

Adsorption of Fe and Pb from Aqueous Solution using Coconut Shell Activated Carbon

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Abstract: The wastewater produced by numerous industrial processes has seriously contaminated the ecosystem. Due to its excellent adaptability and low cost, adsorption is a potential pollutant remediation technology. Different agricultural wastes were used for the adsorption procedure because of their loose and porous structures and included active groups like carboxyl and hydroxyl, which aid in the binding of pollutants. An alternative agricultural by-product that can remove heavy metals is coconut shell. The coconut shell was activated by carbonization for two hours at a temperature of 600°C, followed by impregnation with zinc chloride in a ratio of 1:2 (zinc chloride: adsorbent). The agitation speed, pH, starting concentration, adsorbent mass, and contact time varied throughout the optimization studies on removing Fe and Pb. According to the findings, the adsorption equilibrium of both heavy metals was reached after 240 minutes. Initial concentration, initial pH, and agitation speed were the three elements that contributed to heavy metal removal. The maximum adsorption was demonstrated at pH 7 for Fe and pH 11 for Pb, where the highest adsorption capacities were 9.67 and 10.04, respectively. The results show that the second-order model best expressed the kinetic data for heavy metals adsorption and adsorption. The isotherm data, with R² values of 0.9981 for Fe and 0.9962 for Pb, demonstrated the compatibility of the Freundlich model with isotherm models. Using FTIR, the chemical properties of activated carbon changed both before and after the adsorption process, vanishing and forming a new peak.

Keywords: Heavy Metals, Adsorption, Coconut shell, Activated carbon.

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1. Introduction

Due to its abundance and environmental impact, heavy metal contamination in the aquatic environment has recently generated intense global notice. A significant number of dangerous chemicals, like synthetic dyes and heavy metals, have been dumped into rivers worldwide due to the rapid expansion in the world's population, rising home activity, and agricultural industrial production growth. Additionally, problems with water quality have been linked to metropolitan areas because of the regular practice of discharging toxic household and industrial waste into water bodies, increasing the amount of heavy metals in river water [1–3]. The water's chemistry and the sedimentary substrate's nature frequently correlate with how the

heavy metals react in natural water [4]. Additionally, several speciation modifications that were related to events, including sorption, dissolution, precipitation, and complexation phenomena, affected the behavior and bioavailability of heavy metals [5–6]. The interaction of heavy metals with various geochemical phases during sedimentation significantly impacts the overall responses and behavior of heavy metals in aquatic environments. To maintain a healthy riverine, it is essential to evaluate the concentration and dispersion of heavy metals in the aquatic environment; consequently, predictions of potential contamination and bioavailability frequently used geochemical speciation and the supply of heavy metals in determining chemical fraction [7–8].

Adsorption is typically recognized as a reliable and affordable approach for removing heavy metals from wastewater. Adsorption technology allows for flexible design and simple operation, producing excellent-quality treated effluent [9–12]. The most widely used adsorbent in removing organic contaminants from the aquatic environment, such as dyes and heavy metals, is activated carbon (AC). Generally speaking, its greater micropore and mesopore volumes were the source of its capabilities. Although activated carbon has the same surface area, different activation procedures lead to variable adsorption capabilities, making it challenging to select the best model for adsorption adsorbents with complicated chemical structures. The appropriate carbon adsorption method is frequently dependent on a number of variables, including the metallic species to be removed, the chemistry of the adsorbent surface, and the adsorption circumstances [11–14]. Due to the depletion of its sources, activation carbon prices have recently increased. Since activated carbon has been used frequently but is somewhat expensive, research on cheap and plentiful adsorbents for removing heavy metals has taken center stage.

Research has been done on low-cost adsorbents for considerable metal treatment, including industrial by-products and agricultural wastes [9–10]. Agricultural materials, in particular those containing cellulose, exhibit considerable metal biosorption potential. In the remediation of heavy metals, agricultural materials appear to be a viable alternative as an adsorbent [15–16]. However, the information on which agricultural materials are suitable for heavy metals remediation is still obscure. Though activated carbon plays an important role in the adsorption of dyes [11–13], its ability to remove Pb and Fe is still questioned. This study aimed to determine heavy metal adsorption by the coconut shell-activated carbon understanding of the chemical characteristic of the adsorption mechanism. The kinetic and isotherm models of heavy metals adsorption and the effect of some parameters in the adsorption process were also investigated. Coconut shell-activated carbon could be an effective alternative adsorbent for removing heavy metals from polluted environments.

2. Materials and Methods

2.1. Preparation of activation carbon and heavy metal.

The activated carbon was created using coconut shells from the area. To achieve chemical activation prior to carbonization for two hours at a temperature of 600°C, dehydration and impregnation (1: 2; zinc chloride and adsorbent) were done. At a concentration of 100 mg/L of metal ions, the heavy metal stock solution (Fe and Pb) was created.

2.2. Batch Adsorption Experiment.

As indicated in Table 1, the batch adsorption investigation was carried out by adjusting a number of operating parameters, including contact time, initial heavy metal concentration, adsorbent dosage, pH, and agitation speed. In order to conduct the adsorption experiment, 50 mL of the sample solution was made and used. One (1) g/L of activated carbon was added to the sample solutions. A conical flask containing both the heavy metal sample solution and the adsorbent was put into the orbital shaker and shaken at a speed of 100 rpm for 240 minutes at room temperature (32 °C) until equilibrium was reached and filtered through Sartorius filter paper. Atomic Absorption Spectroscopy was used to calculate the amount of each heavy metal ion in the filtrate (AAS Perkin Elmer PinAAcle 900, United States).

