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Characterization and Adsorption Effectiveness of Coconut Shell-Activated Carbon/Fe_3O_4 as Novel-Adsorbent on Copper Removal from Wastewater

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ABSTRACT: Fe_3O_4-modified activated carbon from coconut shell has arisen as a promising magnetic adsorbent for wastewater treatment applications. This study aims to synthesize activated carbon that meets the Indonesian National Standard (SNI), characterize the physicochemical properties of activated carbon/Fe_3O_4 using Scanning Electron Microscopy-Energy Dispersive X-ray (SEM-EDX) and Fourier Transform Infrared Spectroscopy (FTIR), and evaluate its adsorption efficiency for Cu ion removal under varying operational conditions, including adsorbent mass, contact time, and initial Cu concentration. The adsorbent was synthesized from coconut shell-derived activated carbon, chemically activated with 10% H_3PO_4, and composited with Fe_3O_4 to enhance its magnetic properties. Characterization results confirmed the presence of functional groups and surface morphological features that contribute to improved adsorption performance. Adsorption experiments demonstrated that the optimal conditions were achieved with an adsorbent mass of 3 g, a contact time of 75 min, and an initial Cu concentration of 60 mg/L, yielding a maximum adsorption efficiency of 99.70%. The incorporation of Fe_3O_4 not only enhanced adsorption performance and facilitated easy separation of the adsorbent from aqueous solutions. These findings emphasize the potential of activated carbon/Fe_3O_4 as an efficient and sustainable adsorbent for heavy metal removal in industrial wastewater treatment applications.

KEYWORDS: Activated carbon, activated carbon/Fe_3O_4, wastewater treatment, Cu removal, coconut shell

1.0 INTRODUCTION

Although industrial expansion greatly contributes to environmental degradation, mostly by releasing harmful chemicals in industrial effluents (Rathi et al., 2021; Sathya et al., 2022), it is also essential for economic growth and society improvement. Because of their toxicity and long-term environmental persistence, heavy metals including copper (Cu) especially raise concerns. Although copper is a necessary component for many biological activity, too much of it can cause major health issues including liver necrosis, kidney damage, anemia, and, in extreme conditions, death (Leal et al., 2023; Teschke, 2024). Unlike organic pollutants, heavy metals are non-biodegradable and typically linger in terrestrial and aquatic environments, therefore posing long-term environmental and public health risks (Briffa et al., 2020; Uddin & Cassim, Mohamed Zakeel, MohamedZavahir, 2021; Xu et al., 2024).

Chemical precipitation, ion exchange, membrane filtration, and adsorption are among the spectrum of remedial techniques used to remove heavy metals from industrial effluencies (Saleh et al., 2022; Shrestha et al., 2021). Among these techniques, adsorption has attracted most interest because of its simplicity, economy, and great efficiency in metal ion removal (Hama Aziz et al., 2023; Rashid et al., 2021). Particularly activated carbon is extensively used as an adsorbent because of its great surface area, porosity, and strong affinity for heavy metal (Sultana et al., 2022; Wang et al., 2023). Thanks to their high carbon content and abundance as an agricultural waste, coconut shell-based activated carbon has evolved as an environmentally benign and sustainable substitute in recent years (Deng et al., 2021; Khan et al., 2023; Packialakshmi et al., 2021; Saleem et al., 2025). Conventional activated carbon has various disadvantages despite having a good adsorption capacity including poor structural stability and difficulties in post-treatment separation.

Many changes to activated carbon have been investigated to overcome these limitations and improve its reusability and adsorption effectiveness. Using magnetite (Fe_3O_4) nanoparticles has shown great potential to not only enhance adsorption performance but also help the adsorbent be recovered from treated effluent by external magnetic fields (Zhao et al., 2025). Fe_3O_4 combined with activated carbon produces a composite material with great specific surface area, high adsorption capacity, and magnetic responsiveness—qualities fit for copper ion removal from aqueous environments. Aiming to assess Fe_3O_4-modified activated carbon generated from coconut shell biomass, this work focuses on production and characterisation of this material towards Cu^{2+} ions. Examined methodically will be the physicochemical characteristics of the composite to clarify its structural characteristics and functional performance. The results are likely to help to provide sophisticated and sustainable adsorbent materials for wastewater treatment, therefore providing a practical substitute for heavy metal removal in industrial discharge control.

