Chemical Science International Journal

30(12): 39-50, 2021; Article no.CSIJ.52104
ISSN: 2456-706X
(Past name: American Chemical Science Journal, Past ISSN: 2249-0205)

Comparative Study of the Adsorption of Lead and Cadmium by Activated Carbon from *Jatropha curcas* and *Moringa* Husk

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript

Article Information

DOI: 10.9734/CSJI/2021/v30i1230270

Open Peer Review History:
This journal follows the Advanced Open Peer Review policy. Identity of the Reviewers, Editor(s) and additional Reviewers, peer review comments, different versions of the manuscript, comments of the editors, etc are available here: https://www.sdiarticle5.com/review-history/52104

Original Research Article

Received 20 August 2019
Accepted 25 October 2019
Published 28 December 2021

ABSTRACT

Moringa husks and Jatropha cacus were evaluated using decoloration of methylene blue for the removal of lead (Pb) and cadmium (Cd) from water sample. The results showed that *Moringa* has better properties: physicochemical properties: pH, moisture content (%), ash content (%), carbon content (%), volatile matter (%), surface area (m^2/g), electrical conductivity (µs/cm), bulk and density (g/L), 6.4, 14, 4, 63.26, 10, 787, 867 and 400 respectively, decoloration of methylene blue at 10 to 90 minutes ranges from 11 to 87% , Pb and Cd adsorption capacity 55.7 and 86, % degradation 85 and 92 than the Jatropha cacus 50.2 and 55.2, 78 and 83 respectively. The *Moringa* husk was characterized using Fourier Transform Infrared (FTIR), Scanning Electron Microscope (SEM) and its adsorption properties were studied by Langmuir and Freundlich kinetic isotherms.

Keywords: Activated carbon; adsorption; Jatropha curcas; Moringa husks; methylene blue.

1. INTRODUCTION

Water is the basic component of life; therefore it is important that people should be concerned about the nature of their drinking water especially the classification, evaluation of water weather fit for consumption, physical and chemical properties of the water [1]. Although most health

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problems relating to water can be as a result of pathogens, inorganic elements such as cadmium, lead, arsenic and mercury e.t.c. [2].

Water pollution by heavy metal has been recorded as a major problem in the global context. It occurs due to the direct and indirect discharge of diverse chemicals in to the water bodies without sufficient treatment to reduce and diminish the harmful compound [3]. Pollution of surface and ground water caused by human and industrial activities has been recorded as a major problem in the global context [4]. Water pollution considered as the leading universal cause of 80% of diseases [5]. According to the United Nation Organization reports that there 1.1 billion people still do not have access to safe supply of drinking water, the majority of them are among the world's poorest and developing countries [6].

Chemicals from industrials are usually discharged directly or indirectly with partial or no treatment in to water bodies [7]. Heavy metals even as trace are harmful to human and other living organisms as they tend to accumulate in the tissue of the living organisms [8].

Every day thousands of chemicals are discharged directly and indirectly in to water bodies without further treatment for elimination of the included harmful compound [7]. Heavy metals are without doubt as the most hazardous and harmful metals even if they present as traces, since they accumulate in the tissue of living organisms [8].

Moringa oleifera is a multipurpose tree with variety of applications including coagulant in water treatment. Research has shown that the husk can be converted in to activated carbon by carbonization and activating process [9].

The aim of the research work is to produce activated carbon from Jatropha cacus and *Moringa* husks, evaluated using decoloration of methylene blue and the best activated carbon will be used in the removal of lead and cadmium from water sample obtained from Hunkuyi Dam, Kaduna State which serves as a means of converting agricultural waste in to useful material.

2. METHODS

2.1 Study Area

The study area is National Research Institute for Chemical Technology (NARICT) Zaria, Basawa Town in Sabongari Local Government Area of Kaduna State in Northern Nigeria where plantations of both the *Jatropha* and *Moringa* are situated in the institute and the water sample was collected from Hunkuyi Dam during dry and raining season, 2019.

2.2 Preparation of the Precursor Material

Jatropha curcas and *Moringa* husk was collected from National Research Institute for Chemical Analaysis (NARICT) was washed thoroughly with water, sun drying for 1 hour, crushing and sieving in to workable particles size. The samples was kept in a clean container for carbonization and analysis.

2.3 Chemicals and Impregnating Agent

All the chemicals used were of analytical grade. The impregnating agent for the chemical activation of *Moringa* husk and *Jatropha curcas* was phosphorus acid (H_3P0_4).

2.4 Chemical Activation and Carbonization

The sample materials were carbonized in the absence of air in a muffle furnace at a temperature 550ºC for 60 minutes and the 200 g of carbonized sample was mixed with an aqueous solution of phosphoric acid (Activating agent). The mixture was then subjected to heat at a temperature of 120ºC for 3 hours to vaporize the water. The dried mixture was subjected to heat at a temperature of 650ºC in a muffle furnace to enable activation of the pores of the carbon sample.