Table 1. Operating parameter used in batch adsorption study.

Operating Parameter	Unit
Contact time (min)	5 – 270
Initial heavy metal concentration (mg/L)	10, 15, 20, 25, 30, and 35
Adsorbent dosage (g/L)	1, 2, 3, 4, 5 and 6
pH	3, 5, 7, 9 and 11
Agitation speed (rpm)	50, 100, 200 and 300

The removal and adsorption capacity at a specific time was calculated by using the equation as follows:

$$\text{Adsorption Capacity (mg/g)} = \frac{C_o - C_e}{M} \times V \quad (1)$$

$$\text{Removal Rate (\%)} = \frac{C_o - C_e}{C_o} \times 100 \quad (2)$$

Where M denotes the amount of adsorbent utilized, V is the amount of dye solution used, Co is the amount of dye solution used, and Ce is the amount of dye solution denoted after the adsorption process. Then, a kinetic investigation based on a contact time trial lasting between 5 and 270 minutes was conducted. Utilizing 1-6 g of activated carbon and a heavy metal concentration range of 10-35 mg/L, the impact of the adsorbent dosage was studied. The impact of agitation speed was then researched between 50 and 300 rpm. By adjusting the pH in the range of 2 to 11, the impact of pH on the adsorption of heavy metal was assessed. The heavy metal solution's initial pH value was 5.6. By adding 0.1 M sodium hydroxide or 0.1 M hydrochloric acid (HCl), the pH was changed (NaOH).

2.3. Adsorption isotherm and kinetics.

To the Langmuir model, it was assumed that monolayer adsorption occurs in nature. The Freundlich model, in contrast, is investigated under the presumption that the surface is heterogeneous and that heat is dispersed unevenly along the surface through a process called multiple-layer adsorption. Additionally, the Temkin model was used to study the nature of adsorption, and the Dubinin-Radushkevich model was used to describe how the porous structure affects adsorption. The collected result is plotted along with the linear model equation of the adsorption isotherm models of Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich. The R² value showed the graph's correlation, and the model that has an R² value that is closest to 1 is the one that fits the data the best (Table 2).

Table 2. Various experimental conditions on the adsorption of CV.

Isotherm Model	Linear Form	Reference
Langmuir	I. $\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m}$ II. $\frac{1}{q_e} = \left[\frac{1}{q_m K_L} \right] \frac{1}{C_e} + \frac{1}{q_m}$ III. $q_e = q_m - \left[\frac{1}{K_L} \right] \frac{q_e}{C_e}$ IV. $\frac{q_e}{C_e} = K_L q_m - K_L q_e$	[17]
Freundlich	$\text{Log } q_e = \text{Log } K_F + \frac{1}{n} \text{Log } C_e$	[18]
Temkin	$q_e = \frac{RT}{b_1 T} \ln A_1 T + \left(\frac{RT}{b_1 T} \right) \ln C_e$	[19]
Dubinin-Radushkevich	$\ln q_e = \ln q_s - k_{ad} \epsilon^2$	[20]

Based on adsorption capacity affected by initial heavy metal concentration, Pseudo-first-order and Pseudo-second-order models were used to separate the kinetic equations. When using the Elovich model, it was also assumed that adsorbent surfaces were heterogeneous and that a multilayer adsorption process was involved. The results are presented along with the linear model equations of the pseudo-first-order and pseudo-second-order kinetic adsorption models. The R² value showed the graph's correlation, and the model that had an R² value that was closest to 1 was found to have the best fit.

Table 3. The kinetic model used in this study.

Kinetic models	Equation
Pseudo-first-order	$\ln(q_{e \text{ exp}} - q_t) = \ln(q_{e \text{ cal}}) - k_1 t$
Initial heavy metal concentration (mg/L)	$\frac{t}{q} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e}$
Elovich	$q_t = \frac{1}{b} \ln(ab) + \frac{1}{b} \ln t$

2.4. Fourier-Transform Infrared Spectroscopy (FTIR).

The spectrum of the coconut shell-derived activated carbon was obtained both before and after adsorption, and the functional group on the adsorbent's surface layer was examined using the spectrum table. In the frequency range of 4000 to 450 cm⁻¹, the spectra were examined.

