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Removal Of Heavy Metals Using Activated Carbon Prepared from Coconut Shells as Adsorbent

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ABSTRACT

This study explored the use of activated carbon prepared from coconut shells for the removal of heavy metals in battery wastewater. Battery waste water is an industrial effluent that contains heavy metals such as lead, copper, iron and manganese. The characterisation of the water showed concentrations less than 5 mg/L for each of the metal ions. Generally, heavy metal ions are toxic even at low concentrations, hence this study. Two key parameters were studied and the trend showed that manganese was removed at a higher percentage than all the other metal ions and this can be attributed to its small atomic radius leading to it occupying more sites faster. Five gram was chosen as the optimum dosage for metal ion removal in battery water while 180 minutes was required for contact between the adsorbent and adsorbate. The pseudo first order kinetic model and the Langmuir isotherm both fitted well the kinetic data and the values of RL in the Langmuir indicated a favourable sorption. The mechanism of adsorption was found to be physical because most metal ions in the study achieved a removal of more than 50% without requiring any activation energy.

KEYWORDS

Adsorption
Activated Carbon
Battery Wastewater
Coconut Shells
Heavy Metals

1. INTRODUCTION

Heavy metals present in the environment, particularly those originating from industrial effluents, pose significant challenges due to their detrimental impacts on human and aquatic life. Various heavy metals are commonly found in industrial wastewater and must be reduced to their threshold limit values (concentration) before disposal to mitigate environmental effects. Wastewater from industries such as paint manufacturing, battery production, mining, and metal plating often contains these heavy metals (Kulkarni, 2020). This issue is a major concern for researchers and environmentalists because it significantly contributes to the metal load in natural ecosystems (Mishra *et al.*, 2019). The proper disposal of effluents containing heavy metals is a pressing concern for governments, industrialists, and environmentalists alike (Ilyas *et al.*, 2019). High concentrations of heavy metals can have harmful environmental effects and pose potential risks to human health. For example, lead can cause cognitive impairment, kidney damage, anaemia, and reproductive system harm. Cadmium exposure at high levels can lead to nephrotoxic effects and bone damage after prolonged exposure. Additionally, copper has been linked to body weakness and other health issues (Fu *et al.*, 2020).

Effluent from battery manufacturing industries is a notable source of heavy metals. Wastewater from these industries contains toxic heavy metals that can pollute the environment, including lead, mercury, manganese, iron, cadmium, and chromium (Ahmed and Ahmaruzzaman, 2016; Lekgoba *et al.*, 2020; Ribeiro *et al.*, 2018). The discharge of heavy metals in battery wastewater has raised significant concerns among the public. Therefore, removing heavy metals from this wastewater is crucial to mitigate their harmful effects. Techniques such as ion exchange, reverse osmosis, precipitation, ultrafiltration, and electrodialysis are employed to reduce dissolved heavy metals in effluent (Chaemiso and Nefo, 2019). However, these methods are energy-intensive and require advanced operations, which can be inaccessible or unaffordable for low-income countries.

Recently, research has focused on cost-effective methods for metal removal. Efforts have included using inexpensive and readily available agricultural and forest wastes, such as coconut shells, rice husks, orange peels, corn cobs, sawdust, and peanut husks, as adsorbents to remove heavy metals from wastewater (Solangi *et al.*, 2021; Chaemiso and Nefo, 2019). Adsorption has emerged as a prominent and cost-efficient method for removing heavy metals from wastewater due to its simplicity, effectiveness, and the ready availability of materials to prepare the adsorbent (Hussain *et al.*, 2021; Ahmed and Ahmaruzzaman, 2016). This technique is advantageous for separation and purification because of its low energy consumption, design flexibility, material availability, and the possibility of recycling used adsorbents (Adam *et al.*, 2022; Hussain *et al.*, 2021). The adsorption capacity of an adsorbent is influenced by its surface area and pore size distribution, as the former affects the dissolution rate and the latter affects diffusion rates (Suresh Kumar *et al.*, 2019).

