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Isotherm, Kinetic and Thermodynamic Investigation on The Biosorption Removal of Pb (II) Ion From Solution Onto Biochar Prepared From Breadfruit Seed Hull

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Abstract: *This study investigates the adsorption of lead(II) ions from aqueous solutions using biochar derived from breadfruit seed hulls, addressing the growing concern of lead contamination in water due to its toxic effects on human health and the environment. The aim of the study was to assess the efficiency of breadfruit seed hull biochar in removing lead ions, with a focus on understanding the adsorption mechanisms and the influence of key parameters. Batch adsorption experiments were conducted, examining the effects of solution pH (2.0–11.0), initial lead concentration (10–50 mg/L), temperature (300 K to 323 K), and adsorbent dosage (0.02–0.1 g). The results showed a significant increase in removal efficiency from 50% to 84.5% as the pH rose from 2.0 to 11.0, with the highest removal occurring at pH 11.0, though pH 6.0 was selected as the optimal condition to minimize the formation of metal hydroxides. Removal efficiency decreased from 83% to 47.6% as the initial lead concentration increased from 10 mg/L to 50 mg/L. Temperature also positively impacted the adsorption, with the removal percentage increasing from 72.5% to 82% as the temperature rose from 300 K to 323 K, suggesting an endothermic adsorption process. The adsorption capacity increased from 4.2 mg/g to 11.9 mg/g with the rise in lead concentration. The Langmuir model provided the best fit for the adsorption data, with a high coefficient of determination (R^2 = 0.99595) and a separation factor (RL) ranging from 0.1 to 0.41, indicating favourable adsorption. The Freundlich model also indicated favourable adsorption with an n value of 2.622, though it showed a lower fit compared to the Langmuir model. The study concluded that breadfruit seed hull biochar is an effective biosorbent for lead removal, with the adsorption process primarily governed by monolayer adsorption and chemisorption. It is recommended that breadfruit seed hull biochar be considered for water treatment applications targeting heavy metal removal, with further research into its regeneration and large-scale application for environmental cleanup.*

Keywords: Lead removal, breadfruit seed hull biochar, adsorption isotherms, water treatment, biosorption

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1.0 Introduction

Heavy metal poisoning of water bodies, particularly lead (II) ions, is a serious global environmental issue due to its established toxicity, persistence, and bioaccumulation, which can have catastrophic ecological and human health effects. (Chukwuemeka-okorie *et al*., 2023; Chima *et al*., 2022; Matter *et al*., 2024). . Lead (II) ions can lead to severe health problems even at low exposure, including neurological damage, developmental problems, and cardiovascular ailments. Industrial operations, mining operations, and improper waste disposal practices contribute to the pollution of lead (II) ions in wastewater. Therefore, this signifies the need for effective and sustainable remediation methods (Almanassra *et al*., 2022; Kelle *et al*., 2022; Sirijaree & Praipipat, 2023). Several traditional treatment techniques have been utilized in various industries for removing heavy metals from wastewater. These technological techniques include chemical and electrochemical precipitation, adsorption, ion exchange, membrane filtration, electrolysis, coagulation and solvent extraction. (Imran-Shaukat *et al*., 2022). However, they often suffer from high costs and the generation of secondary pollutants. Adsorption has emerged as a promising alternative for removing pollutants at low concentrations due to its simplicity, cost-effectiveness, and potential for utilizing readily available materials.(Chukwuemeka-okorie, *et al*., 2018). Various materials have been utilized as potential adsorbents for sequestrating heavy metals from aqueous solutions. (Akpomie and Dawodu 2015). These adsorbents include activated carbon, carbon nanotube polymers, and graphene; however, their application is limited due to high costs and the need for regeneration (Imran-Shaukat *et al*., 2022). A variety of agricultural wastes have been explored as cost-effective adsorbents for the removal of heavy metals, including banana peel, tangerine, kiwi peel (Al-Qahtani, 2016), watermelon shells (Gupta & Gogate, 2016), raw pomegranate peel (Ben-Ali *et al*., 2017) and groundnut husk (Gupta & Sen, 2017). However, researchers have reported that these materials have a low adsorption capacity. As a result, chemical modification or activation is frequently necessary to increase their adsorption capabilities. Modifying these materials with surfactants, alkaline, acidic, and organic chemicals considerably enhanced the adsorption capacity of agricultural waste (Akpomie & Dawodu, 2016; Chukwuemeka-Okorie *et al*., 2018). However, treating agricultural waste with chemicals causes secondary pollution. As a result, it becomes vital to research additional methods to enhance agricultural waste's adsorption capabilities.