2.5 Determination of Moisture Content

Thermal drying method was used in the determination of moisture content of the sample. 1.0 g of dried activated carbon was weighed in triplicate and placed in crucible, washed, dried and weighed crucible. The crucibles was then placed in an oven and dried at 105ºC to constant weight for 4 hours according to the method of [10].

The percentage moisture content was calculated as:

$$\text{Moisture (\%)} = \frac{loss\ in\ weight\ on\ drying\ (g)}{initial\ sample\ weight\ (g)} \times 100 \quad (1.1)$$

2.6 Determination of Ash Content

Crucibles was oven washed, oven dried at 105ºC and allowed to cool at room temperature in a desiccator. The crucible was weighed and 1.0 g of the sample introduced and placed in muffle furnace until the temperature rose to 500ºC maintained for 3 hours, then finally cooled in desiccator, reweighed to determine the ash content of the sample.

The percentage ash content was calculated using equation 1.2

$$\text{Ash (\%)} = \frac{Ash\ weight\ (g)}{Oven\ dry\ weigh\ (g)} \times 100 \quad (1.2)$$

2.7 Adsorption Capacity

2.0 g of the prepared activated carbon was added to 100 ml raw water shake thoroughly and allow to stand for the period of an hour to establish adsorption-desorption equilibrium, filtered and then analyze the presence of lead, pb and cadmium, cd both before and after the interaction with the activated carbon using adsorption spectroscopy.

The adsorption capacity was calculated using the equation below

$$Qt = \frac{(Ci - Ct)V}{W} \quad (1.3)$$

Where:

Qt = adsorption capacity at time, t (mg/g)
Ci = concentration of metal before interaction with the activated carbon (mg/L)
Ct = concentration of metal after interaction with the activated carbon (mg/L)
V = volume of the effluent (L)
W = weight of the activated carbon (g) [11]

2.8 Surface Area of the Activated Carbon

The specific surface area of the prepared activated carbon was determined using Sear's method by agitating 1.5 g of the activated carbon sample in 100 ml diluted hydrochloric acid at pH = 3, 30 g of sodium chloride was added while stirring the suspension, then the volume was made up to 150 ml using de-ionized water. The solution was titrated with 0.1N sodium hydroxide to raise the pH from 4 to 9 and the volume recorded

The surface area was calculated below

$$S = 32V - 25 \quad (1.4)$$

Where:

S = Surface area of the activated carbon
V = volume of sodium

2.9 Scanning Electron Microscope (SEM) Analysis

Scanning electron microscopy (SEM) analysis: A Hitachi X-650 scanning electron micro-analyzer was used to take the micrographs of the samples, at an accelerating voltage of 10.0 KV and a working distance of 10.0 mm, the activated carbon was mounted on aluminum stubs using conductive glue and then coated with a thin layer of carbon paste tape on copper pegs and coated with a film of evaporated gold, prior to the observation.

2.10 UV-visible Spectroscopic Analysis

A computer based UV-Vis spectrophotometer was used for the determination of concentration of samples. The system was switched on and warm up to 30 minutes, thoroughly clean quartz cuvettes was use. One of the cuvettes was fill with the reference compound and the other one with compound whose absorbance were measure at maximum wavelength. To get the relationship between concentration and absorbance of the compound, a calibration curve was plotted. Calibration solutions were made from standard solutions of known concentration. The absorbance was plotted against concentration of the calibrated samples. These calibration curves was stored in the system itself and the concentration of the unknown sample was calculated directly from the absorbance.

2.11 Particle Size Distribution

Exactly 20 g of the prepared activated carbon was sieved using sieves of different mesh sizes (2 mm, 1 mm, 800 µm, 700 µm, 500 µm, 400 µm, 300 µm, 200 µm, 100 µm, 50 µm and pan) placed on electrical vibrator in descending order for 5 minutes and the fractions collected, weighed.

2.12 Degradation of Methylene Blue Solution

A 100 ml/L stock solution of methylene blue was prepared using distilled water for all the experiments.

Exactly 100 ml of solution of methylene blue solution was taken from the stock solution, put in a beaker; 2.0 g of *Moringa* husk activated carbon was added. The suspension was kept in a dark at room temperature with continuous stirring using magnetic stirrer. After an hour in the dark, an aliquot of 5 mL was taken from the beaker at interval of 10 minutes for 1 hour; the catalysts were filtered then analyzed using UV-Vis Spectrophotometer to evaluate the degradation of the methylene blue. Same procedure was repeated using *Jatropha curcas* activated carbon.

3. RESULTS

3.1 Characterization of *Moringa* husk and *Jatropha curcas* Activated Carbon

The activated carbon produced from *Moringa* husk and *Jatropha curcas* was characterized using proximate analysis (Table 2).

