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Synthesis and Application of Coconut Shell Activated Carbon Fe_3O_4 Composite for Zn^{2+} Metal Ion Adsorption from Wastewater

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ABSTRACT. Industrial wastewater containing heavy metals such as Zn^{2+} ions is one of the hazardous pollutants that can pose serious risks to the environment and human health. The presence of Zn^{2+} in aquatic systems at high concentrations can lead to acute toxicity, thus requiring effective and sustainable treatment methods. Adsorption has become one of the most widely developed treatment approaches due to its advantages, including high efficiency, low operational cost, and environmental friendliness. This study aims to synthesize and characterize Fe_3O_4/activated carbon composites as adsorbents for the removal of Zn^{2+} ions from wastewater. Fe_3O_4 was synthesized through a coprecipitation method using $FeCl_3 \cdot 6H_2O$ and $FeSO_4 \cdot 7H_2O$ precursors, and subsequently combined with thermally activated coconut-shell carbon to enhance surface area and adsorption capacity. The composite was characterized using X-Ray Diffraction (XRD). Adsorption tests were conducted using various composite masses 0.04 g; 0.05 g; 0.06 g; 0.07 g, and 0.08 g to examine the effect of adsorbent dosage. The best performance was obtained at an adsorbent mass of 0.08 g with an optimum contact time of 75 minutes. Under these conditions, an initial Zn^{2+} concentration of 30 ppm decreased significantly, achieving a maximum removal efficiency of 91% and 84.63% under other test conditions. These findings indicate that the Fe_3O_4/activated carbon composite exhibits high effectiveness and selectivity in adsorbing Zn^{2+} ions and holds strong potential for development as a wastewater treatment material based on renewable and environmentally friendly local resources.

1. INTRODUCTION

Industrial activities have increased significantly in recent decades, resulting in greater environmental pollution, particularly heavy-metal contamination in liquid waste streams. One of the most commonly detected heavy metals in industrial effluents is zinc (Zn^{2+}), which originates from metal plating, battery manufacturing, and paint production. At elevated concentrations, Zn^{2+} is toxic and can cause physiological disorders in aquatic organisms as well as adverse health effects in humans due to bioaccumulation along the food chain [1]. Consequently, the development of effective treatment technologies for Zn^{2+}-containing wastewater is of critical importance.

Among various treatment technologies, adsorption has emerged as one of the most effective and economically feasible methods for heavy metal removal due to its simplicity, high efficiency, and adaptability to low metal concentrations [2]. However, the adsorption performance is strongly influenced by the physicochemical properties of the adsorbent, such as surface area, pore structure, and surface functional groups. Therefore, recent research has increasingly focused on developing advanced composite adsorbents that integrate high adsorption capacity with easy separation and reusability [4]. In this context, magnetic carbon-based composites, particularly Fe_3O_4-modified activated carbon, have attracted considerable attention due to their synergistic adsorption behavior and magnetic recoverability [8].

Activated carbon is widely recognized as an effective adsorbent due to its high surface area, well-developed porosity, and chemical stability [4]. The use of biomass waste such as coconut shells as a precursor for activated carbon has gained increasing attention, given its abundance, low cost, and environmental sustainability [5]. Meanwhile, Fe_3O_4 (magnetite) is an inorganic material with superparamagnetic properties, enabling easy separation from aqueous media using an external magnetic field. Fe_3O_4 has also demonstrated strong potential as a heavy-metal adsorbent due to its reactive surface oxide groups [6].

Several studies have reported the fabrication of Fe_3O_4-based composites with carbonaceous materials for heavy-metal removal. Wang et al. [7] demonstrated the effectiveness of Fe_3O_4–biochar composites for Cu^{2+} and

Pb^{2+} adsorption, while Qing Su et al. [8] reported enhanced Cr(VI) removal using granular activated carbon modified with Fe_3O_4. However, research focusing specifically on Fe_3O_4/activated-carbon composites derived from coconut shell biomass for Zn^{2+} adsorption remains limited.

Moreover, previous studies have often lacked a comprehensive approach integrating morphological, structural, and functional-group characterization to elucidate the adsorption mechanisms involved [9]. In addition, several works have not yet evaluated adsorption kinetics and isotherms in a systematic manner, limiting the understanding of adsorbent performance and selectivity toward Zn^{2+} ions [10].

Based on these research gaps, this study aims to synthesize and characterize a Fe_3O_4/activated carbon composite derived from coconut shells and to evaluate its adsorption performance for Zn^{2+} removal from aqueous solutions. The composite was synthesized using a coprecipitation method and characterized by X-ray Diffraction (XRD) to confirm the formation of the magnetic composite structure. This research seeks to develop a cost-effective and environmentally friendly adsorbent based on locally available biomass, with potential application in wastewater treatment systems for Zn^{2+}-contaminated effluents.