Adsorbents like activated carbon and zeolite are typically designed with specific pore size distributions for particular separation techniques. This paper investigates the use of activated carbon derived from coconut shells to remove heavy metals from battery wastewater. It explores the impact of varying adsorbent dosage and contact time, details the processes of carbonization and activation, characterizes the developed carbon adsorbent, and analyses the removal capacity of heavy metals along with the relevant kinetic models and isotherms.

2. MATERIALS AND METHODS

2.1. Materials

The raw material used in this research work was coconut shell and this was collected from Kure market Minna, Niger state. The sulphuric acid and distilled water were purchased from Panlac chemicals store Minna, Niger State. Effluent from a lead-acid battery from Company X, Industrial Layout, beside ITC Tobacco Co. Ltd, Ilorin, Kwara state, Nigeria.

2.2. Equipment

A digital pH meter (model: C.B 256015) was used to measure the pH of the solution and was calibrated using buffer solutions. A Memmart 600 Electric oven was used to dry the coconut shells in preparation for their crushing and grounding to finer particle sizes.

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The crushed and grounded particles were sieved using a laboratory test sieve (1 mm mesh size). Carbonisation of the powdered coconut shells was done in a Muffle furnace and to check the surface area of the adsorbent, BET analysis was done using a Quantachrome NOVA 4200e. An LS2-3016R mechanical shaker was used to shake the samples for good contact between the battery wastewater and activated carbon. Concentration of heavy metals in the filtrates was measured using Bulk Scientific AAS Modelis Accusys 211.

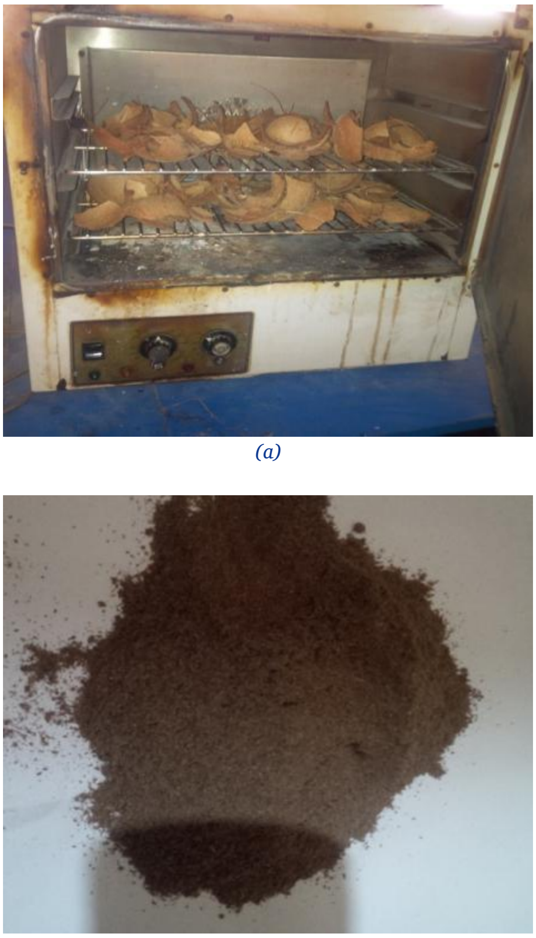
2.3. Preparation Of Activated Carbon from Coconut Shells

The procedure followed for the preparation of the activated carbon from coconut shells was similar to that conducted by (Bernard *et al.*, 2013).

The steps include washing and oven drying the coconut shells at 130 ˚C to reduce moisture content, followed by calcination of the powdered shells at temperatures of 500 ℃, 600 ℃, and 700 ℃ to optimize the production process of the activated carbon, ensuring that the final product is highly efficient in removing heavy metals from the battery wastewater and then activating each sample using 1 molar (1M) solution of sulphuric acid.

