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## Preparation and characterization of *Pericopsis elata* adsorbent for the treatment of heavy metal ions in a simulated waste water

Baba Nwunuji Hikon \*, Godwin Ogbaji Egah, Garindo Boro, Hyelalibiya Ataitiya and Aneshi Arose

Department of Chemical Sciences, Federal University Wukari, P.M.B 1020 Wukari, Taraba State, Nigeria.

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### Abstract

The research aimed at using *Pericopsis elata* adsorbent to elute heavy metals in waste water. The *Pericopsis elata* was charred and activated with 0.1 M HNO<sub>3</sub>. The results of Fourier Transform Infrared (FTIR) indicates broad band at 3742.03 and 3688.2 cm<sup>-1</sup> (ranging from 3000 - 4000 cm<sup>-1</sup>) and were attributed to OH group stretch from alcohol and the water molecules adsorbed at the surface of the adsorbent that interact with the oxygen atoms and internal hydroxyl groups. This suggests that the adsorption was done on the OH bending of water. Zeiss EVO 50 Scanning Electron Microscope (SEM) was used in this study, the result showed that the structure of the *Pericopsis elata* adsorbent was observed at magnification of 10, 100,000 times with virtually unlimited depth of field. The SEM micrographs indicated macropores in the *Pericopsis elata* adsorbent showed large cavity. SEM results showed the surface morphology feature of the adsorbents which is an indication that important interaction occurred between the adsorbate and adsorbent granule interface in the experimental conditions. X-Ray Fluorescence Characterized the Adsorbent and the results showed the following chemical composition based on atomic concentration; carbon 89.39%, oxygen 6.56%, calcium 0.96%, nitrogen 2.19%, aluminium 0.47%, magnesium 0.22%, iron 0.09%, Phosphorus 0.05%, Silicon 0.04%, Sulfur 0.03%. Langmuir and Freundlich isotherm provided information on the capacity of adsorbent. The results indicated that an important interaction occurred between the adsorbate and adsorbent-granule interface.

**Keywords:** Interface; Adsorbent; Heavy metals; Carbon; Adsorbate; *Pericopsis elata*

### 1 Introduction

*Africa Teak* also known as Marsonia or Madrid, Afrormosia, nyinasa, asamela, assamela etc. The botanical name of the plant is *Pericopsis elata* (*P. elata*). It a medium to large tree that can grow up to 50 meters tall, found in relatively dry deciduous forests in West and Central Africa. The wood of *P. elata* is very valuable and durable timber that is often used in Africa. *P. elata* has been an important commercial timber species for over 60 years, and heavy exploitation of the tree has led to concerns about the species' ability to persist in the wild. The largest remaining stands of the tree are located in Cameroon, Congo-Brazzaville, Côte d'Ivoire, Democratic Republic of Congo, Ghana and Nigeria.

The use of *Pericopsis elata* waste as raw material for the development of porous adsorbent is associated with its internal porosity and properties such as surface area, pore volume and pore size distribution. In addition, the internal pore structure, surface characteristics, degree of surface reactivity and presence of functional groups on pore surfaces play an important role in adsorption processes [1].

Heavy metal ions stand out among the inorganic pollutants due to their persistence and toxicity. Heavy metals are generally defined as metals with relatively high densities, atomic weights, or atomic numbers. These metals include cadmium, iron, copper, tin, zinc, chromium. Heavy metals enter the environment by natural and anthropogenic means,

\* Corresponding author: Baba Nwunuji Hikon; Email: [bahahikon@fuwulari.edu.ng](mailto:babahikon@fuwulari.edu.ng)

such as natural weathering of the earth's crust, mining, soil erosion, industrial discharge, urban runoff, sewage effluents, pest or disease control agents applied to plants, air pollution's fallout, etc., [2]. Cadmium is a chemical element with symbol Cd and atomic number 48. This soft, bluish-white metal is chemically similar to the two other stable metals in group 12, zinc and mercury. Cadmium occurs as a minor component in most zinc ores and is a byproduct of zinc production. Cadmium was used for a long time as corrosion-resistant plating on steel, and cadmium compounds are used as red, orange and yellow pigments, to color glass, and to stabilize plastic. Cadmium use is generally decreasing because it is toxic. Although cadmium has no known biological function in higher organisms, a cadmium-dependent carbonic anhydrase has been found in marine [3].

