iron and manganese can also have anthropogenic sources including industrial effluents, landfill leakages and acid mine drainage. Well casing, pump parts, piping and storage tank can also contribute iron and manganese to water (Siabi, 2003). Remediation of contaminated ground water has been practiced using activated carbon adsorption. The removal of organics in water that are weakly adsorbed and present in trace concentration require an activated carbon with a predominance of high energy pores. Activated carbons are processed carbon materials that are capable of adsorbing various substances from gas and liquid streams, because of their highly developed pore structure and large internal specific surface areas (Abdul and Aberuagba, 2005).

Generally, the raw materials for the production of AC are those with high carbon but low inorganic contents such as wood, lignite, peat and coal. Besides that, lots of agricultural waste and byproducts have successfully been converted to activated carbon (AC) for examples macadamia nutshell, coconut shells, wood, cotton stalk, almond shells, rice husk,

date pits (Fierro *et al.*, 2010). In this research, a local agricultural waste which is groundnut shells will be used to produce an activated carbon using chemical activation and will subsequently be applied to remove iron and manganese in raw water.

2.0 Materials and Methods

2.1 Adsorbate Sample

Raw water samples were taken from *Alau Dam* and iron and manganese content of the sampled raw water were measured using Automatic Smart Spectrophotometer (ASS) and it shows that, the sample has high concentration of iron (3.11 mg/l) and manganese (1.32 mg/l) which is in agreement with the findings of Dammo and Sangodoyin (2014) and that of Iduwo *et al.*, (2004) respectively. Hence, the raw water from *Alau Dam* was used as an adsorbate sample in this research. The impregnating agent used for the chemical activation of groundnut shells was H_3PO_4 .

2.2 Characterization of AC

Activated carbon was prepared and various properties of the prepared activated carbon were characterized by following the standard procedures: The moisture content was found by oven-drying test method (ASTM D2867 - 09). The percentage of volatile matter of the AC sample was determined by the standard method (ASTM D5832 - 98). To determine the ash content (ASTM D2866 - 94) method was followed. The pH, Electrical conductivity and Bulk density were determined as described by Ahmedna *et al.*, (2000). The surface morphological changes of activated carbon samples were investigated using a Scanning Electron Microscope (SEM) operated at 25kV. The sample was then analyzed using Shimadzu 8300 spectrometer and the spectrum was recorded in a spectral range (FTIR). The specific surface area of activated carbon was estimated using Sear method. The yield and the adsorption capacity were also calculated using equation 2.1 and 2.2 respectively.

$$\text{Yield (\%)} = \frac{\text{weight of AC after carbonization}}{\text{weight of raw material}} \times 100 \text{----- 2.1 (Malik et al., 2006)}$$

$$\text{Adsorption capacity } Q_t = \frac{(C_i - C_t)V}{W} \text{----- 2.2 (Hameed, 2009)}$$

Q_t = adsorption capacity at time t (mg/g).

C_i = concentration of metal before interaction with the activated carbon (mg/l).

C_t = concentration of metal after interaction with the activated carbon (mg/l).

V = volume of the effluent (l).

W = weight of the activated carbon (g). (Hameed, 2009)

2.3. Adsorption Process

2.3.1. Adsorption Experiment Based on Adsorbent Dose

Hundred milliliter of the adsorbate solution was measured and contacted with varied masses (1, 2, 3, 4, 5, 6, 7 and 8g) of the adsorbent for an equilibrium adsorption time of 1hr at room temperature followed by intermittent stirring. The solution was filtered and analyzed with Automatic Smart Spectrophotometer (ASS) analysis.

2.3.2. Adsorption Experiment Based on Contact Time

The effect of contact time on adsorption was studied. 3g of AC was added to water sample at pH 6.8. The mixture was shaken at room Temperature. Aliquots of the clear solution were pipette out at different time intervals until equilibrium was reached.