2. MATERIALS AND METHODS

The characterization of materials was conducted using advanced analytical instruments, including an X-Ray Diffraction (XRD) system for crystalline phase identification, a Fourier Transform Infrared Spectroscopy (FTIR) spectrometer for functional group analysis, and a Scanning Electron Microscope (SEM) for morphological examination. All chemical reagents were of analytical grade and used without further purification. $FeCl_3 \cdot 6H_2O$ (≥99%, Merck), $FeSO_4 \cdot 7H_2O$ (≥99%, Sigma-Aldrich), NaOH (≥98%, Merck), and H_3PO_4 (85 wt%, Merck) were used as received. The procedures for data collection, processing, and interpretation are systematically presented in the subsequent subsections to provide a clear and coherent methodological framework.

2.1 Material Preparation and Composite Synthesis

The preparation of raw materials began with the collection, washing, and drying of coconut shells to remove impurities. The dried shells were carbonized at high temperatures to produce charcoal. Chemical activation was carried out by soaking the charcoal in H_3PO_4 solution to increase pore development and improve adsorption capability. After activation, the Fe_3O_4/activated carbon composite was synthesized using the co-precipitation method. Solutions of $FeCl_3 \cdot 6H_2O$ and $FeSO_4 \cdot 7H_2O$ were mixed in a controlled molar ratio, then added to a suspension of activated carbon. A NaOH solution with 5 M in concentration was introduced dropwise until a black precipitate formed, indicating the creation of Fe_3O_4 particles attached to the carbon matrix. The precipitate was subsequently filtered, washed to neutrality, and dried to obtain the final composite[11]. Characterization using XRD, FTIR, and SEM was performed to identify crystalline structure, chemical functional groups, and surface morphology. The average crystallite size (D) of the Fe_3O_4 component was estimated using the Scherrer equation (1)[14].

$$D = \frac{K\lambda}{\beta \cos \theta'} \quad (1)$$

2.2 Adsorption Testing and Equations

Adsorption performance was evaluated using synthetic Zn^{2+} solutions with variations in adsorbent dosage in a range between 0.04 g; 0.05 g; 0.06 g; 0.07 g; 0.08 g. The concentration of the waste water is fixed at 30 ppm, while the contact time among them is 75 minutes. The residual concentration of Zn^{2+} after adsorption was measured, and the data were analyzed to determine adsorption capacity and removal efficiency. The equilibrium adsorption capacity q_e was calculated according to (2), where C_0 is the initial concentration (mg/L), C_e is the equilibrium

concentration (mg/L), V is the solution volume (L), and m is the mass of adsorbent (g). following standard adsorption evaluation methods [3,10].

$$q_e = \frac{(Co-Ce)\,.\,V}{m} \quad (2)$$

The removal efficiency was determined using (3), which expresses the percentage of Zn^{2+} removed from the aqueous solution [3,10].

$$\%R = \frac{(Co-Ce)}{Co} \text{ x } 100\% \quad (3)$$

Both parameters were used to evaluate the adsorption behavior of the synthesized Fe_3O_4/activated carbon composite and to determine the extent to which the material conforms to theoretical adsorption models. By analyzing these parameters, the study aimed to assess the dominant adsorption mechanism occurring on the composite surface and to identify which model provides the best statistical and physicochemical representation.

3. RESULTS AND DISCUSSION

Present and elaborate your research results here. Although not everything needs to be disclosed, a good paper must contain new, useable, and fully described information. Authors should expect to be challenged by reviewers if the results are not supported by adequate data and critical details.

3.1 Structural Characterization of the Fe_3O_4/Activated Carbon Adsorbent

To elucidate the crystalline structure and confirm the hybridization of Fe_3O_4/activated carbon nanoparticles with the activated carbon support, the prepared material was subjected to X-ray Diffraction (XRD**)** analysis.