Chemisorption (or chemical adsorption) is adsorption in which the forces involved are valence forces of the same kind as those operating in the formation of chemical compounds. The problem of distinguishing between chemisorption and physisorption is basically the same as that of distinguishing between chemical and physical interaction in general. No absolutely sharp distinction can be made and intermediate cases exist, for example, adsorption involving strong hydrogen bonds or weak charge transfer. [4].

## 2 Material and methods

### 2.1 Sample Collection and Preparation

*Pericopsis elata* or Marsonia waste woods were collected randomly at six different locations within Wukari Local Government Area of Taraba State in the month of April, 2019. The production of activated carbon from marsonia waste was adopted from [5] the removal of red weight of each raw material was introduced into the furnace at 350 °C in the absence of air for one hour thirty minutes and to cool at room temperature.

#### 2.1.1 Chemical Activation

The carbonated materials (carbon) from *Pericopsis elata* wood were crushed into powder using a crusher and sieved with a 1.88 mm sieve to obtain a uniform particle size. Measured weights of each carbonated materials were soaked in nitric acid of 0.1 M in a crucible. Each sample solution was stirred until the mixture turned to paste. The pastes were heated in a muffle furnace at 800 °C for 2 h in the absence of air to increase the surface area of the samples for adsorption purposes. The activated carbons were cooled to room temperature, washed with distilled water until their pH approximates 7 (no change in colour when tested with a red litmus paper) indicating no trace of nitric acid, dried for 3 h and stored in an air-tight nylon [5].

#### 2.1.2 Classical Characterization of *Pericopsis elata* Adsorbent

The physiochemical properties of the activated carbons such as the yield of Carbon, Bulk Density, Porosity, yield of carbon and pH were determined using standard procedures. All results were the average of duplicate analysis.

##### Determination of pH

The pH of the adsorbents was determined by method adopted by [6]. A 1.0 g of the adsorbent was weighed into 100 mL distilled water in a beaker and kept on a magnetic stirrer for 1 h at 120 rpm. The pH was then measured and recorded. The pH readings were taken in triplicates using HI 8014 pH meter (Hanna instruments).

##### Bulk density of adsorbents

The bulk density was determined using the tamping method of [7]. A 5 g portion of each adsorbent was packed in a 10 mL measuring cylinder and tamped until it occupied a minimum volume. The apparent volume is read to the nearest graduated unit. The bulk density was measured as:

$$\text{Bulk density} \left( \frac{\text{g}}{\text{cm}^3} \right) = \frac{\text{Mass of the adsorbent (g)}}{\text{Apparent Volume of adsorbent (cm}^3\text{)}} \dots\dots\dots \text{(i)}$$

##### Yield of Carbon

The char material cooled after carbonization was weighed and used to obtain the percentage yield of carbon using the formula;

$$\% \text{ Yield of Carbon} = \frac{\text{weight of char}}{\text{Weight of raw materials}} \times 100 \dots\dots\dots \text{(ii)}$$

### Porosity Determination

The porosity of the activated carbon was obtained using the formula;

$$\text{Porosity} = \frac{\text{pore volume}}{\text{Total volume [volume of centrifuge tube]}} \dots\dots\dots \text{(iii)}$$

The pore volumes of the activated carbons were obtained using the formula:

$$\text{Pore Volume} = \frac{\text{Bulk Density of activated Carbon}}{\text{Density}}$$

$$\text{Therefore the porosity is } \frac{\text{Bulk Density of Activated Carbon}}{[\text{Density of water}] \times \{\text{total volume}\}} \dots\dots\dots \text{(iv)}$$

