

Removal of lead ions from wastewater using raw and activated carbon of *Pterocarpus santalinoides* shell

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Abstract

Removing toxic metals from the environment is a primary concern for researchers because of their hazardous effects. In this study, raw and activated *Pterocarpus santalinoides* shell was utilized in removal of lead (Pb) metal from an aqueous solution. The prepared adsorbents were analysed by Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM), and mineral composition to identify their structural composition and understand the adsorption mechanism. The influence of various adsorption variables such as dose of adsorbents, initial concentration of lead metal, contact time, solution temperature, and adsorption isotherms, kinetics, and thermodynamic parameters was studied. Adsorption efficiencies and capacities for the adsorbents were calculated. Based on the results generated, the two different adsorbents utilised showed a good alternative to commercial materials, with removal efficiencies greater than 80%. The Langmuir isotherm and pseudo-second-order model showed good fits to the experimental data. Moreover, the thermodynamic results revealed that the adsorption of Pb ions via the raw and activated carbon of *Pterocarpus santalinoides* shell was an endothermic and spontaneous process. According to this research, *Pterocarpus santalinoides* shell, being an agricultural waste, might be explored as an efficient adsorbent for extracting Pb ions from wastewater.

Keywords: Lead; Water Pollution; Adsorption; Agricultural Waste; *Pterocarpus santalinoides*; Thermodynamics; Isotherms; Kinetics.

1. Introduction

The modernization and rapid technological advancement of society have given rise to environmental pollution in the ecosystem as a result of the introduction of several undesirable pollutants into the environment (Ulah *et al.*, 2020; Zada *et al.*, 2018).

Globally, water contamination is a severe environmental issue, mostly brought on by climate change and agricultural and industrial activities. The ecosystem is at risk when heavy metal-polluted wastewater that has not been fully or even partially treated is discharged to the environment (Abdel-Shafy and El-Khateeb, 2019). This situation leads to a large rise in the number of heavy metals in surface and ground waters, which affect aquatic bodies (Bayuo *et al.*, 2022). The most frequent pollutants in water are heavy metals. The occurrence of heavy metals in the environment is a major concern to researchers as a result of their harmfulness to both plants and animals (Kulbir *et al.*, 2018).

Lead metal is frequently found in the environment and is highly hazardous (Demay *et al.*, 2016). Lead (II) ions are commonly present in wastewater from industries such as batteries, mines, paint, pesticides, and smelting. When lead (II) ions built up in the body due to exposure to contaminated water and other sources, it resulted in a wide range of

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health issues, including hypertension, impaired blood production, severe stomach aches, brain and kidney damage, and miscarriages in pregnant women. (Bayuo *et al.*, 2019). Lead (II) is a highly toxic metal, even at very low concentrations, and is a non-essential element because it has no known benefit to human health or other living things. The World Health Organisation's (WHO) desired limit of lead (Pb) concentrations in drinking water is 0.01 mg/l (Ahmed *et al.*, 2023). The greatest strategy to prevent toxic metal pollution and consequent human poisoning is to treat heavy metal-contaminated wastewater before discharging, even though lead metal toxicity might be clinically diagnosed and treated. (Raut *et al.*, 2021).

Various chemical and physical methods have been probed for heavy metal removal from wastewater before it is discharged to the environment (Ullah *et al.*, 2020). These include chemical precipitation, coagulation, flocculation, membrane filtration, electrochemical techniques, and ion exchange. However, many of these removal techniques have shown good results; they have high removal efficiency, are expensive, and can generate secondary pollutants (Bolisetly *et al.*, 2019). Therefore, researchers are concentrating on developing cheap and efficient methods for treating heavy metal-polluted wastewater. The adsorption technique is regarded as one of the most effective methods for heavy metal removal due to its flexibility in design and operation, low energy consumption, minimization of sludge and by-product production, the possibility of regenerating adsorbents, and high removal efficiency even at very low metal concentrations (Fiyadh *et al.*, 2019). Activated carbon (AC) is the most utilised and recognised heavy metal adsorbent. It has shown to be more advantageous in heavy metal removal because of its high metal uptake capacity, high rate removal efficiency, and ease of working principle, but because of the cost of preparation and the impossibility of its regeneration, its usage at large-scale applications has been limited (Tsade *et al.*, 2020; Danish and Ahmad, 2018).