Biochar, defined as a carbon-rich material generated by the pyrolytic conversion of multiple organic feedstocks, such as agricultural residues, food waste, forest residues, sludge, and animal dung, has emerged as a strong and efficient technology for the upgradation of agricultural waste and recovery of heavy metals. (Park *et al*., 2019; Shakoor *et al*., 2021). Biochar are readily abundant and accessible globally, and its disposal is associated with problems; hence, converting this waste into biochar is a sustainable solution for waste management and resource recovery. (Qiu *et al*.., 2022). It is known to have a high surface area, a porous structure, and an abundance of surface functional groups. Biochar has been used in water treatment as a low-cost adsorbent, replacing high-cost activated carbon. Pesticides, dyes, heavy metals, polycyclic aromatic hydrocarbons, volatile organic compounds, and medicines are some of the pollutants it eliminates. (Qiu *et al*., 2022). This study investigates breadfruit seed hull biochar as a novel adsorbent for removing lead (II) ions from aqueous solutions. Breadfruit (Artocarpus altilis) is a common tropical fruit; its waste is an easily available but underutilized biomass resource. Converting breadfruit seed husk into charcoal is a sustainable waste management solution that produces a valuable adsorbent material. This study examines the efficacy of breadfruit seed husk biochar in removing lead under various experimental conditions, including pH, adsorbent dosage, solution temperature, contact time, and initial lead concentration. The adsorption mechanisms are explored, and the potential of breadfruit seed hull biochar as a low-cost and sustainable solution for lead remediation from wastewater is evaluated.

2.0 Materials and Methods

2.1 Materials and Reagents

Analytical grade lead(II) nitrate [$Pb(NO_3)_2$], nitric acid (HNO_3), hydrochloric acid (HCl), and sodium hydroxide (NaOH) were procured from Sigma-Aldrich and used without further purification. Breadfruit used in this study was obtained from Ogbete Main Market, Enugu State, Nigeria.

2.2 Preparation of Breadfruit Seed Hull Biochar

Breadfruit seed hulls were manually separated, washed with tap water to remove surface impurities, and pre-dried under sunlight for 24 hours. They were further oven-dried at 80 °C for 48 hours, ground using a mortar and pestle, and pyrolyzed in a muffle furnace at 350 °C for 3 hours in a closed porcelain crucible to obtain biochar. The resulting biochar was washed with distilled water to remove residual ash and oven-dried at 100 ± 2 °C for 30 hours. It was sieved through a 100 µm mesh and stored in airtight containers for subsequent use.

2.3 Batch Adsorption Experiments

Batch adsorption was conducted to evaluate the removal efficiency of Pb(II) ions. A 1000 mg/L Pb(II) stock solution was prepared by dissolving $Pb(NO_3)_2$ in distilled water and diluting it to obtain working concentrations of 10–50 mg/L. Solution pH was adjusted to values between 2 and 11 using 0.1 M NaOH or HCl. Adsorption tests were performed in 100 mL glass containers by mixing 0.02 g of biochar with 10 mL of Pb(II) solution. Parameters varied include pH (2–11), initial Pb(II) concentration (10–50 mg/L), biochar dose (0.02–0.1 g), temperature (300–323 K), and contact time (5–180 minutes).

Specific conditions for each parameter test were:

pH effect: 20 mg/L Pb(II), 120 min contact time.

Initial concentration: pH 6.0, 120 min.

Biochar dose: 20 mg/L Pb(II), pH 6.0, 120 min.

Contact time: 20 mg/L Pb(II), pH 6.0.

Post-adsorption, supernatants were analyzed for residual Pb(II) concentration using an Atomic Absorption Spectrophotometer (AAS, Buck Scientific 210VGP). The amount of Pb(II) adsorbed was calculated using:

$$\%\text{Uptake} = \left(\frac{(C_o - C_e)}{C_o}\right) * 100 \qquad (1)$$

$$qe = \frac{(C_o - C_e)V}{m} \qquad (2)$$

where m (g) is the mass of the biochar used, and V (L) is the volume of the solution, Co and Ce represent the initial and final concentrations of lead ions in the solution, respectively, in mg/L. All experiments were conducted in duplicates, and the mean values were reported. Error bars in Fig.s 1 and 2 represent standard deviations.

2.4 Equilibrium Isotherm Models

Adsorption equilibrium data were fitted to Langmuir, Freundlich, Temkin, and Dubinin–

Radushkevich isotherms using the following equations:

Langmuir:

$$\frac{C_e}{q_e} = \frac{1}{q_L K_L} + \frac{C_e}{q_L} \quad (3)$$

Freundlich:

$$\log q_e = \log K_F + \left(\frac{1}{n}\right) \log C_e \quad (4)$$

Temkin:

$$q_e = B \ln A + B \ln C_e \quad (5)$$

Dubinin–Radushkevich:

$$lnq_e = lnq_m - \beta\varepsilon^2 \quad (6)$$

where Ce is the equilibrium concentration (mg/L), q_e is the amount adsorbed (mg/g), qLq_LqL and K_L are Langmuir constants, K_F and n are Freundlich constants, and B=RT/b is a Temkin constant. In the Dubinin–Radushkevich model, q_m is the theoretical capacity (mg/g), β (mol^2/J^2) is a constant, and ε\varepsilonε is the Polanyi potential:

$$\varepsilon = RT \ln(1 + \left(\frac{1}{Ce}\right) \quad (7)$$

2.5 Kinetic Models

Adsorption kinetics were evaluated using pseudo-first-order (PFO), pseudo-second-order (PSO), intraparticle diffusion (IPD), and liquid film diffusion (LFD) models:

PFO:

$$\log(q_e - q_t) = \log q_e - \frac{K_1}{2.303} t \quad (8)$$

PSO:

$$\frac{t}{q_t} = \frac{1}{K_2 {q_e}^2} + \frac{t}{q_e} \quad (9)$$

IPD:

$$q_t = K_d t^{1/2} + C \quad (10)$$

LFD:

$$Ln(1 - F) = D - K_{FD} t \quad (11)$$

where qt and qe (mg/g) are adsorption capacity at time t (min) and equilibrium, respectively, while Kfd, Kd, K2, and K1 are LFD, IPD, PSO, and PFO rate constants, respectively. C and D are LFD and IPD model intercepts. F is the equilibrium fractional attainment.

2.6 Thermodynamic Analysis

Thermodynamic parameters—Gibbs free energy (ΔG^0), enthalpy (ΔH^0), and entropy (ΔS^0) were calculated using:

$$\Delta G^0 = -RT \ln K_c \quad (12)$$

$$\ln K_C = -\left(\frac{\Delta H^0}{RT}\right) + \left(\frac{\Delta S^0}{R}\right) \quad] \ (13)$$

T (K) is the absolute temperature, Kc is the equilibrium constant, and ΔG°, ΔH°, and ΔS° represent the changes in free energy, enthalpy, and entropy, respectively. (Al-Musawi *et al*., 2021)

2.7 Statistical Analysis

Model fits were evaluated using the coefficient of determination (R^2) and the sum of squared errors (SSE), computed with OriginPro 2019b software. A model was considered optimal when R^2 was high and SSE low. All experiments were repeated, and average values were reported. Standard deviations were used to represent variability in data through error bars.

3. 0 Results and Discussion

3.1 Influence of solution pH

Fig. 1(a) illustrates the influence of initial solution pH on the adsorption of lead (Pb^{2+}) ions from aqueous solution using breadfruit seed hull biochar. The acidity or alkalinity of the solution is a key parameter that affects the surface charge of the biochar and the solubility/precipitation of lead ions (Chukwuemeka-Okorie *et al*., 2023). Experimental results revealed a progressive increase in removal efficiency from 50% to 84.5% as pH increased from 2.0 to 11.0.

At pH 2.0, the high concentration of H^+ ions leads to intense competition with Pb^{2+} ions for available adsorption sites, thereby inhibiting the adsorption capacity (Almanassra *et al*., 2022). As the solution becomes less acidic and approaches pH 6.0, this competition reduces, allowing more Pb^{2+} ions to bind to the adsorbent surface (Şenol & Arslanoğlu, 2024). Although the highest removal efficiency was recorded at pH 11.0, the possibility of $Pb(OH)_2$ precipitation at alkaline conditions may interfere with the adsorption mechanism (Liang *et al.*, 2007). Hence, pH 6.0 was selected as the optimum pH for further experiments to ensure that adsorption is driven primarily by surface interactions rather than precipitation.

