

The Use of Nanoparticle as Absorbent in the Remediation of the Physico-Chemical Properties of Crude Oil and Waste Water Contaminated Environment

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Abstract: In order to remove lead from an aqueous solution taken from industrial effluent and soil contaminated by crude oil, the study used snail shell nanoparticles, a plentiful and inexpensive resource. The adsorbent was described. The adsorbent was subjected to Fourier transform infrared (FTIR) examination. The morphological properties of the adsorbent were examined using a scanning electron microscope (SEM). Experimental data was optimization using Mathematica software 11.0.2.1. Temperature, contact time, and concentration were examined. Thermodynamics, kinetics, and equilibrium adsorption isotherms were examined. Langmuir, Freundlich, and isotherm models were used to assess the experimental data. Pseudo-first order, pseudo-second order, and intra-particle models were fitted to the kinetics data. The adsorption isotherm developed fits the Freundlich isotherm model well, according to the results, with the maximum correlation coefficient (R²) of 0.966. According to the thermodynamic study, the lead removal process by the snail shell nanoparticles was feasible, exothermic, and spontaneous. These findings suggest that lead can be successfully removed from aqueous solutions using snail shell nanoparticles as an adsorbent.

Keywords: Nanoparticle, Snail Shell, Lead Adsorption, Waste Water, Crude Oil Remediation.

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I. INTRODUCTION

Traces of heavy metals have been found in environments with a history of crude oil spills and industrial wastewater deposition. These heavy metals, which are contaminants, have posed a lot of unbearable effects on natural environmental activities. These heavy metals include cobalt, cadmium, iron, copper, lead, etc. Researchers in science and engineering have taken time to identify traces of these toxic elements, their effects on the ecosystem, and the various means of remediation from the environment. It is known that environmental heavy metals can contaminate biological populations, including humans, animals, and plants, both acutely and over time [1, 2]. Natural soil erosion, volcanic eruptions, atmospheric precipitation, and wastewater discharge from a variety of industries, such as melting, plating, plastics, photography, tanning, and the

production and consumption of materials containing metals and dyes, are some of the ways that these heavy metals enter the natural ecosystem [3]. One of the most dangerous heavy metals for aquatic and human contamination is lead, which may be removed by adsorption, which is a practical way to restore the health of ecosystems.

The properties and removal of cadmium (Cd) by powdered golden apple snail shell and related mechanisms were investigated. And findings demonstrated that the amount of Cd removal complied with the Langmuir model and that the Cd removal efficiency from the solution grew gradually and achieved an equilibrium state as the concentration, pH, temperature, and contact time of shell powder increased significantly. Additionally, the thermodynamic analysis indicated that the powdered golden apple snail shell was an endothermic, spontaneous process for

the elimination of Cd. The quantity of powdered golden apple snail shell then had a significant impact on the pH solution [4] (Zhao et al., 2016). A research work that used chitosan derived from snail shell (SSC) for adsorption in order to remove Pb^{2+} from wastewater. The study employed the intraparticle diffusion, pseudo-second order, and pseudo-first order models. According to their findings, the adsorption kinetic data was best represented by the pseudo-second-order model. The thermodynamic characteristics showed that the adsorption of lead into SSC was exothermic and non-spontaneous, according to additional analysis of their findings [5] (Oyedeko et al., 2025). Another study found that calcining golden apple snail shells at 1050°C could remove up to 30% of copper (II) ions, 88% of cadmium (II) ions, and 56% of lead (II) ions at pH 6. A stable pseudo-second-order reaction equation was used to remove the metal, and the adsorbing material offered two benefits: a lesser volume of golden apple snail shell and an affordable adsorbent [6] (Ketwong et al., 2018). An analysis comparing the use of periwinkle and activated snail shells for the extraction of heavy metal ions from aqueous solutions. According to the data, activated carbon derived from periwinkle shells had higher porosity, pore volume, pH, and moisture content than activated snail shells. Using both adsorbents, the ideal adsorbent dose and contact time for removing these ions were noted and observed, respectively. The pseudo-second-order kinetic model showed good conformity with the adsorption kinetics data [7] (Nwajei et al., 2023). In laboratory continuous shaking studies, the use of raw and acid-pretreated bivalve mollusk shells (BMSs) to extract metals from aqueous solutions containing a single or combined metal was assessed at several BMS dosages, pH levels, and temperatures. Ultimately, the removal efficiencies (REs) of the raw BMSs for Fe, Zn, and Cu were reported when the BMSs were employed to treat electroplating wastewater, while the REs of the BMSs that had been acid-pretreated were also disclosed [8] (Liu et al., 2009). Lead, cobalt, and copper ions were extracted from synthetic and industrial effluent using a bioadsorbent powder made from Solamen Vaillant snail shell. Additionally, the analysis took into account the impact of many characteristics, including temperature, pH, and contact time. The findings show that the optimal adsorption efficiency for cobalt, lead, and copper pH was determined. Additionally, the temperature and contact time were shown for an initial ion concentration and dose of the adsorbent. The Langmuir model yielded the highest adsorption capacity values for lead, cobalt, and copper, respectively. The exothermic, spontaneous, and practicable nature of the adsorption process was further demonstrated by the thermodynamic parameter values [9] (Esmaili et al., 2020). A recent study used snail shell dust as a biosorbent to remove heavy metals like copper, nickel, and lead from artificially made pertinent heavy metal solutions. And by examining a number of variables, including contact time, adsorbent doses, adsorbent size, test solution pH, and beginning heavy metal content, the effectiveness of freshwater snail shell dust was calculated. The monolayer mode of the adsorption mechanism was reflected in the trends that followed the Langmuir isotherm, which was used for analysis. The maximum adsorptive capacities for nickel, copper, and lead were 28.95 mg/g, 29.14 mg/g, and 16.35 mg/g, respectively