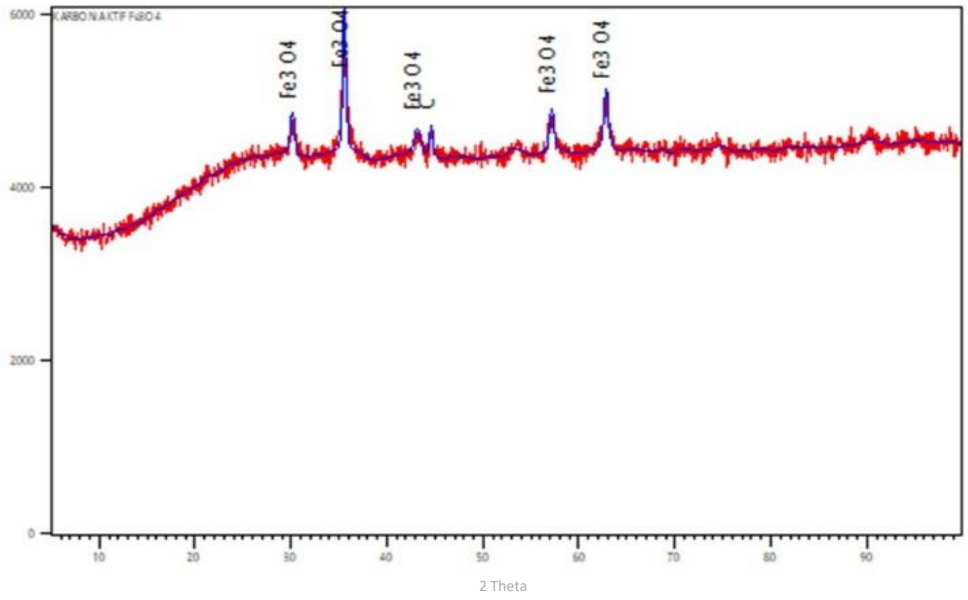

Figure 1. XRD of Fe_3O_4/activated carbon composites

The X-ray Diffraction (XRD) pattern of the Fe_3O_4/activated carbon composites (derived from coconut shell) confirms the successful synthesis and presence of the target phases. The diffraction pattern exhibits several sharp and well defined characteristic peaks. The major diffraction peaks are observed at 2Θ values of approximately 30.1°; 35.5°; 43.1°; 57.0° and 62.6°, corresponding to the crystallographic planes (220), (311), (400), (511), and (440), respectively. The distinct sharpness and high intensity of these peaks suggest a highly crystalline nature for the Fe_3O_4 component within the composite.

Crucially, the pattern also features a broad, low-intensity halo spanning the 2Θ range of roughly 15° to 30°. This characteristic signature is attributed to the amorphous nature of the Activated Carbon matrix originating from the coconut shell. This indicates that the Fe_3O_4 nanoparticles are successfully intercalated or dispersed within the non-graphitic carbon support material, forming the desired composite structure. No significant peaks corresponding to iron impurities such as Fe_2O_3 or FeO were observed, validating the high purity of the synthesized magnetite phase [12].

Based on the Scherrer equation, the calculated crystallite size of the Fe_3O_4 phase was below 100 nm, indicating that the material can be classified as nanocrystalline. Crystallite sizes in this range are generally defined as nanocrystalline according to established X-ray diffraction theory [14]. The lack of peak shifting or significant lattice distortion, when compared to bulk Fe_3O_4, suggests that the formation of the composite with activated carbon did not significantly alter the fundamental crystal structure of the magnetite core. Based on the peak broadening analysis using the Scherrer equation, the Fe_3O_4 crystallites are classified within the nanocrystalline range, typically below 50 nm. Materials in this size regime are known to exhibit enhanced surface reactivity and improved adsorption performance due to the increased surface-to-volume ratio, which is favorable for heavy metal ion interaction [12].