Table 1. Physicochemical properties of water sample from Hunkuyi Dam

Parameter assessed	Water samples	Permissible limit (WHO)
pH	11.12±1.24	6.0 – 9.0
Temperature (^{0}C)	36±1.30	**<**40
Electric conductivity (µS/cm)	3.03±0.57	2.0
Turbidity	6.35±0.69	5
Total dissolved solids (mg/L)	500±3.42	500
Total solids (mg/L)	270±2.89	200
Biochemical oxygen demand (mg/L)	37.7±1.92	30
Dissolved oxygen (mg/L)	37±0.55	10
Nitrate (mg/L)	20.0±2.03	20
Sulphate (mg/L)	86.2±3.90	500
Phosphate (mg/L)	4.32±0.69	5.0
Chromium (ppm)	0.48±0.04	0.5
Copper (ppm)	0.059±0.01	0.1
Zinc (ppm)	2.81±0.56	<1
Lead (mg/L)	0.085±0.006	0.05
Cadmium (mg/L)	0.036±0.0039	0.01

Table 2. Physicochemical properties of *Jatropha curcas* and *Moringa* Husk activated carbon

Parameter	Results	
	Moringa	**Jatropha**
pH	6.4	6.8
Moisture content (%)	14	15
Ash content (%)	4	4
Carbon content (%)	63.26	40.260
Volatile matter (%)	10	8
Surface area (m^2/g)	896	787
Electrical conductivity (µs/cm)	867	824
Bulk density (g/L)	400	397
Adsorption capacity of pb and cd (mg/g)	55.7 and 86	50.2 and 55.2
% adsorption of pb and cd	85 and 92	78 and 83

Table 3. Particle size distribution of *Jatropha curcas* and *Moringa* Husks activated carbon

Mesh sizes (mm or µm)	Particle size distribution	
	Moringa(mm)	Jatropha (mm)
2mm	0.71	0.52
1mm	0.54	0.35
800 µm	2.35	2.22
700 µm	2.31	2.11
500 µm	2.20	2.06
400 µm	2.07	1.90
300 µm	2.00	1.80
200 µm	0.81	0.61
100 µm	0.17	0.42
50 µm	0.00	0.00
Pan	0.00	0.00

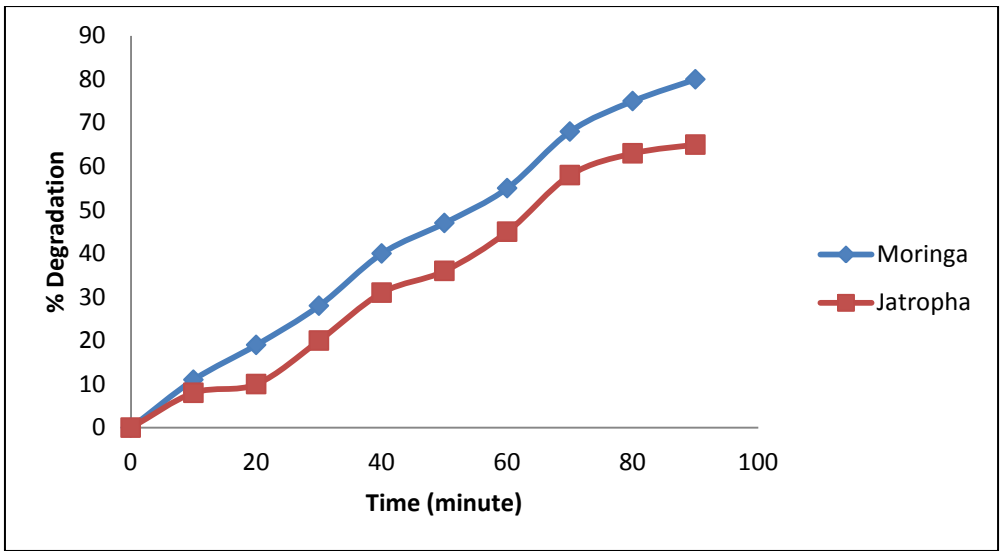


Fig. 1. Percentage degradation of methylene blue using *Moringa* Husk and *Jatropha curcas* activated carbon