[10] (Sharma and Davi, 2024). To determine how well the $Pb(ii)$, $Cd(ii)$, and $Ni(ii)$ ions in wastewater from pharmaceutical plants might be absorbed, carbonized *Archachatina marginata* was employed. Using DESIGN EXPERT Version 7.0.0 software, the adsorption efficiencies of these heavy metals were investigated using the Response Surface Method (RSM) and Central Composite Design (CCD). As the dosage of the adsorbent was raised, the percentage removal of $Pb(ii)$ and $Ni(ii)$ rose, but that of $Cd(ii)$ fell. As the temperature and time increased, so did the removal of all three [11] (Olanipekun et al., 2019). An alien freshwater snail (*Physa acuta*) was tested for its capacity to extract cadmium from tainted water using its shell dust (PSD). The tests' results show that PSD can be employed as an effective, affordable, and eco-friendly biosorbent for removing cadmium from aqueous solutions [12] (Hossain and Aditya, 2013). Cadmium was used as a model metal to evaluate the freshwater snail (*Lymnaea luteola*) shell dust's capacity for metal biosorption. An Artificial Neural Network (ANN) model was built for presenting the biosorption process under diverse situations. According to [13] Hossain et al. (2015), shell dust from the snail *L. luteola* (LSD), a waste biomaterial, may be utilized as an inexpensive and environmentally benign biosorbent to remove cadmium from aqueous solutions. In order to remove harmful heavy metals like $Pb(II)$ ions, the biosorptive ability of cylindrical paper shell mussel (*Anodontoides ferussacianus*) shell biomass has been examined in this study. The maximum amount of $Pb(II)$ removed was 200 mg/L. Sorbents have adjusted a number of physico-chemical parameters for the biosorptive capabilities of sorbates. Furthermore, several electronegative functional groups on the surface of *A. ferussacianus*' shell were revealed by Fourier Transform Infra-Red Spectroscopic (FTIR) research. These groups may provide the binding sites for the cations that are being studied [14] (Shahzad et al., 2017).

The objectives of this study is to use snail shell nanoparticles of *Archachatina marginata* species as an inexpensive adsorbent in the restoration of the physicochemical properties of industrial wastewater and crude oil-contaminated soil by the extraction of the toxic lead element of the Bonny Local Government Area, Rivers State, Nigeria. The impact of several factors, including temperature, pH, contact time, metal ion concentration, and adsorbent dosage, was investigated in order to accomplish this. Furthermore, the behavior of the adsorption process was examined using kinetic, equilibrium, and thermodynamic models.

II. MATERIALS AND METHOD

➤ Apparatus and Equipment

A digital weighing scale, a sample bottle, a conical flask, a measuring cylinder, filter paper, a beaker, a volumetric flask, a spatula, a funnel, a sieve, a water bath, an x-ray diffractometer, and a scanning electron microscopy machine were among the tools utilized in this work.

➤ Reagents

Samples of the chemicals used in this experiment were acquired from Nizeh Chemicals Ltd., located in Rivers State,

Nigeria, near Mile 3 Diobu, Port Harcourt. Nickel sulphate and lead nitrate, which are present in industrial waste water and environments contaminated by crude oil, were used to recover nickel and lead aqueous solutions. Deionized water was then used to dilute the stock solution to a predetermined concentration. The Federal Polytechnic of Oil and Gas Bonny, located in Bonny Rivers State, Nigeria, provided that information.

➤ Preparation of Sample

The snail shell of the *Archachatina marginata* species were obtained from big market bonny island, Bonny, Rivers State, Nigeria. They were then baked for 30 minutes at a temperature between 100°C and 110°C. To guarantee that the shells' surface was well coated with acid, 50g of shells were mixed with a precise volume of 95% phosphoric acid at a volume ratio of 1:10 in the following step. The temperature gradually increased to 400°C in less than two hours after the mixture was placed in the furnace. The sample was then calcined in a furnace set to 400°C for an hour. The furnace was then turned off and allowed to cool to room temperature. After that, the powder was cleaned until it had a pH of 6.5. The final product was again baked at 120°C for an hour to guarantee complete drying. After drying, the shells were crushed and ground into powder using an industrial crusher. To create a fine powder, the powdered shell was subsequently sieved via a 25–50 nm sieve.

➤ Preparation of Lead Solution

3.2 grams of lead nitrate Pb (NO₃) were dissolved to create 1000 parts per million of lead (Pb). The standard solution was serially diluted to create two in 100 milliliters of deionized water. Other solutions were serially diluted from the stock solution.

➤ Design of Experiment

Three independent factors were taken into consideration: contact duration, temperature, and concentration. The adsorption experiment was designed using central composite design, a subset and optimization tool of response surface technique, to investigate the effect of the independent variable on the adsorption process.

➤ Adsorption Experiments

• Variation of Time

Five separate 100ml beakers containing five grams of the adsorbent (powdered snail shell) were filled with 50ml of 100ppm, and the mixture was thoroughly mixed with a spatula. After letting the combination stand at 84.1°C for 10 minutes, it was filtered, and the filtrate's lead elimination was assessed using AAS. For 30 minutes, 60 minutes, 90 minutes, and 120 minutes, respectively, the aforementioned process was repeated.

• Variation of Concentration

Five grams of the adsorbent were weighed and placed into five beakers with varying concentrations of lead solution (100 ppm to 500 ppm). After being mixed and left to stand at 84.1°C for 101 minutes, the mixtures were filtered through filter paper.

• Variation of Temperature

By adding 50 milliliters of a 100-ppm concentration of lead solution to five separate beakers containing five grams of the adsorbent, the temperature was changed. The mixture was then stirred and left to stand at 26.4°C for 101 minutes before being filtered and the filtrate subjected to AAS analysis. For varying temperatures of 40°C, 60°C, 80°C, and 100°C, respectively, the same process was performed.

