Carbon-magnetic composites from agricultural waste and natural resources: A sustainable adsorbent for copper removal

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ABSTRACT

This study reports on the synthesis and characterization of the Fe_3O_4/activated carbon composites derived from oil palm empty fruit bunch (EFB) waste and natural iron sand for copper (Cu^{2+}) adsorption. Composites with ratios of 1:1, 5:3, and 3:5 (EFB carbon:iron sand) were prepared. Iron sand was processed by co-precipitation to produce Fe_3O_4 magnetite, while EFB carbon was activated using 1 N CH_3COONa. The structural and functional properties were analyzed by XRD, FTIR, and SEM-EDS, while adsorption efficiency was measured using AAS. Results confirmed Fe_3O_4 formation with a cubic structure ($a = 8.3750$ Å), particle sizes of 79.97–98.91 μm, and excellent copper removal efficiency of 99.8% with an adsorption capacity of 0.377 mg/g.

Keywords: adsorption, CH_3COONa 1 N, Fe_3O_4/C, Co-precipitation, oil palm empty fruit bunches.

INTRODUCTION

Water is an essential element in life. The water supplies on Earth, whether obtained from underground sources or from above-ground sources, must meet the requirements for the use of the water, such as for daily life (Dewi & Hadisoebroto, 2021). Good water is the water that meets health requirements, namely bacteriological, chemical, radioactive and physical requirements. In the chemical requirements stated, clean water does not contain chemicals in the quantities that exceed the limit (Rolia et al., 2023). Clean water, which is essential for daily life, is becoming increasingly scarce. The main cause is the pollution of fresh water, which occurs when industrial waste, chemicals, agricultural fertilizers and pesticides, as well as household and municipal waste, enter the soil and water sources (Senthil Kumar et al., 2019). The discharge of industrial waste without proper management, containing hazardous organic pollutants, such as dyes, has caused contamination of soil and waterways (Khue et al., 2025). Heavy metals are the chemicals often found in water.

Heavy metals in water are sorted from most to least abundant, namely Pb > Cu > Cd > Cr (Ummah, 2019). However, even in small amounts heavy metals still endanger health if they continue to accumulate in the bodies of living things. The water polluted by copper (Cu) will generally change yellowish to greenish color according to the concentration level, smell, and create a metallic taste in water. Copper, as a heavy metal, is needed for the development of the human body (Siringoringo et al., 2022). The removal of heavy metals in water has been carried out by various conventional methods including precipitation, reduction, oxidation, solvent extraction, ion exchange, and adsorption (Kardiman et al., 2020). The process by which molecules

or particles stick to the surface of a solid substance known as an adsorbent is known as adsorption. However, some of the above methods have disadvantages related to high costs and low efficiency at metal concentrations of 1 mg/l – 100 mg/l (Cherono et al., 2021). In a previous study conducted by (Sembiring et al., 2023), the results of research on the synthesis of natural iron sand for heavy metal absorption materials showed that 99.86% was produced as a heavy metal absorption material (Rianna et al., 2024), with an efficiency of 96.26% in the absorption of Cu and Cr. Processing agricultural waste is not only cheaper for wastewater treatment, but also helps reduce disposal problems. Many studies utilize rice husks, sugarcane bagasse, coconut shells, and palm oil waste as materials for making adsorbents (Salamah et al., 2025). Natural iron sand has the potential to be used as an adsorbent, because it can absorb heavy metals, such as Cu, Cr, Cd, and Pb. The presence of iron oxide compounds in it plays a role in expanding the pore surface of the adsorbent. In addition, as the temperature increases, the maximum capacity of natural iron sand in the absorption process also tends to increase (Danarto, 2021). The coprecipitation technique works through a precipitation process, in which soluble substances are absorbed and form deposits. This process is often used in the production of natural iron sand and the separation of analytes from contaminants. This method can produce micro and nano particles at room temperature, in a short time, and with high-quality materials (Lestari, 2023).