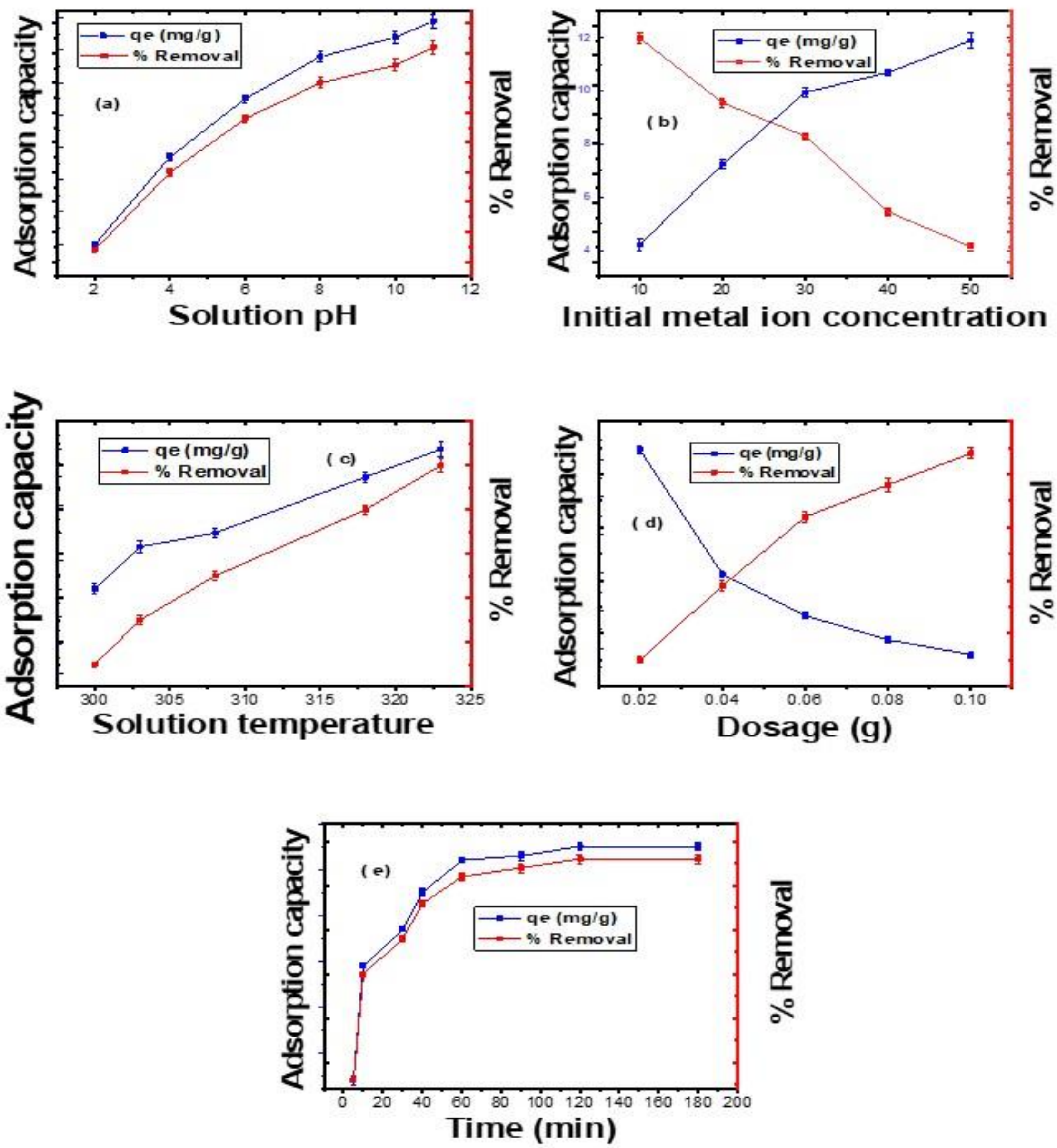

Fig 1. (a) Solution pH, (300K, 20 mg/L, 120 min, 0.02g) (b) Influence of initial metal ion concentration (300K, pH 6, 120 min, 0.02g) (c) solution temperature (120 min, 20 mg/L, pH 6, 0.02g) (d) adsorbent dose (300K, 20 mg/L, 120 min, pH 6)) and (e) influence of contact time (pH 6, 20 mg/L, 300 K, 0.02 g).

The effect of initial Pb^{2+} concentration (10–50 mg/L) on adsorption efficiency is presented in Fig. 1(b)**.** Results show a negative relationship between concentration and removal efficiency, which decreased from 83% at 10 mg/L to 47.6% at 50 mg/L. This trend is attributed to the saturation of available active sites at higher ion concentrations (Chukwuemeka-Okorie *et al*., 2021; Çiftçi *et al*., 2023). Conversely, the adsorption capacity (mg/g) increased with increasing concentration, from 4.2 to 11.9 mg/g, due to the greater driving force provided by steeper concentration gradients (Akpomie & Dawodu, 2015).

This outcome is supported by previous findings (Khan *et al*., 2017; Almanassra *et al*., 2022), which indicate that higher ion availability promotes enhanced utilization of adsorbent binding sites. A working concentration of 20 mg/L was selected for subsequent tests to strike a balance between realistic environmental relevance and reliable performance evaluation.

Fig. 1(c) shows the effect of temperature (300–323 K) on Pb^{2+} adsorption efficiency. Removal increased from 72.5 to 82%**,** indicating an endothermic adsorption process. This behaviour is typical of chemisorption, where higher temperatures provide additional kinetic energy, facilitating stronger interactions between Pb^{2+} ions and active sites on the biochar surface (Eddy *et al*., 2010; Eddy *et al*., 2024b). The trend aligns with the findings of Boulaiche *et al*. (2019), further confirming that increased temperature enhances lead removal efficiency.

The effect of varying biochar dosage (0.02–0.1 g) on Pb^{2+} removal is shown in Fig. 1(d)**.** As dosage increased, removal efficiency rose from 72.5 to 92.0%**,** indicating that more active sites became available for ion binding (Chukwuemeka-Okorie *et al*., 2023). This result is consistent with earlier studies (Islam *et al*., 2017; Dawodu & Akpomie, 2014), which reported similar positive correlations between adsorbent dosage and metal removal efficiency.