The amount of lead removed was determine with the below equation.

$$q = \frac{(C_i - C_f)}{w} \times v$$

Where q is the equilibrium amount of dye adsorbed in mg/g

v is the volume of the solution in ml

w is the mass of the lead used in grams

C_i and C_f are the initial and final concentration in g/ml respectively.

Efficiency of adsorption was determined using the equation below.

$$\%_{ads} = \frac{C_i - C_f}{C_i} \times 100$$

III. RESULT AND DISCUSSION

➤ Characterization

Fourier Transform Infra-Red (FT-IR) of Ground snail shell.

• Functional Group of Adsorbent

The FT-IR spectra can provide information about the chemical composition of the material. The result of the FT-IR is shown in Figure 1. They were interpreted and the observed functional group and corresponding intensities of absorbance are shown in table 1.

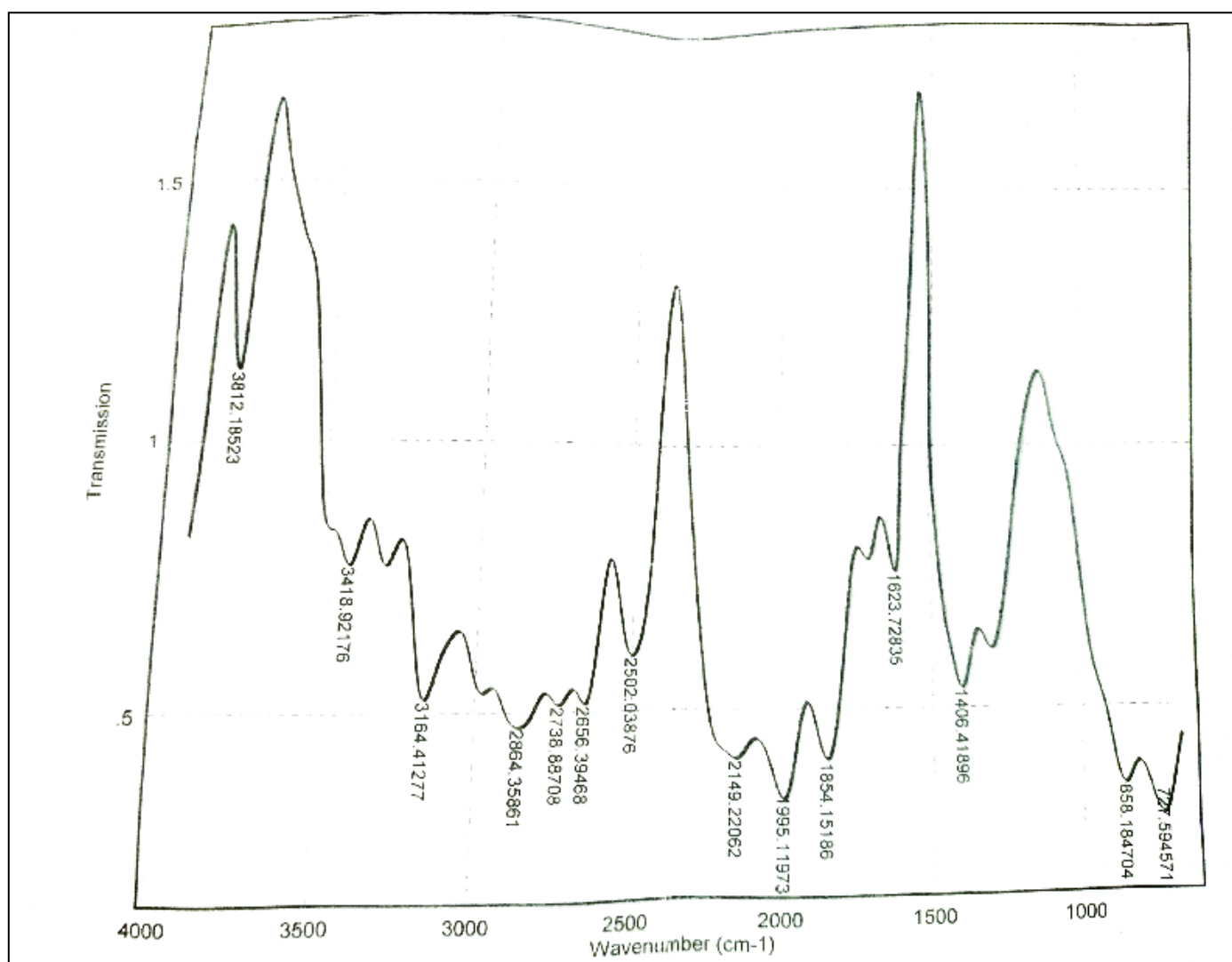


Fig 1 FT-IR Spectrum of Ground Snail Shell

Tabel 1 FTIR Interpretation of Ground Snail Shell

| S/N | Wave number(cm ⁻¹) | Functional group | Compounds |
|-----|--------------------------------|----------------------|---------------------|
| 1 | 727.5946 | C = C Bending | alkene |
| 2 | 858.1847 | | |
| 3 | 1406.419 | O - H Bending | Carboxylic |
| 4 | 1623.728 | C = C Stretching | Alkene |
| 5 | 1854.152 | C = O Stretching | Anhydride |
| 6 | 1995.12 | C = C = C Stretching | Allene |
| 7 | 2149.221 | C ≡ C Stretching | Akyne |
| 8 | 2502.039 | S - H Stretching | Thiol |
| 9 | 2656.395 | C - H Stretching | Aldehyde |
| 10 | 2738.887 | C - H Stretching | Aldehyde |
| 11 | 2864.359 | O - H Stretching | |
| 12 | 3164.413 | O - H Stretching | |
| 13 | 3418.922 | O - H Stretching | |
| 14 | 3812.185 | O - H Stretching | O-H Alcohol stretch |