Adsorption is a process in which molecules or particles attach themselves to the surface of a solid material called an adsorbent. The use of nanoadsorbents is considered a relevant issue and is the main focus in heavy metal adsorption research (Zarabadipour et al., 2025). Simple and cost-effective adsorption, such as adsorption using appropriately modified agricultural waste, can increase the absorption capacity of heavy metals (Fitasari et al., 2022). Empty palm fruit bunches have great potential to be utilized as a promising renewable carbon source (Sisuthog et al., 2022). Their abundant availability also supports renewable production (Tussa Balsia et al., 2024). 23% of fresh fruit bunches consist of lignocellulose which is the main component of TKKS, which has the ability to adsorb heavy metals because TKKS has active groups -OH and -COOH (Muhammad, A.G., Oktasari, 2019).

Natural iron sand is a type of natural material that is easy to obtain. Natural iron sand contains many minerals, such as iron (Fe), titanium (Ti), and silica (Si) and contains 10–35% magnetic, namely magnetite, hematite, maghemite and dominant is magnetite (Melinia et al., 2022). In the research conducted by (Naat, 2022), iron sand extraction can be carried out using the sol-gel, alkalifusion, hydrothermal, and coprecipitation methods. In this study, the researcher used the coprecipitation method. Synthesis of natural iron sand by coprecipitation method and activation of TKKS carbon with CH_3COONa 1N. Coprecipitation is a simple method with the use of short time and low temperature namely, <100 °C (Lubis, 2022), as well as affordable costs compared to other conventional methods. Previous research by (Sumila et al., 2023), on the performance testing of palm oil activated carbon with CH3COONa 1 N activator found that TKKS activated carbon with sodium acetate can absorb iron (Fe) up to 91%. This condition indicates that CH_3COONa 1 N is optimal for heavy metal absorption. Magnetic biochar is a new type of material that combines magnetic properties with biochar, has larger pores, and is able to provide ample space to absorb and remove pollutants (Wang et al., 2023).

The effectiveness of biochar (BC) in extracting heavy metals from soil has attracted widespread attention among researchers. However, the adsorption capacity of BC for heavy metals tends to decrease over time, accompanied by a relatively low affinity for certain types of metals and limitations due to reaching saturation point. In addition, the addition of BC has the potential to modify the physical and chemical properties of soil, which in turn can affect the stability of heavy metals within it. To overcome these constraints, various BC modification strategies have been studied, such as the addition of functional groups and combination with other nanomaterials (Wang et al., 2025). Porous Fe_3O_4/C derived from metal–organic framework (MOF) materials (Kavoosi & Masoudpanah, 2025). In this study, researchers combined TKKS with other natural materials, namely natural iron sand, so that the expected absorption of copper can be more optimal. The test used FT-IR, XRD, SEM-EDS, and AAS.

METHODS

Preparation of natural iron sand

The natural iron sand that has been separated from other materials was crushed and passed through a 100 mesh sieve, then washed with ethanol and distilled water, precipitated for 30 minutes and then washed until the pH was neutral. It was filtered, then dried in an oven at a temperature of 200 $^{\circ}$C for 1 hour, then crushed again to produce natural iron sand powder then tested XRF to determine the elemental content in it. Various synthesis methods considered for producing magnetite (Fe_3O_4), nanoparticles, and Fe_3O_4-based nanocomposites include coprecipitation, green synthesis, hydrothermal, sol-gel, and electrochemical approaches (Shah et al., 2025).

Preparation of carbon from oil palm empty fruit bunches

Empty palm oil bunches were washed thoroughly, cut and dried in the sun for 3 days. TKKS in the furnace with a temperature of 500 °C for 1 hour. After cooling, TKKS was crushed with a mortar and passed through a 100 mesh sieve. TKKS was activated with CH_3COONa 1N activator stirred with a magnetic bar on a hot plate at 120 °C for 1 hour. After activation, it was washed until the pH was neutral and dried in an oven using a temperature of 100 °C for 1 hour, crushed using a mortar and pestle and then produced activated carbon powder of empty palm oil bunches.

Incorporation of natural iron sand and empty palm oil bunches

Fe_3O_4 powder and TKKS activated carbon powder were combined in ratio of 1 : 1, 5 : 3, 3 : 5 into a beaker glass. Than ball milling at 500 rpm for 1 hour at room temperature. The magnetic properties of the sample increased after undergoing the grinding process (Rianna et al., 2025). Calcination was carried out at 800 °C for 1 hour. Then the sample was crushed and the resulting Fe_3O_4/C powder.