Fig. 1(e) displays the effect of contact time on Pb^{2+} adsorption. The removal was rapid during the initial phase and slowed down as equilibrium was approached. The high initial rate is due to the abundance of available binding sites, which progressively become occupied over time (Madala *et al*., 2017; Boulaiche *et al*., 2019). Equilibrium was reached at 120 minutes, which was adopted as the optimum contact time for further experiments, consistent with previous findings (Chukwuemeka-Okorie *et al*., 2018).

3.2 Equilibrium Isotherm analysis

The adsorption isotherm provides important information about the affinity between the adsorbent and the adsorbate molecules in solution. It sheds light on adsorbent surface features as well as the adsorption process. The equilibrium isotherm modelling of lead ion adsorption onto breadfruit seed hull was investigated using the Dubini-Raduskevich, Langmuir, Temkin, and Freundlich models. The theories of these isotherm models are thoroughly defined (Akpomie *et al*., 2023; Chukwuemeka-Okorie *et al*., 2023), and the estimated isotherm parameters are presented in Table 1. Fig. 2(a) illustrates the Langmuir isotherm model for the adsorption of lead (II) ions onto biochar. This model, which is limited to homogeneous monolayer adsorption on the adsorbent surface, provided a strong fit to the adsorption data, as evidenced by its high coefficient of determination (R^2 value of 0.99595) and low sum of squared errors (SSE). Furthermore, a Langmuir model separation parameter ($R_L = 1/[1 + K_L Co]$) is related to the dimensionless constant, R_L which is an index for predicting the feasibility of the Langmuir type of adsorption (Almanassra *et al*., 2022) while the dimensionless— Generally, R_L represents a favourable when 0 < RL < 1, irreversible when $R_L = 0$, unfavourable when

$R_L > 1$ and linear when $R_L = 1$ adsorption process. (Ajala *et al*., 2024). The adsorption data yielded a separation factor ranging from 0.1 to 0.41, which affirms that the Langmuir model predicts favourable adsorption for the adsorption of heavy metal ions. Also, the agreement of the adsorption data with the monolayer adsorption type, support the chemisorption mechanism as predicted earlier (Eddy *et al*., 2024a).