Table 2 Isotherm Parameters for Lead Metal Removal

| Isotherm model | Equation | Slope | Intercept | R ² |
|----------------|--------------------|----------|-----------|----------------|
| langmuir II | y = 4.271x + 2.918 | 4.271 | 2.918 | 0.878 |
| Freundlich | y = 0.744x - 2.106 | 0.744 | -2.106 | 0.964 |
| Temkin | y = 0.018x + 0.069 | 0.018 | 6.90E-02 | 0.856 |
| DR | y = 2E-12x - 4.137 | 2.00E-12 | -4.137 | 0.164 |

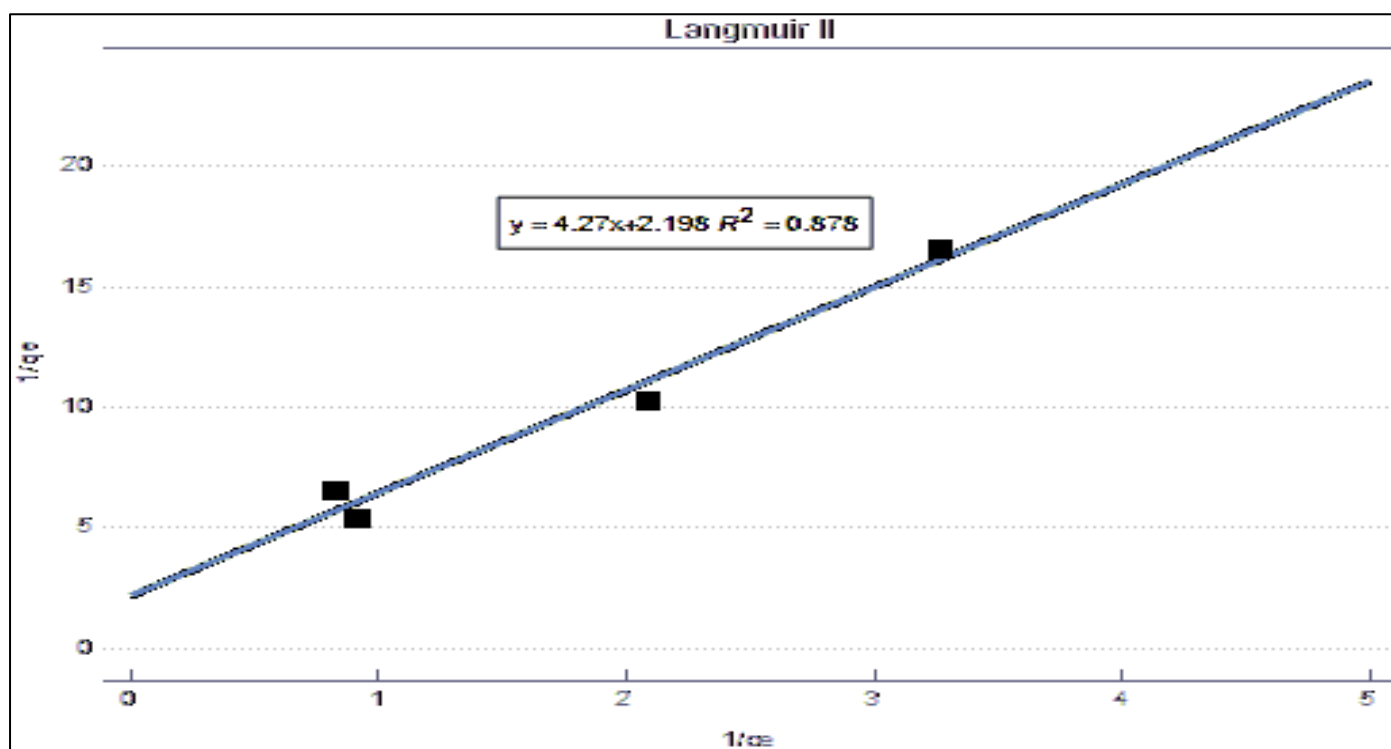


Fig 2 Langmuir (II) Isotherm for Lead Metal Adsorption

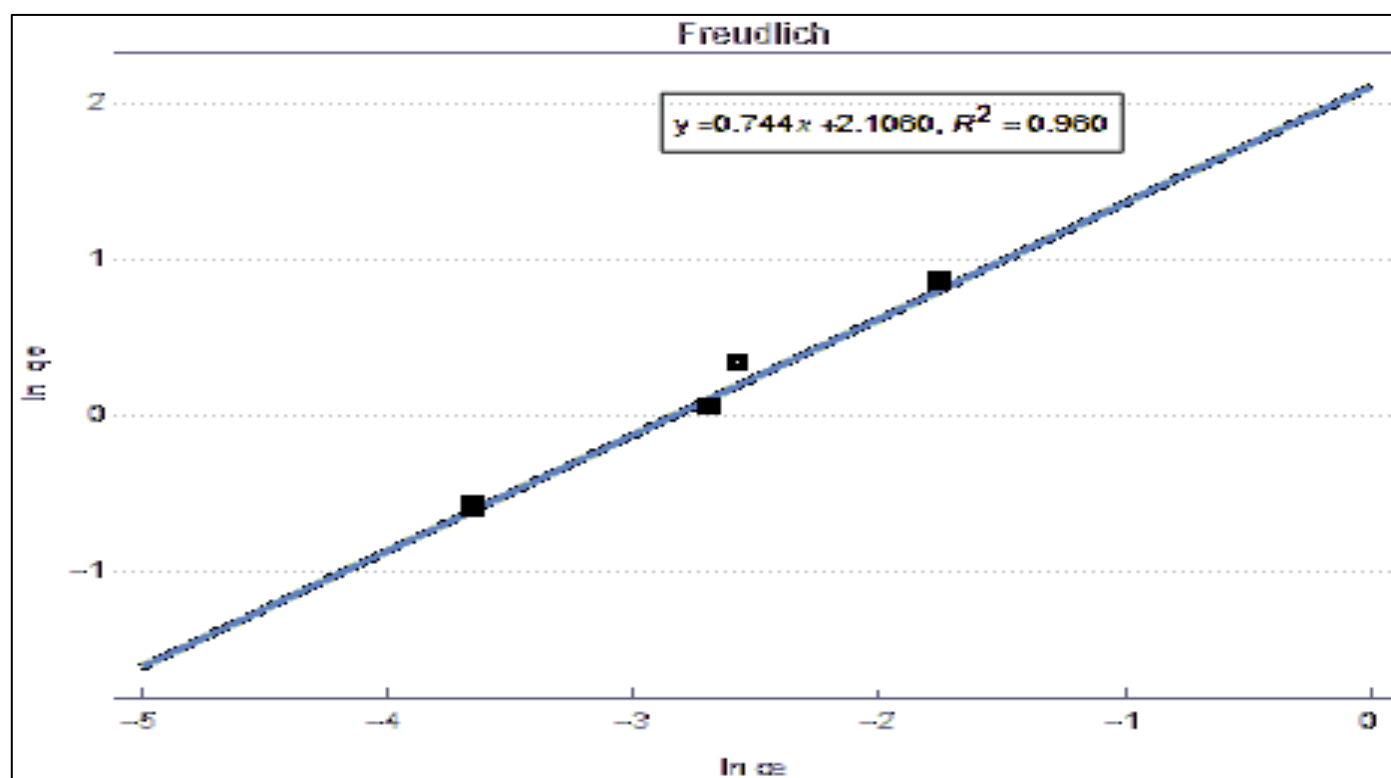


Fig 3 Fredlich Plot for Lead Adsorption

Table 3 Thermodynamic Parameter for Adsorption of Lead.

| S/N | Equation | R^2 | Temp (k) | $\Delta H(\text{J/mol})$ | $\Delta S(\text{J/mol/k})$ | $\Delta G(\text{J/mol})$ |