2.3.1. Dehydration, Crushing and Grounding

The coconut shell collected was washed with ordinary tap water, sundried for two days and further oven dried at a temperature of 130 ˚C for 4 hours to ensure the moisture has been removed. The dried shell was crushed, ground and sieved to obtain 1.0 mm finer dust particle size (Figure 1). It was then placed in airtight plastic bag to prevent re-absorption of moisture from the atmosphere.



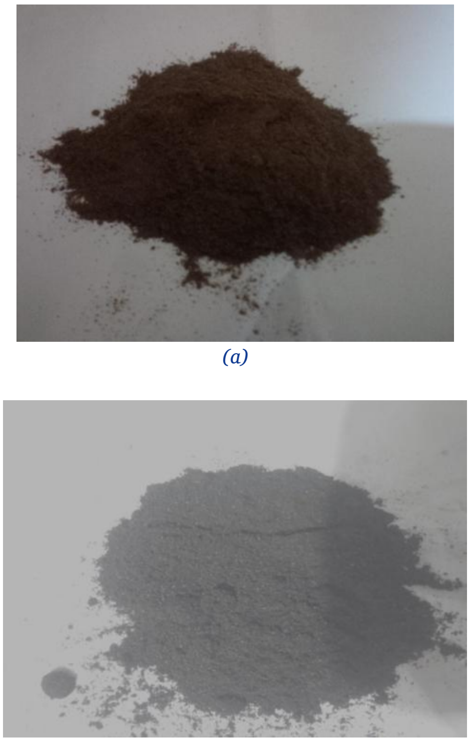
(a)

(b)

Figure 1: Coconut shell samples used in the study in (a) pre-processed state (b) ground state

Carbonisation (Calcination)

Powdered coconut of 135.17 g was placed in the furnace for carbonization at a specified temperature (Figure 2). The furnace temperature was set at 500 ˚C, 600 ˚C and 700 ˚C for periods of 30, 60 and 90 minutes respectively. This is to get rid of inorganic volatile matter in the precursor leaving behind the more stable carbon and ash forming minerals.



(a)

(b)

Figure 2: The Calcination process showing (a) Sample before calcination (b) Sample after calcination

2.3.2. Activation

After carbonization, the samples were activated with 100 mL of 1M of sulphuric acid each and allowed an activation time of 24 hours after which it was washed with distilled water to remove excess activating agent from the carbon surface until the pH of the mixture of water and the sample was within 6.0 and 7.0, measured using a pH meter. The sample was re-introduced into the oven at a temperature of 100℃ for 30 minutes and then preserved in a sample container where it was ready to be used for the adsorption process. Figure 3 shows the sample after activation.

Figure 3: Sample after activation (activated carbon)

2.4. Characterisation of the Activated Carbon

2.4.1. *BET Surface Area Analysis*

In BET surface area analysis, nitrogen is usually employed due to its availability and its strong interaction amid some solids. Properly calibrated and accurate pressure transducers examine the pressure changes due to the process of adsorption. After the formation of adsorption layers, the sample is removed from the nitrogen atmosphere and heated to cause the adsorbed nitrogen to be free from the material. The collected data is displayed in the form of a BET isotherm, which plots the amount of gas adsorbed as a function of the relative pressure. The Langmuir theory relates the monolayer adsorption of gas molecules (Swenson and Stadie, 2019), also called adsorbents, onto a solid surface to the gas pressure of an average above the solid surface at a predetermined temperature.

2.4.2. *Determination of Moisture Content*

Thermal drying technique was used to determine moisture content of the samples. Percentage moisture content (%MC) was obtained using the formula in Equation 1

$$Moisture\ content = \frac{loss\ of\ weight\ on\ drying}{initial\ sample\ weight} \times 100 \qquad [1]$$

2.4.3. *Determination of Ash Content*

Ash content is given as

$$Ash\ content = \frac{W_i - W_f}{W_i} \times 100 \qquad [2]$$

where W_i and W_f represent the initial and final crucible weight and sample respectively.