Characterization Fe_3O_4/C

XRD testing was carried out to see the crystal phase, lattice parameters and degree of crystallinity. SEM-EDS testing to analyze the surface and mapping of the constituent elements of natural iron sand samples and activated carbon TKKS. FT-IR testing to determine the functional groups of the samples formed. AAS testing to determine the percentage of adsorbent adsorption and adsorption efficiency against copper (Cu).

RESULTS AND DISCUSSION

XRF analysis of Fe_3O_4/C composite

XRF characterization was used to analyze the elemental content of natural iron sand synthesized using the coprecipitation method. The Fe_3O_4 content was varied in a controlled manner to adjust the physicochemical, mechanical, and magnetic properties of the hydrogel (Viteri et al., 2025). Table 1 presents the XRF test results for natural iron sand.

On the basis of the XRF test results, it was found that the main element in natural iron sand is Fe with a concentration of 49.52% wt. This result is similar to previous research conducted by (Rahmayeni et al., 2023), who identified the Fe content in natural iron sand resulting from the concentration process using a sluice box, found that Fe was the most dominant element at 65.725%, and the research by (Khasanah et al., 2021) found that the

Table 1 Elemental content of natural iron sand using XRF testing

No.	Element	Concentration (%wt)	Compound
1	Fe	49.52	Iron
2	Al	4.75	Aluminum
3	Si	3.98	Silicon
4	Ti	3.23	Titanium
5	Mg	2.43	Magnesium
6	Mn	0.52	Manganese
7	Ca	0.34	Calcium
8	V	0.20	Vanadium
9	K	0.29	Potassium
10	Zr	0.16	Zirconium
11	Zn	0.06	Zinc
12	Cl	0.04	Chlorine
13	Bi	0.02	Bismuth
14	P	0.01	Phosphorus
15	Rb	0.02	Rubidium
16	Ta	0.01	Tantalum
17	Cr	0.01	Chromium

dominant element was Fe at 70.75%, with other constituent elements being Al, Si, Mn, and Ti.

XRD analysis of Fe_3O_4/C composite

X-ray diffraction is one of the material testing tools currently used to observe the crystalline phase and atomic structure in a material using a technique that does not damage the sample (Samik et al., 2023)(Table 2, Figure 1).