Over the last few decades, Using and creating heavy metal removal methods have represented a great challenge to researchers, especially in developing countries (Chai *et al.*, 2021). Recently, agricultural waste has been one of the many sources of inexpensive adsorbents that are attracting scientific interest for heavy metal removal due to its availability and abundance, biodegradability in natural and environmental settings, higher ability to adsorb metal ions, high surface area, compatibility, being easily renewable, being eco-friendly, and being more specific in nature (Gupta *et al.*, 2021; Elgarahy *et al.*, 2021). Several agricultural wastes such as rice husk, peanut husk, sawdust, groundnut husk, and potato peel have been studied (Simon *et al.*, 2022; Tsade *et al.*, 2020). Plant-based waste is made up of cellulose, hemicelluloses, and lignin and has various functional groups like carboxyl groups, phenolics, hydroxyls, amides, ethers, methyls, and amines that can act as binding sites on their surfaces to which the metal ions can be effectively adsorbed under normal circumstances through various mechanisms (Bilal *et al.*, 2022; Nleonu *et al.*, 2017).

Pterocarpus santalinoides is a species of the Fabaceae family known to have various uses in the daily lives of local populations. In Nigeria, its leaves are used in human food as a vegetable, while its fruits have no economic value. Studies have shown that *Pterocarpus santalinoides* is rich in phytochemicals (Ayena *et al.*, 2021; Nwokorie *et al.*, 2015). Therefore, the selection of a carbon-rich adsorbent is very important in heavy metals removal from wastewater via complexation or electrostatic attraction between the metal ions and various oxygen-carrying functional groups present on the surface of raw or activated carbon adsorbents (Huang *et al.*, 2017). Several investigations have revealed Pb (II) ions adsorption potentials of natural and treated agricultural-based materials through multiple sorption mechanisms involving ion exchanges, precipitation with inorganic components, interactions with electrons, and complexation with oxygen-containing functional groups (Han *et al.*, 2017).

This paper focuses on the study of lead (II) ion removal from wastewater using raw and activated carbon from *Pterocarpus santalinoides* shell as adsorbents. The efficiency of the prepared adsorbents on Pb (II) ions removal as influenced by adsorbent dosage, contact time, initial metal ion concentration, and temperature were studied. The obtained results were used to study the isotherm, kinetic, and thermodynamic parameters for the lead (II) ions adsorption processes.

2. Materials and Methods

2.1. Chemical and Reagents

Analytical-grade reagents were used in this study. Stock solutions of 100 mg/l of Pb (II) ions were prepared using deionized water by dissolving the required amounts of PbCl₂ (a product of Loba Chemie PVT. Ltd., India). The corresponding dilutions used in the adsorption experiments were prepared from the stock solutions. The activated carbon was activated using 0.1 M HCl (98 %), a product of Sigma Aldrich with 98% purity.

2.2. Preparation of Adsorbent (Raw and Activated Carbon)

Pterocarpus santalinoides fruits were collected from the botanical farm of the Federal Polytechnic, Nekede Owerri, Imo State. The fruits were first washed thoroughly with running water, followed by distilled water to remove extraneous materials, and air dried for 14 days. The dried material had its seeds removed, leaving the shell. The shell was ground into powder. The powdered samples were separated into two portions, of which one was used to prepare activated carbon.

Activated carbon from *Pterocarpus santalinoides* shell was prepared by treating the ground-dried materials with 0.1 M HCl and putting them into a muffle furnace at 500 °C for 3 hours. Under vacuum, the muffle furnace was cooled to ambient temperature. Then, using distilled water, the produced products were rinsed until the pH of the filtrate reached 7. The activated carbon from *Pterocarpus santalinoides* shell was then dried at 1000 °C. The ground raw and activated carbon were ground to a particular size of 250 mesh to obtain a fine powder. The powder of the individual adsorbents was labeled, kept in airtight plastic bottles, and stored in desiccators for further use.

2.3. Adsorbent Characterization

The mineral content of the adsorbents was determined following the guidelines of the ASTM E1755-01 standard (ASTM, 2020). The functional groups present on the surface of the raw adsorbent were determined using Fourier transform infrared (FTIR) spectroscopy (Happ-Genzel). The surface morphology of the raw and activated carbon of the adsorbents was studied with the help of a scanning electron microscope (phenom prox).

2.4. Batch Adsorption Experiment

Batch adsorption studies were used to investigate Pb²⁺ ion removal from aqueous solutions using raw and activated carbon from *Pterocarpus santalinoides* shells. An aqueous solution of Pb²⁺ ions was prepared. The adsorption experiments were studied in sample bottles containing Pb²⁺ ion solution at the given initial concentrations. The mixture of the metal solution and adsorbent was kept under constant stirring throughout the adsorption process until equilibrium, except where contact time is being studied. After 60 min, the mixture was filtered, and the filtrate was analysed for the detection of unadsorbed Pb (II) ions using an atomic absorption spectrophotometer. A number of experimental parameters influencing the adsorption process, such as the contact time (20–120 min), adsorbent dose (0.1–0.5 g/100 ml), initial metal ion concentration (10–50 mg/l), and temperature (30–70 °C) were studied to evaluate the optimum conditions for the removal of Pb²⁺ ions from the 100 ml aqueous solution over the applied adsorbents. The metal removal percentage was calculated from equation (1), and the removal capacity of each adsorbent (q_e) was calculated from equation (2):

$$\text{Removal percentage (\%)} = \frac{(C_0 - C_e)}{C_0} \times 100 \quad (1)$$

$$q_e \left(\frac{\text{mg}}{\text{g}} \right) = \left(\frac{C_0 - C_e}{m} \right) V \quad (2)$$

Where C₀ and C_e (mg/l) are the initial and final concentrations of Pb²⁺ ions in solution, respectively, V (L) is the volume of solution, and M (g) is the adsorbent mass.