2.0 MATERIALS AND METHODS

2.1 Materials

This work synthesizes and characterizes the adsorbent using a set of components. Undergoing a series of washing, drying, and high-temperature carbonization, coconut shell was the main precursor for activated carbon generation. Procedures of cleaning and diluting employed demineralized and distilled water. Aiming at increasing surface porosity, activation of the carbon was carried out with 10% phosphoric acid (H_3PO_4). Fe_3O_4 was generated for the manufacture of the magnetic composite from precursors sodium hydroxide (NaOH), ferric chloride hexahydrate ($FeCl_3{\cdot}6H_2O$), and ferrous sulfate heptahydrate ($FeSO_4{\cdot}7H_2O$). Copper(II) sulfate pentahydrate ($CuSO_4{\cdot}5H_2O$) was utilized in adsorption investigations to imitate Cu^{2+}-contaminated wastewater [10]. Scanning electron microscopy combined with Energy Dispersive X-Ray Spectroscopy (SEM-EDX) was used to analyze the morphological properties and elemental composition of the synthesized adsorbent, while Fourier Transform Infrared (FTIR) spectroscopy was employed to identify functional groups. Cu^{2+} concentrations both before and after the treatment allowed one to estimate the adsorption performance using an Atomic Absorption Spectrophotometer (AAS). The choice of these materials and methods sought to evaluate the adsorption capacity of the activated carbon/Fe_3O_4 composite and their possible use in copper ion removal from wastewater holistically.

2.2. Methods

The experimental process consisted in many phases: fabrication of activated carbon, synthesis of the activated carbon/Fe_3O_4 composite, formulation of synthetic copper-containing wastewater, and performance of adsorption tests. Coconut shells were first sun-dried for two to three days, then oven-dried at 300°C for one hour after first cleaning to eliminate surface impurities. The dried biomass was then carbonized in a muffle furnace at 500°C for three hours, carefully covered in aluminum foil to reduce oxygen exposure. Ground and sieved to produce homogeneous particles with a mesh size of 150, the resultant biochar was Soaking in a 10% H_3PO_4 solution for 24 hours chemically activated the carbon; it was then filtered and rinsed under distilled water to produce a neutral pH (6–7). After that, the activated carbon dried two hours at 100°C in an oven.

Dissolving 6.5 grammes of activated carbon in 300 mL of demineralised water and heating the mixture to 70°C produced the activated carbon/Fe_3O_4 composite. Separately, 100 mL of demineralized water was used to dissolve 7.8 grams (28 mmol) $FeCl_3{\cdot}6H_2O$ and 3.9 grams (14 mmol) $FeSO_4{\cdot}7H_2O$; agitated until homogenous; then added to the activated carbon solution at 70°C. After 30 minutes of mixing, a black precipitate developed from the dropwise addition of 100 mL of 5M NaOH solution. The precipitate was rinsed with demineralized water until the pH was neutral; then, it was dried in an oven at 100°C for three hours. Dissolving 0.196 grammes of $CuSO_4{\cdot}5H_2O$ in 500 mL of demineralised water produced a stock solution of Cu ions. Diluting this solution allowed one to get Cu concentrations of 10, 20, 30, 40, 50, and 60 mg/L for adsorption studies.

Adsorption effectiveness was assessed under several conditions including adsorbent mass, contact duration, and Cu concentration. Combining 0.5, 1, 1.5, 2, 2.5, and 3 grams of activated carbon/Fe_3O_4, agitated at 150 rpm for 60 minutes, then separated using a magnet the mg/L Cu solution. Three grams of activated carbon/Fe_3O_4 were added to 100 mL of 10 mg/L Cu solution and agitated for 15, 25, 30, 45, 60, and 75 minutes at 150 rpm before separation in the contact time variation. Three grams of activated carbon/Fe_3O_4 were added to one hundred millilititer Cu solution at initial concentrations of 10, 20, 30, 40, 50, and 60 mg/L in the Cu concentration variation. After 75 minutes at 150 rpm, the mixture separated.