The sharp peaks indicate that the magnetite synthesis has good crystallization performance forming cubic crystals. These results correspond to magnetite JCPDS 00-019-0629 with lattice parameter α = 8.347Å with miller index peaks of [202], [220], [311], [400], [511], [440] respectively. In addition, it can be seen that the widened peak in the diffraction angle range of 18–28° indicates the presence of diffraction peaks of amorphous carbon layers. This confirms that the Fe_3O_4/C composite has been successfully synthesized (Liu et al., 2019). At a ratio of 1:1 obtained a degree of crystallinity value of 76.35%, in the ratio of 5 : 3 obtained a degree of crystallinity value of 63.43%, and in the ratio 3 : 5 obtained a degree of crystallinity value of 78.86%. The value of the degree of crystallinity obtained shows a high number, indicating the level of regularity of the atomic structure in the material is increasingly organized, which affects its magnetic properties. The degree of crystallinity decreased especially in the 5 : 3 ratio with 5 g carbon and 3 g natural iron sand. This indicates that carbon inhibits crystal growth and increases the amorphous part of the material. The different crystallinity values are due to the difference in the amount of carbon added to the natural iron sand. Different amounts of activated carbon in each sample will prevent the nanoparticle from moving, which reduces agglomeration. In addition, the more carbon added, the wider the diffraction peak, which results in a decrease in the degree of crystallinity. In a previous study, observed how the amount of carbon in Fe_3O_4@ZnO-C nanocomposites affects the structure and magnetic properties. It was explained that a smaller crystal size indicates lower crystallinity of the material, due
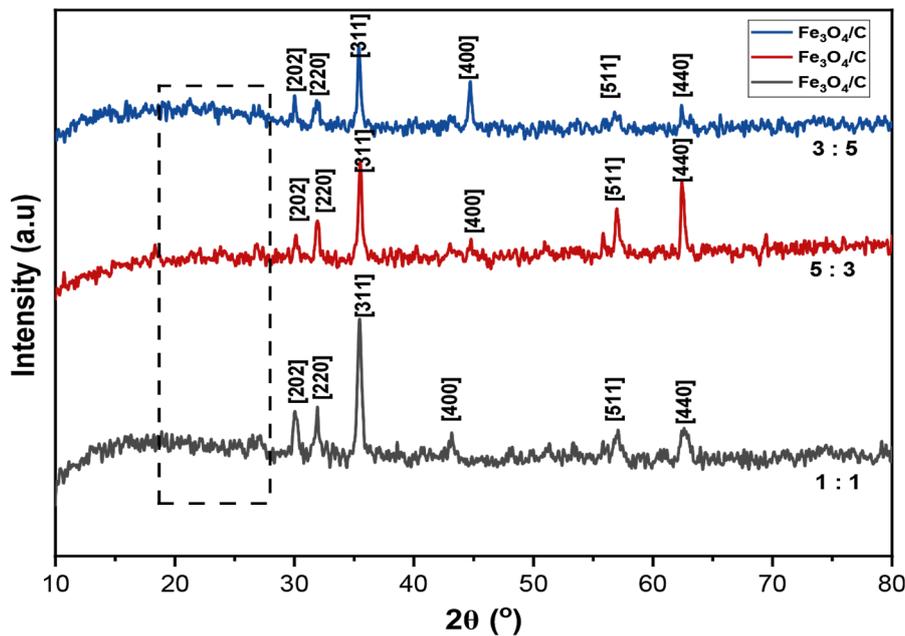

Figure 1. Diffraction pattern of Fe_3O_4/C

Table 2. Crystal size, lattice parameters, degree of crystallinity

Sample name	2θ (deg)	FWHM (deg)	θ(rad)	Crystal size (nm)	Lattice parameters (nm) a = b = c	Degree of crystallinity (%)
C1	35.45	0.0071	0.3093	20.5	0.8399	76.35
C2	35.41	0.0050	0.3090	29.13	0.8407	63.43
C3	35.50	0.0052	0.3098	28.02	0.8388	78.86

to variations in carbon addition in each sample, so that the magnetic properties of the material used also change (Astuti et al., 2022).

FT-IR analysis of Fe₃O₄/C composite

To detect the chemical structure of a compound in a sample, the Fourier Transformed Infrared (FTIR) method is used, which utilizes molecular vibrations. FT-IR is a tool that can detect functional groups and analyze mixtures from the sample being analyzed without damaging the sample during the analysis process (Abriyani et al., 2024)(Figure 2). Specimens in KBr tablets, sodium chloride, or liquid samples or suspensions in liquid paraffin can be used to measure solid spectra (Mani et al., 2023). The absorption in the wavelength range of 1680–1620 identifies C=C stretching vibrations which are Alkena functional groups. In the wavelength range 2300–1800, C=O stretching vibrations were identified as carbonyl functional groups. In the range of wavelength absorption areas 2700–2500 at a wavelength of 2653 in each sample identifies the presence of O-H stretching vibration the intensity of the band widens. The 3700–3600 wavelength absorption region range in the sample was identified as O-H stretching. In the 750–550 cm⁻¹ absorption band wavelength range, the interactions between metal and oxygen were identified. The Fe-O vibrations were observed to cause strain in sample C3, with sharpened band intensity, while samples C1 and C2 showed broadening (Husain et al., 2021). This may occur due to the addition of Fe₃O₄ to activated carbon with different compositions, causing changes in the functional group vibrations of the activated carbon, and the interaction between the components to occur successfully (Fisli et al., 2018). Higher temperatures will cause changes in functional groups, namely shifts, loss of absorption peaks and decreases in absorption levels. Unstable radical compounds will be formed which then react, forming new compounds (Maslahat et al., 2022).