2.3.3. Adsorption Isotherm Study

The experimental data were used with the linearized equations of the two parameters model Langmuir and Freundlich as given in Equations 3.3 and 3.4 respectively to obtain the isotherm parameters.

$$\frac{1}{Q_e} = \frac{1}{Q_o} + \frac{1}{bQ_o C_e} \text{ ----- 2.3}$$

$$\log Q_e = \log K + \frac{1}{n} \log C_e \text{ ----- 2.4}$$

3.0. Results and Discussion

3.1. Characterization of the Activated Carbon Produced

Adsorbent was characterized for its proximate and ultimate analysis (Table 3.1).

Table: 3.1. Characterization of the groundnut shell AC

S/No	Parameters	Results
1	pH	6.8
2	Moisture content (%)	15
3	Volatile matter (%)	11
4	Ash content (%)	6
5	Carbon content (%)	68
6	Bulk density (g/L)	420
7	Electrical conductivity (μS/cm)	824
8	Surface area (m ² /g)	975
9	Adsorption capacity of Mn and Fe (mg/g)	55.5 and 87
10	Percentage adsorbed of Mn and Fe (%)	84 and 90

From the proximate analysis, it was observed that moisture, ash and volatile matter % was high which may be due to its plant origin (Malik *et al.*, 2006). Surface area is very high for groundnut shell based adsorbent which shows its high adsorption capacity. The pH of the produced activated carbon was found to be 6.8, which falls within the range of pH of most agricultural by products. The density of a generated activated carbon plays a great role on adsorbate uptake. The bulk density was found to be 420 g/l. Generally, higher density carbons hold more adsorbate per unit volume.

3.2. Scanning Electron Microscopy of the Activated Carbon



Fig. 3.1. SEM Micrograph of Groundnut Shell before and after Activation

To study the effect of activation on porosity Scanning Electron Microscopy (SEM) technique was employed to observe the surface physical morphology of the groundnut shell-derived activated carbon. Figure 3.1 show the SEM micrographs of the raw material before and after chemical activation. SEM micrograph of char (before activation) shows the presence of some flaky structure and rudimentary pores but is not rich in porous cavity while SEM micrograph of activated carbon shows the presence of wide pores due to chemical activation with acid which is highly corrosive and dehydrating in nature.

3.3. FTIR Spectrum of Activated Carbon

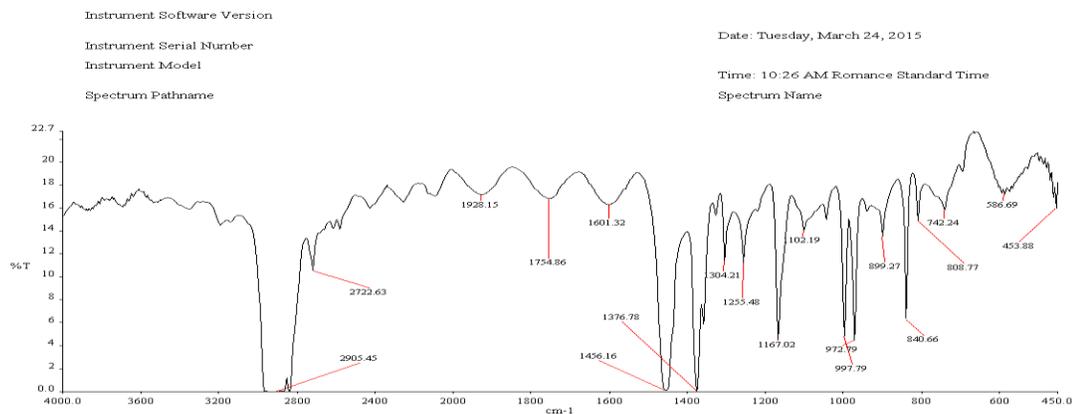


Fig. 3.2. Result of FTIR Spectrum of Activated Carbon

Fourier Transform Infrared Spectroscopy (FTIR) was carried out in order to identify the functional groups present in activated carbon. Functional groups of adsorbents not only affect the adsorption behaviour, but also dominate the adsorption mechanism (Zheng *et al.*, 2014). The peaks appearing in the FTIR spectrum were assigned to various functional groups according to their respective wave numbers. Figure 3.2 shows the FTIR spectrum for the activated carbon produced, spectra peaks representing phenols, carboxyl and

carbonyl were observed in 2905.45 cm⁻¹, 2722.63 – 1456.16 cm⁻¹, and 1376.78 – 997.79 cm⁻¹, respectively.