To evaluate adsorption efficiency and capacity, each sample's final Cu concentration was determined using an Atomic Absorption Spectrophotometer (AAS). FTIR Spectroscopy identified functional groups on the adsorbent surface, while SEM-EDX characterized the activated carbon/Fe_3O_4 composite by surface morphology and elemental composition.

2.2. Data Analysis

The data analysis in this study includes the determination of moisture content, ash content, iodine adsorption capacity, adsorption percentage, and adsorption capacity of the activated carbon/Fe_3O_4 adsorbent. According to Shahrokhi-Shahraki et al. (Shahrokhi-Shahraki et al., 2021), the adsorption capacity may be determined with the equation 1:

$$Q = \frac{(Co-Ce)V}{m} \qquad (1)$$

Where:

Q = Adsorption capacity (mg/g)
Co = Initial solution concentration (mg/L)
Ce = Final solution concentration (mg/L)
V = Volume of solution (L)
m = Mass of adsorbent (g)

3.0 RESULT AND DISCUSSION

3.1 Activated Carbon Specification

The coconut shells utilized in this work were chemically activated using a 10% H_3PO_4 solution. Table 1 summarises the findings of tests of the activated carbon's quality against SNI (Indonesian) criteria.

Table 1. Parameter contents of activated carbon

Parameter	Test Results	SNI Standard	Results
Moisture Content	2.48 %	Max 15 %	Qualified
Ash Content	1.29 %	Max 10 %	Qualified
Iodine Adsorption	767.51 mg/g	Min 750 mg/g	Qualified

The results signify that the activated carbon produced meets the SNI standards, making it suitable for use as an adsorbent.

3.2 Characterization Of Activated Carbon And Activated Carbon/Fe_3O_4

Examining the surface form and functional groups of the adsorbent, SEM-EDX and FTIR helped to define it. The porous structure with uneven surfaces seen in the SEM images (Figure 1) suggested that the activation procedure employing H_3PO_4 was efficient in producing pores. With white particles covering the surface and showing the presence of Fe_3O_4, the SEM pictures of activated carbon/Fe_3O_4 revealed smaller particles than those of the activated carbon.

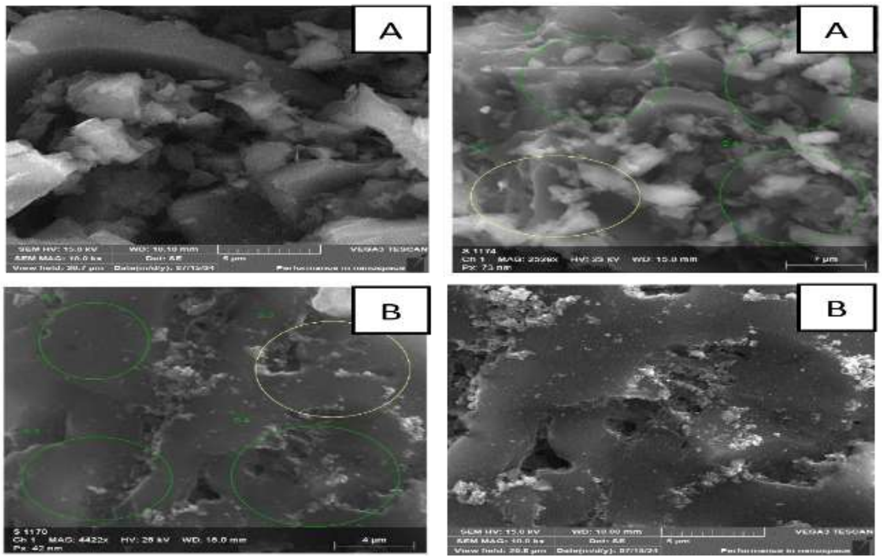

Figure 1. Surface morphology of (a) activated carbon and (b) activated carbon/Fe3O4

