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RESEARCH PAPER

Preparation and Characterization of Magnetite-Modified Coconut Shell Biochar Nanocomposites for Efficient Pb(II) Removal from Water

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ABSTRACT

Lead (Pb(II)) is a highly toxic, persistent, and non-biodegradable heavy metal that poses substantial risks to environmental and human health. Given these challenges, the development of sustainable, low-cost, and efficient adsorbents derived from renewable biomass has emerged as a particularly promising strategy for mitigating heavy metal contamination in aquatic environments. In this study, activated biochar (AB) was synthesized from coconut shells via oxygen-limited pyrolysis at 500 °C and subsequently functionalized with magnetite (Fe_3O_4) nanoparticles to produce a magnetic biochar nanocomposite (MBC). The incorporation of Fe_3O_4 was intended to enhance adsorption performance while simultaneously enabling rapid magnetic separation of the adsorbent following water treatment. The Pb(II) adsorption behavior of both AB and MBC was systematically investigated by examining the effects of solution pH, adsorbent dosage, initial metal ion concentration, and temperature. The morphological and structural properties of the synthesized materials were characterized using field-emission scanning electron microscopy (FE-SEM). Results revealed that the activated biochar possessed a well-developed porous structure, while the uniform deposition of Fe_3O_4 nanoparticles onto its surface generated a magnetic nanocomposite featuring nanoscale pores with diameters ranging from approximately 14 to 25 nm, thereby increasing the availability of active adsorption sites. Across all experimental conditions examined, the magnetic biochar exhibited superior Pb(II) adsorption performance relative to the unmodified biochar. A maximum adsorption capacity of 505 mg g^{-1} was achieved at an adsorbent dosage of 0.08 g L^{-1} and an initial Pb(II) concentration of 50 mg L^{-1}. Conversely, increasing the adsorbent dosage to 0.40 g L^{-1} led to a marked decrease in adsorption capacity to 62.25 mg g^{-1}, primarily attributable to particle aggregation and incomplete utilization of the available adsorption sites. Beyond its high adsorption efficiency, the magnetic biochar demonstrated facile recovery from aqueous solution through the application of an external magnetic field, thereby eliminating the need for conventional solid–liquid separation methods and substantially enhancing its practical applicability. Collectively, these findings indicate that magnetite-modified coconut shell biochar constitutes an efficient, environmentally sustainable, and economically viable adsorbent for Pb(II) removal from contaminated water. The combination of high adsorption capacity, straightforward magnetic recovery, and the valorization of renewable agricultural waste underscores its considerable potential for large-scale wastewater treatment applications and sustainable environmental remediation.

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INTRODUCTION

Water pollution caused by toxic contaminants has become one of the most critical environmental challenges worldwide. Rapid industrialization, urbanization, and population growth have significantly increased the discharge of hazardous substances into aquatic ecosystems, resulting in the deterioration of both surface water and groundwater quality. Among these contaminants, heavy metals, pesticides, volatile organic compounds (VOCs), petroleum hydrocarbons, pharmaceuticals, and synthetic dyes are frequently detected in industrial effluents. Unlike many organic pollutants, heavy metals are non-biodegradable, persistent, and capable of accumulating in living organisms, posing long-term ecological and public health risks [1–3].

Lead (Pb) is one of the most toxic and widely distributed heavy metals in the environment. Owing to its desirable physicochemical properties, including high malleability, corrosion resistance, low melting point, and ease of processing, lead is extensively used in battery manufacturing, metal plating, pigments, mining, smelting, cable production, ceramics, and electronic industries. Consequently, wastewater generated from these industrial activities often contains elevated concentrations of Pb(II), which can be discharged into aquatic environments if inadequately treated [1–3]. Once released into the environment, lead is highly persistent and readily accumulates in soils, sediments, and aquatic organisms, thereby entering the food chain through bioaccumulation and biomagnification.

Lead exposure has severe toxicological effects on both humans and wildlife. Even at relatively low concentrations, Pb(II) can damage the nervous, renal, cardiovascular, reproductive, and hematopoietic systems. Children are particularly vulnerable because their developing nervous systems are highly sensitive to lead toxicity, which may result in cognitive impairment, learning disabilities, behavioral disorders, reduced intelligence quotient (IQ), and irreversible neurological damage. Owing to these adverse health effects, the World Health Organization (WHO) and environmental regulatory agencies have established stringent limits for lead concentrations in drinking water and industrial wastewater [4,5].

Various technologies have been developed for the removal of heavy metals from contaminated water, including chemical precipitation, coagulation-flocculation, ion exchange, membrane filtration, electrochemical treatment, solvent extraction, and adsorption [6,7]. Although many of these methods exhibit satisfactory removal efficiencies, they are often associated with significant operational limitations, such as high energy consumption, sludge generation, membrane fouling, complex operation, and elevated capital and maintenance costs. Consequently, adsorption has emerged as one of the most attractive treatment technologies because of its operational simplicity, high removal efficiency, ease of regeneration, and economic feasibility, particularly for the treatment of wastewater containing low concentrations of heavy metals [7].

Biochar has recently attracted considerable attention as a sustainable adsorbent for environmental remediation. It is a carbon-rich porous material produced through the thermochemical conversion of biomass under oxygen-limited conditions. The physicochemical properties of biochar, including surface area, pore structure, mineral composition, surface functional groups, and aromaticity, are strongly influenced by the feedstock type and pyrolysis conditions [8–10]. Owing to its highly porous structure and the presence of oxygen-containing functional groups, such as hydroxyl, carboxyl, and carbonyl groups, biochar exhibits excellent adsorption capability toward a wide range of inorganic and organic pollutants through ion exchange, electrostatic attraction, surface complexation, pore filling, and precipitation mechanisms [8–10].

Among the various biomass feedstocks, coconut shells represent an abundant, renewable, and inexpensive agricultural by-product with high carbon content and excellent thermal stability. Converting coconut shell waste into biochar not only provides an environmentally friendly adsorbent but also contributes to agricultural waste valorization and supports circular economy principles. Nevertheless, pristine biochar generally exhibits limited adsorption capacity and presents practical challenges during post-treatment separation because of its fine particle size and low density.

To overcome these limitations, considerable research has focused on modifying biochar with metal oxides and magnetic nanoparticles. Among these materials, magnetite (Fe_3O_4) has

received particular attention because it possesses excellent magnetic properties, chemical stability, environmental compatibility, and abundant surface hydroxyl groups that can participate in heavy-metal adsorption. The incorporation of Fe_3O_4 nanoparticles into the biochar matrix not only enhances the number of active adsorption sites but also enables rapid magnetic separation of the spent adsorbent from treated water, thereby simplifying operation and improving the economic viability of the treatment process.

Although numerous studies have reported the application of magnetic biochar for heavy-metal removal, adsorption performance varies considerably depending on the biomass precursor, synthesis method, surface characteristics, and operating conditions. Furthermore, information regarding magnetite-modified coconut shell biochar for Pb(II) removal remains relatively limited, particularly concerning the relationship between structural modification and adsorption performance.