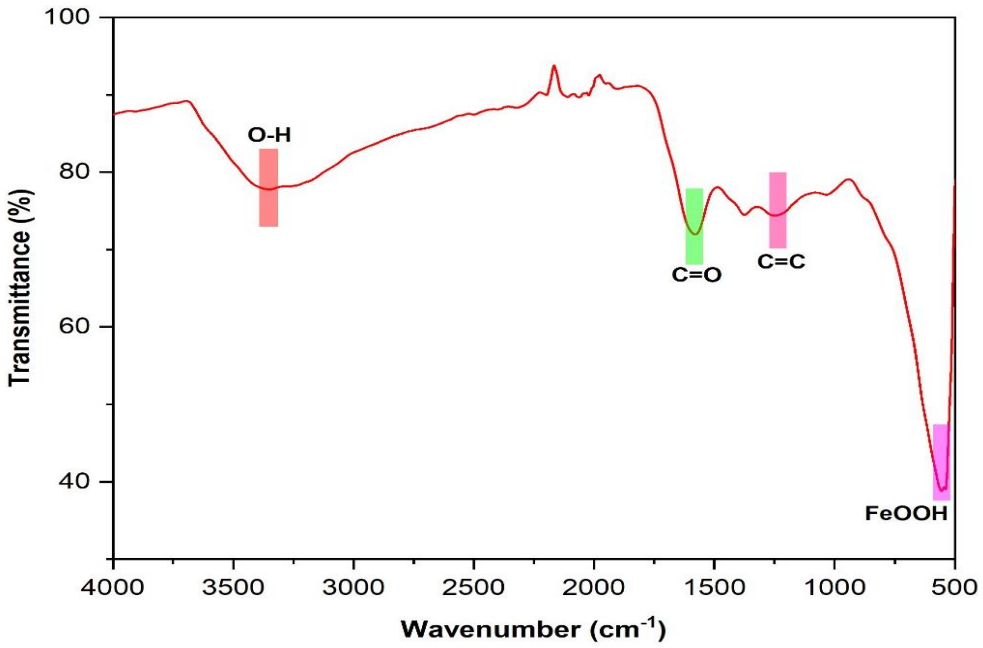

Figure 2. FTIR of Fe_3O_4/activated carbon composites

The Fourier Transform Infrared (FTIR) spectrum of the Fe_3O_4/activated carbon composite is presented in Figure 2 and provides important information regarding the surface functional groups involved in Zn^{2+} adsorption. A broad absorption band observed in the region of approximately 3200–3600 cm^{-1} is attributed to the stretching vibration of O–H groups, originating from hydroxyl functionalities on the activated carbon surface as well as surface hydroxyl groups associated with iron oxides. Similar broad O–H bands have been widely reported for Fe_3O_4-based carbon composites and are known to play a crucial role in heavy metal adsorption through hydrogen bonding and surface complexation mechanisms [6]. The absorption band detected around 1550–1650 cm^{-1} corresponds to the stretching vibration of C=O functional groups, such as carbonyl and carboxyl groups present on the activated carbon surface. Previous studies have demonstrated that these oxygen-containing functional groups significantly enhance the adsorption of divalent metal ions, including Zn^{2+}, by providing negatively charged sites that promote electrostatic attraction and coordination bonding [9].

In addition, a distinct band observed in the range of 1000–1200 cm^{-1} is assigned to C=C stretching vibrations, which are characteristic of aromatic structures in biomass-derived activated carbon. The presence of these bands is consistent with reports on coconut shell–based activated carbon and confirms the successful formation of a stable aromatic carbon framework after activation [5]. A strong absorption band appearing in the low wavenumber region around 500–600 cm^{-1} is attributed to Fe–O vibrations, associated with magnetite (Fe_3O_4) phases. Similar Fe–O absorption bands have been observed in Fe_3O_4/activated carbon composites reported in the literature, confirming the successful incorporation of magnetic iron oxide into the carbon matrix. This observation is in good agreement with the crystalline phases identified by XRD analysis [12].

3.2 Fe_3O_4/activated carbon Performance

Table 1. Adsorption performance of the composite adsorbent with artificial Zn wastewater at 30 ppm

Adsorbent Mass (mg)	Zn^{2+} Initial Concentration (mg/L)	Zn^{2+} Concentration After Adsorption (mg/L)
40	30	4.60
50	30	4.12
60	30	3.6
70	30	3.17
80	30	2.7

The preliminary examination of the adsorption data, as comprehensively detailed in Table 1, serves as the foundational evidence for the composite's efficacy as an adsorbent. The investigation was systematically conducted by challenging the Fe_3O_4/Activated Carbon composite with a constant initial zinc ion concentration Zn^{2+} of 30mg/L, while incrementally varying the adsorbent mass from 40 mg up to 80 mg. A compelling trend of decreasing residual Zn^{2+} concentration was consistently observed in the effluent solution as the mass of the adsorbent increased [13]. For instance, the concentration of Zn^{2+} remaining in the solution after the adsorption equilibrium was reached decreased markedly from 4.60 mg/L at the lowest dosage 40 mg to a significantly lower value of 2.70 mg/L at the maximum dosage 80 mg. This reduction in final equilibrium concentration explicitly demonstrates a dose dependent effect, unequivocally confirming that a larger quantity of the magnetic composite translates into a greater capacity to extract and immobilize the dissolved zinc ions from the artificial wastewater. This quantitative evidence firmly establishes the fundamental role of available surface sites, which are directly proportional to the adsorbent mass, in governing the overall effectiveness of the removal process.