#### 2.1.3 Instrumental Characterization of Adsorbents

##### Scanning electron micrographs (SEM) analysis

Scanning electron microscopy (SEM) is used to determine the surface structure and shape of an adsorbent [8]. The SEM imaging was carried out at National Institute for Research and Chemical Technology Zaria. Scanning electron microscope (SEM - JEOL, JSM 7600 F) was used to determine the surface texture and porosity of the adsorbent. A thin layer of platinum was sputter-coated on the adsorbent for charge dissipation during SEM imaging. The sputter coater (Eiko IB-5 Sputter Coater) was operated in an argon atmosphere using a current of 6 mA for 3 min. The coated samples were then transferred to the SEM specimen chamber and observed at an accelerating voltage of 5 kV, eight spot size, four aperture and 15 mm working distance.

##### Fourier transformed infra-red (FTIR) analysis

The FT-IR was also carried out at National Institute for Research and Chemical Technology Zaria. This method of analysis was conducted to determine the existence of the surface functional groups of the adsorbents. The Fourier transform infrared (FTIR) spectra were collected in the range of 650-4000  $\text{cm}^{-1}$  using the KBr disk method [9].

##### X-ray fluorescence (XRF) analysis.

The XRF was carried out at National Institute for Research and Chemical Technology Zaria. Sky-ray Instrument (EDX3600B X-ray fluorescence) was used. Method of analysis was used to determine the chemical composition of the adsorbents. In sample preparation, the sample was pulverised to fine homogeneous size and then pelletized before analysis [9].

## 3 Results and discussion

The results of the physicochemical properties of *Pericopsis elata* adsorbent indicated that the yield of Carbon produced was 89.00% while the porosity of the adsorbent was 0.818. Bulk density of the adsorbent was 8.16  $\text{g}/\text{cm}^3$  and the pH obtained was 7.40.

**Table 1** Some Physicochemical Properties of Adsorbent

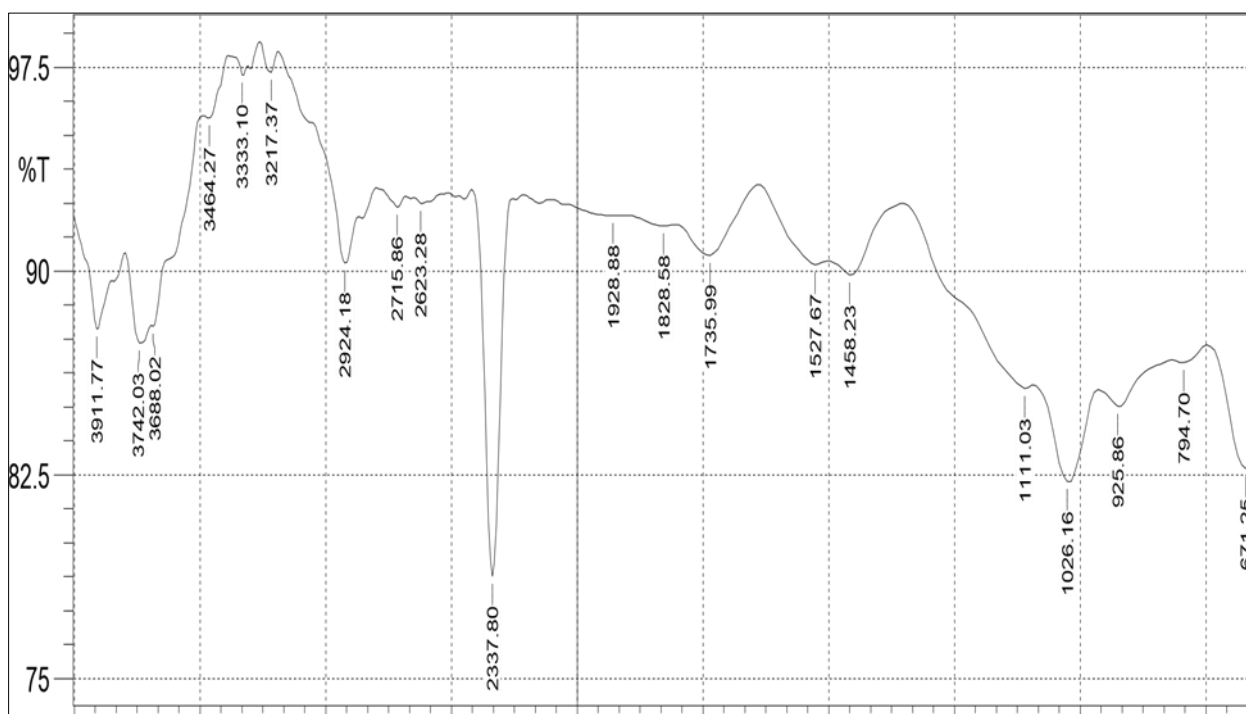
Properties	Adsorbent ( <i>Pericopsis elata</i> )
Yield of Carbon (%)	89.00
Porosity Determination	0.818
Bulk-density ( $\text{g}/\text{cm}^3$ )	8.16
pH	7.40