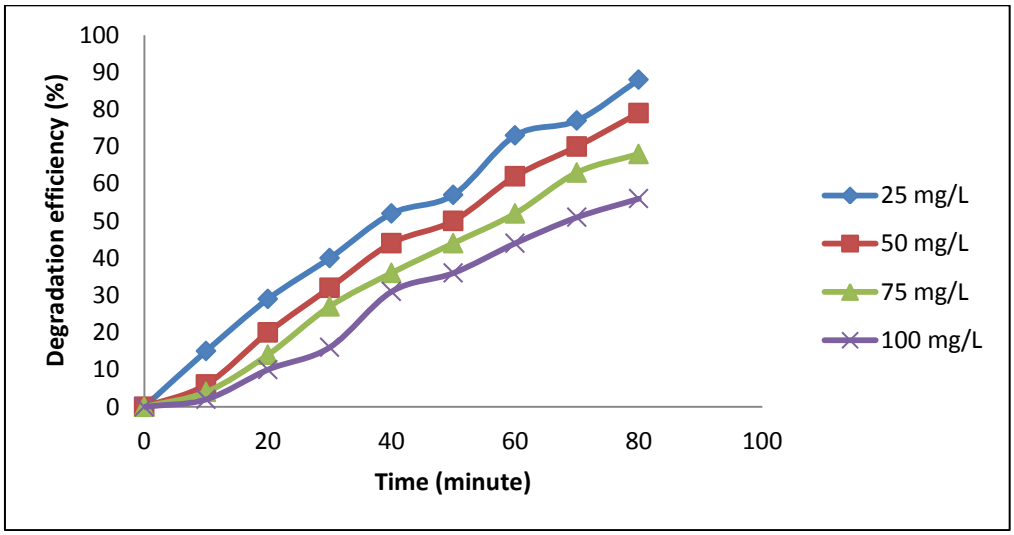


Fig. 2. Effect of methylene blue concentration on its degradation using *Moringa* Husk activated carbon

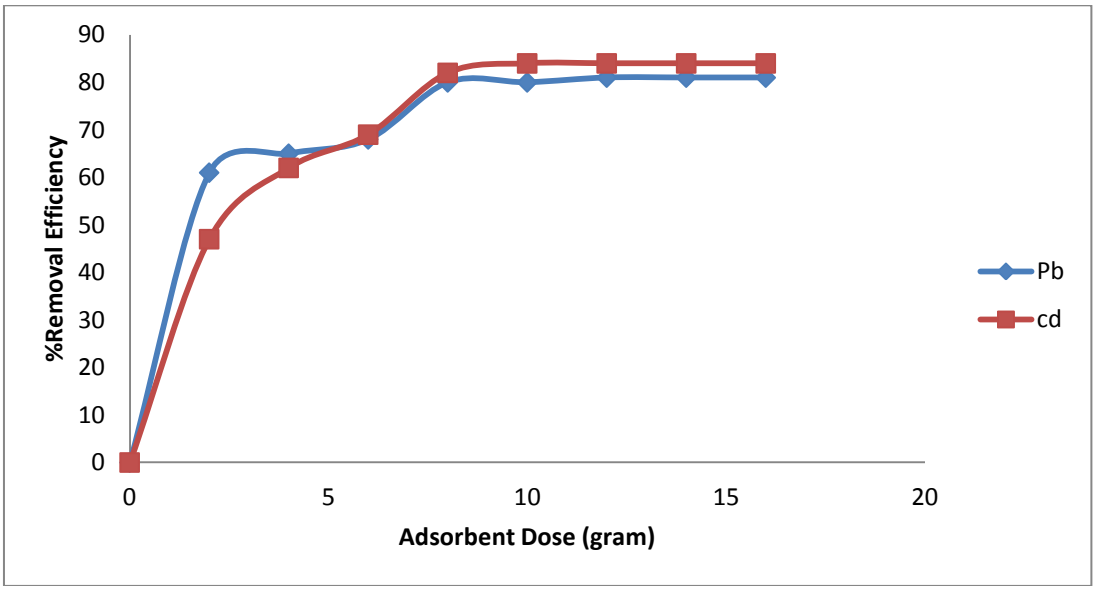


Fig. 3. Efficiency of Lead (pb) and Cadmium (cd) removal in relation to adsorbent dose using *Moringa* Husk activated carbon