2.5. Adsorption Study

The experiment was carried out by measuring 100 mL of battery waste water and pouring it in a 250 mL conical flask. Different doses (3 – 6 g) of the produced activated carbon were tested by adding them to the battery waste water to determine the optimum dosage needed. Batches of solution and adsorbent were also run at different contact times (0 – 300 minutes). The conical flask containing the adsorbent and battery waste water was positioned in the mechanical shaker operated at room temperature (32 0C) and speed of 250 rpm to ensure equilibrium or stability as shown in Figure 4. The suspension obtained was filtered using Whattman a filter paper and the Flame Atomic Absorption Spectrophotometer (AAS) was used to analyse the amount or concentration of the different heavy metals present in the filtrate.

Concentrations or amount of heavy metal absorbed by the adsorbent was evaluated using Equation 3:

$$qt = \frac{(Co - Ct)v}{w} \qquad [3]$$

where C_o and C_t represent the initial and final concentration of the heavy metals present in battery waste water before and after adsorption at time t respectively, and C_e represents the concentration of heavy metals in battery waste water (mg/l) when stability was attained.

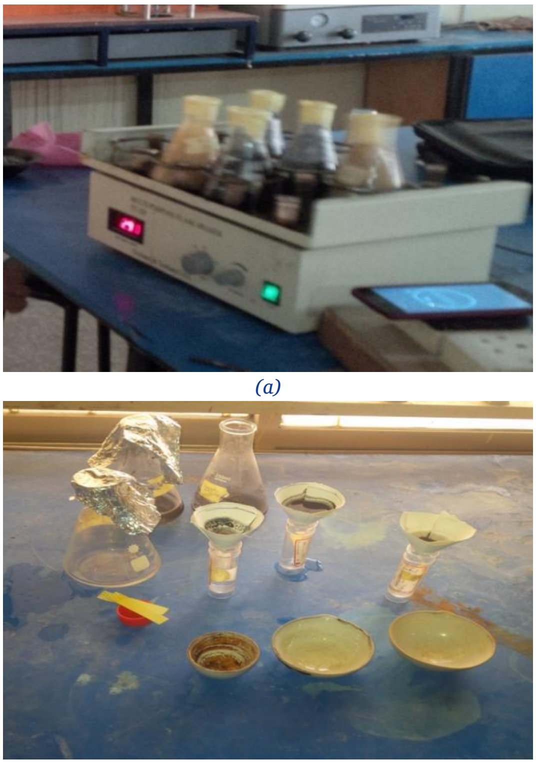
Volume of the battery waste water used is represented by V (ml), and W represents the mass (g) of the adsorbent used. The percentage of metals removed was obtained using Equation 4.

$$R\% = \frac{(C_o - C_i)}{C_i} \times 100 \qquad [4]$$

where (R%) is the ratio of difference in metals concentration before and after adsorption.

2.6. Adsorption Kinetic Models

The progress of adsorption with time is better explained by the adsorption kinetics. Reactions are generally described by the order and the rate of reactions at which it is occurring is described by these models. The models that explain the rate kinetics are the Pseudo first order, Pseudo second order and the intra – particle diffusion.


(a)

(b)

Figure 4: The sample preparation process, showing (a) Samples in a mechanical shaker (b) Filtration process of the samples

2.6.1. *Pseudo First Order Model*

The linear expression of the pseudo first order model is given by:

$$\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \qquad [5]$$

where q_e and q_t are the amount of metal adsorbed (mg/g) at equilibrium and at any time, t and k1 (min-1) is the first order rate constant.

2.6.2. *Pseudo Second Order Model*

The pseudo second order describes the kinetics through the following expression:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \qquad [6]$$

where q_e and q_t are the amount of metal adsorbed (mg/g) at equilibrium and at any time, t and k2 (g/mg.min) is the second order rate constant.