Therefore, the objective of the present study was to prepare activated biochar from coconut shells and modify it with magnetite (Fe_3O_4) nanoparticles to produce a magnetic biochar nanocomposite for efficient Pb(II) removal from aqueous solutions. The adsorption performances of activated biochar and magnetic biochar were systematically compared by investigating the effects of solution pH, adsorbent dosage, initial Pb(II) concentration, and temperature. In addition, the synthesized materials were characterized using field-emission scanning electron microscopy (FE-SEM) to evaluate their surface morphology and structural characteristics. The findings of this study provide valuable insights into the development of sustainable, low-cost, and magnetically recoverable biochar-based adsorbents for the remediation of lead-contaminated wastewater.

The present study aimed to develop an environmentally sustainable and highly efficient magnetic biochar-based adsorbent for the removal of Pb(II) ions from aqueous solutions. The specific objectives were as follows:

1. To synthesize a magnetite-modified biochar (MBC) nanocomposite using coconut shell (Cocos nucifera) biomass as an abundant, renewable, and low-cost carbon precursor.

2. To characterize the surface morphology of activated biochar (AB) and magnetite-modified biochar (MBC) using field-emission scanning electron microscopy (FE-SEM) in order to evaluate the structural changes induced by magnetite incorporation.

3. To investigate the adsorption performance of the synthesized MBC for Pb(II) removal from aqueous solutions and determine its maximum adsorption capacity while assessing its magnetic recoverability after the adsorption process.

4. To elucidate the adsorption mechanism through equilibrium isotherm, adsorption kinetic, and thermodynamic analyses.

5. To optimize the adsorption process using Response Surface Methodology (RSM) based on a Box–Behnken Design (BBD) by evaluating the effects of solution pH, adsorbent dosage, initial Pb(II) concentration, and contact time on Pb(II) removal efficiency.

MATERIALS AND METHODS

Materials

Collection of Raw Materials

Coconut shells (Cocos nucifera) were obtained from a local market in Hilla, Babylon Governorate, Iraq, and were used as the biomass precursor for biochar production. Water samples used throughout this study were collected from several locations in Hilla City and stored in clean, pre-labeled polyethylene containers prior to use to prevent contamination.

Preparation of Coconut Shell Biochar

The collected coconut shells were thoroughly washed with distilled water to remove adhering dust and impurities. The cleaned shells were crushed into small pieces and dried in a laboratory oven at 110 °C for 1 h to remove residual moisture before chemical activation and pyrolysis.

Preparation of Activated Biochar (AB)

Activated biochar (AB) was prepared using a chemical activation-assisted pyrolysis method. The dried coconut shell powder was impregnated with concentrated sulfuric acid at a biomass-to-acid ratio of 1:2 (w/v). Following impregnation, the material was repeatedly washed with distilled water until a neutral pH was achieved to eliminate residual acid. The washed biomass was subsequently dried at 80 °C for 2 h.

Pyrolysis was carried out in a tubular furnace at 500 °C for 60 min under a continuous argon atmosphere (20 mL min^{-1}) with a heating rate of 10 °C min^{-1}. After naturally cooling to room

temperature, the carbonized product was finely ground and stored in airtight polyethylene containers for subsequent characterization and adsorption experiments [6].

Preparation of Magnetite-Modified Biochar (MBC)

Magnetite-modified biochar (MBC) was synthesized by depositing Fe_3O_4 nanoparticles onto the surface of activated biochar using the co-precipitation method. Briefly, 10 g of activated biochar was dispersed in 400 mL of distilled water. Subsequently, 3.4 g of $FeSO_4{\cdot}7H_2O$ and 6.6 g of $FeCl_3{\cdot}6H_2O$ were dissolved in the suspension, which was stirred continuously at 100 rpm and maintained at 80 °C for 30 min.

A 25% ammonium hydroxide (NH_4OH) solution was then added dropwise until the suspension reached pH 10–11, promoting the in situ precipitation of Fe_3O_4 nanoparticles onto the biochar surface. The reaction mixture was continuously stirred for an additional 30 min to ensure complete formation and homogeneous distribution of magnetite nanoparticles.

The resulting black magnetic composite was separated using an external permanent magnet, washed repeatedly with distilled water until neutral pH was achieved, and dried at 80 °C for 24 h. Finally, the dried material was ground into a fine powder and stored in airtight containers in a dry environment until use. The synthesized adsorbent is hereafter referred to as magnetite-modified biochar (MBC) [21,22].

Morphological Characterization

The surface morphology of activated biochar (AB) and magnetite-modified biochar (MBC) was examined using a field-emission scanning electron microscope (FE-SEM, ZEISS Sigma 300, Germany). FE-SEM analysis was performed to evaluate changes in surface texture, pore structure, particle size, and the distribution of Fe_3O_4 nanoparticles following magnetic modification.

Batch Adsorption Experiments

The adsorption performance of magnetite-modified biochar (MBC) toward Pb(II) ions was evaluated through batch adsorption experiments designed using Response Surface Methodology (RSM). Among the available RSM techniques, the Box–Behnken Design (BBD) was selected because it efficiently evaluates the individual and interaction effects of multiple variables while requiring a relatively small number of experimental runs.

Four independent variables were investigated: solution pH (5, 7, and 9), contact time (5, 10, and 15 min), initial Pb(II) concentration (30, 40, and 50 mg L^{-1}), and adsorbent dosage (0.08, 0.24, and 0.40 g L^{-1}). The experimental design matrix generated using Design-Expert® software is presented in Table 1.

For each experimental run, a predetermined amount of MBC was added to Pb(II) solutions prepared at the desired initial concentrations. The suspensions were agitated under the conditions specified by the experimental design until equilibrium was attained. Following adsorption, the magnetic adsorbent was rapidly recovered using an external permanent magnet, thereby eliminating the need for filtration or centrifugation.

The residual Pb(II) concentration in the aqueous phase was determined using atomic absorption spectroscopy (AAS). The equilibrium adsorption capacity, (q_e) (mg g^{-1}), and Pb(II) removal efficiency (%) were subsequently calculated using Eqs. (2) and (3), respectively [8].

RESULTS AND DISCUSSION

Coconut Shell as a Precursor for Biochar Production

Coconut shell (Cocos nucifera) is an abundant lignocellulosic agricultural by-product with high carbon content, making it an excellent precursor for the production of biochar. Its composition, which is primarily cellulose, hemicellulose, and lignin, provides structural stability during pyrolysis and promotes the formation of a highly carbonized

Table 1. Experimental variables and levels employed in the Box–Behnken Design (BBD).

Variable	Symbol	−1	0	+1
Contact time (min)	A	5	10	15
Solution pH	B	5	7	9
Initial Pb(II) concentration (mg L^{-1})	C	30	40	50
Adsorbent dosage (g L^{-1})	D	0.08	0.24	0.40

porous material. In addition, naturally occurring inorganic constituents, including potassium and calcium, contribute to the physicochemical properties of the resulting biochar and may enhance its adsorption performance.