3. Results and Discussion

3.1. Effect of contact time.

Figure 1 heavy metal of Pb and Fe removal and adsorption capacity with different contact times, which varies of 5-270 min. The experimental results indicate that the adsorption of Pb ions exhibited an accelerated rate within the first 20 minutes. Furthermore, it was observed that the adsorption capacity of the activated carbon increased proportionally with the starting concentration of Pb. In addition, it is noteworthy that the adsorption of iron (Fe) onto activated carbon exhibited a comparable effect. Specifically, during the initial 40 minutes of the experiment, the adsorption process of Fe by activated carbon saw a quick acceleration, leading to a significant rise in adsorption capacity. A heightened driving force seen during the beginning phase of the experiment led to a quick adsorption process. This was attributed to the

rapid transfer of the metal surface of the adsorbent particles and the existence of exposed surface area, which contained active sites [14–15, 21]. Moreover, the exposed surface area and the number of active sites would decrease over time. The decrease in the driving force leads to a prolonged period for the metal ions to reach equilibrium, thus resulting in a decrease in the rate of adsorption [21]. The attainment of equilibrium state for activated carbon, with an initial concentration of 10 mg/L, requires a time period of 240 minutes for both heavy metals. According to the research, the process of adsorption of heavy metals involves the initial arrival of heavy metal molecules at the boundary layer, followed by their interaction with the adsorbent surface and subsequent penetration into the porous structure of the adsorbent [22–23]. The present study yielded comparable results, indicating that the adsorption of Pb and Fe required a significantly extended duration of contact.

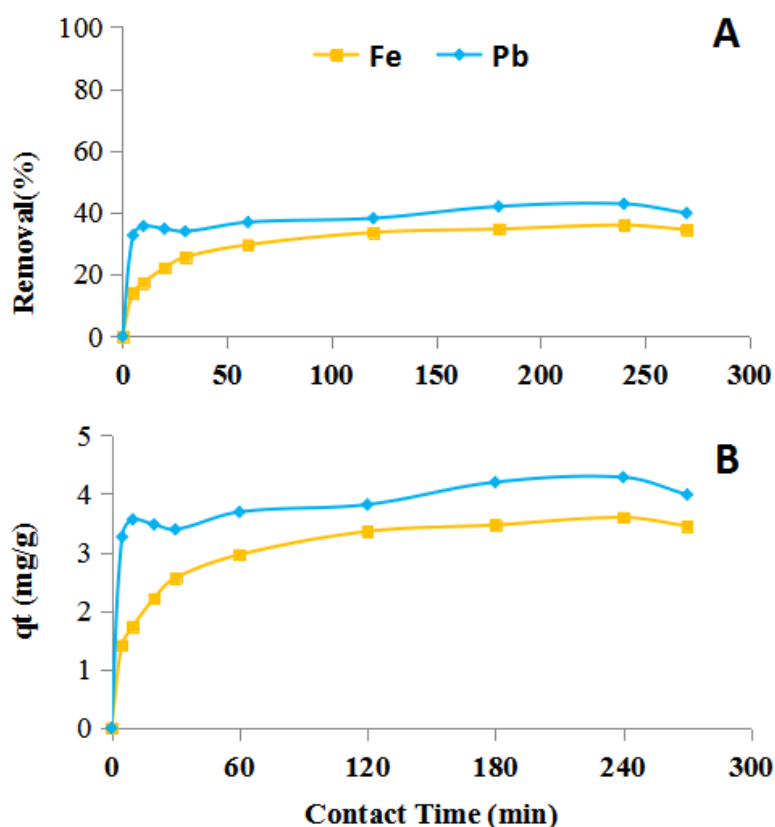


Figure 1. The Effect of Contact Time on The Removal (A) and Adsorption.

3.2. Effect of initial concentration.

To investigate the effects of initial concentration on the removal efficiency and adsorption capacity of the activated carbon, various initial concentrations of heavy metal were prepared, ranging from 10 mg/L to 35 mg/L: 15 mg/L, 20 mg/L, 25 mg/L, 30 mg/L, and 35 mg/L. For each heavy metal solution, the following experimental variables were fixed: contact time of 240 m, agitation speed of 100 rpm, dosage of 1 g/L, and all solutions at their natural pH with a volume of 50 milliliter. Figure 2 revealed that the highest Fe and Pb removal efficiencies were 35.93% and 42.7%, respectively. In addition, the greatest adsorption capacities for Fe and Pb were 10.18 mg/g and 12.54 mg/g, respectively. In addition, it was found that the initial concentration of heavy metal is directly proportional to the adsorption capacity, as an increase in the initial concentration of heavy metal would increase the adsorption capacity. The removal of heavy metals is dependent on the initial concentration; the

greater the accessibility of heavy metal ions in the solution, the quicker the adsorption due to the enhancement of dynamic contact between the adsorbent and the adsorbate. The outcome was comparable to the previous investigation [22–23]. Furthermore, the initial concentration of heavy metal is inversely proportional to its removal efficiency, such that an increase in the initial concentration of heavy metal would reduce its removal efficiency. The fraction of exposed surface decreased with increasing coverage; consequently, heavy metal ions must compete with one another for adsorption sites, resulting in a slower adsorption rate [24].