The EDX analysis disclosed the existence of carbon (C), oxygen (O), silica (Si), phosphorus (P), and aluminum (Al) (Figure 2). The carbon content was the highest at 90.04%, followed by oxygen at 9.87%. The EDX analysis confirmed the presence of iron (Fe) and chlorine (Cl), which are components of $FeCl_3.6H_2O$ and $FeSO_4.7H_2O$ used in the synthesis. The carbon content was 83.12%, and iron content was 15.44%. It can be concluded that the collection of small spheres observed in Figure 1.B. indicates that Fe_3O_4 particles have coated the activated carbon, making the pore structure of the activated carbon no longer visible (Bahador et al., 2021; Wakshum et al., 2024)

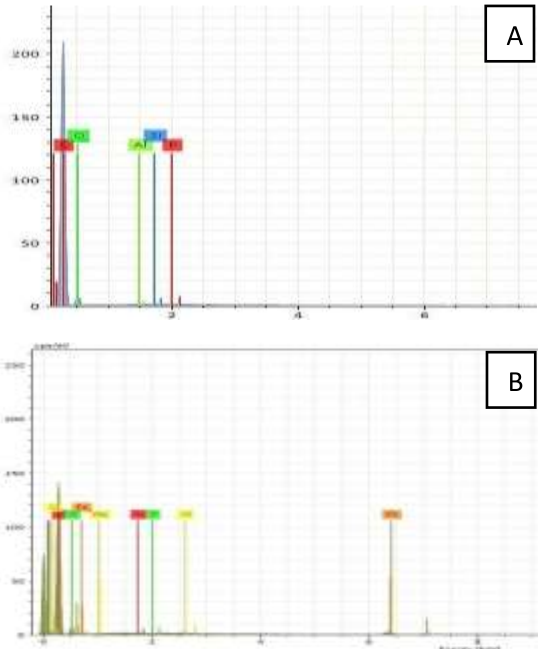

Figure 2. (a) Activated carbon and (b) activated carbon/Fe3O4 element graph

The FTIR analysis of activated carbon revealed key functional groups that contribute to its adsorption capabilities. As shown in Figure 3, the spectrum showed strong absorption peaks at 3744.01 cm^{-1}, corresponding to O-H stretching vibrations indicative of alcohol groups, and at 2362.60 cm^{-1}, representing C≡C stretching vibrations from alkyne groups. Additionally, peaks at 1692.70 cm^{-1} and 1564.31 cm^{-1} were observed, indicating the presence of C=O (ketone) and C=C (aromatic or alkene) stretching vibrations, respectively. These functional groups highlight the polar nature of the activated carbon, which enhances its ability to adsorb contaminants. For activated carbon/Fe_3O_4, the FTIR spectrum displayed similar peaks, including O-H stretching at 3738.27 cm^{-1} and C≡C stretching at 2359.01 cm^{-1}, along with a distinct peak at 555.15 cm^{-1}, which corresponds to Fe-O stretching vibrations.

The existence of a distinctive peak guarantees the effective integration of Fe_3O_4 into the activated carbon matrix, therefore giving the composite magnetic characteristics and improving its adsorption performance. Attributed to Fe–O stretching vibrations, the FTIR spectra of the activated carbon/Fe_3O_4 composite displayed a clear absorption band at 552.28 cm^{-1} during the adsorption process, therefore indicating the effective adsorption of Cu^{2+} ions onto the adsorbent surface. Furthermore, the identification of O–H and C≡C functional groups suggests that π–π interactions and hydrogen bonding would have helped to explain the adsorption process. With their functional groups and magnetic properties acting in concert to enable copper ion removal from aqueous environments, the FTIR results collectively justify the potential of the activated carbon/Fe_3O_4 composite as an efficient adsorbent.