During pyrolysis, the thermal decomposition of lignocellulosic components generates a carbon-rich framework with a porous architecture and numerous surface functional groups. These characteristics make coconut shell biochar an attractive adsorbent for environmental remediation applications. Owing to its large surface area, porous structure, and oxygen-containing functional groups, coconut shell biochar has demonstrated excellent potential for the removal of heavy metals, dyes, pharmaceuticals, and other organic pollutants from contaminated water.

Besides wastewater treatment, coconut shell biochar has received increasing attention as a renewable carbon material for soil improvement, carbon sequestration, catalyst support, and energy production. Consequently, converting coconut shell waste into biochar not only provides a value-added adsorbent but also contributes to sustainable biomass utilization and circular economy practices. Fig. 1 illustrates the raw coconut shell used as the precursor material and the activated biochar produced after pyrolysis. Fig. 1. (A) Raw coconut shell (Cocos nucifera). (B) Activated biochar (AB) produced by pyrolysis.

Surface Morphology of the Adsorbents

The surface morphology of activated biochar (AB) and magnetite-modified biochar (MBC) was investigated using field-emission scanning electron microscopy (FE-SEM), and the corresponding micrographs are presented in Fig. 2.

The FE-SEM image of activated biochar (Fig. 2A) revealed a heterogeneous porous structure with irregular cavities and interconnected channels generated during the pyrolysis process. The removal of volatile organic compounds during thermal decomposition created numerous pores that are expected to provide abundant adsorption sites for Pb(II) ions. The preserved fibrous architecture of the coconut shell indicates that the biomass maintained its structural integrity while developing a porous carbon framework suitable for adsorption.

Following magnetite modification, considerable changes in surface morphology were observed (Fig. 2B). Numerous Fe_3O_4 nanoparticles were uniformly deposited on the biochar surface, resulting in a rougher texture and increased

(A) (B)

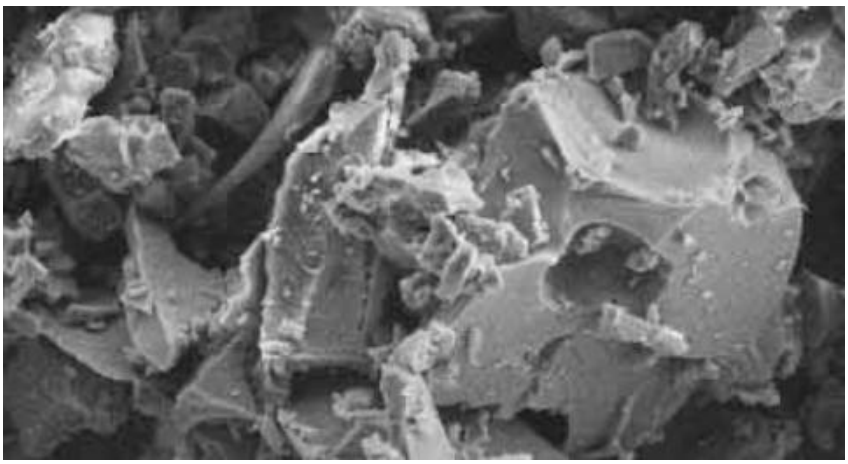
Fig. 1. [A] Coconut shell particle, [B] Coconut shell Biochar.

surface heterogeneity. The nanoparticles were well distributed throughout the pore network with only minor agglomeration, indicating successful immobilization of magnetite onto the carbon matrix. The intimate contact between the magnetic nanoparticles and the biochar surface is expected to increase the number of available active adsorption sites while simultaneously imparting magnetic properties that facilitate rapid recovery of the adsorbent after treatment.

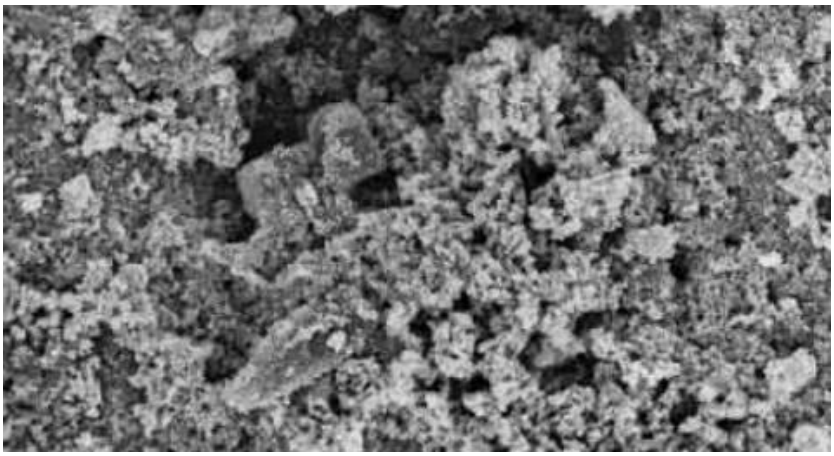
The improved surface roughness and development of additional nanoscale pores after Fe_3O_4 incorporation are expected to enhance Pb(II) adsorption through increased surface area and stronger interactions between the metal ions and surface functional groups. These observations confirm the successful synthesis of the magnetic biochar composite and support its improved adsorption performance observed in subsequent experiments.

FTIR Analysis Before and After Pb(II) Adsorption

Fourier transform infrared (FTIR) spectroscopy was employed to identify the surface functional groups responsible for Pb(II) adsorption and to evaluate the changes occurring after adsorption. The FTIR spectra of the adsorbent before and after Pb(II) uptake are presented in Fig. 3.



(A)



(B)

Fig. 2. FE-SEM micrographs of (A) activated biochar (AB) and (B) magnetite-modified biochar (MBC).

Before adsorption, several characteristic absorption bands were observed. The bands between 667 and 999 cm^{-1} were assigned to out-of-plane bending vibrations of alkene (=C–H) groups. Peaks located at approximately 1030, 1227, 1418, and 1454 cm^{-1} were attributed to C–O and C–O–C stretching vibrations associated with cellulose, hemicellulose, and lignin-derived functional groups. The absorption bands at 1516 and 1539 cm^{-1} corresponded to aromatic C=C skeletal vibrations, while the peak at approximately 1645 cm^{-1} was attributed to carbonyl (C=O) and amide (N–H) vibrations. Strong absorption bands observed at 2855 and 2924 cm^{-1} were assigned to aliphatic C–H stretching vibrations.

After adsorption of Pb(II), noticeable shifts in both the positions and intensities of several absorption bands were observed. These spectral changes indicate that hydroxyl, carbonyl, aromatic, and nitrogen-containing functional groups actively participated in the adsorption process. The decrease in peak intensity together with the shift in characteristic absorption bands suggests the formation of coordination bonds between Pb(II) ions and oxygen-containing functional groups on the adsorbent surface.

These results demonstrate that Pb(II) adsorption occurred through multiple mechanisms, including surface complexation, ion exchange, electrostatic attraction, and coordination interactions involving hydroxyl, carboxyl, carbonyl, and aromatic functional groups. The FTIR analysis therefore provides strong evidence that the abundant oxygen-containing functional groups present on the biochar surface play a dominant role in Pb(II) immobilization.

BET Surface Area and Porosity Analysis

The textural properties of activated biochar and magnetite-modified biochar were evaluated using Brunauer–Emmett–Teller (BET) surface area analysis. The results indicate that magnetite modification substantially improved the porous characteristics of the adsorbent.