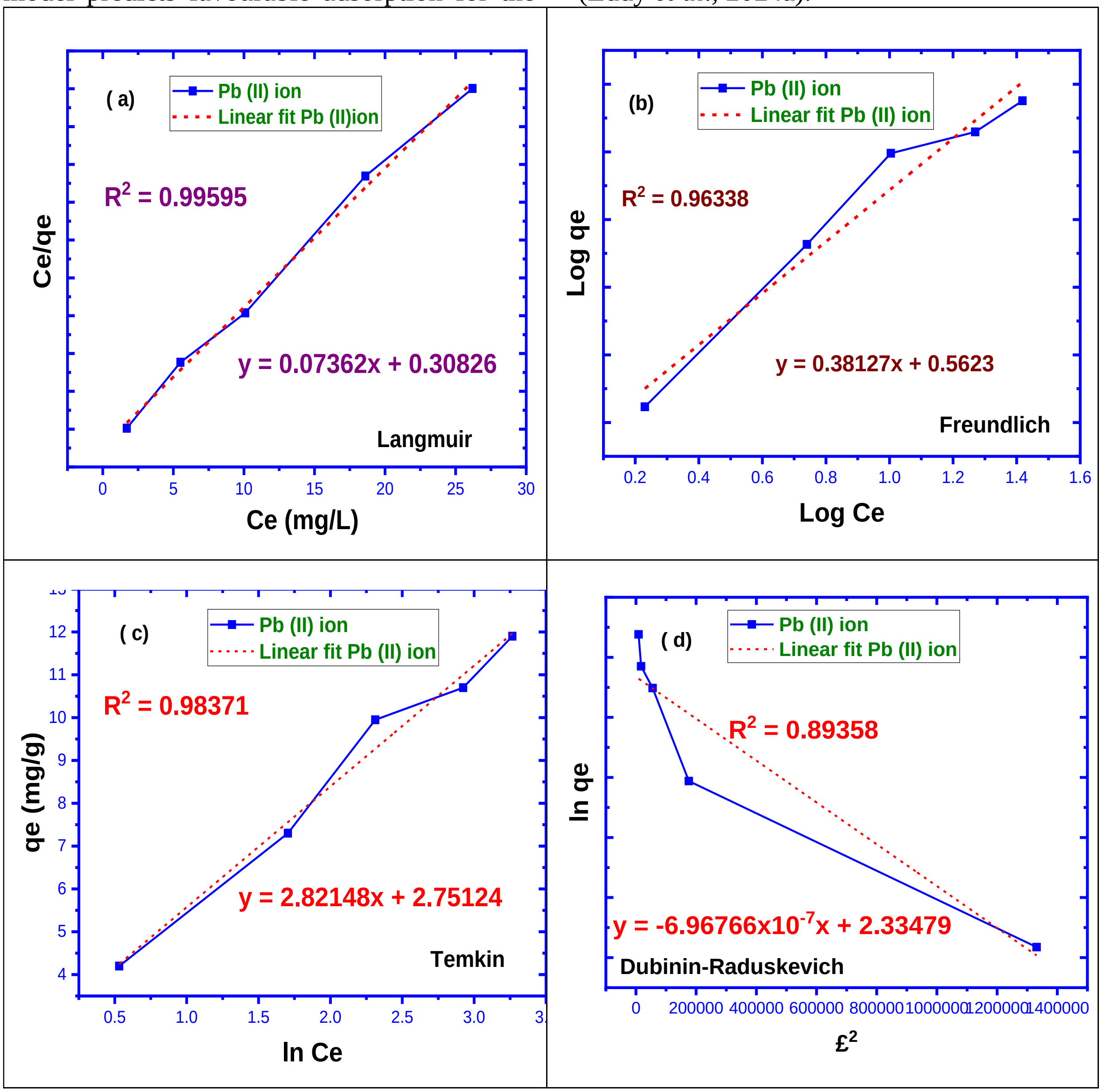

Fig. 2: Fitting of isotherms for the adsorption of Pb^{2+} based on (a) Langmuir (b) Freundlich (c) Temkin and (d) Dubinin-Raduskevich adsorption models

Fig. 2(b) shows the Freundlich model, which describes a multilayer heterogeneous adsorption process. The Freundlich model yielded an adsorption intensity (n) of 2.622, which also indicates favourable adsorption (n > 1), but the lower R^2 value suggests a poorer fit than Langmuir (Eddy *et al*., 2024b; Sirijaree & Praipipat, 2023). Also, the Freundlich n value indicates the nature of adsorption and correlates to favourable adsorption when n is between 1 and 10 (Sirijaree & Praipipat, 2023). The Freundlich model yielded n = 2.622, indicating that lead (II) ions adsorb well onto the biochar. The favourable R_L and n values suggest that the carbon-based material derived from breadfruit seed hull could be used as an effective biosorbent. The Temkin model exhibited a higher coefficient of determination (R^2) for the adsorption of lead (II) ion compared to the Freundlich and Dubinin-Radushkevich models. The Temkin model (R^2 = 0.98371) implies adsorbate–adsorbent interactions, hinting at chemisorption (Ogoko *et al*., Ogoko *et al*., 2023). The D–R model, with the lowest R^2, supports physisorption based on the mean free energy (E = 12.30 kJ/mol). These findings are visually summarized in Fig. 1, which displays the linear plots for each isotherm model. The Langmuir plot stands out with the highest degree of linearity, reinforcing the model's suitability.

Table 1: Equilibrium isotherm constants for the adsorption of Pb (II) ion on BFSHBC

Model	Parameter	Value
Langmuir	q_L (mg/g)	13.58
	K_L (L/mg)	0.238
	R^2	0.99595
	SSE	0.00873
Freundlich	K_F	3.650
	1/n	0.38127
	n	2.622
	R^2	0.96338
	SSE	0.00489
Temkin	A (L/g)	2.651
	B (mg/g)	2.821
	R^2	0.98371
	SSE	0.61843
Dubinin-Raduskevich	qm (mg/g)	10.327
	B (mol^2/J^2)	-6.967E-7
	R^2	0.89358
	SSE	0.07531

3.3 Kinetic model adsorption

The kinetic behaviour of Pb(II) adsorption was studied using pseudo-first-order (PFO), pseudo-second-order (PSO), intraparticle diffusion (IPD), and liquid film diffusion (LFD) models. The kinetic constants and regression values are summarized in Table 3. while the plots are illustrated in Fig. 3. Based on the coefficient of-determination and the sum of squared errors (SSE) for the different kinetic models, the PSO model best fits the experimental data (R^2 = 0.99979), and its calculated qe (7.4 mg/g) closely aligns with the experimental qe (7.25 mg/g), indicating that chemisorption may be the rate-limiting step (Ogoko *et al*., 2023). . The PFO model shows a significant deviation between calculated and experimental q_e and a lower R^2 value, indicating a less suitable degree of fitness. Also, the IPD model suggests that while intraparticle diffusion contributes to adsorption, it is not the only controlling mechanism, as the line does not pass through the origin. Similarly, the LFD model implies the significance of boundary layer diffusion.