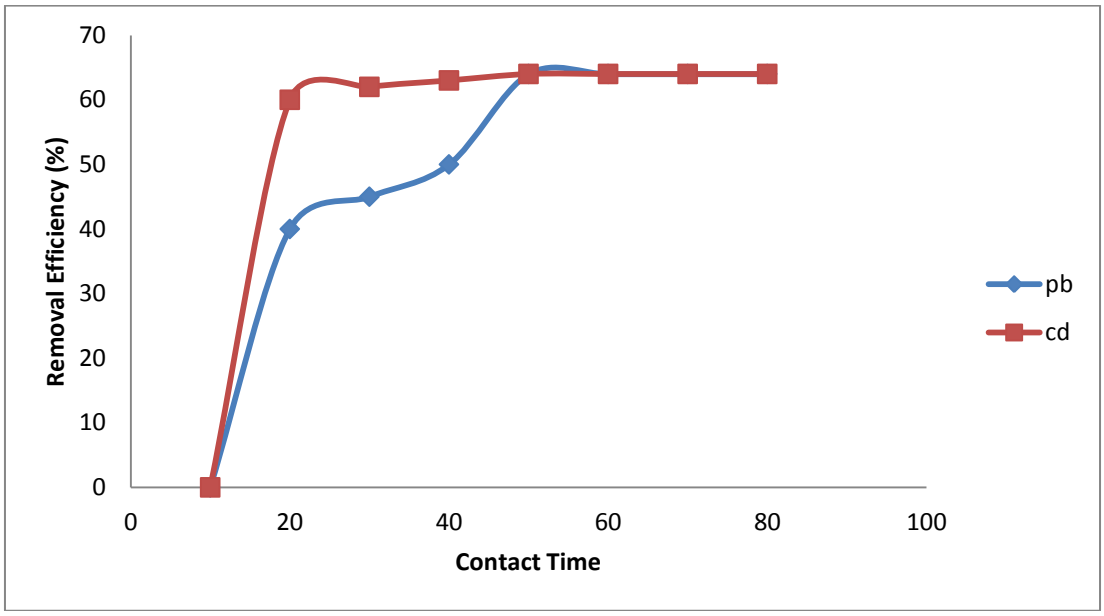


Fig. 4. Percentage removal of Lead (pb) and Cadmium (cd) based on contact time using *Moringa* Husk activated carbon

Table 4. Some properties of raw and treated water sample from Hunkuyi Dam

Samples	pH	Turbidity (NTU)	TDS (mg/L)	BOD (mg/L)	COD (mg/L)	Pb (mg/L)	Cd (mg/L)
Raw	11.12	6.35	510	37.7	40.9	0.085	0.036
Treated via *Jatropha curcas*	10.03	5.50	489	34.5	36.7	0.06	0.015
Treated via *Moringa* husks	8.00	4.90	445	27.4	30.0	0.039	0.0071

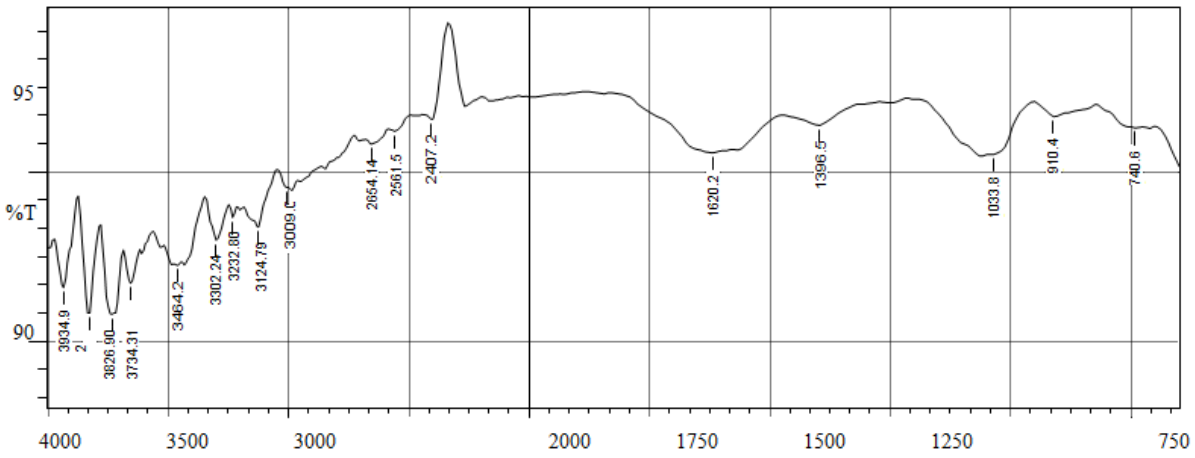


Fig. 5. Fourier Transform Infrared (FTIR) spectra of the pure *Moringa* husks

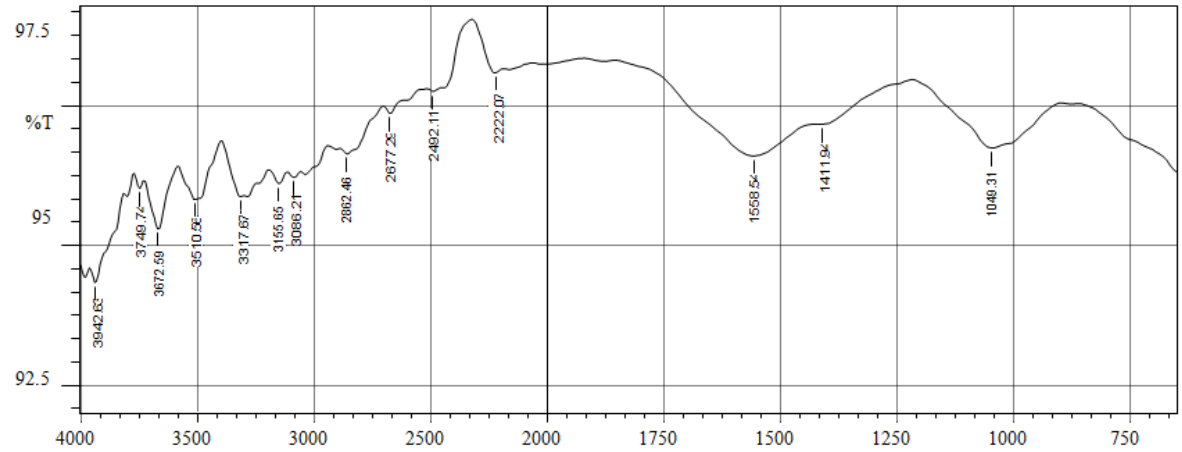


Fig. 6. Fourier Transform Infrared (FTIR) spectra of the activated *Moringa* husks