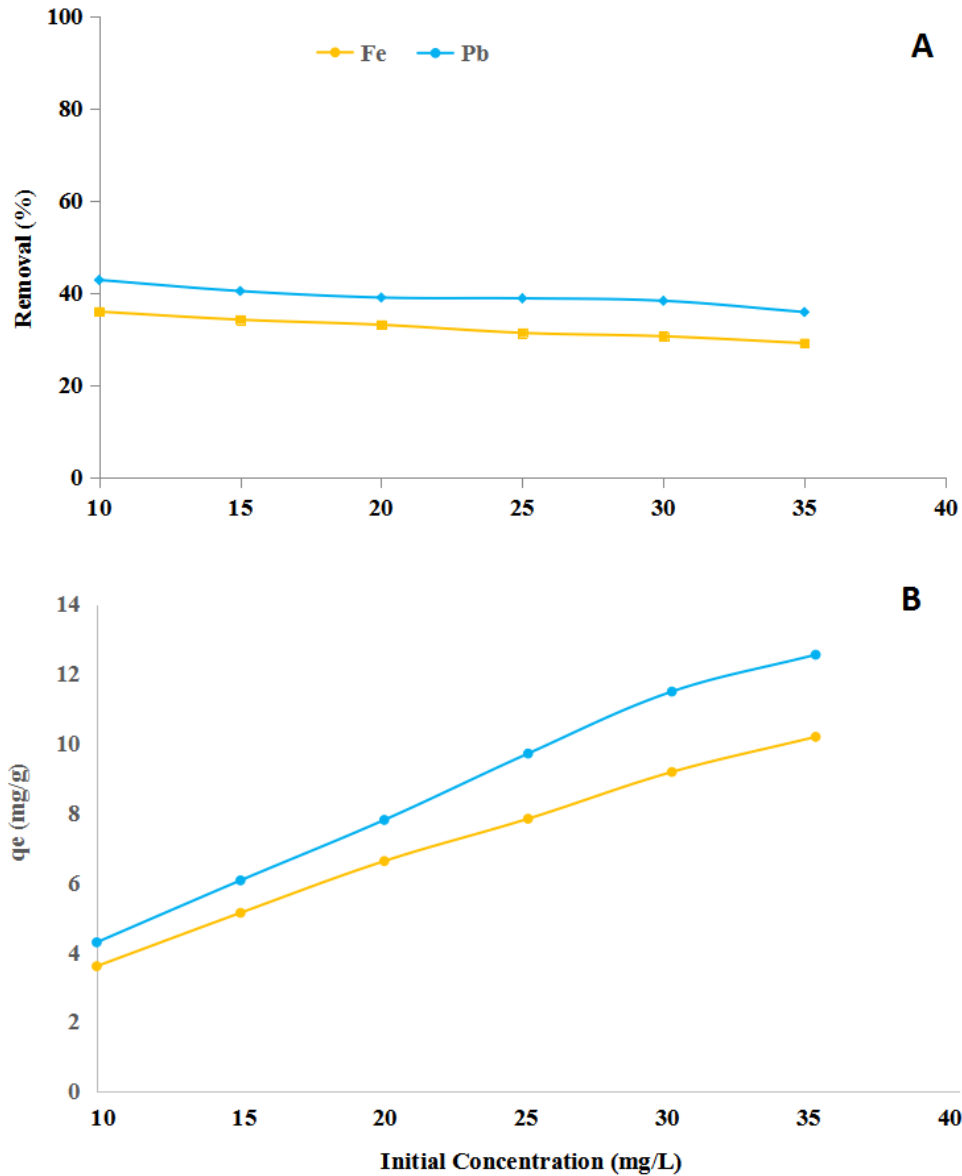


Figure 2. The effect of initial concentration on the removal (A) and Adsorption Capacity (B) of Fe and Pb.

3.3. Effect of adsorbent dosage.

Preparation of different adsorbent dosages ranging from 1 g/L, 2 g/L, 3 g/L, 4 g/L, 5 g/L, and 6 g/L in order to examine the effect of adsorbent dosage on the removal efficiency and adsorption capacity of the activated carbon. For each heavy metal, the constant variables contact time of 240 m, agitation speed of 100 rpm, initial concentration of 10 mg/L at its natural pH, and solution volume of 50 ml were fixed. Figure 3 revealed that the highest Fe and Pb removal efficiencies are 57.31% and 62.31%, respectively. In addition, the highest adsorption capacities attained for Fe and Pb are 3.69 mg/g and 4.19 mg/g, respectively. It has been

demonstrated that the adsorbent dosage is directly proportional to the removal efficiency, with an increase in adsorbent dosage increasing removal efficiency. This may be due to the presence of additional active adsorption sites and functional groups supplied by additional availability of pore volume and greater surface area of the adsorbent, which was discovered in higher adsorbent dosage, resulting in a higher removal percentage of heavy metal ions [25]. Nonetheless, the adsorbent dosage is inversely proportional to the adsorption capacity, such that an increase in adsorbent dosage would decrease adsorption capacity. In addition, an increase in adsorbent dosage above 1 g/L demonstrated a decline in adsorption capacity. This may be due to the fact that all active sites are fully exposed and utilized at lower levels of activated carbon. Increasing the dosage of the adsorbent would only result in certain active sites being exposed and occupied by the heavy metal ions, thereby decreasing its adsorption capacity. The obtained result is comparable and consistent with the previous study [26–27].