**Table 2** FT-IR Analysis of Adsorbents

Observed Frequency (cm <sup>-1</sup> )	Functional groups	Pe	Assignment
4000-3000	Alcohol	3688.02	Free O-H stretch
	Alcohol	3742.03	O-H stretch
	Alcohol	3688.02	O-H stretch
	Alcohol	3688.02	N-H stretch
3000-2500	Alkane	2924.19	C-H stretch
2500-2000	Carbondioxide	2337.80	O=C=O stretch
2000-1000	Alkoxy	1026.16	C-O stretch
1000-500	Alkene	671.25	C=C bending
	Halo-compound	671.25	C-Cl

Pe: *Pericopsis elata* adsorbent.

From the FTIR result, the broad band at 3742.03 and 3688.2 cm<sup>-1</sup> (ranging from 3000 4000 cm<sup>-1</sup>) are attributed to OH group stretch from alcohol and from the water molecules adsorbed at the surface of the adsorbent that interact with the oxygen atoms and internal hydroxyl groups [10]. This suggests that the adsorption may have been done on the OH bending of water. 1026.16cm<sup>-1</sup> and 2337.80 cm<sup>-1</sup> carboxyl O=C=O and alkoxy group respectively CO. The band at 671.25 cm<sup>-1</sup> is the unsaturated bending of C–C bonds ascribed to aromatic C=C and 2924.18671.25 cm<sup>-1</sup> alkane stretching of C-H.

**Figure 1** FTIR Spectral of *Pericopsis elata* adsorbent

The chemical compositions of *Pericopsis elata* adsorbent are presented in Table 3 below. The results shows that the sample was mainly composed of carbon, oxygen, calcium, nitrogen, aluminium, magnesium, iron. Basically, the major component is carbon which has 89.39%.