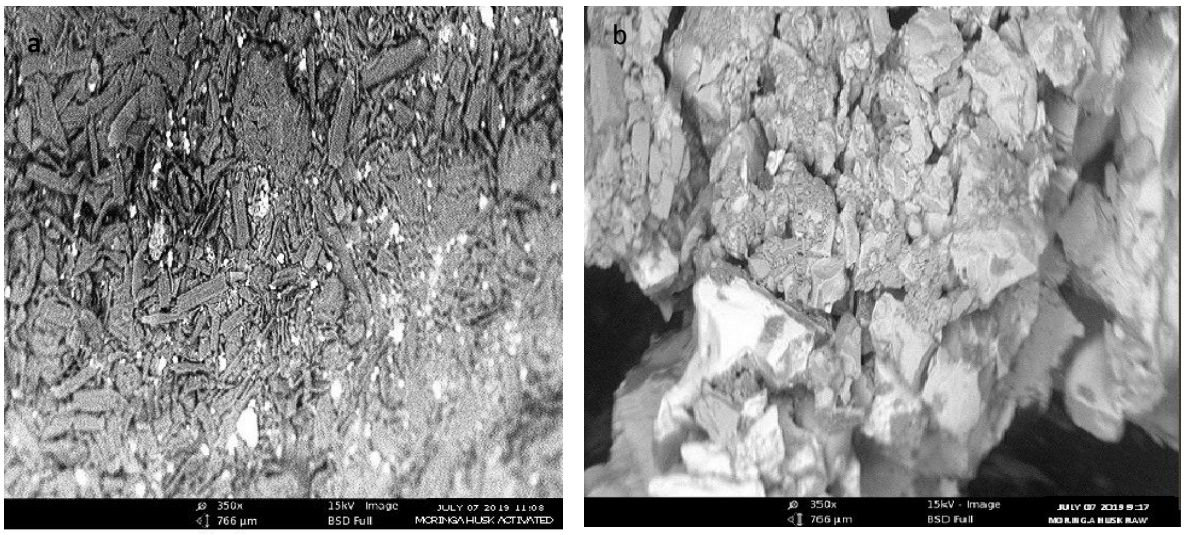


Plate 1. Image of scaning electron microscope of (a) Pure *Moringa* husk (b) Activated *Moringa* husks

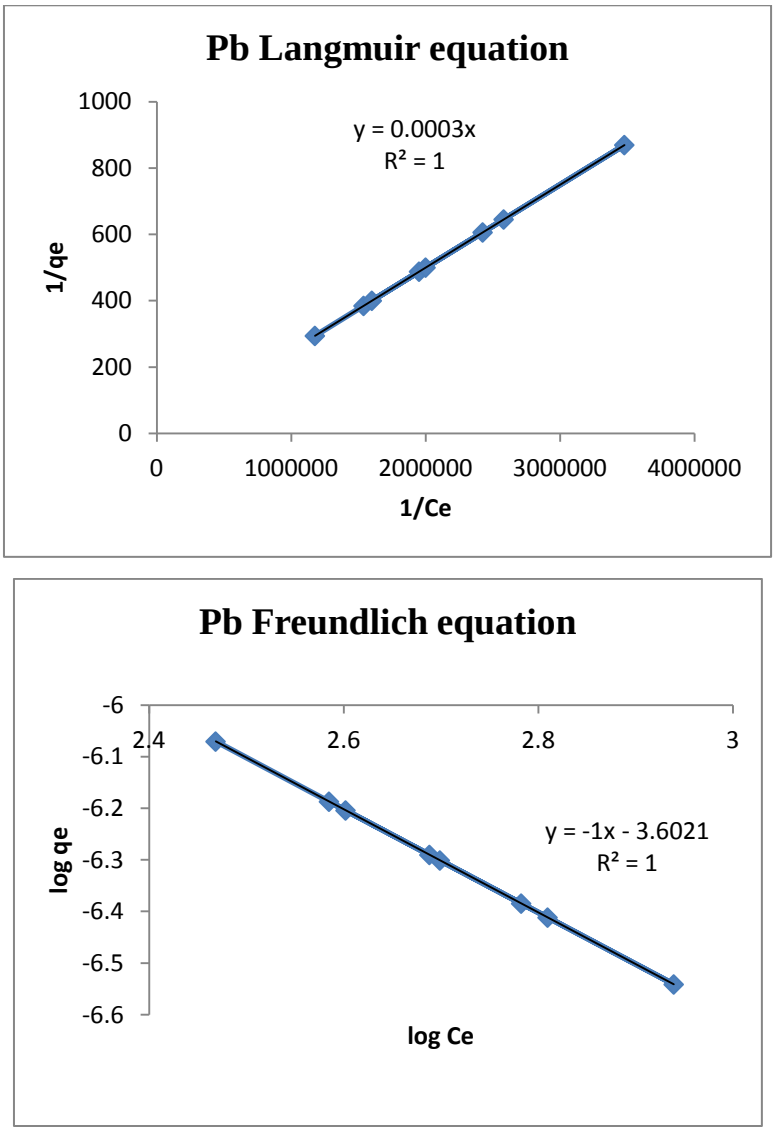
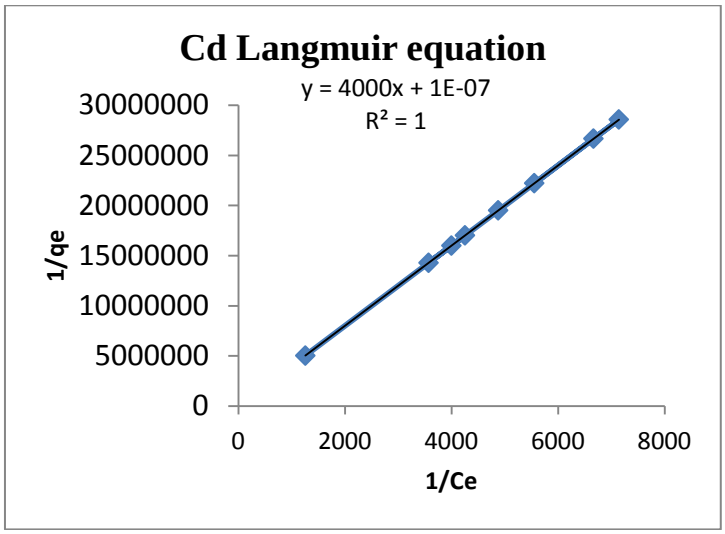