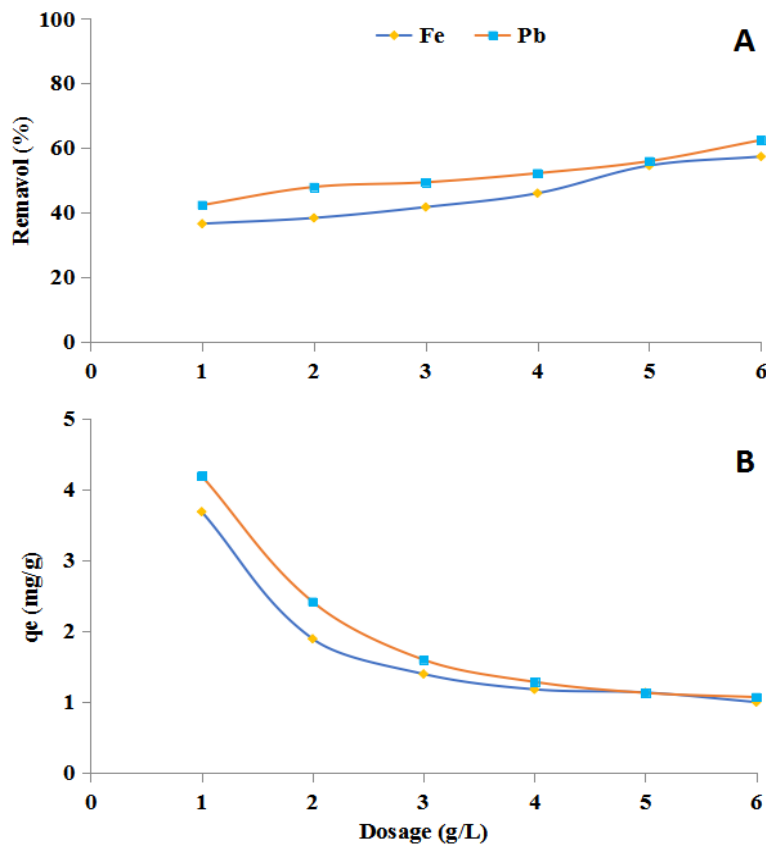


Figure 3. The effect of adsorbent dosage on the removal (A) and adsorption capacity (B) of Fe and Pb.

3.4. Effect of pH.

Figure 4 demonstrates that the highest Fe adsorption capacity is 9.67 mg/g, while the highest Pb adsorption capacity is 10.04 mg/g, and the highest Fe and Pb removal efficiencies are 99.84% and 99.12%, respectively. According to a previous study [28], the reduced adsorption rate of the heavy metal Pb at pH values below 7 is due to the excessive protonation of the carbon surface. Due to the reduced competition between proton (H^+) and the positively charged metal ion that was located at the surface sites, the repulsion of adsorbing metal ions was decreased as the pH increased. At a constant pH, the initial concentration of metal ions affected the removal efficiency of metal ions, with an increase in concentration decreasing the removal efficiency of heavy metal ions. In addition, a pH greater than 7 induces competition

between the carbon surface and the OH^- solution for Pb ions due to an excess of OH^- , resulting in a decrease in heavy metal ions adsorption on the carbon surface. After a pH greater than 7, metal hydrolysis and precipitation reached their maximum, demonstrating that pH significantly impacts adsorption capacity. Due to the amphoteric nature of iron in aqueous solution [29], the metal ions of the heavy metal Fe were completely adsorbed by activated carbon at a pH of 5 or greater.

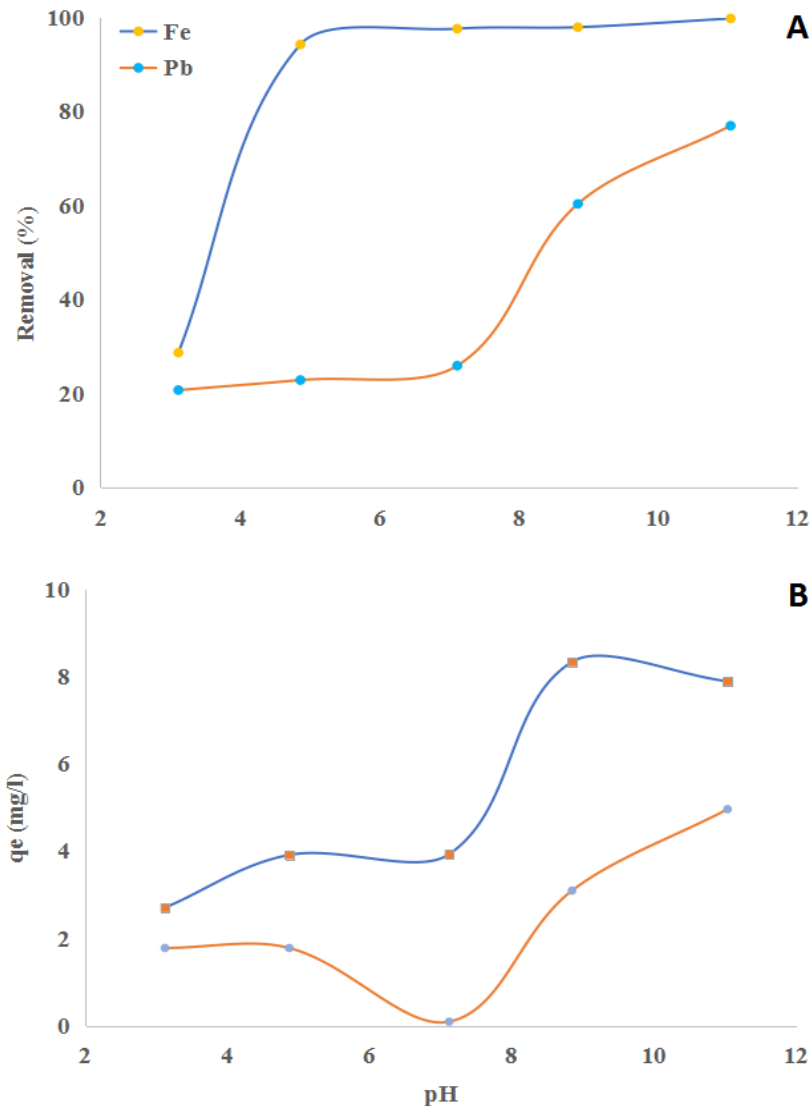


Figure 4. The Effect of pH on The Removal (A) and Adsorption Capacity (B) of Fe and Pb.