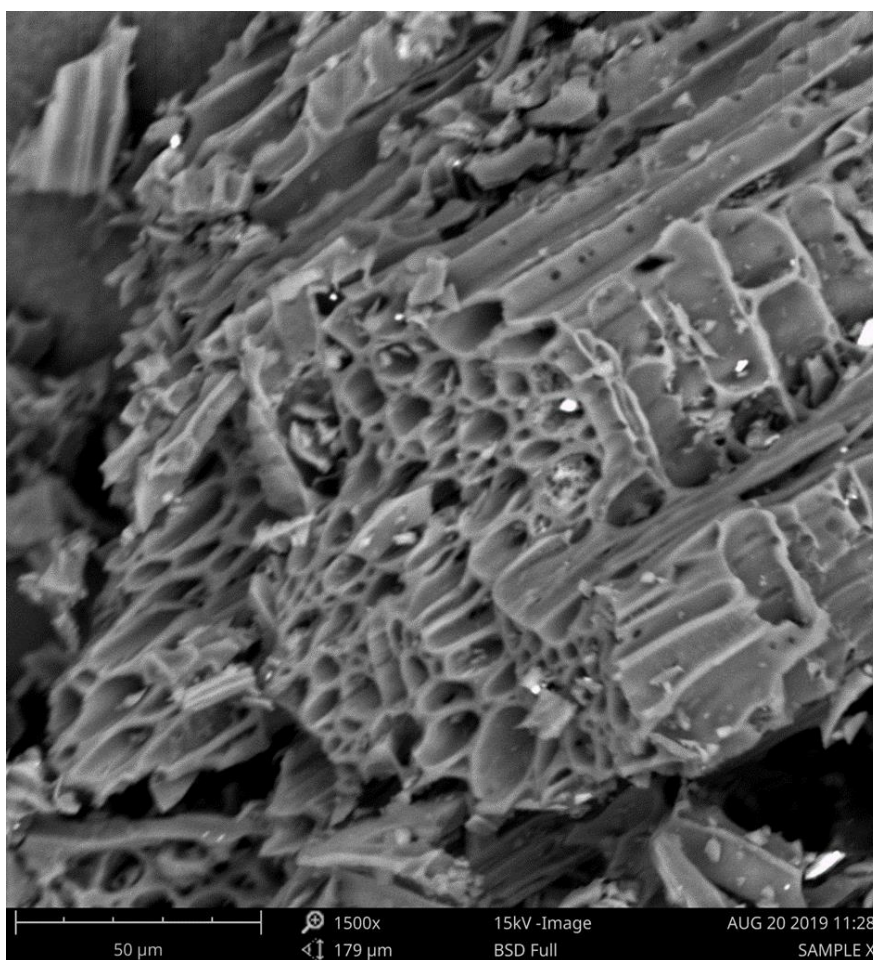
This implies that the *Pericopsis elata* is a cellulose material with high carbon content. Others are impurities which are present in their oxide form.

**Table 3** X-Ray Fluorescence Characterization of the Adsorbent

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
6	C	Carbon	89.39	84.24
8	O	Oxygen	6.56	8.23
20	Ca	Calcium	0.96	3.03
7	N	Nitrogen	2.19	2.41
13	Al	Aluminium	0.47	0.99
12	Mg	Magnesium	0.22	0.43
26	Fe	Iron	0.09	0.40
15	P	Phosphorus	0.05	0.12
14	Si	Silicon	0.04	0.09
16	S	Sulfur	0.03	0.07

### 3.1 SEM Analysis

SEM: A Zeiss EVO 50 Scanning Electron Microscope (SEM) was used in this study. The structure of the raw material (*Pericopsis elata* wood waste) was observed at magnification of 10 100,000 times with virtually unlimited depth of field. The SEM micrographs indicated macropores in the *Pericopsis elata* adsorbent showed large cavity

**Figure 2** SEM Porosity

SEM results show the surface morphology feature of the adsorbents which is an indication that important interaction can occur between the adsorbate and adsorbent-granule interface in the experimental conditions [8]. The plate shown above showed a porous, rough and irregular surface with crystalline structure which indicated more macro pores for adsorption. Similar observation was reported by [10]. It can be concluded that the *Pericopsis elata* adsorbent provides an external surface of porous and more reactive sites which enhanced its adsorption capacity.

### 3.2 Adsorption Isotherms

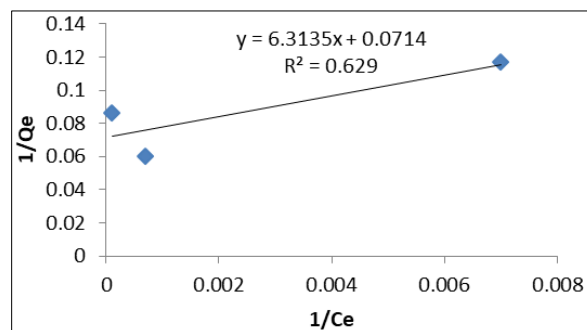
**Table 4** Isotherm Parameters for Chromium and Cadmium Adsorption on Adsorbent

Adsorbent	Models	Adsorbates	
		Cr	Cd
<i>Pericopsis elata</i>	<b>Langmuir</b>		
	$Q_m(\text{mg/g})$	13.032	14.084
	$b(\text{L/mg})$	0.787	0.011
	$R^2$	0.9712	0.722
	$R_L$	0.654	0.752
	<b>Freundlich</b>		
	$1/n$	1.727	1.810
	$n_F$	0.579	0.721
	$R^2$	0.629	0.522
	$K_F((\text{mg/g})\text{L/mg})^{1/n}$	2.922	2.731

Isotherms models were studied for the adsorption process. Langmuir and Freundlich isotherm provide information on the capacity of absorbent.