The specific surface area increased from 3.06 m^2 g^{-1} for activated biochar to 7.86 m^2 g^{-1} after Fe_3O_4 modification. Likewise, the total pore volume increased from 0.0101 cm^3 g^{-1} to 0.0554 cm^3 g^{-1}, whereas the average pore diameter increased from 13.24 nm to 28.22 nm.

The increase in surface area and pore volume can be attributed to the uniform deposition of Fe_3O_4 nanoparticles, which generated additional mesoporous structures and increased the accessibility of adsorption sites. The enlarged pore network facilitates the diffusion of Pb(II) ions into the internal structure of the adsorbent, thereby improving mass transfer and enhancing adsorption efficiency.

Furthermore, the combination of a porous carbon matrix with magnetite nanoparticles provides a synergistic effect by increasing both the number of available active sites and the affinity of the adsorbent toward Pb(II) ions. The improved textural properties observed by BET analysis are consistent with the FE-SEM results and explain the superior adsorption capacity of magnetite-modified biochar compared with activated biochar.

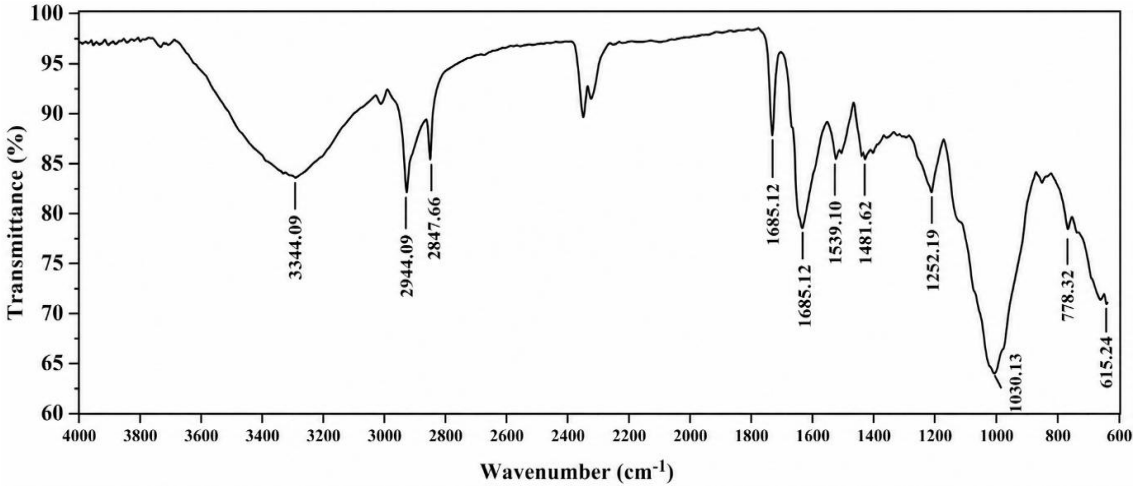


Fig. 3. FTIR spectra of activated biochar before and after Pb(II) adsorption.

Overall, the BET analysis confirms that Fe_3O_4 modification significantly enhances the physicochemical properties of coconut shell biochar, making the synthesized magnetic biochar an efficient adsorbent for Pb(II) removal from aqueous solutions.

Effects of Operational Variables on Pb(II) Adsorption

The adsorption performance of magnetite-modified biochar (MBC) toward Pb(II) ions was evaluated using a Box–Behnken Design (BBD). The removal efficiency and equilibrium adsorption capacity were significantly influenced by the interaction among solution pH, contact time, initial Pb(II) concentration, and adsorbent dosage.

Statistical analysis demonstrated that the initial Pb(II) concentration and adsorbent dosage were the most influential variables governing the adsorption process. Increasing the solution pH enhanced Pb(II) removal over the investigated range, which can be attributed to the progressive deprotonation of oxygen-containing functional groups on the MBC surface. At low pH values, the adsorption sites are extensively protonated, resulting in strong competition between H^+ ions and Pb(II) ions for the available binding sites. As the pH increased, electrostatic repulsion decreased and more negatively charged functional groups became available for metal complexation, thereby enhancing adsorption efficiency.

Contact time also exerted a positive effect on Pb(II) removal. Rapid adsorption occurred during the initial stage owing to the abundance of vacant active sites on the adsorbent surface. As equilibrium was approached, the adsorption rate gradually decreased because the remaining active sites became progressively occupied and intraparticle diffusion became the controlling mechanism. Equilibrium was essentially attained within 15 min, demonstrating the rapid adsorption kinetics of the synthesized magnetic biochar.

Adsorbent dosage markedly affected both Pb(II) removal efficiency and adsorption capacity. Increasing the adsorbent dosage improved the overall removal efficiency because a greater number of adsorption sites became available. However, the equilibrium adsorption capacity (q_e) expressed on a mass basis decreased with increasing adsorbent dosage. This behavior is commonly attributed to the incomplete utilization of adsorption sites resulting from particle aggregation and overlapping active sites at higher adsorbent concentrations.

Conversely, increasing the initial Pb(II) concentration increased the equilibrium adsorption capacity because the larger concentration gradient provided a stronger driving force for mass transfer between the aqueous phase and the adsorbent surface. Under the optimum experimental conditions, the maximum adsorption capacity reached approximately 505 mg g^{-1}, demonstrating the exceptional affinity of MBC toward Pb(II) ions. This high adsorption performance can be attributed to the combined effects of the porous biochar matrix, abundant oxygen-containing functional groups, and uniformly dispersed Fe_3O_4 nanoparticles, which collectively increased the number of accessible adsorption sites.

The adsorption performance achieved in this study compares favorably with many previously reported agricultural biomass-derived adsorbents. Coconut shell-derived MBC exhibited substantially higher adsorption capacity than numerous chemically activated biochars and agricultural residues reported in the literature, confirming the beneficial synergistic effect of magnetite incorporation. These findings indicate that MBC is a promising, low-cost, and environmentally sustainable adsorbent for Pb(II) removal from contaminated water.

Response Surface Analysis of Operational Parameters

Response surface methodology (RSM) was employed to evaluate the interactive effects of the operating variables on Pb(II) removal efficiency. The three-dimensional response surface plots (Figs 4–9) illustrate the combined influence of solution pH, contact time, initial Pb(II) concentration, and adsorbent dosage.

The response surfaces revealed that solution pH exerted the greatest positive influence on Pb(II) removal. Increasing the pH from 5 to 9 consistently enhanced the adsorption efficiency throughout the investigated experimental domain. This behavior is attributed to the reduction in competition between hydrogen ions and Pb(II) ions for the available adsorption sites and the increased availability of negatively charged functional groups capable of coordinating metal ions.

The interaction between solution pH and

contact time demonstrated that Pb(II) removal increased rapidly during the initial adsorption period and gradually approached equilibrium after approximately 15 min. The rapid initial uptake indicates that adsorption primarily occurred on readily accessible surface sites before intraparticle diffusion became the dominant transport mechanism.