3.5. Effect of agitation speed.

To investigate the effect of agitation speed on the removal efficiency and adsorption capacity of activated carbon (ZnCl_2), samples were prepared with 50 rpm, 100 rpm, 200 rpm, and 300 rpm agitation speeds, respectively. The constant conditions were a contact time of 240 minutes, an adsorbent dosage of 1 g/L, a concentration of 10 mg/L for each heavy metal at its natural pH, and a solution volume of 50 ml. Figure 5 demonstrates that the highest removal capacity for Fe is 36.82%, and for Pb, it is 45.87%. In addition, the highest adsorption capacities for Fe and Pb are 3.75 mg/g and 4.70 mg/g, respectively. Due to the fact that an increase in agitation speed decreased the film boundary layer surrounding the particles, the adsorption capacity and the external film transfer coefficient increased. Based on these conditions, the

boundary layer develops gradually into a very thin layer, which approaches the laminar sublayer [14–15, 20].

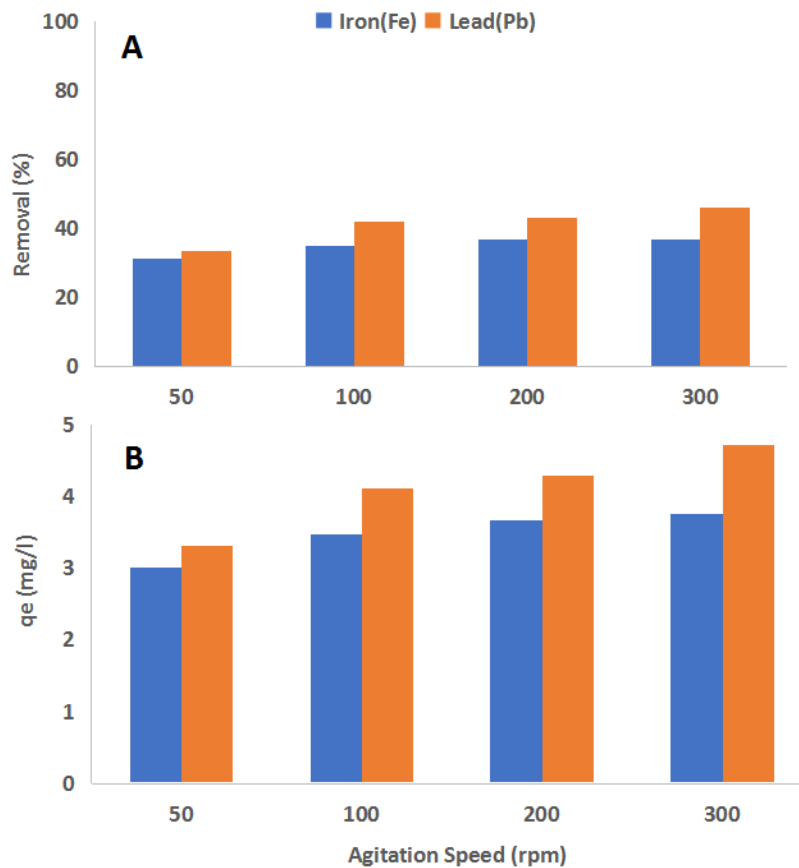


Figure 5. The Effect of Agitation Speed on The Removal (A) and Adsorption Capacity (B) of Fe and Pb.

3.6. Isotherm and kinetic studies.

To fully optimize the adsorption conditions for heavy metals, it is important to establish the most precise equilibrium curve correlation. Interaction between the solute-solution and the accumulation intensity of adsorbate on the adsorbent determined the adsorption capacity. The adsorption of heavy metals (Iron and Lead) onto ZnCl₂ synthesized activated carbon was investigated using four (4) isotherm models: Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich. Figures 6 and 7 depict the plot of isotherm parameters in the linearized equation for heavy metals (iron and lead). For Iron (Fe), the correlation coefficients (R²) follow the trend Freundlich > Langmuir > Temkin > Dubinin-Radushkevich, while for Lead (Pb), the trend is Freundlich > Temkin > Langmuir > Dubinin-Radushkevich. On the basis of the obtained experimental data, Freundlich isotherm was determined to be the best-fitting model for both Fe and Pb, with an R² of 0.9981 and 0.9962, respectively. The adsorption constant, exponent in the Freundlich isotherm model, where a value greater than 1 indicates the adsorption of the synthesized activated carbon, indicates the preferability of adsorption [9]. The Freundlich isotherm model indicates the presence of heterogeneity on the adsorbent surface, where the adsorptive surface of activated carbon is composed of small heterogeneous adsorption patches [14–15].