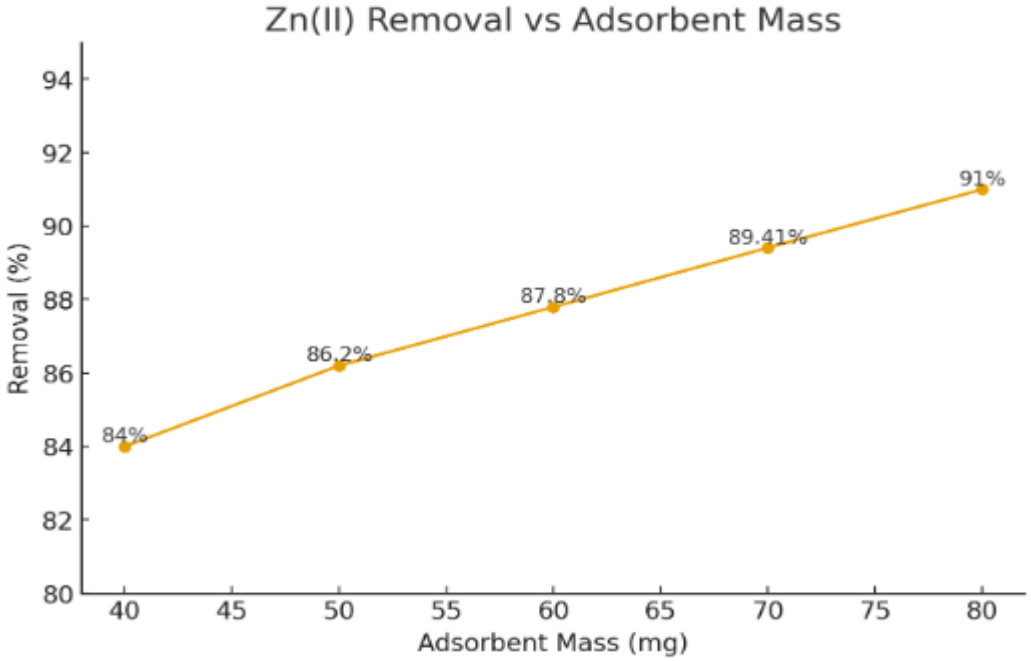

Figure 2. % Removal of Zn(II) based on adsorbent mass

Further corroboration and a clearer visualization of this mass-dependent phenomenon are provided by Figure 2, which graphically illustrates the percentage removal of Zn^{2+} as a function of the adsorbent mass. The figure establishes a strong positive linear relationship across the entire range tested, emphasizing the direct correlation between the solid-phase concentration and the system's performance. The removal efficiency saw a tangible and significant rise, escalating from a robust baseline of 84.63% at 40 mg to an impressive peak efficiency of 91% when the adsorbent mass reached 80 mg. When compared with similar magnetic carbon-based adsorbents reported in previous studies, the Fe_3O_4/activated carbon composite synthesized in this work demonstrates comparable or superior Zn^{2+} removal efficiency. For instance, Mirjavadi et al. [12] reported Zn^{2+} removal efficiencies of approximately 89% using Fe_3O_4-based nanocomposites, while Su et al. [8] achieved similar performance using Fe_3O_4-loaded granular activated carbon.

The high efficiency observed in this study can be attributed to the synergistic combination of porous activated carbon and nanocrystalline Fe_3O_4. With a fixed volume of solution and a constant number of zinc ions, the addition of more adsorbent material exponentially increases the total surface area, thereby enhancing the kinetic driving force and the probability of successful contact and binding between the pollutant ions and the composite's surface moieties. The observed enhancement in Zn^{2+} removal with increasing adsorbent mass can also be explained from a mass transfer perspective. A higher adsorbent dosage increases the total surface area and number of active adsorption sites, thereby reducing mass transfer resistance between the liquid phase and the solid surface. This facilitates external film diffusion and accelerates the transport of Zn^{2+} ions toward the active sites on the composite surface, leading to higher removal efficiency

4. CONCLUSION

Adsorption experiments showed a clear dose-dependent behavior, where increasing the adsorbent mass from 40 mg to 80 mg enhanced Zn(II) removal efficiency to a maximum of 91%. This performance is comparable to, and in some cases higher than, those reported in previous studies on Fe_3O_4-based and carbon-supported magnetic adsorbents. For instance, Mirjavadi et al. reported Zn(II) removal efficiencies in the range of 85–90% using Fe_3O_4-based nanocomposites, while Su et al. achieved comparable efficiencies using Fe_3O_4-loaded granular activated carbon. The competitive adsorption performance observed in this study can be attributed to the synergistic interaction between the porous activated carbon derived from coconut shell and the nanocrystalline Fe_3O_4 phase, which enhances surface reactivity and mass transfer.

Overall, the Fe_3O_4/activated carbon composite developed in this work represents a cost-effective and environmentally friendly adsorbent derived from renewable biomass, with adsorption efficiency comparable to established magnetic composites reported in the literature. These results support its potential application in wastewater treatment systems for the remediation of Zn(II)-contaminated effluents.

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