3. Results and Discussion

3.1. Characterization of the Adsorbent

3.1.1. Mineral Composition

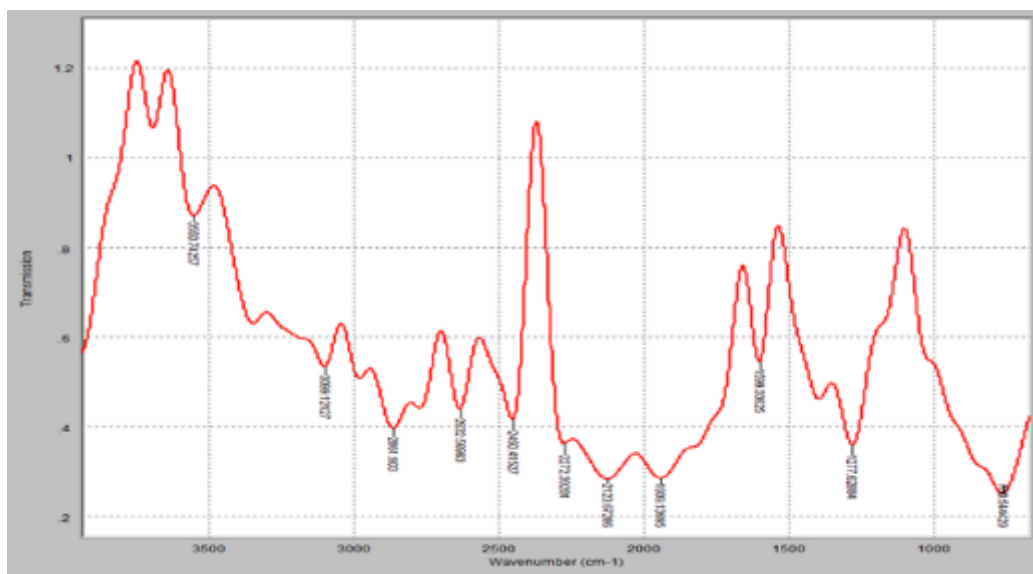
The result of the mineral composition of the raw and activated carbon of *Pterocarpus santalinoides* shell reveals that sodium was highest in both the raw (0.3549 mg/g) and activated carbon (0.2336 mg/g) shells, leaving magnesium (0.1566 mg/g) for the raw shell and potassium (0.0803 mg/g) for the activated carbon as the least mineral, respectively (Table 1). These results are comparable to those presented in the literature. Previous studies have shown that the presence of minerals such as K⁺, Na⁺, Ca²⁺, and Mg²⁺ plays an important role in the removal of metals from aqueous solutions because of their tendency to act as exchangeable cations in solution (Onyemenonu et al., 2015).

Table 1 Mineral composition of *Pterocarpus santalinoides* Shell

Adsorbents	Calcium (mg/g)	Magnesium (mg/g)	Potassium (mg/g)	Sodium (mg/g)
Raw shell	0.2123	0.1442	0.1566	0.3549
Shell activated carbon	0.1121	0.0942	0.0803	0.2336

3.2. FTIR Analysis

It was feasible to determine the existence of functional groups in the adsorbent responsible for the metal adsorption mechanism, such as by complexation or electrostatic force using Fourier transform infrared spectroscopy. (Simon *et al.*, 2022; Nleonu *et al.*, 2017). The FTIR spectra for the raw biomass studied are shown in Figure 1. The large number of IR bands observed is associated with the complex nature of agricultural plant materials. It is clear that the absorption peaks observed at 1277.62 and 2632.57 cm^{-1} indicate the presence of C-O and O-H groups, respectively, due to the carboxylic functional groups. The peaks observed between 2123.67 - 2272.39 cm^{-1} show the presence of nitrile groups. Similarly, the peaks observed in 1599.33 cm^{-1} show the presence of N-H due to the amine functional group. These results may be an indication that the adsorption sites offered by these functional groupings are responsible for the removal of the studied heavy metal via electrostatic attraction or complexation between the metal ions and different oxygen-containing functional groups carried by the surface of raw *Pterocarpus santalinoides* shell.