|-----|------------------|-------|----------|--------------------------|----------------------------|--------------------------|
| 1 | $-6251x + 7.912$ | 0.87 | 299.4 | -51970.814 | 65.780368 | -71665.46 |
| 2 | | | 313 | | | -72560.07 |
| 3 | | | 333 | | | -73875.68 |
| 4 | | | 353 | | | -73875.68 |
| 5 | | | 373 | | | -75191.28 |

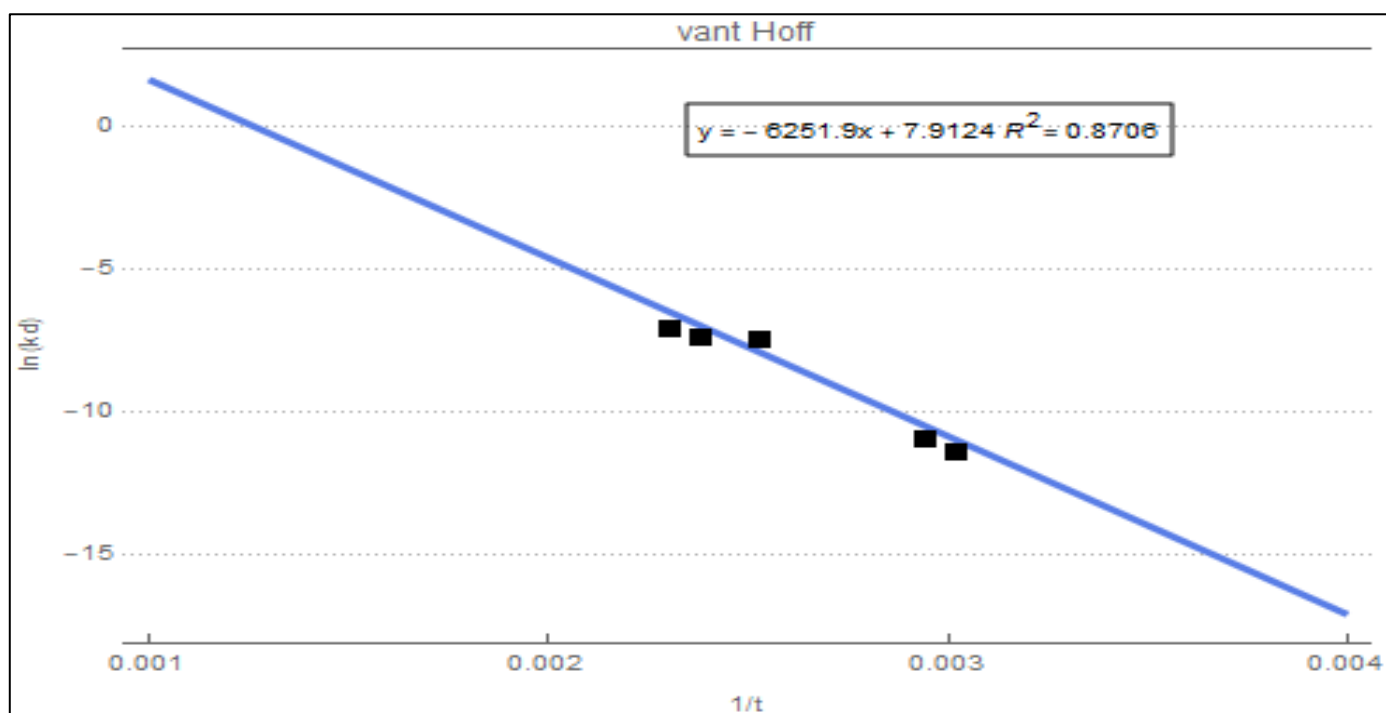


Fig 4 Vant Hoff Thermodynamic Plot of Lead Adsorption

Table 4 Kinetic Parameter of Lead Adsorption

| S/N | Kinetic | Equation | R^2 | Parameters |
|-----|--------------------------|------------------------|-------|---|
| 1 | First order | $y = 0.001x - 7.100$ | 0.614 | $q_e = -1.00E - 03$ $k_1 = 0.00081853$ |
| 2 | Second order | $y = 7.578x - 9321$ | 0.99 | $q_e = -8.13E - 04$ $k_1 = 0.13196094$ |
| 3 | Intra-particle diffusion | $y = 2E - 05x + 0.055$ | 0.648 | $k_{id} = 2.00E - 05$ $C = 0.055$ |