Fig. 7. Application of langmuir and freundlich equation for the removal of Pb using *Moringa* activated carbon



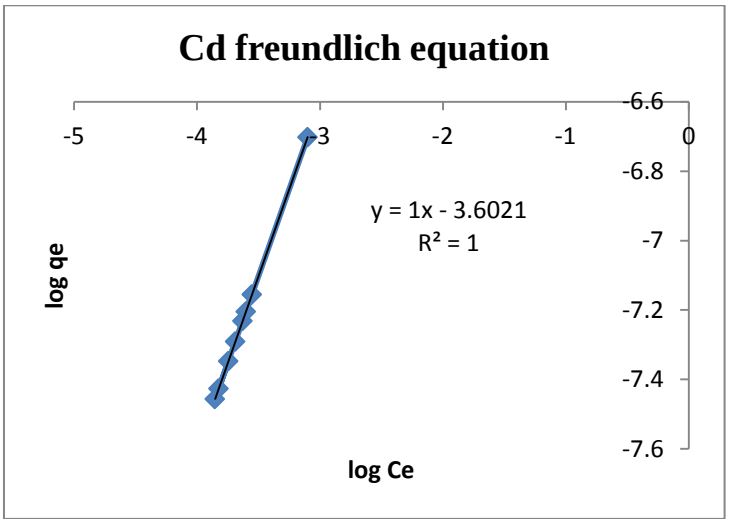


Fig. 8. Application of langmuir and freundlich equation for the removal of Cd using *Moringa* activated carbon

4. DISCUSSION

The physicochemical properties of water sample (Table 1) obtained from Hunkuyi dam exceeds the permissible limits set by World health organization [12] with the exception of temperature, total dissolved solids, nitrate, phosphate, suphate, chromium and copper while cadmium and lead showed significantly high value. According to Naseem et al. [13] revealed that drinking water contaminated with lead and cadmium above the recommendation limits can lead to renal failure, kidney diseases and even death.

It was observed from the proximate analysis (Table 2) that the moisture, ash, carbon content and the volatle matter % of both the *Moringa* husks and Jatropha cacus are high, according to (Malik et al., 2006) the high percentages of the mositure, ash, carbon and volatile matter can be due to the origin of the plants. The high absorption capacity of the lead and cadmium using *moringa* husk activated carbon can be attributed to the surface area, bulk density which are very high and the density of the generated activated carbon as it also play a vital role on adsorbate uptake. It was also observed that the pH of both the *Moringa* husk and Jatropha activated carbon were within the permissible limits set by World Health Organization.

The performance of the *moringa* husk and jatropha cacus activated carbon was first evaluated using decoloration of methylene blue.Methylene blue is used as a text model in evaluating the activity of serries of activated carbon (Disanto et al.,1972). Fig. 1 shows that *Moringa* activated carbon has high percentage of degradation than Jatropha cacus activated carbon for an instance, at 90 minutes *moringa* has 80% degradation while Jatropha cacus has 65 % degradation of methylene blue respectively. Therefore *moringa* husk acitvated carbon has better performance in the removal of lead and cadmium from Hunkuyi dam than Jatropha cacus activated carbon.