3.4. Effect of Adsorbent Dose and Contact Time on Adsorption

The effect of different amounts of adsorbent on the removal of Fe and Mn was considered. Experiments were conducted using different adsorbent concentrations viz., 0.5, 1, 2, 3, 4, 5, 6, 7 and 8g. While, the effect of contact time is an important parameter in determining the equilibrium time that should be allowed for the maximum uptake of an adsorptive by a given adsorbent.

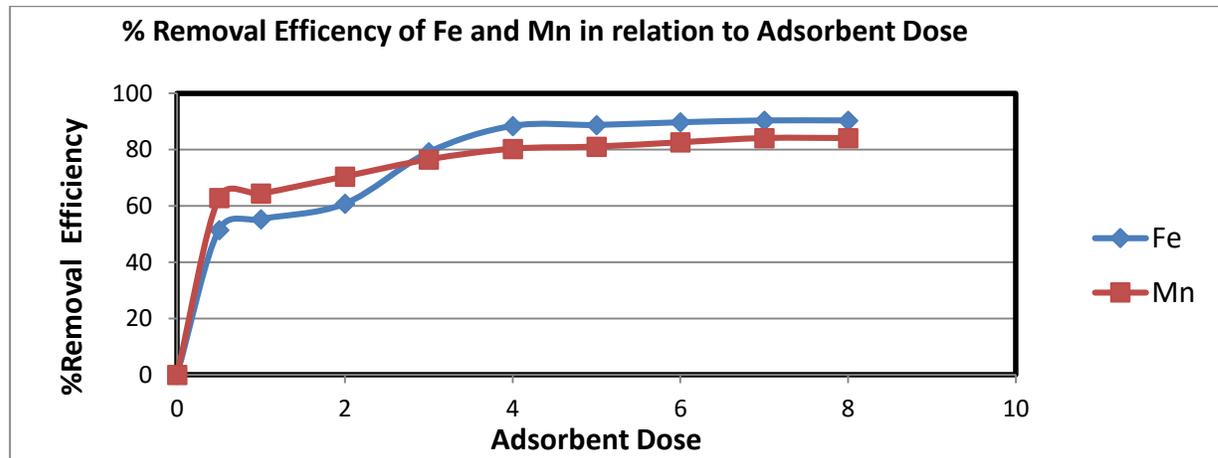


Fig.3.3. Efficiency of Iron (Fe) and Manganese (Mn) Removal in relation to Adsorbent Dose

Figure 3.3 shows that, the percent adsorption of iron (Fe) and Manganese (Mn) increased from 51.45 to 90.35% and 62.88 to 84.09% respectively, as the adsorbent dose was increased from 0.5 to 8g. Although, percent adsorption increases with increase in adsorbent dose but amount adsorbed per unit mass decreases. This statement can be supported from the results, that the trend in percent adsorption shows very sharp increase from 0.0 to 79.09% in the case Fe and 76.52% in the case of Mn, at the dose increments from 0.5 to 3g but on further increment of the dose the percent efficiency increased slowly. So, it's apparent that increasing the adsorbent dose increases adsorption but the amount adsorbed per unit mass decreases.