2.6.3. *Intra – Particle Diffusion Model*

The intra – particle diffusion model was proposed by Webber and Morris (1963). The plot of qt versus t1/2 should result in a straight line passing through the origin then the model is followed otherwise film diffusion plays and important role during the adsorption process. The model is given by the following equation:

$$q_t = k_{ID} t^{1/2} + I_D \qquad [7]$$

where qt is the amount of metal adsorbed (mg/g) at at any time, t, k_{ID} (mg/g.min-1/2) is the intra – particle diffusion rate constant and ID is the intercept through which the line passes.

2.7. Adsorption Isotherms

Adsorption isotherms analyse the adsorbate concentration in the solution and the amount adsorbed by a specific mass of adsorbent. They determine the adsorption capacity of the adsorbent and depend upon the nature and type of system.

2.7.1. Langmuir Model

The model is assumed to be applicable to monolayer adsorption and the sites are homogenous with equal affinity towards the adsorbate. The linearized equation of the model is given by:

$$\frac{C_e}{q_e} = \frac{1}{q_o b} + \frac{C_e}{q_o} \qquad [8]$$

where C_e is the equilibrium concentration (mg/L), q_e is the amount of adsorbed per unit mass of adsorbent at equilibrium (mg/g). q_o and b are the Langmuir constants related to the adsorption capacity and the energy of adsorption, respectively.

2.7.2. Freundlich Model

The model is applied for heterogeneous surfaces and the logarithmic form of the model is expressed by:

$$\log q_e = \log k_f + n \log C_e \qquad [9]$$

where kf and n are the Freundlich constants related to the adsorption capacity and the intensity, respectively.

3. RESULTS AND DISCUSSION

3.1. Pore Structure Characterisation – Activated Carbon

Tables 1 to 3 show the BET results obtained for the surface area, pore volume, and pore size of raw coconut shell and activated carbon produced from coconut shell at 500˚C, 600˚C, and 700˚C. The untreated samples showed a lower BET surface area but a larger pore size, which is important because it affects the dissolution rate and adsorption capacity (Suresh Kumar *et al.*, 2019). The BET method with the highest specific surface area of 10110.0 m^2/g (Table 1) was obtained at a temperature of 700˚C using the Langmuir method. This method proposes a monolayer adsorption approach (Foo and Hameed, 2010; Kapur and Mondal, 2014). The presence of micropores contributed significantly to the increased surface area. The pore size distribution affects the diffusion rate and the surface area per unit volume (Suresh Kumar *et al.*, 2019). The pore volume and pore size were higher at 157.1 cc/g and 6.399 nm, respectively (Tables 2 and 3), as determined using the Dubinin-Radushkevich (D-R) method.

Table 1: Surface area (m^2/g) for the raw coconut shell and the activated coconut shell at different temperatures

		Activation Temperature (°C)		
Surface Area Method (m^2/g)	Raw Coconut Shells (25 °C)	500	600	700
Single point BET	261.0	672.1	1640.0	1721.0
Multiple point BET	356.9	1138.0	1750.0	2083.0
Langmuir	925	9984.0	3135.0	10110.0
DR method micro pore area	442.0	1204.0	2553.0	2654.0
DFT cumulative surface area	106.5	258.8	294.84	520.4
BJH method cumulative adsorption	446.0	1247.0	1795.0	2348.0

NB: rtp = room temperature and pressure

The D-R method is commonly used for characterizing microporous materials and assumes a Gaussian distribution of pore energies. It provides insights into the adsorption mechanism, favouring a monolayer adsorption process (Benzaoui *et al.*, 2018). In addition, methods like the Density Functional Theory (DFT) and Barrett-Joyner-Halenda (BJH) are commonly employed for pore structure analysis. The DFT method uses a statistical thermodynamic approach to model pore size distributions based on adsorption isotherms, providing detailed information on micro- and mesopores. The BJH method, on the other hand, is suitable for analyzing mesoporous materials, focusing on pore size distributions within the 2–50 nm range. The range of particle pore sizes in this study, between 2–50 nm, indicates that the particles are mesoporous (Suresh Kumar *et al.*, 2019). These high BET values indicate a highly developed pore network within the carbon. The increase in surface area is attributed to the creation of new micropores formed during carbonization.