**Figure 1** The FT-IR spectrum of *Pterocarpus santalinoides* shell

3.3. Scanning Electron Microscopy Analysis

SEM images were used to analyse the morphological characteristics, like texture, topography, and surface, of the adsorbents. The SEM images of the two studied adsorbents prior to and after the Pb^{2+} ions adsorption are presented in Figure 2. The analysed raw shell adsorbent showed a fibrous microstructure with an irregular and rough surface with aggregation and cavities that form a network of roles and fibres, while the image of activated carbon from the shell revealed a non-fibrous microstructure with a regular and rough surface without aggregation. These morphological characteristics can facilitate the adsorption of lead metal. Significant changes were noticed in the surface morphology of the two adsorbents caused by the interaction with lead (II) ions after the adsorption of the metal.

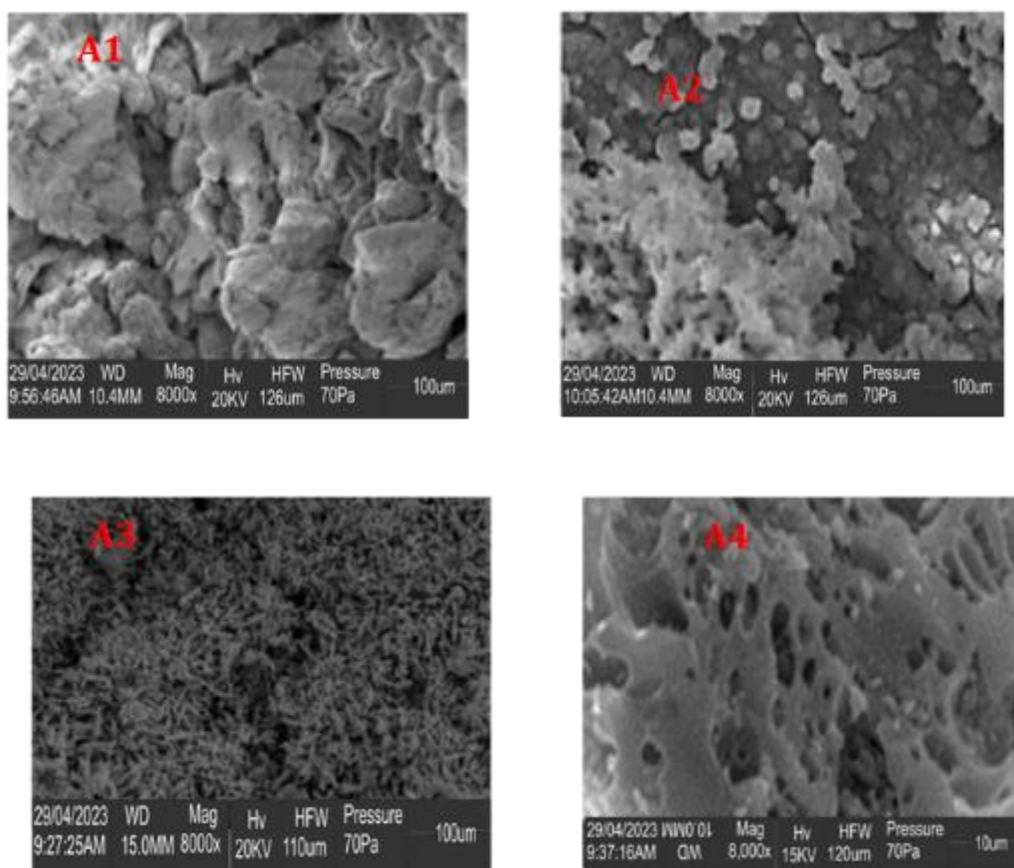


Figure 2 The SEM images of the adsorbent surface: (A1) raw shell before adsorption (A2) raw shell after lead (A3) shells activated carbon before adsorption (A4) after lead adsorption.

3.4. Adsorption Process Analysis

3.4.1. The Effect of Contact Time

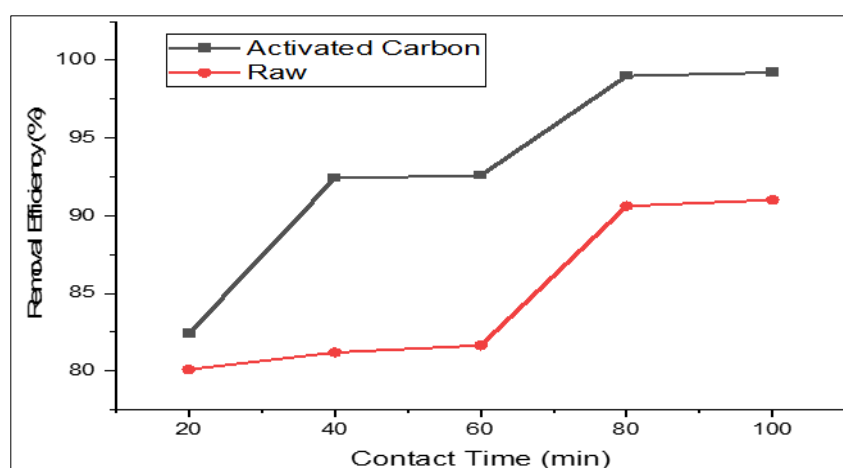


Figure 3 Effect of contact time on the removal of Pb by raw shell and shell activated carbon of *Pterocarpus santalinoides*