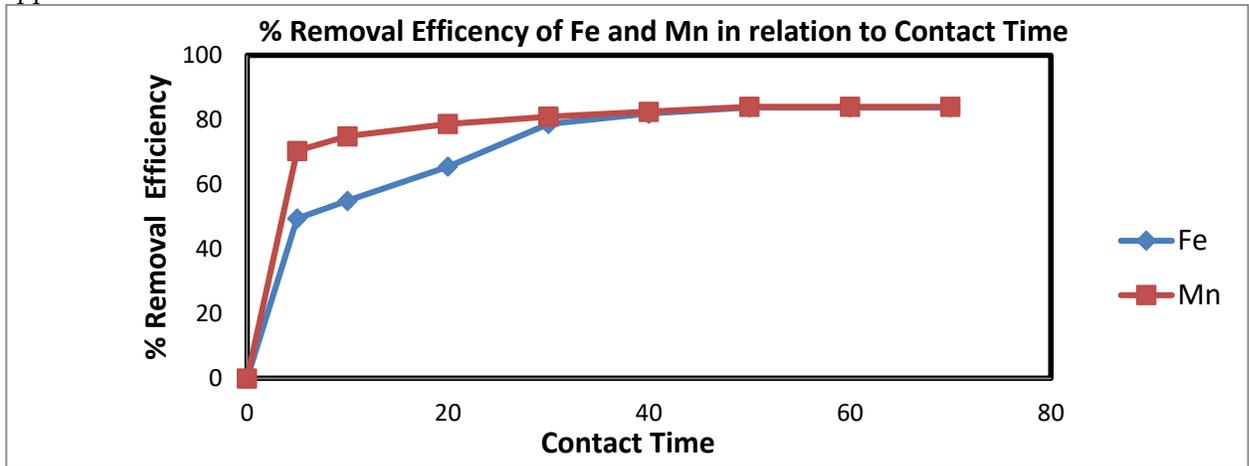


Fig. 3.4 Percent Removal Efficiency of Fe and Mn Based on Contact Time

The percent efficiency of iron and manganese removal based on contact time is shown on Figure 3.4. It can be seen on the figure that about 83.92% removal of iron was achieved at about one hour. This percentage was not increased from 50 - 70 minutes, despite the increase in time signifying the attainment of equilibrium point. So increasing contact time after reaching equilibrium may be time wasting. In batch adsorption, monolayer of adsorbate is normally formed on the surface of adsorbent and the rate of removal of adsorbate species from aqueous solution is controlled by the rate of transport of the species to the empty sites (Elaigwu *et al.*, 2010).

3.5 Adsorption Isotherm Curves

The adsorption isotherms data were fitted to both the Freundlich and Langmuir isotherm equations.