Table 1:Pore volume obtained for the raw coconut shell and the activated coconut shell at different temperatures

		Activation Temperature (°C)		
Pore Volume (cc/g)	Raw coconut shells (25 °C)	500	600	700
DR method	157.1	42.79	90.73	83.65
DFT method	11.74	31.40	78.62	62.61
DH method cumulative adsorption	21.98	63.43	92.78	1.192
BH method cumulative adsorption pore volume	21.47	62.03	9.039	1.168

Table 2: Values of the pore size for raw coconut shell and activated coconut shell at different temperatures

		Activation Temperature (°C)		
Pore Size (nm)	Raw coconut shells (25 °C)	500	600	700
DR method adsorption pore diameter (mode dv (d))	5.644	6.399	4.145	6.256
DFT pore diameter mode	2.647	2.647	2.769	2.647
DH method adsorption diameter (mode dv (d))	2.121	2.141	1.925	2.128
BJH method adsorption pore diameter	2.121	2.141	2.259	2.128

3.2. Battery wastewater characterisation

Battery water was characterised to identify the present of heavy metals and it was found that it contained more Iron (Fe) content at 4.68 mg/L while Manganese (Mn) was the least at 0.65 mg/L. Table 4 presents the heavy metal concentrations before adsorption.

Table 3: Concentrations of heavy metals in the battery wastewater before adsorption

Heavy metal	Concentration (mg/L)
Copper	0.75
Lead	1.50
Manganese	0.65
Iron	4.68

3.3. Batch Adsorption Experiments

The effects of two parameters (adsorbent dosage and contact time) on the percentage of lead, manganese, copper and iron adsorbed was investigated by varying one parameter while keeping all the others constant. Concentration of heavy metal remaining in solution was then determined.

3.3.1. Effect of contact time

The relationship between the contact time and removal percentage of heavy metals from battery waste water using activated carbon prepared from coconut shell is shown in Figure 5. The effect of the contact time was studied at room temperature (32oC) at 60 minutes interval. From the result obtained, it was shown that heavy metals removal increases as the contact time increases. Lead (Pb), copper (Cu), manganese (Mn), and iron (Fe) were removed using the adsorbent prepared. The percentage of heavy metals removed approached equilibrium at 180 minutes for lead (Pb), 240 minutes for iron (Fe) and manganese (Mn) and 180 minutes for copper (Cu) with percentage removals of 13.2%, 44.6%, 50.2% and 75.88% for lead, iron, copper and manganese respectively. The removal trend shows that a higher percentage of manganese is removed compared to other metal ions (Pb<Fe<Cu<Mn). Figure 5 shows the effect of contact time on adsorption of heavy metals.

3.3.2. Effect of adsorbent dosage

Experiment was carried out with adsorbent dosage of 3, 4, 5 and 6 g per 100 mL of battery wastewater When the adsorbent dose increased, the percentage removal of the metals also increased as shown in Figure 6. The percentage adsorption of lead, manganese, iron and copper increased due to an increase in the number of binding sites which is in line with the study of (Mbugua *et al.*, 2014). Generally, an increase in adsorbent dosage should result in an increase in the percentage removal of all the metal ions. Lead, iron and manganese reached equilibrium or their maximum removal at 5 g with percentage removals of 50%, 50.86% and 91% respectively while copper attained a maximum removal at 4 g with 86.67%. A further increase in the dosage brought no increase in adsorption which is a result of overlapping of adsorption or binding sites due to excess numbers of adsorbent particles (Benzaoui *et al.*, 2018). The removal trend indicates that a higher percentage of manganese is removed compared to other metal ions (Pb<Fe<Cu<Mn). Hence 6 g was chosen as the optimum adsorbent dosage for the removal of lead, manganese, copper and iron and was used for further investigation of the work.