The interaction between adsorbent dosage and initial Pb(II) concentration revealed opposite trends for removal efficiency and adsorption capacity. Increasing the adsorbent dosage enhanced the overall removal efficiency because more adsorption sites became available. In contrast, increasing the initial Pb(II) concentration increased the equilibrium adsorption capacity by providing a greater mass transfer driving force. These findings demonstrate that maximum adsorption capacity and maximum removal efficiency do not necessarily occur under identical operating conditions.

Although alkaline conditions favored Pb(II) removal, excessively high pH values may promote the precipitation of lead hydroxide, resulting in an overestimation of adsorption performance. Therefore, the optimum pH was selected within the range where adsorption remained the dominant removal mechanism while avoiding

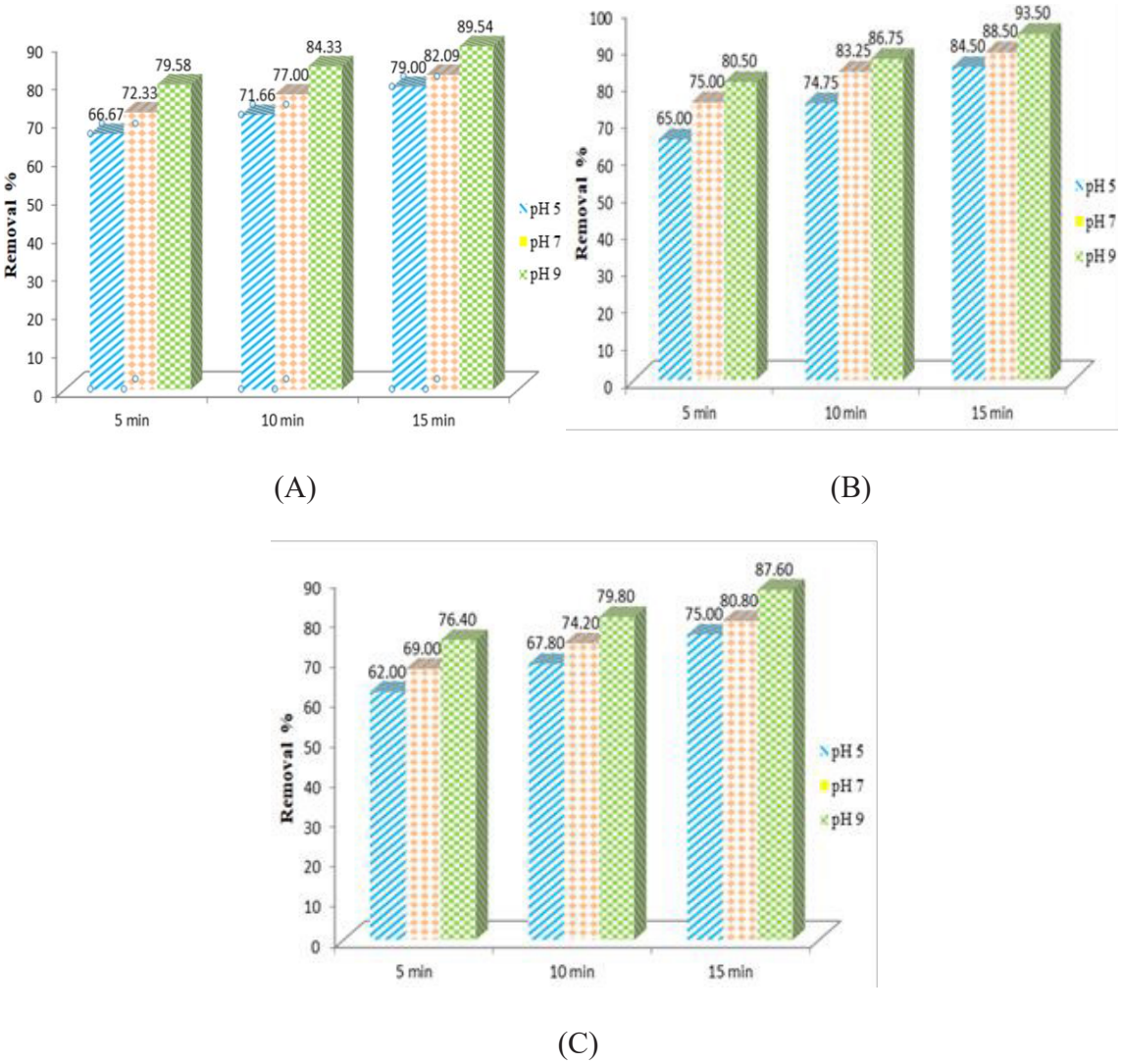


Fig. 4. Three-dimensional response surface plots illustrating the combined effects of solution pH and contact time on Pb(II) removal efficiency using 0.08 g L^{-1} of magnetite-modified biochar (MBC) at initial Pb(II) concentrations of (A) 30, (B) 40, and (C) 50 mg L^{-1}.

significant metal precipitation. The optimized operating conditions predicted by the RSM model were in close agreement with the experimental observations, confirming the reliability and predictive capability of the developed quadratic model.

Adsorption Isotherm Analysis

Adsorption equilibrium studies were conducted to investigate the interaction between Pb(II) ions and the surface of magnetite-modified biochar (MBC). The equilibrium data were analyzed using the Langmuir and Freundlich isotherm models to elucidate the adsorption mechanism.

The adsorption capacity increased progressively with increasing equilibrium Pb(II) concentration until saturation of the available adsorption sites was approached. Under the optimized experimental conditions, the maximum experimental adsorption capacity reached approximately 505 mg g^{-1}, indicating the remarkable affinity of MBC for Pb(II) ions.

The Langmuir isotherm assumes monolayer adsorption on a homogeneous surface containing energetically identical adsorption sites without interactions among adsorbed species. In contrast, the Freundlich model describes multilayer adsorption on heterogeneous surfaces possessing adsorption sites with different energy distributions.