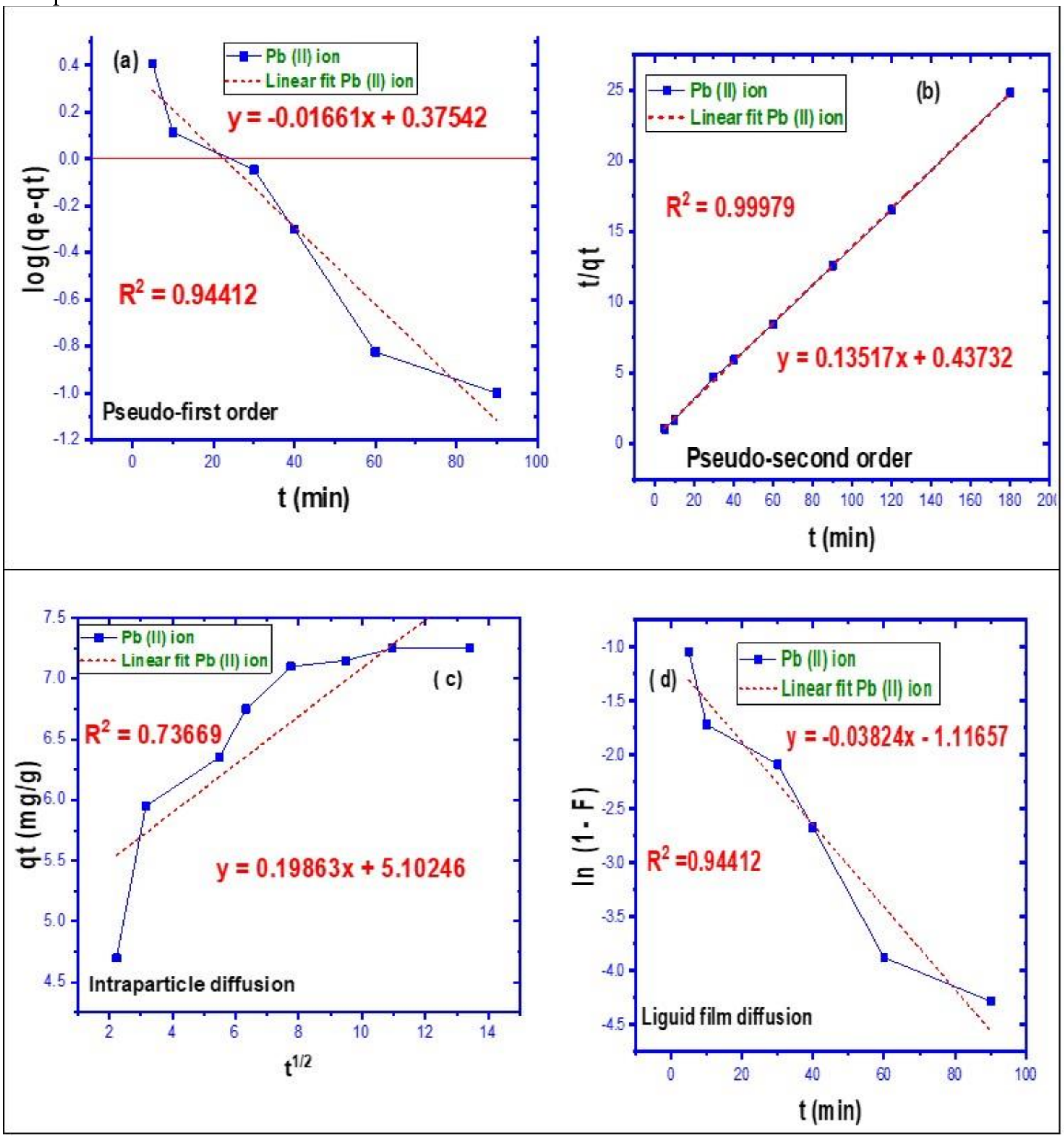

Fig. 2: Kinetic Modelling (a) Pseudo-first-order (PFO), (b) Pseudo-second-order (PSO) (c) Intraparticle diffusion (ITD) (d) Liquid film diffusion (LFD) on the removal of Pb (II) ion onto biochar.

3.4 Thermodynamic of adsorption

The thermodynamics of lead (II) ion adsorption on BFSHBC was also calculated from the Van't Hoff plot to evaluate the feasibility of the adsorption process, as depicted in Fig. 4. The calculated thermodynamic parameters, including changes in entropy (ΔS^o), free energy (ΔG^o), and enthalpy (ΔH^o), were used to analyze the randomness, spontaneity and the physical or chemical nature of lead (II) ion adsorption onto BFSHBC. (Huda *et al*., 2023). The calculated thermodynamic parameters obtained are presented in Table 3. The adsorption of lead (II) ions on the prepared biochar adsorbent is spontaneous, as the ΔG^o values were negative at all temperatures.