Langmuir isotherms relate metal uptake per unit weight,  $q_e$  adsorption capacity,  $C_e$  initial and residual concentration. The SEM results show the surface morphology feature of the adsorbents which is an indication that important interaction occurred between the adsorbate and adsorbent granule interface in the experimental conditions [8]. The plate shown above indicates porous, rough and irregular surface with crystalline structure which indicated more macro pores for adsorption. Similar observation was reported by [10].

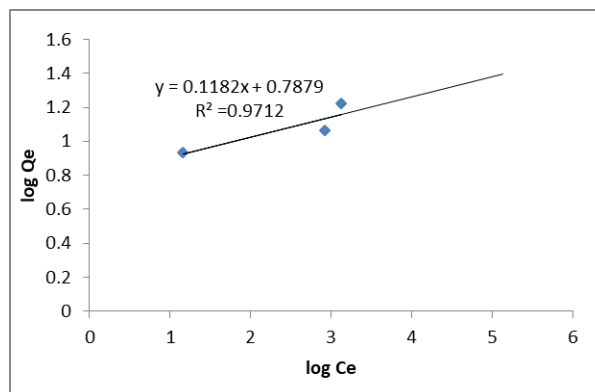
Isothermal adsorption data for adsorbent at 35 °C were plotted and found that the value of correlation coefficients  $R^2$  were 0.9712 and 0.722 for Cr and Cd which clearly suggests the applicability of Langmuir adsorption model. The values of  $q_m$  slope increased with the rise of temperatures, indicating the sorption processes are endothermic in nature. The values of  $b$  intercept increased slightly with increasing temperature. Low values of parameter  $b$  indicate that *Pericopsis elata* absorbent have high affinity for Chromium and cadmium. As shown in figure 3;



**Figure 3** Langmuir isotherms graph

The Freundlich isotherm is based on multilayer adsorption on the heterogeneous surface of the adsorbent containing an unequal amount of energies, the model represented below, Where  $Q_e$  (mg/g) is the amount of adsorbate that is adsorbed per unit mass of the adsorbent and  $C_e$  (mg/L) is the equilibrium concentration.  $K_f$  is the Freundlich constants

related to the adsorption capacity and  $n_f$  is the adsorption intensity, which varies with the heterogeneity of the material.



**Figure 4** Freundlich isotherm graph

Applying Freundlich model, a linear plot of  $\log Q_e$  versus  $\log C_e$  was plotted and it gives the slope of  $n_f$  and intercept  $K_f$ . The constant  $K_f$  is the relative adsorption capacity of the adsorbent and  $n_f$  is the sorption intensity. The value of  $1/n$  obtained was 1.727 which lies in the range of 1 to 10, therefore, it can be concluded that *Pericopsis elata* wood waste is a good multilayer adsorbent with good adsorption capacity according to Freundlich model as reported by [11].

#### 4 Conclusion

This study shows that *Pericopsis elata* adsorbent removed heavy metals in waste water and the adsorption was done on the  $-OH$  bending of water. SEM results showed the surface morphology feature of the adsorbents which is an indication that important interaction occurred between the adsorbate and adsorbent granule interface in the experimental conditions. The X Ray Fluorescence results revealed that carbon has the highest concentration (89.39%), followed by oxygen (6.56%) while the least was recorded in Sulfur as 0.03%. This is an evidence that the *Pericopsis elata* raw material are made up of cellulose compound. The Langmuir and Freundlich isotherm provided information on the capacity of the adsorbent which indicates that an important interaction occurred between the adsorbate and adsorbent-granule interface.

#### Compliance with ethical standards

##### Acknowledgments

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##### Disclosure of conflict of interest

The authors declared no conflict of interest.

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