Comparison of the fitted isotherm parameters

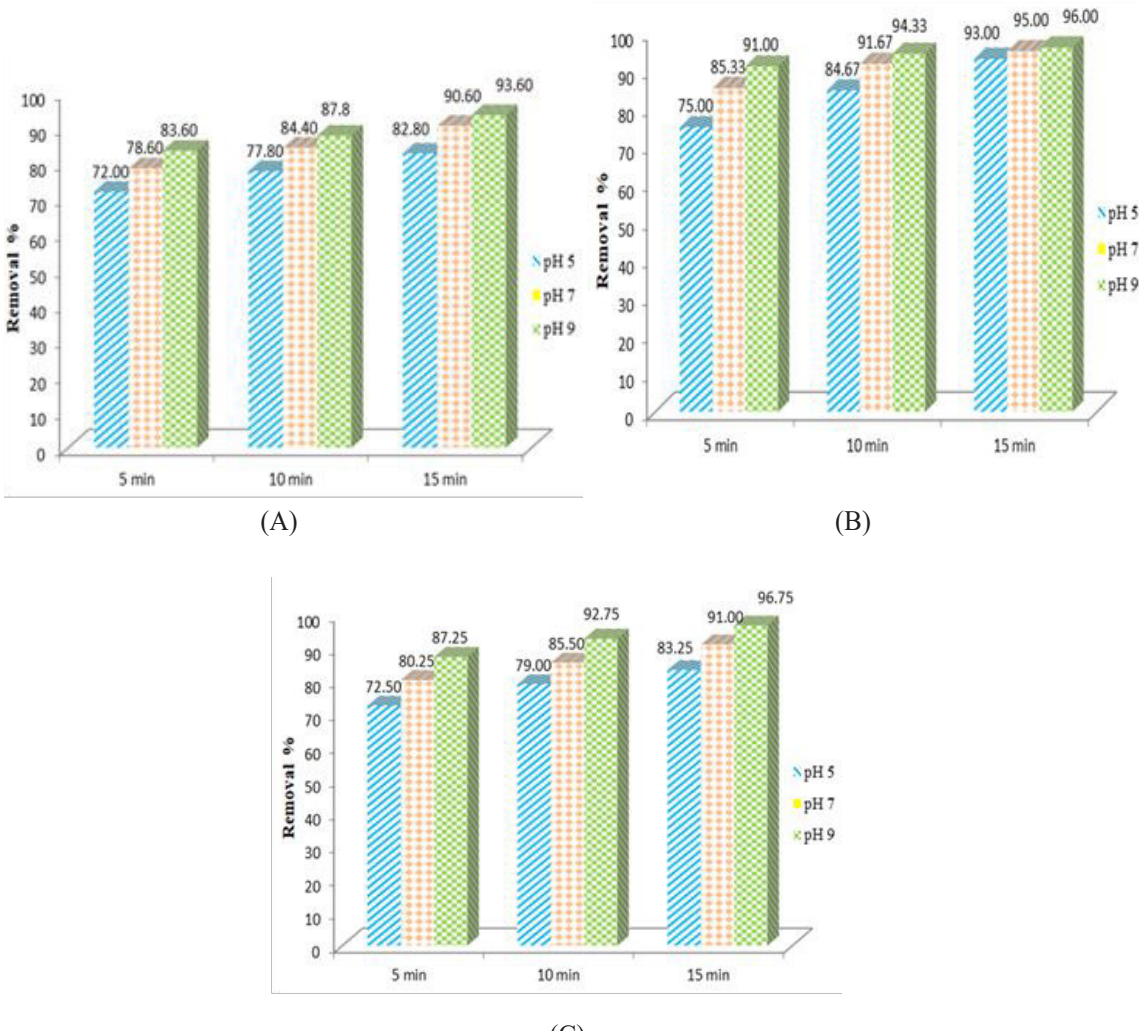


Fig. 5. Three-dimensional response surface plots illustrating the combined effects of solution pH and contact time on Pb(II) removal efficiency using 0.24 g L^{-1} of magnetite-modified biochar (MBC) at initial Pb(II) concentrations of (A) 30, (B) 40, and (C) 50 mg L^{-1}.

showed that the adsorption equilibrium was better represented by the Langmuir model, indicating that Pb(II) adsorption predominantly occurred through monolayer coverage on relatively uniform active sites. The excellent adsorption performance can be attributed to the synergistic combination of several mechanisms, including electrostatic attraction, ion exchange, surface complexation, and coordination between Pb(II) ions and oxygen-containing functional groups distributed throughout the porous carbon matrix.

Furthermore, incorporation of Fe_3O_4 nanoparticles increased both the surface area and the number of chemically active adsorption sites, thereby improving the accessibility of Pb(II) ions to the adsorbent surface. The porous biochar structure promoted rapid mass transfer, whereas the magnetic nanoparticles facilitated efficient adsorbent recovery after treatment without compromising adsorption performance.

Overall, the isotherm analysis confirms that magnetite-modified biochar synthesized from coconut shell biomass possesses excellent adsorption characteristics for Pb(II) removal and represents a highly promising adsorbent for practical wastewater treatment applications.

Influence of Operational Parameters on Pb(II) Removal

The significance of the operational variables affecting Pb(II) adsorption was evaluated using analysis of variance (ANOVA) based on the quadratic response surface model. Although the individual linear effects of solution pH, contact

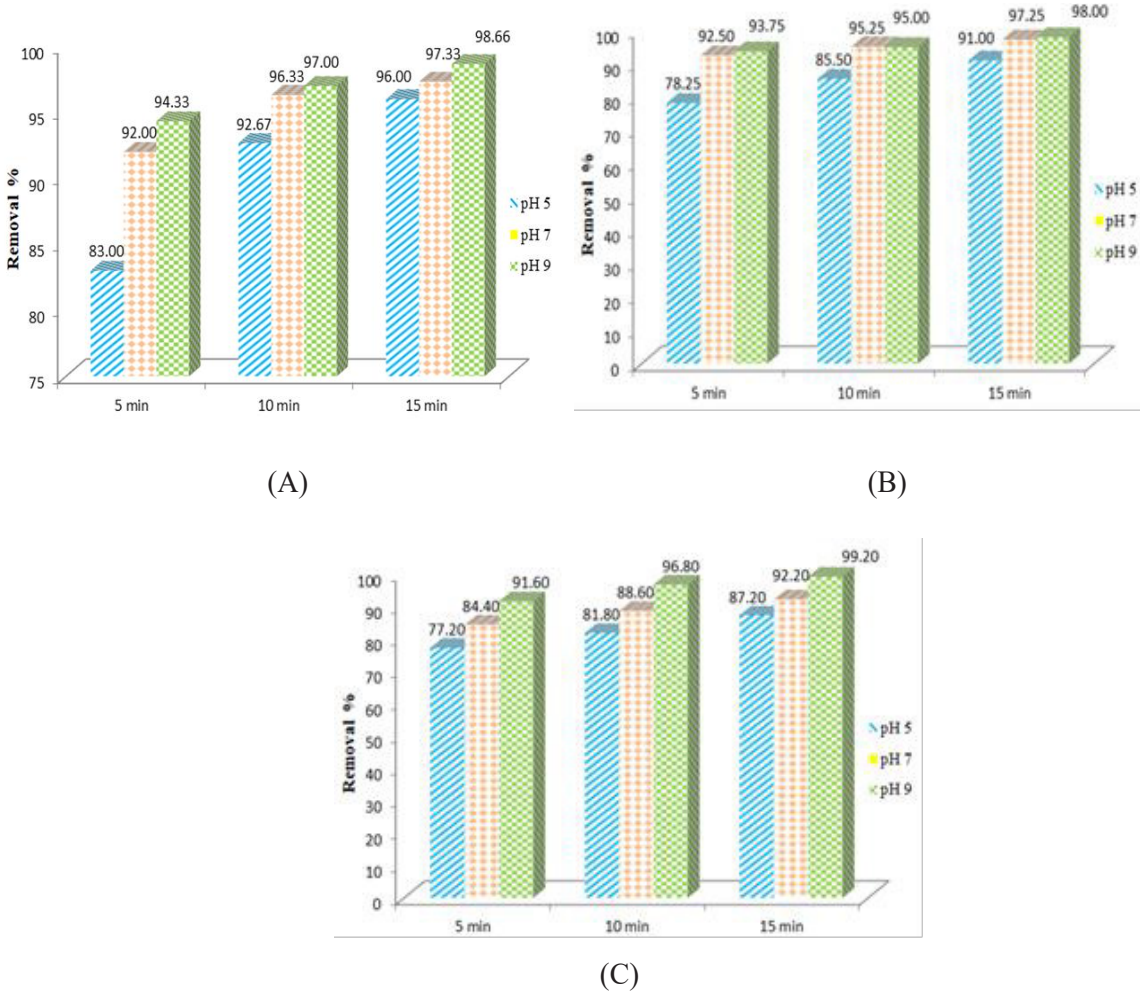


Fig. 6. Three-dimensional response surface plots illustrating the combined effects of solution pH and contact time on Pb(II) removal efficiency using 0.40 g L^{-1} of magnetite-modified biochar (MBC) at initial Pb(II) concentrations of (A) 30, (B) 40, and (C) 50 mg L^{-1}.