3.4. Adsorption Kinetic Models

The pseudo first order kinetic model and the pseudo second order models were tested to identify the mechanism of adsorption for the four metal ions that were adsorbed using coconut shells activated carbon. With reference to Table 5, three (3) of the metal ions being

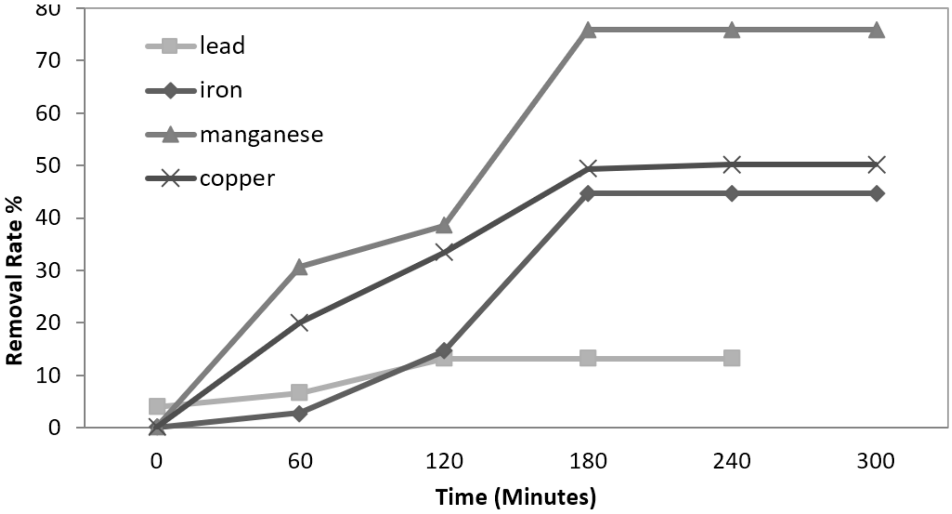


Figure 5: Effect of contact time on adsorption of heavy metals

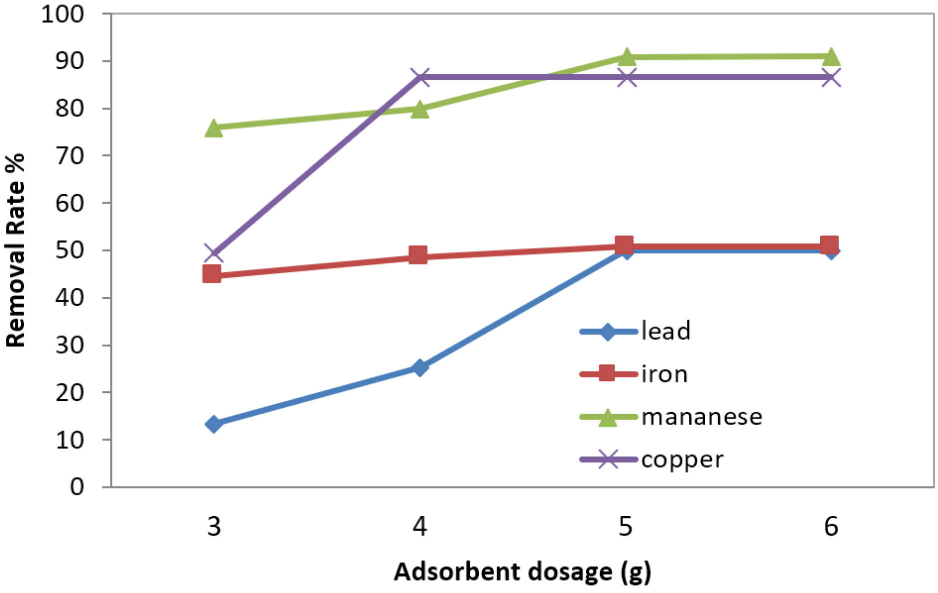


Figure 6: Effect of adsorbent dosage on percentage removal of heavy metals

Table 5: Summary of the kinetic models

Metal ion		Lead	Iron	Manganese	Copper
Pseudo First Order	q_e (mg/g)	0.0346	1.3589	0.0807	0.0459
	k_1 (min^{-1})	0.0263	0.0392	0.0267	0.0246
	R^2	0.826	0.802	0.791	0.886
Pseudo Second Order	q_e (mg/g)	0.0207	0.0282	0.0335	0.0217
	k_2 (g/mg. min)	1.63E-08	6.50E-08	1.40E-07	5.58E-08
	R^2	0.477	0.530	0.667	0.911
Intra-Particle Diffusion	K_{id}	0.0006	0.0078	0.0012	0.0009
	I_D	0.0021	0.0536	0.0029	0.0010
	R^2	0.874	0.857	0.844	0.892

Lead, Iron and Manganese fitted well with the pseudo first order at R2 values of 0.826, 0.802 and 0.791 respectively while copper fitted well with the pseudo second order at R2 = 0.911. The pseudo first order suggests that a system requires low to no activation energy and proposes that the mechanism of adsorption is physical adsorption (physisorption). Intra – particle diffusion plays a larger role in the system because the values of ID are very close to zero (Bouhamed *et al.*, 2012; Kapur and Mondal, 2014).

3.5. Adsorption Isotherms

The coefficient of correlation (R2) was used to determine the models and isotherms that best fit the system and the best fit is determined by how close the R2 is to unity. Table 6 Lead, Manganese and Copper fitted well with the Langmuir isotherm with R2 values of 0.9478, 0.9208 and 0.998 respectively while Iron fitted well with the Freundlich at R2 = 0.876. The Langmuir isotherm suggests