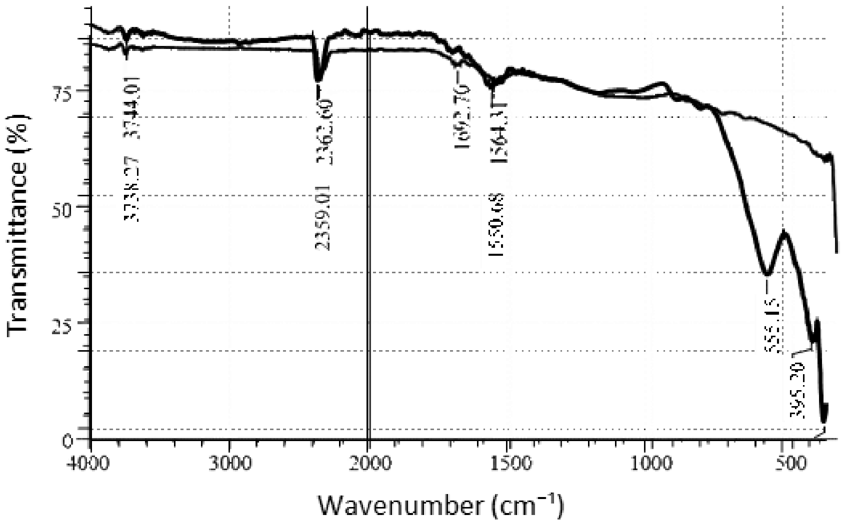

Figure 3. FTIR Characteristics of Activated Carbon and activated carbon/Fe_3O_4

3.3 Adsorption Effectiveness

The adsorption efficacy of the activated carbon/Fe_3O_4 composite for Cu^{2+} ion removal was systematically assessed by manipulating critical operational variables such as adsorbent dosage, contact duration, and initial copper ion concentration (as shown in Table 2). The findings revealed that the use of 3 g of adsorbent resulted in the highest removal efficiency of 98.10%, corresponding to an adsorption capacity of 0.327 mg/g. The presence of O-H and C=O functional groups, as confirmed by Fourier-transform infrared (FTIR) spectroscopy, suggests that activated carbon possesses a polar surface, enhancing its affinity for metal ions (Liu et al., 2022).

The relationship between adsorbent mass and adsorption efficiency demonstrated that increasing the mass of activated carbon/Fe_3O_4 led to improved Cu ion removal due to the availability of additional active adsorption sites. However, the adsorption capacity per gram of adsorbent exhibited a slight decline, likely due to inter-site competition and possible agglomeration effects reducing surface accessibility (Tan et al., 2024).Regarding contact time, an equilibrium state was observed at 75 min, with an adsorption efficiency of 98.30% and an adsorption capacity of 0.3276 mg/g. The first fast adsorption phase can be ascribed to the prevalence of accessible active sites. In contrast, the subsequent plateau phase suggests saturation of the adsorbent surface and a reduction in the adsorption rate as equilibrium is approached. The effect of initial Cu concentration revealed that an optimal concentration of 60 mg/L resulted in the highest adsorption efficiency of 99.70% and an adsorption capacity of 1.994 mg/g. While higher Cu concentrations generally contributed to increased adsorption capacities, a slight decline in efficiency was observed at intermediate concentrations, likely due to saturation of adsorption sites and the formation of competing interactions between metal ions (Hong et al., 2019; Ma et al., 2020).

These results highlight the remarkable effectiveness of the activated carbon/Fe_3O_4 composite in removing Cu^{2+} ions, reinforcing its potential as a viable adsorbent for wastewater treatment applications. The adsorption behavior demonstrated in this study is consistent with prior findings, thereby supporting the applicability of magnetically modified activated carbon in the remediation of heavy metal-contaminated effluents.