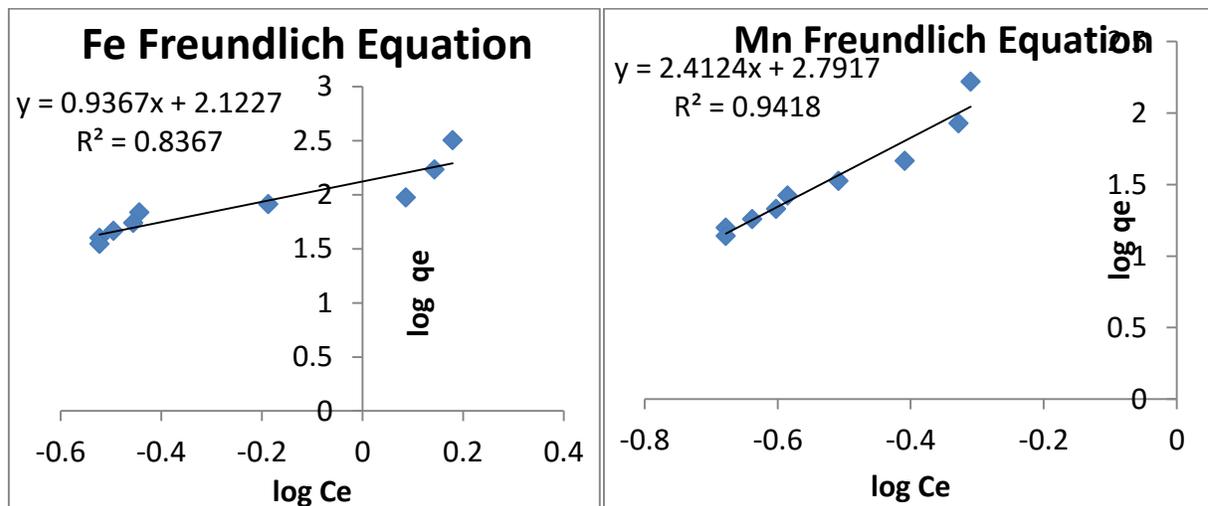


Fig. 3.5 Application of Freundlich Equation for the adsorption of Fe and Mn

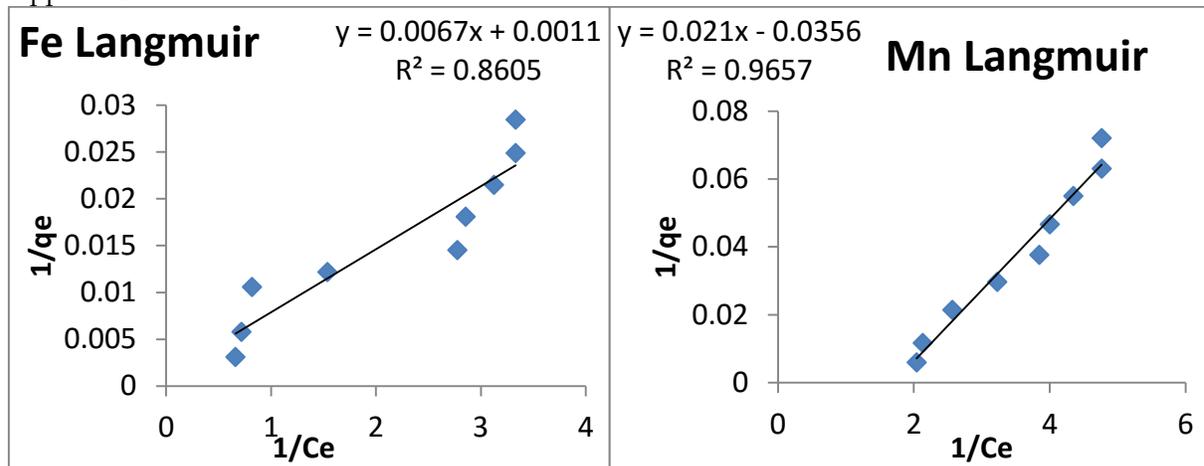


Fig. 3.6 Application of Langmuir Equation for the Adsorption of Fe and Mn

Isotherm data were analyzed using Langmuir and Freundlich adsorption equations. The results show that adsorption process could be described well with both Freundlich and Langmuir isotherms. The experimental equilibrium data fitted well to Langmuir equations with correlation coefficient values greater than 0.9 as reported in a related work (Kehinde *et al.*, 2008). Although both isotherms describe the adsorption process but Langmuir isotherm, for each sample of adsorbate fitted better with higher correlation coefficient recorded for each sample. The Langmuir model was also found to give a better fit in the adsorption of phenol by water hyacinth ash (Uddin *et al.*, 2007).

4.0 Conclusion

Removal of Iron (Fe) and Manganese (Mn) from the solution using activated carbon prepared from groundnut shell has been achieved. The physiochemical parameters of the AC produced are very well comparable with those prepared from similar agricultural wastes reported in the literature. This is an indication that Groundnut Shell could be an excellent precursor for preparation of AC for iron and manganese uptake in water. The data obtained show that the metal ion uptake by the activated carbon increased with increasing both adsorbent dose and contact time, with equilibrium being established in about one hour. Results from the analysis indicated that, contact time and initial adsorbent concentration (AC Dose) have some significant effect on the removal of iron and manganese. The adsorption isotherm was studied and the equilibrium adsorption data fitted to the two models, but gave a better fit to the Langmuir model, as was evidenced from the higher value of R^2 of 0.941 and 0.965 respectively.

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