time, initial Pb(II) concentration, and adsorbent dosage were not statistically significant at the 95% confidence level ($p > 0.05$), several interaction and quadratic terms exhibited statistically significant effects, demonstrating that Pb(II) adsorption was governed by the combined influence of the investigated variables rather than by any single parameter.

Among the interaction terms, the combined effect of solution pH and adsorbent dosage (BD) was statistically significant ($p = 0.0145$), indicating that adsorption performance depended strongly

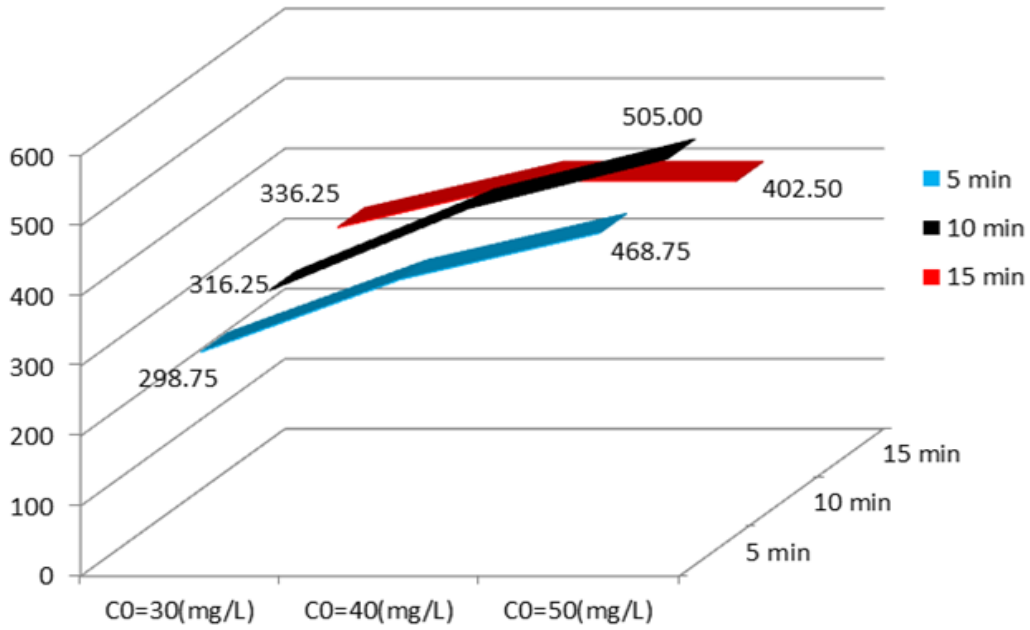


Fig. 7. Equilibrium adsorption capacity of magnetite-modified biochar (MBC) for Pb(II) at different initial Pb(II) concentrations using an adsorbent dosage of 0.08 g L^{-1} (solution pH = 9).

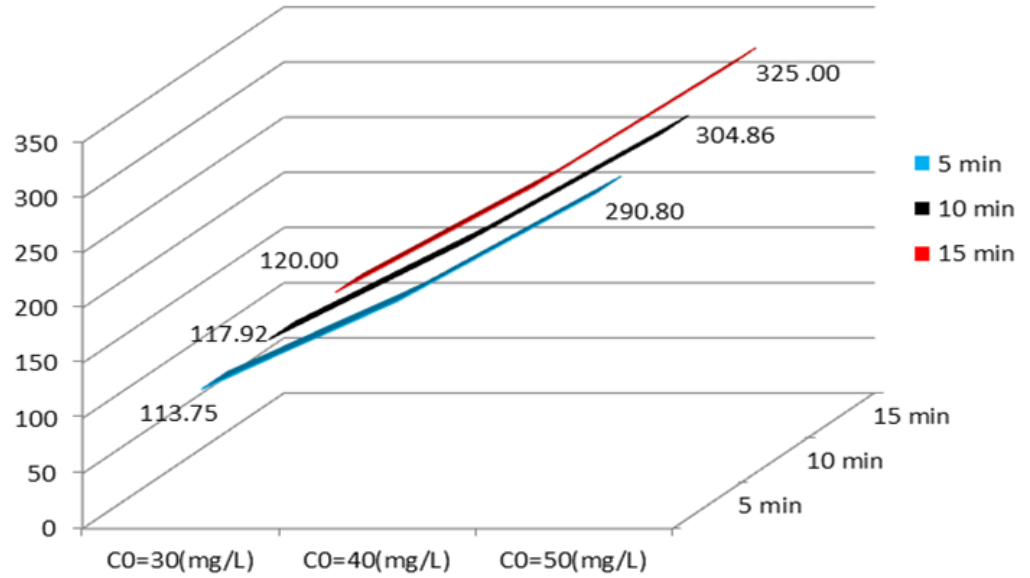


Fig. 8. Equilibrium adsorption capacity of magnetite-modified biochar (MBC) for Pb(II) at different initial Pb(II) concentrations using an adsorbent dosage of 0.24 g L^{-1} (solution pH = 9).

on the simultaneous optimization of these two variables. Likewise, the interaction between initial Pb(II) concentration and adsorbent dosage (CD) was significant ($p = 0.0128$), suggesting that the availability of adsorption sites relative to the metal-ion concentration plays a crucial role in determining adsorption efficiency.

The quadratic terms associated with the initial Pb(II) concentration (C^2) and adsorbent dosage (D^2) also exhibited significant effects, confirming the presence of nonlinear relationships within the experimental domain. These results indicate that increasing either parameter beyond its optimum level does not necessarily improve adsorption performance because excessive adsorbent dosage may promote particle aggregation, whereas excessively high Pb(II) concentrations may approach saturation of the available adsorption sites.

The response surface analysis predicted a maximum Pb(II) removal efficiency of approximately 99.2% under the optimized operating conditions. The close agreement between the predicted and experimental responses confirms the adequacy of the developed quadratic model and demonstrates that Response Surface Methodology is an effective tool for optimizing the adsorption process while minimizing the number of experimental trials.

Overall, the statistical analysis demonstrates that efficient Pb(II) removal depends on the synergistic interaction among the operating variables, emphasizing the importance of process optimization for achieving maximum adsorption performance.