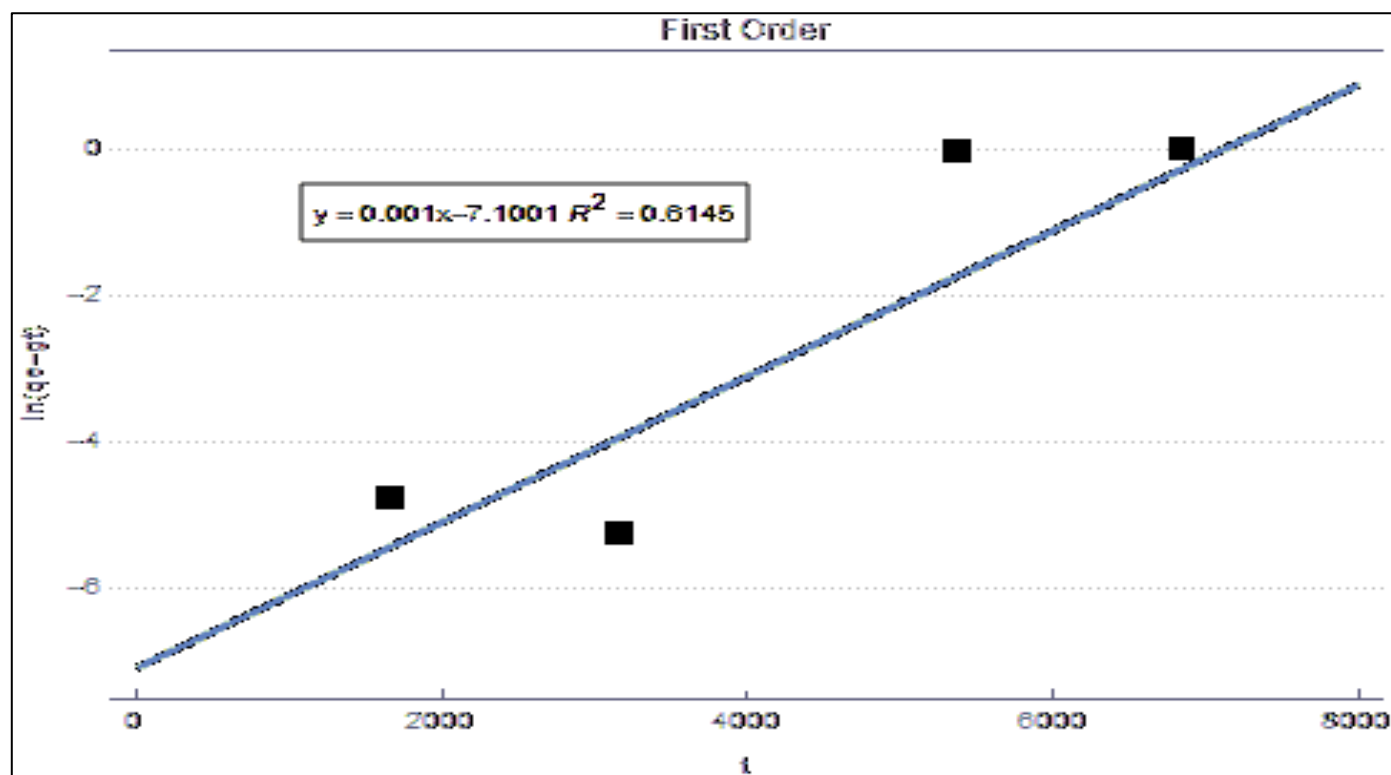


Fig 5 First Order Kinetic Parameter for Lead Adsorption

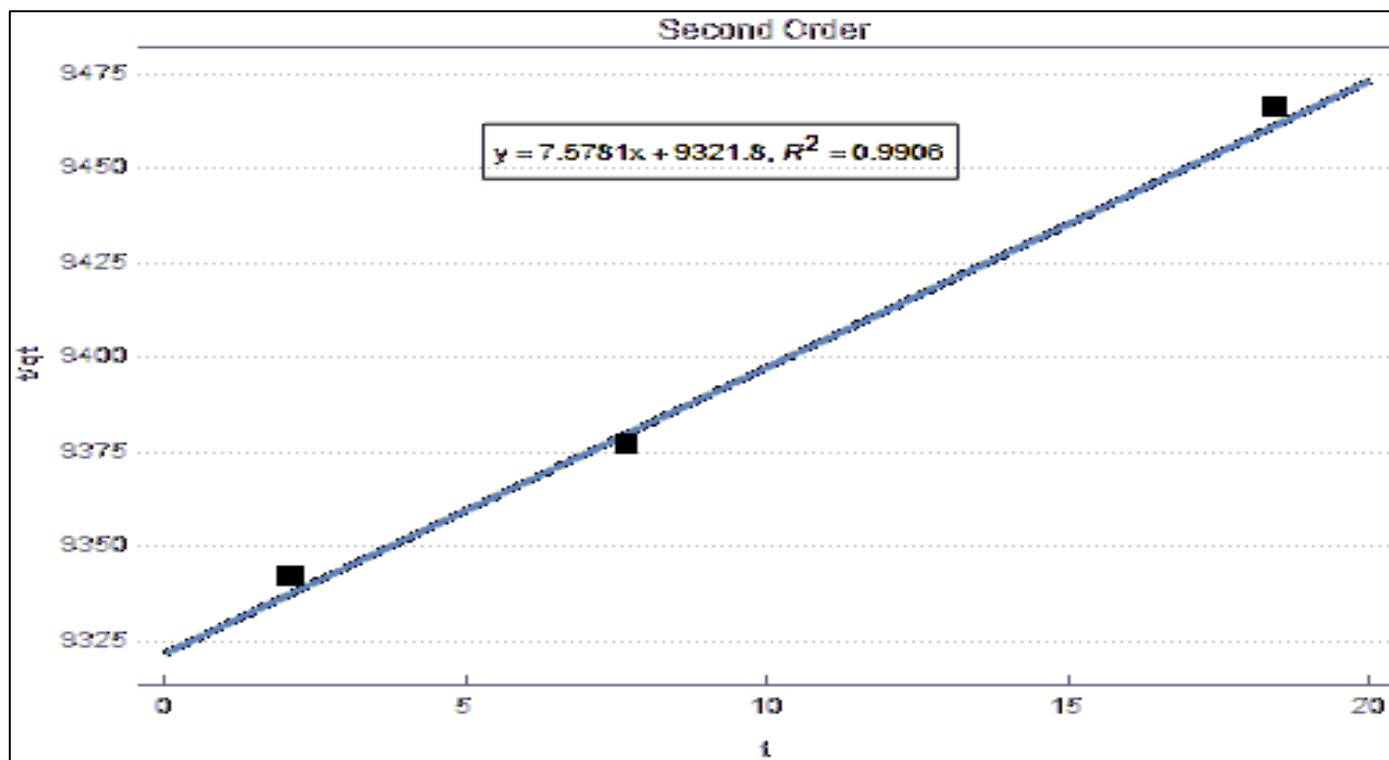


Fig 6 Second Order Kinetic Parameter for Lead Adsorption

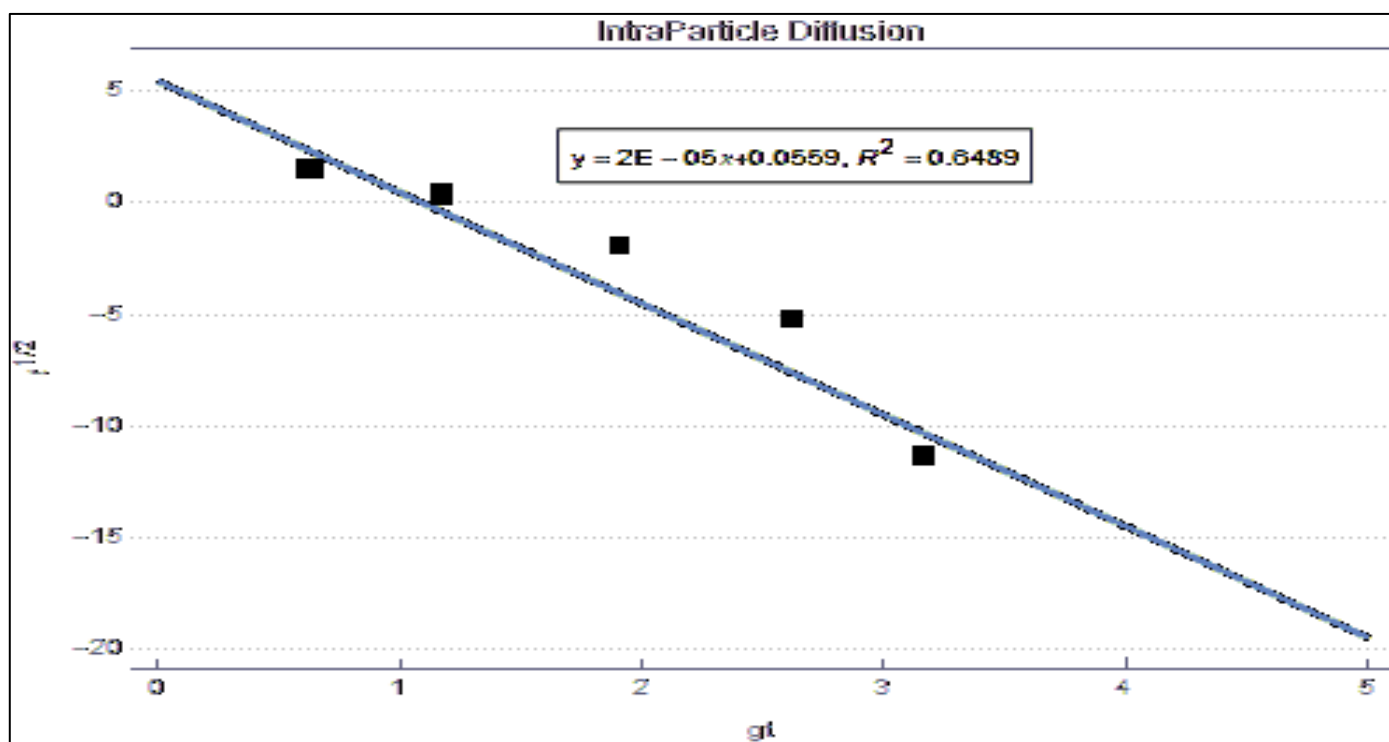


Fig 7 Intraparticle Kinetic Parameter for Lead Adsorption

➤ Assessment of Adsorption Isotherm

The uniform (homogeneous) adsorption of the adsorbed with the same energy on all adsorbent surfaces is the foundation of the Langmuir isotherm model. Equation 1 provides the Langmuir isotherm model in its linear version.