The removal of Pb (II) ions was studied under a variable contact time (20, 40, 60, 80, and 100 min) using an optimised amount of 0.2 g of raw and activated carbon from *Pterocarpus santalinoides* shell and a metal ion concentration of 40 mg/l. According to the results of percentage removal and adsorption capacity from Figures 3 and 4, it was observed that as the contact time increases, the percentage removal and adsorption capacity increase in the presence of both

adsorbents. The system attains equilibrium for lead removal with raw adsorbent at 40 min and 80 min under activated carbon adsorbent. Raw adsorbent shows the highest removal efficiency of 99.28%, while activated carbon shows 99.22%. The close removal efficiency observed shows that both adsorbents have related characteristics and affinity for the metal ions on their adsorption sites (Petrella *et al.*, 2018). Therefore, the raw and activated carbon shell adsorbents show higher adsorption efficiency for lead ions under the studied conditions. The high rate of removal percentage of the studied adsorbent observed in this study will draw more attention from researchers in the future for wastewater treatment.

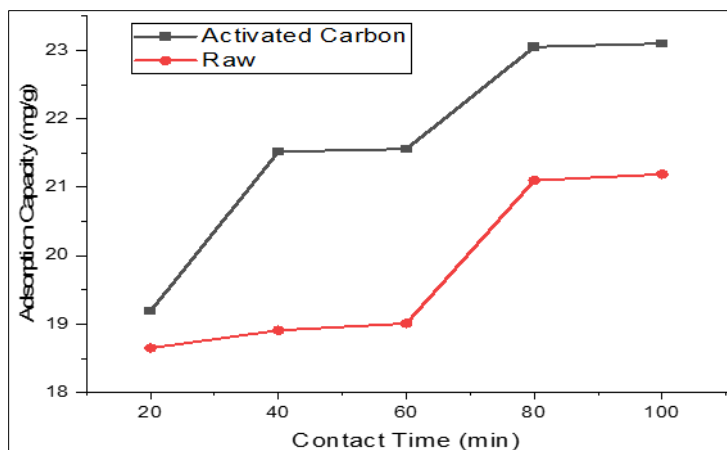


Figure 4 Effect of contact time on the adsorption capacity of raw shell and shell activated carbon of *Pterocarpus santalinoides* for Pb.

3.5. The Effect of Initial Metal Ion Concentration

The effects of the initial concentration on Pb percentage removal and adsorption capacity at a constant adsorbent dose of 0.1 g/100 ml and a contact time of 60 min are presented in Figures 5 and 6, respectively. The studies were carried out at various adsorption process concentrations of Pb ions (10, 20, 30, 40, and 50 mg/l). The results obtained revealed that the percentage removal efficiency and adsorption capacity decreased with an increase in metal ion concentration. This occurred because Pb ions are quickly adhered to the adsorbent sites at lower concentrations of the metal ions and are removed frequently when compared to the available adsorbent binding sites. Interestingly, the adsorption capacity of activated carbon shells was observed to be higher when compared to raw shells on Pb adsorption due to the different characteristics in their morphology, as explained in the scanning electron microscopy result.

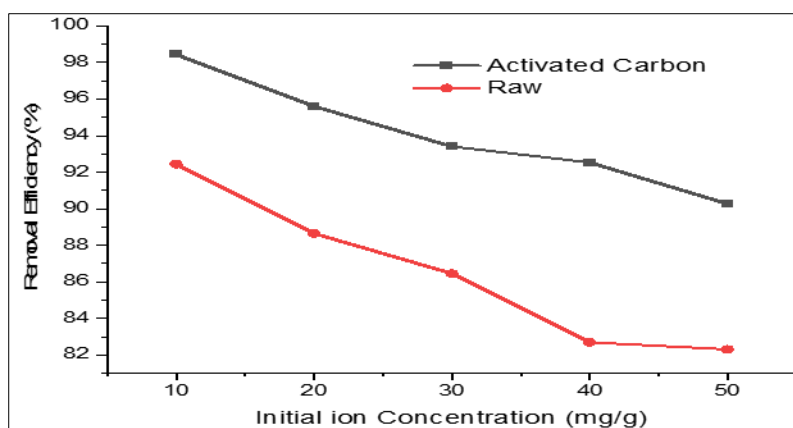


Figure 5 Effect of initial metal ion concentration on the removal of Pb by raw shell and shell activated carbon of *Pterocarpus santalinoides*.