Table 2: Kinetic model constants for the adsorption of Pb (II) ion onto biochar

Model	Parameter	Value
Pseudo-first-order	qe_{exp} (mg/g)	7.25
	qe_{cal} (mg/g)	2.4
	K_I (min^{-I})	0.04
	R^2	0.94412
	SSE	0.0836
Pseudo-second-order	qe_{cal} (mg/g)	7.4
	h (mg/g min)	2.30
	K_2 (g/mg min)	4.2×10^{-2}
	R^2	**0.99979**
	SSE	0.09909
Intraparticle diffusion	K_d (mg/g min$^{1/2}$)	0.19863
	C	5.10246
	R^2	0.73669
	SSE	1.44921

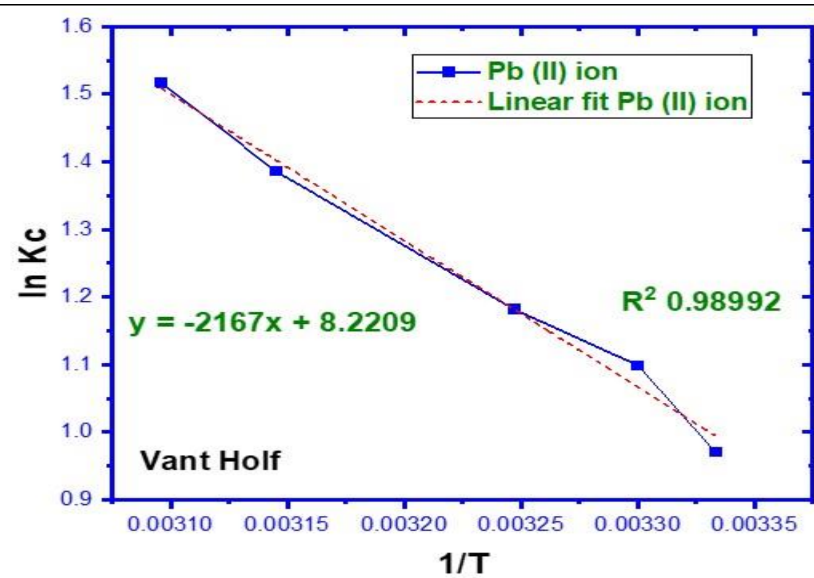

Fig. 4. Van't Hoff plot for the adsorption of Pb (II) ion onto BFSHBC.

The negative ΔG° values indicate that the adsorption process is spontaneous at all studied temperatures. The increase in the magnitude of the negative values with temperature also indicates that the adsorption becomes more spontaneous as the temperature increases. The positive ΔH° (18.016 kJ/mol) confirms that the process is endothermic, and the positive ΔS° value (68.349 J/mol·K) reflects increased randomness at the solid–solution interface. The magnitude of ΔH° falls within the range typically associated with physisorption (2–40 kJ/mol), supporting the conclusion that physical adsorption must have occurred before chemisorption.

Table 3: Thermodynamic parameters for Pb (II) ion adsorption onto BFSHBC

Temp (K)	Kc	ΔGº(kJ/mol)	ΔHº(kJ/mol)	ΔSº(J/mol K)	R^2
300	2.64	-2.42	18.016	68.349	**0.98992**
303	3.00	-2.77			
308	3.26	-3.03			
318	4.00	-3.67			
323	4.56	-4.07			

5.0 Conclusion

The study investigated the adsorption of lead(II) ions onto biochar derived from breadfruit seed hull using various isotherm models, including Langmuir, Freundlich, Temkin, andDubinin–Radushkevich. The results revealed that the Langmuir isotherm model provided the best fit for the experimental data, with a high coefficient of determination (R^2 = 0.99595) and low error values, suggesting monolayer adsorption on a homogeneous surface and indicating a chemisorption mechanism. The dimensionless separation factor (RL) values ranging from 0.10 to 0.41 further confirmed the favorability of the adsorption process. Although the Freundlich model also indicated favourable adsorption with an adsorption intensity (n) of 2.622, its lower R^2 and higher SSE compared to the Langmuir model implied a less accurate fit, supporting the predominance of monolayer adsorption. The Temkin model, with a relatively high R^2, indicated the presence of adsorbent–adsorbate interactions, while the Dubinin–Radushkevich model, having the lowest R^2, suggested the possibility of physisorption based on the calculated mean free energy. These findings highlight the suitability of breadfruit seed hull biochar as an effective biosorbent for lead ion removal from aqueous solutions. It is concluded that the adsorption mechanism is best described by the Langmuir model, indicating a uniform distribution of active sites and monolayer coverage. Based on these findings, it is recommended that breadfruit seed hull biochar be further developed and optimized for use in water purification systems, particularly for the removal of heavy metals such as lead. Additionally, scale-up studies and regeneration tests should be conducted to evaluate the reusability and long-term performance of the biosorbent under real environmental conditions.

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