a monolayer approach and homogenous surfaces in the adsorption sites while the Freundlich proposes a multilayer approach and heterogeneous systems. In the Langmuir system, there is no interaction between the adsorbed species and that proposes a chemical adsorption mechanism (chemisorption) (Foo and Hameed, 2010; Kapur and Mondal, 2014). The fitting of the Langmuir is also validated by the values of RL which are in the ranges of 0 < RL < 1, and according to Foo and Hameed (2010) this shows a favourable sorption.

Table 6: Summary of the Kinetic Isotherms

Metal ion		Lead	Iron	Manganese	Copper
Langmuir Isotherm	q_o (mg/g)	4.248	17.889	19.920	12.594
	b	1.885	0.310	20.917	198.5
	R_L	0.547	2.229	0.069	0.007
	R^2	0.9478	0.7402	0.9208	0.998
Freundlich Isotherm	n	0.834	0.247	3.032	17.730
	K_f	2.699	1.195	4.247	2.941
	R^2	0.9126	0.876	0.8523	0.1038

4. CONCLUSION

In conclusion, the research demonstrated that the capacity of the adsorbent depends on both contact time and adsorbent dosage. The study found that the adsorbent's capacity increased with contact time, indicating a proportional relationship until equilibrium was reached, suggesting the saturation of the adsorbent's pore spaces. Additionally, increasing the dosage of the adsorbent enhanced its capacity, meaning the amount of adsorbate also increased. The dominant mechanism of adsorption in this study appeared to be physical adsorption, as three of the metal ions followed the pseudo-first-order model, and the correlation coefficients for both the Langmuir and Freundlich isotherms were close, indicating the system could follow either isotherm. However, copper showed a tendency to follow a multilayer approach through the pseudo-second-order model and Langmuir isotherm, which typically describes chemical adsorption.

Declaration Of Competing Interest.

The authors report no declaration of interest.

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