Table 2. Table of adsorption comparison

Parameter	Optimal value	Adsorption efficiency	Adsorption capacity
Mass of adsorbent	**3 g**	**98.10%**	**0.327 mg/g**
Contact time	**75 min**	**98.30%**	**0.3276 mg/g**
Concentration	**60 mg/L**	**99.70%**	**1.994 mg/g**

FTIR analysis of the activated carbon/Fe_3O_4 composite following the adsorption process (Figure 4) exhibited notable spectral changes, confirming the effective uptake of Cu^{2+} ions. A distinct absorption band at 552.28 cm⁻¹ was identified, corresponding to Fe–O stretching vibrations, which indicates the critical role of Fe–O bonds in facilitating metal ion adsorption. This absorption arises from the stretching vibrations within the Fe–O bonds present in the Fe_3O_4 crystal structure (Rasheed et al., 2025). Moreover, distinctive peaks at 2362.60 cm^{-1} linked with C≡C stretching and at 3680.18 cm^{-1} linked with O–H stretching. These results imply that hydrogen bonding and π-bond interactions are involved in the adsorption process. Especially, the O–H functional group is probably in charge of facilitating hydrogen-bond interactions among adsorbed water molecules, so improving the affinity of the adsorbent surface toward Cu^{2+} ions.

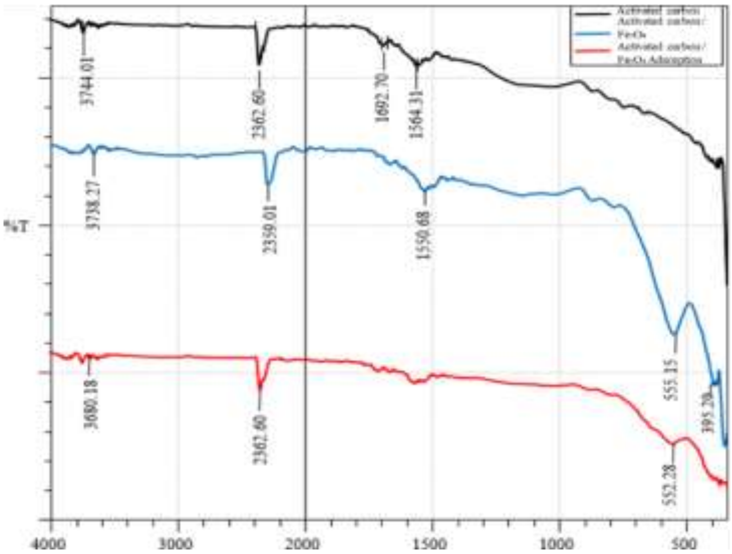

Figure 4. Comparison of FTIR characterization results of activated carbon/Fe_3O_4 after adsorption

The appearance of these peaks confirms the functional groups' influence on activated carbon and the magnetic properties of Fe_3O_4 on the adsorption process. The FTIR data following adsorption generally shows the efficiency of activated carbon/Fe_3O_4 in Cu ion collecting capacity. Its adsorption capability is much raised by the Fe-O bonds and functional groups (Wen et al., 2015; Xie et al., 2019). With best performance under some mass, contact duration, and concentration, the results table and FTIR analysis indicate that activated carbon/Fe_3O_4 is an outstanding adsorbent for Cu ions. Fe-O bonds and functional groups like O-H and C≡C improve its adsorption characteristics, so this material is a possible one for wastewater treatment.

4.0 CONCLUSION

The characterisation and adsorption performance of activated carbon/Fe_3O_4 show its efficiency for removing Cu ions. Coconut shell-derived activated carbon satisfied SNI criteria for moisture content (2.48%), ash content (1.29%), and iodine adsorption capability (767.51 mg/g). SEM-EDX investigation showed a porous structure, while FTIR validated functional groups like Fe-O, indicating Fe_3O_4 inclusion. Optimal adsorption conditions were obtained with 3 g of adsorbent, 75 minutes of contact time, and an initial Cu concentration of 60 mg/L, resulting in a maximum adsorption efficiency of 99.70% and a capacity of 1.994 mg/g. These findings emphasize activated carbon/Fe_3O_4 as a very effective and readily separable adsorbent for Cu removal. Future research should investigate pH and temperature modification to improve its efficacy in wastewater treatment.

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