This study employs three (3) kinetic models to explain the mechanism behind the adsorption of the heavy metals iron and lead onto activated carbon. The linear form of the kinetic models was used to calculate and tabulate Table 4's specific adsorption constants. On

the basis of the table, it was determined that the Pseudo-second-order model best fit the experimental result. The R^2 for Iron (Fe) is 0.9956, while the R^2 for Lead (Pb) is 0.9968 when heavy metals are removed with activated carbon. The adsorption capacity (q_e) values obtained from the Pseudo-second-order equation, which are 3.6 mg/g for Iron (Fe) and 4.272 mg/g for Lead (Pb), are in a similar agreement with the experimental adsorption obtained, and the result was comparable to the previous study [30–33]. The sorption reaction between heavy metal and activated carbon follows a pseudo-second-order kinetic model, whereas the overall rate of the metal adsorption process is determined by a chemical process involving iron exchange or complexation [30–33].

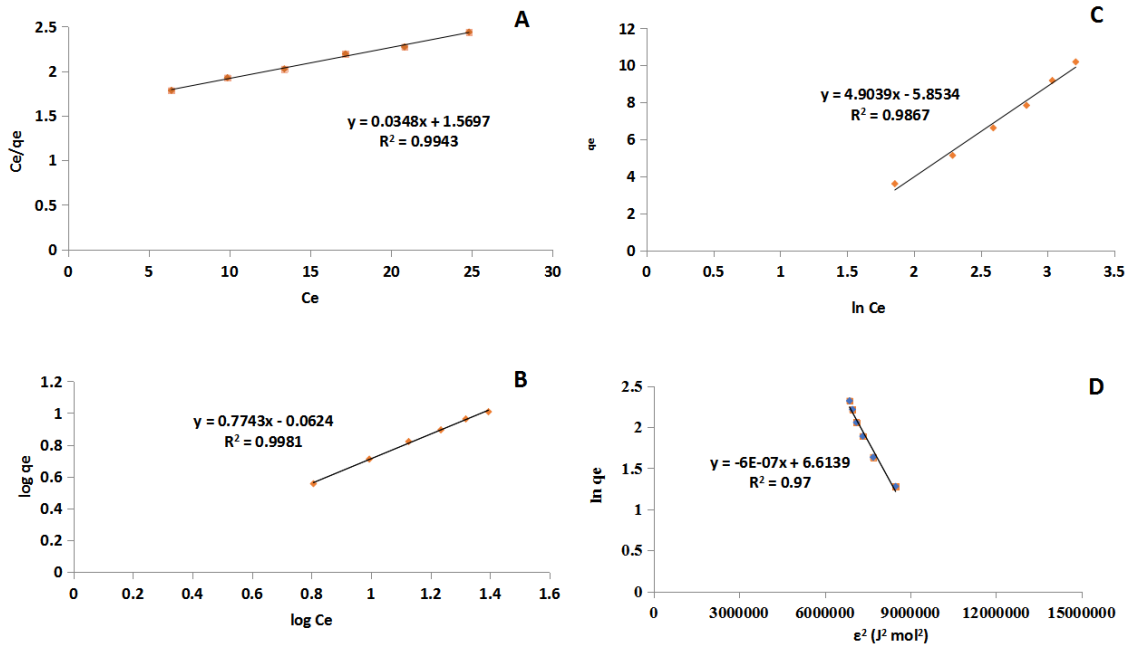


Figure 6. Isotherm Studies of Fe Adsorption on Coconut Shell Activated Carbon: Langmuir (A); Freundlich; Temkin (C); Dubinin-Radushkevich (D).