$$\frac{C_e}{q_e} = \frac{1}{K_m q_m} + \frac{1}{q_e} C_e \quad 1$$

where C_e is the concentration of the adsorbed material in the solution following the adsorption process in mg/L , q_m is the maximum adsorption capacity in mg/g , K_L is the isotherm constant, and q_e is the adsorbed component per gram of adsorbent in mg/g . The graph's gradient and y-intercept can be computed by withdrawing the C_e/q_e curve in terms of C_e [15, 16]. The multi-layer adsorption on heterogeneous surfaces and the unequal energy distribution on the

adsorbent's active sites serve as the foundation for the Freundlich isotherm model. Equation 2 represents the Freundlich isotherm model in its linear version.

$$\ln(q_e) = \ln K_f + \frac{1}{n} \ln C_e \quad 2$$

where C_e is the equilibrium concentration (mg L^{-1}), q_e is the adsorption capacity at equilibrium time (mg L^{-1}), and K_f and n is the Freundlich model constant [16,1]. The maximal adsorption capacity of the adsorbent for lead, as determined by the Langmuir isotherm model plotted in Figure 2, was 26.04. Furthermore, the experimental findings suited the Langmuir model well presented in Figure 3, as evidenced by the correlation values (R^2) for the lead adsorption process that the Langmuir isotherm model produced, which were 0.878. According to the Langmuir isotherm model, the adsorbent's maximum adsorption capacity for lead was 26.04. Additionally, the Langmuir isotherm model yielded correlation values (R^2) for the lead adsorption process of 0.878, indicating that the experimental results fit the Langmuir model well.

➤ Thermodynamic Study

The thermodynamic parameters include changes in enthalpy (ΔH°), entropy (ΔS°) and Gibbs free energy (ΔG°). Eq. (8) is used to calculate ΔG° :

$$\Delta G^\circ = -RT \ln K_c$$

where R denotes the universal gas constant (8.314 J/mol-K), T is the absolute temperature (K) and K_c is the equilibrium constant obtained from.

$$K_c = \frac{C_{ad,eq}}{C_{eq}}$$

where the concentrations of metal ions adsorbed on the adsorbent surface at equilibrium state (mg/L) and those still in solution at equilibrium state (mg/L) are denoted by $C_{ad,eq}$, and C_{eq} , respectively. The expression can be used to determine ΔH° and ΔS° from the slope and intercept of $\ln K_c$ against $1/T$.

$$\ln K_c = \frac{-\Delta G^\circ}{RT} = \frac{-\Delta H^\circ}{RT} + \frac{\Delta S^\circ}{R}$$

Given the values in Table 3, the adsorption of Pb (II) using the adsorbent was naturally occurring and possible since the Gibbs free energy was negative at all temperatures. Figure 5 displays the vant Hoff thermodynamic plot of $\ln K_d$ against $1/T$. Table 2 displays the isotherm parameters derived from the slope and intercept; negative values of Gibbs free energy (ΔG) in the table suggest that the process of adsorption was possible and natural. A negative enthalpy value (ΔH) signifies an exothermic adsorption process, while a positive entropy value (ΔS) indicates an increase in disorderliness as the lead ion is absorbed. As the temperature rises, Gibbs free energy falls, resulting in negative values indicating the adsorption process was practical and naturally occurring.

➤ Kinetic Study

Pseudo-first-order, pseudo-second-order, and intraparticle diffusion kinetic models were employed to examine the kinetic behavior of lead adsorption. Equation 3 expresses the pseudo-first-order kinetic model, often known as the Lagrangian model, in linear form:

$$\ln(q_e - q_t) = \ln(q_e) - k_1 t \quad 3$$

The linear form of the pseudo-second-order kinetic model is also given by equation 4.

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad 4$$

Also, the linear form of the intraparticle diffusion model is presented in equation 5:

$$q_t = K_i t^{1/2} + I \quad 5$$

where K_i and I represents the intraparticle diffusion rate ($\text{mg/g-min}^{1/2}$), and boundary layer thickness which is determined from the slope and intercept of q_t against $t^{1/2}$ according to Fig. 7. The pseudo-second-order kinetic model was shown to be able to better characterize the kinetic behavior of the adsorption process based on the correlation coefficients derived from the pseudo-first-order, pseudo-second-order, and intraparticle diffusion kinetic models for concentrations of 10, 20, and 50. Based on the findings from the intraparticle diffusion model, it can be concluded that the adsorption mechanism consists of a single, steeply sloping step where heavy metal ion molecules are transported straight from the layer to the surface of the adsorbent at a high rate. Plots of $\ln(q_e - q_t)$ against t and t/q_t against t in Figures 5 and 6 demonstrate the pseudo first and second order. Table 4 displays the values of q_e and k_1 , which were derived from the plot's intercept and slope, respectively. The pseudo second order model has a higher R^2 value of 0.99 than the pseudo first order model.

IV. CONCLUSION

The impact of many factors, including pH, contact time, temperature, the initial concentration of metal ions, and adsorption dosage, was investigated in order to wrap up the study. With a pH of 5, a temperature of 25°C, a contact time of 60 minutes, a metal ion concentration of 10 mg/L, and an adsorbent concentration of 2g/L, the findings indicated that the best lead removal efficiency was 94.4. Additionally, the highest lead removal efficiency from industrial wastewater was achieved at 85%. The snail shell nanoparticle's favorable potential in eliminating metal ions from industrial wastewater and crude oil contaminations is confirmed by its high adsorption efficiency and inexpensive cost.

The equilibrium behavior of the adsorption of metal ions was ascertained using the Langmuir and Freundlich isotherm models. The Freundlich isotherm model was less consistent with the experimental results than the Langmuir isotherm model, which had higher correlation coefficients.

Conclusions showed that the pseudo-second-order kinetic model could more accurately capture the kinetic behavior of the adsorption process when compared to pseudo-first-order, pseudo-second-order, and intraparticle diffusion kinetic models.

Thermodynamic parameter data indicated that the use of snail shell nanoparticles as a bioadsorbent for the adsorption of lead from crude oil-contaminated soil and industrial wastewater was suitable, spontaneous, and exothermic.

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