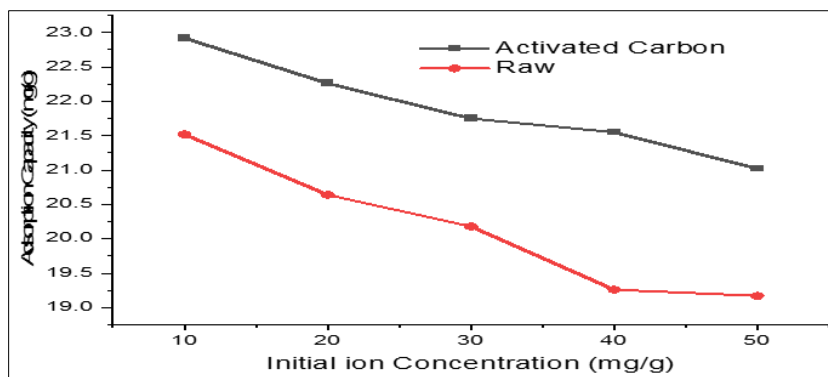


Figure 6 Effect of initial metal ion concentration on the adsorption capacity of raw shell and shell activated carbon of *Pterocarpus santalinoides* for Pb.

3.6. The Effect of the Adsorbent Dose

In this study, different doses of both adsorbents (0.1, 0.2, 0.3, 0.4 and 0.5 g) were applied under metal ion concentration (40mg/l) and contact time (60min). The adsorption removal efficiency and adsorption capacity of Pb are shown in Figures 7 and 8, respectively. The experimental findings proved that the removal efficiency increases as adsorbent dose increased due to a greater availability of surface area at a higher adsorbent concentration. The adsorption capacity of both adsorbents decreased with increase in adsorbent dose. The decreasing rate of adsorption capacity obtained in this study may be due to over lapping of adsorbent sites as a result of crowding together of adsorbent particles (Ahmed *et al.*, 2023). Also it is clear that the adsorbent made from the raw shell shows closed removal efficiency and adsorption capacity with the activated carbon shell as supported by the mineral composition result.

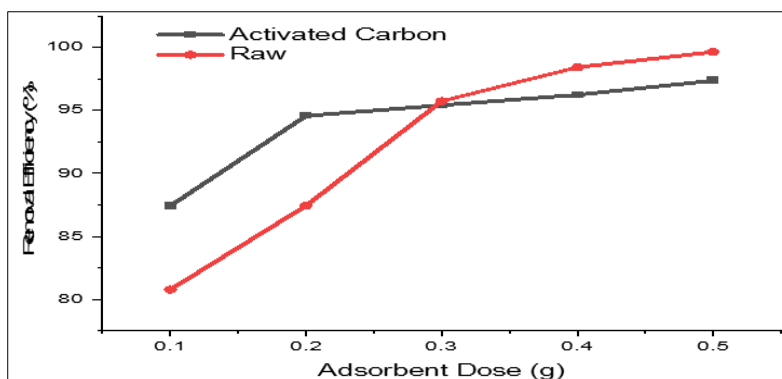


Figure 7 Effect of adsorbent dose on the removal of Pb by raw shell and shell activated carbon of *Pterocarpus santalinoides*.

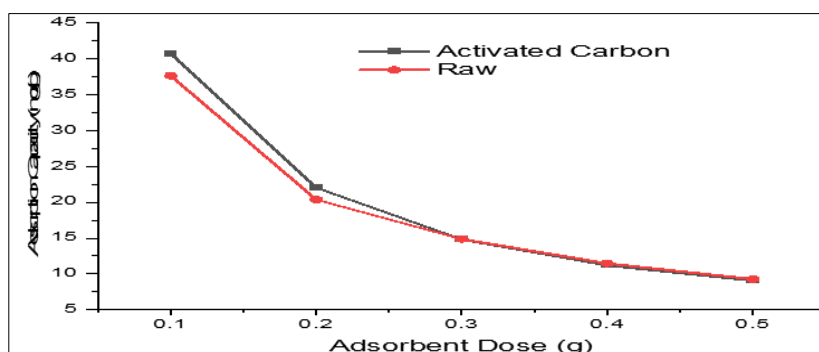


Figure 8 Effect of adsorbent dose on the adsorption capacity of raw shell and shell activated carbon of *Pterocarpus santalinoides* for Pb.