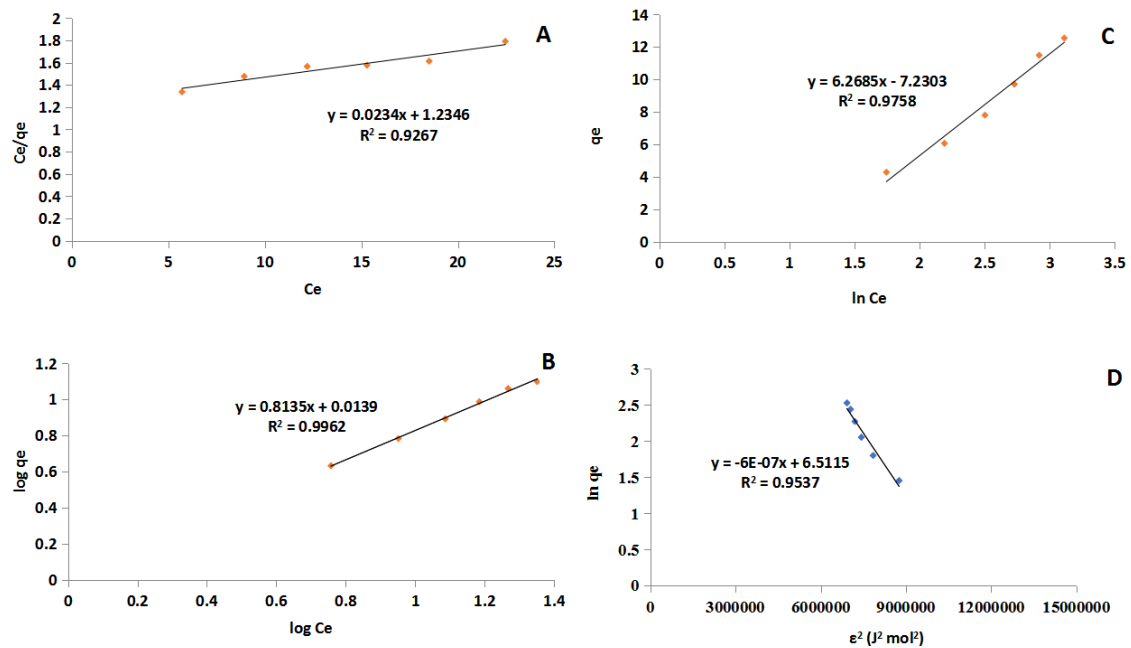


Figure 7. Isotherm Studies of Pb Adsorption on Coconut Shell Activated Carbon: Langmuir (A); Freundlich; Temkin (C); Dubinin-Radushkevich (D).

Table 4. Kinetic Parameter for Fe and Pb Adsorption on Coconut Shell Activated Carbon.

Heavy Metal	Kinetic Models								
	Pseudo-first-order			Pseudo-second-order			Elovich		
	q _e (mg/g)	K ₁ (1/min)	R ²	q _e (mg/g)	K ₂ (g/mg min)	R ²	b (g/mg)	a (mg/g min)	R ²
Fe	2.23	0.0172	0.9508	3.6	0.031	0.9956	2.211	0.6692	0.9222
Pb	2.071	0.0242	0.8238	4.274	0.049	0.9968	4.196	32566	0.5389

3.7. FTIR.

Table 5 provides a summary of the FTIR spectra before and after the adsorption of heavy metals (Fe and Pb). Before and after adsorption, the different functional groups were identified on the surface of the activated carbon. In addition, various changes in conditions, such as shifting, vanishing, and the formation of new peaks, are observed after the absorption of heavy metals. After adsorption, new peaks appear at 3369.51 cm⁻¹ for Fe and 3280.06 cm⁻¹ for Pb, while the peak at 2669.32 cm⁻¹ (H-C=O: C-H stretch) for Pb disappears, and the peak at 2586.77 cm⁻¹ (H-C=O: C-H stretch) for Fe shifts slightly. Peaks shifted from 2124.58 cm⁻¹ to 2161.86 cm⁻¹ and 2184.22 cm⁻¹ after the adsorption of Fe and Pb, for example. In addition, additional shifting has occurred between 1586.57 cm⁻¹ and 1632.57 cm⁻¹ for both Fe and Pb, and the remaining peaks at 1103.29 and 946.74 cm⁻¹ have undergone continuous shifting.

Table 5. FTIR Spektra Characteristic of Fe and Pb Adsorption on Coconut Shell Activated Carbon.

AC Before adsorption (cm ⁻¹)	After Fe adsorption (cm ⁻¹)	After Pb adsorption (cm ⁻¹)	Functional Group	Compound Class
-	3369.51	3280.06	O-H stretch, H-bonded, N-H stretch, O-H stretch	alcohols, phenols, 1°, 2° amines, amides, carboxylic acids
2669.32	2586.77	-	H-C=O: C-H stretch	aldehydes
2124.58	2161.86	2184.22	-C≡C- stretch	alkynes
1997.85	1938.22	1990.40	C-H bending, C=O stretching	aromatic compound, anhydride
1586.57	1632.57	1632.57	N-H bend	1° amines
1103.29	1066.02	1140.57	C-N stretch, C-O stretch	aliphatic amines, aromatic amines, alcohols, carboxylic acids, esters, ethers
946.74	961.65	969.11	=C-H bend	alkenes

4. Conclusions and Recommendations

The activated carbon derived from coconut shell was a suitable adsorbent for ion (Fe) and lead (Pb) due to its capacity to retain heavy metal ions in aqueous solution at given concentrations. In accordance with the results of both experiments, the adsorbent reached equilibrium adsorption for both heavy metals within 240 minutes of contact. The adsorption capacity for heavy metal ions Fe and Pb decreased as the adsorbent dosage increased. In addition, the percentage of removal for both heavy metal ions decreased with increasing initial concentration, whereas the adsorption capacity of the adsorbent increased rapidly with increasing initial concentration. In addition, a pH greater than 5 demonstrated that the removal percentage for both heavy metal ions has exponentially increased close to 100 percent, with an adsorption capacity of 9.67 mg/g for Fe and 10.04 mg/g for Pb. In addition, the removal percentage has gradually increased as the agitation speed has increased. Heavy metal ion adsorption depends on time, initial concentration, adsorbent dosage, pH, and agitation speed. Pseudo-first-order, Pseudo-second-order, and Elovich models suggested that the best-fitted

model was a second-order model that provided the best correlation of sorption and suggests that the rate-limiting step may be chemical sorption rather than diffusion. Using four isotherm models (Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich), Freundlich demonstrated that the surface of activated carbon is heterogeneous for both heavy metals. Therefore, activated carbon from coconut shells could be a cost-effective adsorbent for removing heavy metals, particularly Fe and Pb. However, additional research is required to enhance the absorption capacity of activated carbon.

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Conflicts of Interest

The authors declare no conflict of interest.

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