The effect of concentration of methylene blue on its degradation using *Moringa* husk activated carbon was investigated by varying the concentration of the methylene blue from 25 to 100 mg/L (Fig. 2) which shows that the percentage removal of methylene blue decreased with increasing methylene blue concentration, when the conccentation varried from from 25 to 100 mg/L, the percentage degradation of methylene blue decreased from 88% to 45%. At higher concentation, the methylene blue molecules absorbed some of the incident radiation which will decrease the amount of available light photons that will drive the photocalytic process (Jaafar et al. 2012).

The effect of the different amount of *Moringa* activated carbon on its percentage removal of lead, Pb and cadmium, cd was investigated by varying the concentration of the activated carbon from 2, 4, 6, 8, 10, 12, 14 and 16 grams. Fig. 3 shows that the percentage removal of the lead,

pb and cadmium, cd increase with increase in the adsorbent dose from 2 g to 10 g at 47 and 60% to 82 and 84% removal of lead, pb and cadmium, cd respectively however, the increases was sharply from 0 to 79 % in the case pb and 0 to 81% in the case of cd at dose of 8 g but slower with further increment of dose until it remained contend, the significant slower of a sample containing increment of dose from 10 g to 16 g can be attributed to decreased in amount of dose absorbed per unit mass, similar trend was reported in the work of Aji MM et al. [14].

The adsorption desorption equilibrium of the *Moringa* activated carbon was investigated on the Percent Removal Efficiency of pb and cd Based on Contact Time. Fig. 4 shows that about 65% removal of pb and cd was achieved in an hour. It also shows that the percentage removal remained constant at 60 – 80 minutes; this signified that equilibrium is reached at 60 minute in which is the maximum required time for the removal the lead, pb and the cadmium, cd. Similar trend was reported in the work of Aji MM et al., [14]. 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 [15].

Fourier Transform Infrared Spectroscopy (FTIR) was used to identify the functional groups present in the pure and prepared activated *Moringa* husks used for the absorption of lead (Pb) and cadmium (Cd) (Figs. 4 and 5). Each peak in the FTIR spectrum were assigned to respective functional group observed at 3934.9 – 1620.2 cm-1, and 1396.5 cm-1 (Fig. 4), while, 3942.63 – 1049.31 cm-1 (Fig. 5) which can be assigned to alcohol, aldehyde, alkene and phenol respectively for the pure *Moringa* husks, while Alcohol, aldehyde, phenol and nitro-compound respectively for the activated *Moringa* husks. According to Zheng et al. [16] functional groups present in a compound play a vital role on the absorption feature of the particular compound. Also according to Fan and Wang [17] the chemical nature of the compound also an important factor in understanding the absorption process of a particular compound.

The morphological characteristic of the pure and activated *Moringa* husks were investigated using Scanning Electron Microscope (SEM) analysis (plate 1a and b). Non compact arrangement of the non-particles with shape rice of which the particles are not aggregated being like shapes of small particles of wood with little or no pores making it difficult to determine it exact shape and sizes (plate 1a) this is also observed in the work of [18], while plate 1b shows the microgram of the activated *Moringa* husks that most of the particles are aggregated and exhibited a compact arrangement in the form of rocks.

The removal isotherms of the heavy metals: lead (Pb) and cadmium (Cd) were represented using Langmuir and Freundlich equations. The analysis of the data for the removal of Pb and Cd were graphically shown in Figs. 7 and 8 which shows that the kinetic isotherms of the Langmuir and Freundlich perfectly fitted and described the absorption processes of the lead (Pb) and cadmium (Cd) using activated carbon from *Moringa* husks. Both the Langmuir and Freundlich equations has the same correlation coefficient greater than 0.5, that is 1 which were well fitted in to isotherms equations equilibrium and also fitted better for sample of each adsorbate, this is also observed in the work of Kehinde et al. [19] also in the work of Mohammed et al. [20] which showed that the Langmuir isotherms has better adsorption equilibrium than the Freundlich isotherm equation in the removal of iron (Fe) and manganese (Mn). Under adsorption process the plot of Langmuir equation usualy predicts the favorability of a system in batch processes which is normally described by separation factor [21].

5. CONCLUSION

Moringa husks and *Jatropha curcas* were activated for removal of lead and cadmium in water sample but was evaluated using decoloration of methylene blue. The results obtained shows that *Moringa* has better properties: physicochemical properties, particle size distribution, adsorption of lead and cadmium than the activated *Jatropha curcas*. The *Moringa* husks was then characterized using Fourier Transform Infrared (FTIR), Scanning Electron Microscope (SEM) and its adsorption properties were then studied by Langmuir and Freundlich kinetic isotherms. The serves as a means of converting agricultural waste in to useful materials.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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