3.7. The Effect of Temperature

The temperature has a great effects on the adsorption behavior of metal ions in solution, as an increase in temperature affects the intermolecular forces between the adsorbate and adsorbent particles by either weakening the bond formed between the adsorbate and adsorbent sites or creating a large surface area for adsorption. This phenomenon either results in a decrease or an increase in the amount of metal ions adsorbed (Nleonu *et al.*, 2023). However, Figures 9 and 10 revealed that the rate of percentage removal of Pb by both adsorbents decreases as the solution temperature increases. The result explained that the strength of intermolecular forces between the adsorbate and adsorbent is strongly dependent on the solution temperature. The impact of the temperature study confirmed that the process of Pb²⁺ ions adsorption by both adsorbents was an endothermic process based on the thermodynamic adsorption data.

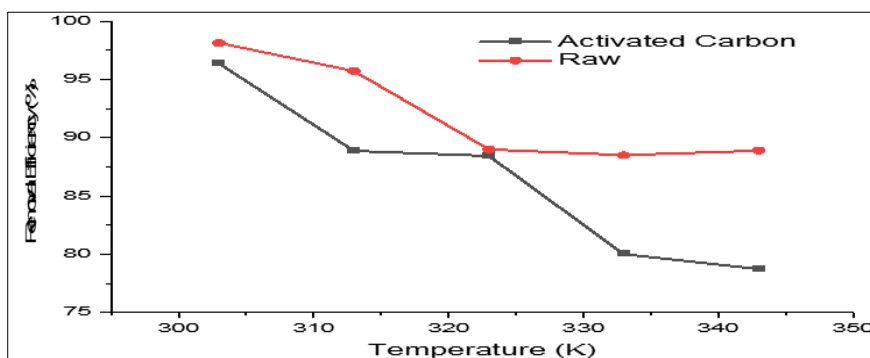


Figure 9 Effect of temperature on the removal of Pb by raw shell and shell activated carbon of *Pterocarpus santalinoides*

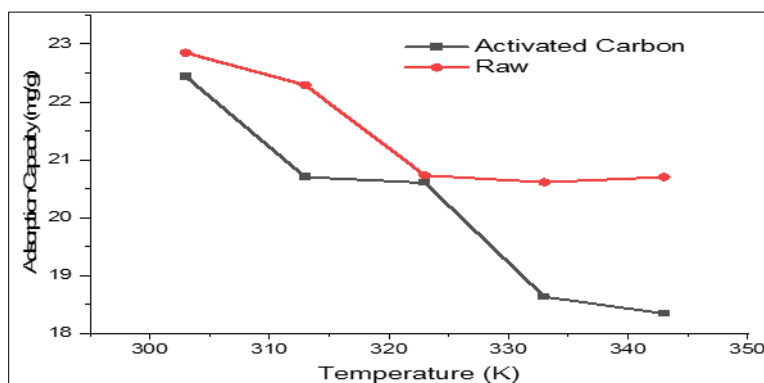


Figure 10 Effect of adsorbent dose on the adsorption capacity of raw shell and shell activated carbon of *Pterocarpus santalinoides* for Pb.

3.8. Adsorption Kinetics

The adsorption kinetic study was used to determine whether Pseudo-first or pseudo-second order processes were followed during the adsorption process. The first-second-order equation (4) and second-order kinetic equation (5) were utilized:

$$\ln \frac{C_t}{C_0} = - K_1 t \tag{4}$$

$$\frac{1}{C_t} = \frac{1}{C_0} + K_2 t \tag{5}$$

Where C₀ is the initial concentration and C_t is the residual metal ion concentration of metal ion (mg/l) at a definite time t (min), K₁ is the pseudo-first order rate constant (min⁻¹), and K₂ is the pseudo-second order rate constant (g/mg.min).

The kinetic adsorption result clarifies that the adsorption of Pb onto the studied adsorbents obeyed the pseudo-second-order model based on the correlation coefficients (R^2), which are positive and close to unity, whereas the values of R^2 for pseudo-first-order were negative, as tabulated in Table 1. This result suggests that the pseudo-second-order model satisfactorily described the best kinetic model for Pb adsorption by raw and activated carbon of *Pterocarpus santalinoides* shell, showing that the adsorption process is controlled by chemical adsorption (Chima *et al.*, 2023).

Table 2 Kinetic adsorption model parameters for lead adsorption by raw and activated carbon of *Pterocarpus santalinoides* shell.