Adsorption Kinetics of Pb(II)

The adsorption kinetics of Pb(II) onto magnetite-modified biochar (MBC) were investigated to elucidate the adsorption mechanism and identify the rate-controlling steps governing metal uptake. The experimental data were analyzed using the pseudo-first-order (PFO), pseudo-second-order (PSO), and Weber–Morris intraparticle diffusion models.

Among the investigated models, the pseudo-second-order model provided the best agreement with the experimental data, exhibiting an excellent correlation coefficient ($R^2 = 0.999$). Furthermore, the equilibrium adsorption capacity predicted by the PSO model (123.92 mg g^{-1}) closely matched the experimentally determined value (123.20 mg g^{-1}), indicating the reliability of the model in describing the adsorption process.

The superior performance of the pseudo-second-order model suggests that Pb(II) adsorption was predominantly controlled by chemisorption involving electron sharing or electron exchange between Pb(II) ions and the

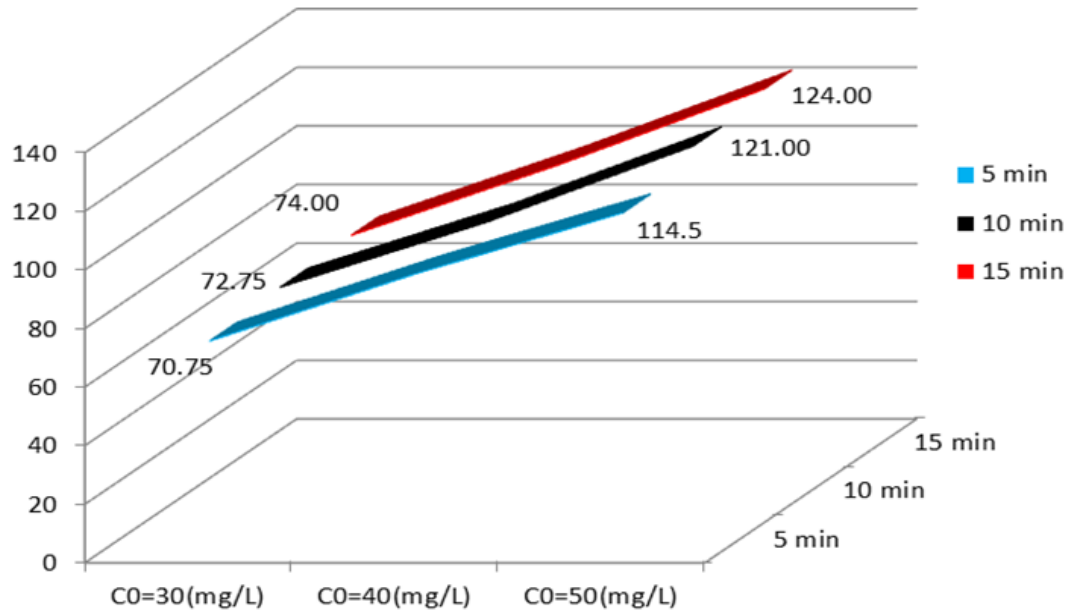


Fig. 9. Equilibrium adsorption capacity of magnetite-modified biochar (MBC) for Pb(II) at different initial Pb(II) concentrations using an adsorbent dosage of 0.40 g L^{-1} (solution pH = 9).

oxygen-containing functional groups present on the MBC surface. These interactions likely involve hydroxyl, carboxyl, carbonyl, and phenolic groups together with surface complexation and ion-exchange mechanisms.

To further investigate the adsorption mechanism, the Weber–Morris intraparticle diffusion model was applied. The resulting plots did not pass through the origin, indicating that intraparticle diffusion was involved in the adsorption process but was not the sole rate-limiting step. Instead, Pb(II) adsorption proceeded through multiple consecutive stages, including rapid external surface adsorption, gradual diffusion of Pb(II) ions into the internal pore structure, and finally equilibrium adsorption on the available active sites.

The rapid initial adsorption observed during the first few minutes can be attributed to the abundance of readily accessible adsorption sites on the external surface of the adsorbent. As these sites became progressively occupied, the adsorption rate decreased because intraparticle diffusion and surface complexation became the dominant mechanisms controlling Pb(II) uptake.

Overall, the kinetic analysis demonstrates that Pb(II) adsorption onto magnetite-modified biochar is a rapid and efficient process governed by a combination of chemisorption, ion exchange, surface complexation, and intraparticle diffusion. The excellent agreement between the experimental data and the pseudo-second-order model further confirms the high adsorption affinity of the synthesized MBC toward Pb(II) ions, supporting its potential application in practical wastewater treatment systems.

CONCLUSION

This study successfully synthesized a magnetite-modified coconut shell biochar (MBC-IO) as an efficient and environmentally sustainable adsorbent for the removal of Pb(II) ions from aqueous solutions. The incorporation of Fe_3O_4 nanoparticles into the biochar matrix significantly improved the surface characteristics, producing a porous magnetic nanocomposite with enhanced adsorption performance and enabling rapid separation of the spent adsorbent from treated water using an external magnetic field.

Physicochemical characterization confirmed the successful formation of the magnetic biochar nanocomposite. FE-SEM analysis revealed the development of a rough and highly porous surface after magnetite modification, while FTIR analysis demonstrated the presence of abundant oxygen-containing functional groups responsible for Pb(II) binding through complexation, ion exchange, and electrostatic interactions. BET analysis further showed that magnetite incorporation increased the specific surface area, pore volume, and average pore diameter, thereby providing a greater number of accessible adsorption sites.

The adsorption experiments demonstrated that Pb(II) removal was strongly influenced by solution pH, contact time, initial metal concentration, and adsorbent dosage. Response Surface Methodology based on the Box–Behnken Design effectively optimized these operating variables and predicted a maximum Pb(II) removal efficiency of approximately 99.2% under the optimum conditions. The highest experimental adsorption capacity reached 505 mg g^{-1}, demonstrating the excellent adsorption capability of the synthesized nanocomposite compared with many agricultural waste-derived adsorbents reported in the literature.

Adsorption equilibrium was best described by the Langmuir isotherm model, indicating predominantly monolayer adsorption on homogeneous active sites. Furthermore, kinetic analysis showed that the pseudo-second-order model provided the best fit to the experimental data, suggesting that chemisorption was the dominant adsorption mechanism. The Weber–Morris diffusion model indicated that intraparticle diffusion contributed to the adsorption process but was not the only rate-controlling step, confirming that Pb(II) uptake occurred through a combination of surface complexation, ion exchange, and pore diffusion.

Overall, the results demonstrate that magnetite-modified coconut shell biochar is a low-cost, sustainable, and highly efficient adsorbent for Pb(II) removal from contaminated water. The combination of high adsorption capacity, magnetic recoverability, simple preparation, and the utilization of abundant agricultural waste makes this material a promising candidate for practical wastewater treatment applications. Future studies should focus on regeneration and reuse of the adsorbent, evaluation in real industrial wastewater, continuous-flow column experiments, long-term stability, and techno-economic assessment to facilitate large-scale implementation.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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