Adsorbents	Pseudo first order		Pseudo second order	
	K	R^2	K	R^2
Raw shell	6.95×10^{-5}	-0.3395	4.72×10^{-2}	0.9999
Shell activated carbon	1.02×10^{-3}	-0.8782	4.21×10^{-2}	0.9987

3.9. Adsorption Isotherms

The adsorption isotherms of Pb metal on *Pterocarpus santalinoides* (raw and activated carbon) shell were studied based on the optimum operating conditions, which were 0.1 g adsorbent dose, 40 mg/l metal ion, and a temperature of 30 °C. Based on the Langmuir and Freundlich mathematical models, the Langmuir and Freundlich parameters were computed and tabulated in Table 2, along with their correlation coefficients for the adsorption data. The Langmuir and Freundlich adsorption isotherm equations are presented in equations 5 and 6, respectively.

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m} \tag{5}$$

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \tag{6}$$

Where q_m and K_L are the Langmuir constants (mg/g), K_f are Freundlich equilibrium constant (mg/g), q_e is the metal dose adsorbed on a specific amount of adsorbent (mg/g), C_e is the equilibrium concentration of the solution (mg/l) and q_m is the maximum dose of metal concentration required to form a monolayer (mg/g).

The experimental result indicated that the R^2 value for the Langmuir model is higher and positive, while the Freundlich model shows negative R^2 values for both adsorbents. The isotherm model that has the best fit for Pb adsorption in the two adsorbents studied is Langmuir, indicating that the adsorption occurred in a homogeneous monolayer.

Table 3 Isotherm model parameters for lead adsorption by raw and activated carbon of *Pterocarpus santalinoides* shell.

Adsorbents	Freundlich Model			Langmuir Model		
	$\frac{1}{n}$	K_f (mg/g)	R^2	q_L	K_f (mg/g)	R^2
Raw shell	0.038	22.67	-0.9999	21.19	0.99	0.9999
Shell activated carbon	0.011	22.85	-0.9980	22.78	186.01	1

3.10. Adsorption Thermodynamics

Thermodynamic variables like standard free energy change (ΔG°), standard enthalpy change (ΔH°), and standard entropy change (ΔS°) were used to study the thermodynamic behaviour of Pb adsorption onto raw and activated carbon of *Pterocarpus santalinoides* shell. The standard free energy change (ΔG°) was calculated using equation (7).

$$\Delta G^\circ = -RT \ln K_d \tag{7}$$

Where K_d , T and R are the equilibrium rate constant (L/g), temperature (K) and gas constant (J/K.mol), respectively. The parameters of thermodynamic were calculated using equation (8).

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ$$

8

From the plot of ΔG° against T , ΔH° and ΔS° were obtained by the intercept and slope, respectively. All thermodynamic parameter values obtained are tabulated in Table 3. It was evident that the ΔS° value was negative (-0.084 KJ/mol.K) for the raw shell and positive (0.044 KJ/mol.K) for the activated carbon derived from the shell of *Pterocarpus santalinoides*. The ΔH° values were positive for the raw and activated carbon for the adsorption of Pb^{2+} ions from aqueous solution, which means that the endothermic process was predominant. The Gibb's free energy values were negative and decreased from -25.32 to -28.66 (KJ/mol) for the raw shell and -13.16 to -14.90 (KJ/mol) for activated carbon, with an increase in the solution temperatures from 303 to 343 K. The thermodynamic result indicates that the adsorption process for lead (II) ions by raw and activated carbon of *Pterocarpus santalinoides* shells is spontaneous and favourable at various solution temperatures.

4. Conclusion

Currently, the prevention of water contamination from heavy metals is of major concern to researchers. Research is currently focusing on the development of suitable technology for the decontamination of heavy metals from wastewater using agricultural waste materials. In this study, raw and activated carbon from *Pterocarpus santalinoides* shells was applied for the removal of lead (II) ions from synthetic aqueous solutions by varying some basic parameters. The physicochemical properties of the adsorbents were analysed, and the results obtained confirmed the adsorption potential of the raw and activated carbon. The adsorption results of the study showed that raw and activated carbon from *Pterocarpus santalinoides* shells can be considered an effective, easily available, and low-cost adsorbent for the removal of Pb^{2+} ions from contaminated wastewater. The removal efficiencies of both adsorbents on the Pb^{2+} ion were strongly dependent on their contact time, initial metal ion concentration, and temperature. The Langmuir isotherm model was well fitted to the experimental data, indicating that the two adsorbents were effective in removing Pb^{2+} ions from aqueous solutions with low sorption energy. The experimental data of Pb^{2+} ions adsorption by the adsorbents studied were fitted with the second-order kinetic model, revealing that adsorption occurs by chemisorption technique. The thermodynamic data disclose that the adsorption process was endothermic, spontaneous, and feasible in nature. This work exposed that raw and activated carbon from *Pterocarpus santalinoides* shells can be considered an alternative for the removal of Pb^{2+} ions from wastewater, which is a low-cost agricultural waste.

Compliance with ethical standards

Acknowledgement

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Disclosure of conflict of interest

The authors declare that they have no conflict of interest.

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