SEM-EDS analysis of Fe₃O₄/C composite

SEM-EDS test results show that the surface of the sample has a non-uniform and uneven particle size of activated carbon. SEM-EDS provides faster and more accurate results, and this method is easy to use with a short research time (Hulungo et al., 2022). TKKS activated carbon and natural iron sand were successfully synthesized, with open pores appearing more clearly. The nature of magnetite makes the force of attraction between particles so that is some parts there is agglomeration due to the entry of Fe₃O₄ particles into the pores of activated carbon which can create new pore gaps; besides that, the difference in atomic radius between activated carbon TKKS and Fe₃O₄ may cause agglomeration. The magnetic properties of magnetite create an attractive force between particles, resulting in agglomeration in some areas due to the entry of Fe₃O₄ particles into the pores of activated carbon, which can create new pore gaps, as in a similar study conducted (Ramadiani & Munasir, 2021). with Fe₃O₄/C composites where the activated carbon came from waste paper and rubber
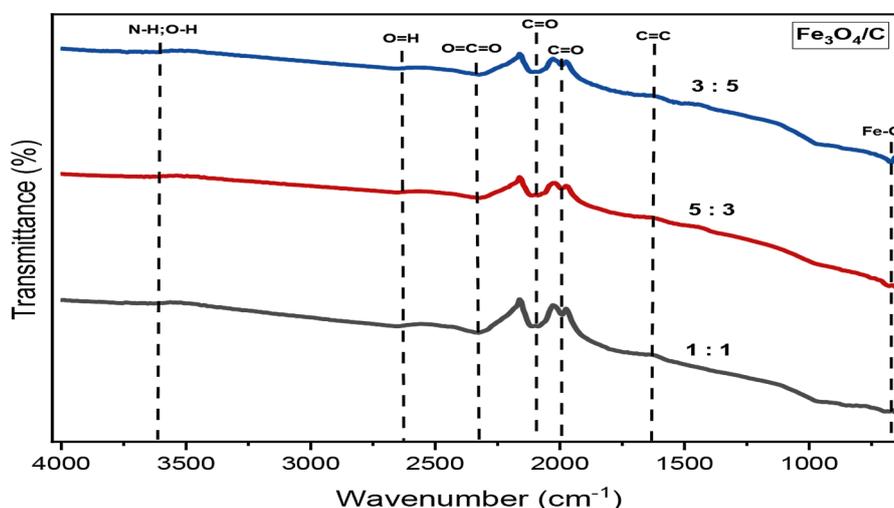

Figure 2. FT-IR results curve of Fe₃O₄/C

wood sawdust, Fe₃O₄ was found to enter and fill the pores of the activated carbon matrix, creating new pore gaps in some areas. In the study by (Rianna et al., 2022), which was about the study and characterization of Fe₃O₄ synthesized from natural iron sand, agglomeration also occurred due to the High Energy Milling (HEM) mixing process, which was also carried out in this research process. With a ratio of 1:1 Fe_3O_4/C shows the elemental composition of C = 82.8%, O = 11.8%, Fe = 2.8%, Si = 1.1%, Na = 0.7%, Al = 0.5%, Mg = 0.3% and has a particle size of 79.97 μm. With a ratio of 5 : 3 shows the elemental composition Fe_3O_4/C of C = 77.8%, O = 15.8%, Fe = 2.2%, Si = 2.0%, Na = 0.9%, Al = 0.7%, Mg = 0.6%. With a ratio of 3 : 5 shows the

elemental composition Fe_3O_4/C of C = 76.0%, O = 16.3%, Fe = 4.3%, Si = 1.4%, Na = 1.1%, Al = 0.6%, Mg = 0.3% (Figure 3).

AAS analysis of Fe_3O_4/C composite

Empty palm oil bunches activated carbon adsorbent powder: Natural iron sand was used to adsorb heavy metals. The heavy metal to be adsorbed was cooper ion (Cu). The results of analysis AAS indicate that the concentration of copper ions absorbed was below 0.0025 ppm or 0.025 mg/L, so that the cooper absorption efficiency for each sample was 99.8% and the adsorption capacity was 0.377 mg/g. Previous studies Naufal et al., 2025 have reported the synthesis of magnetic
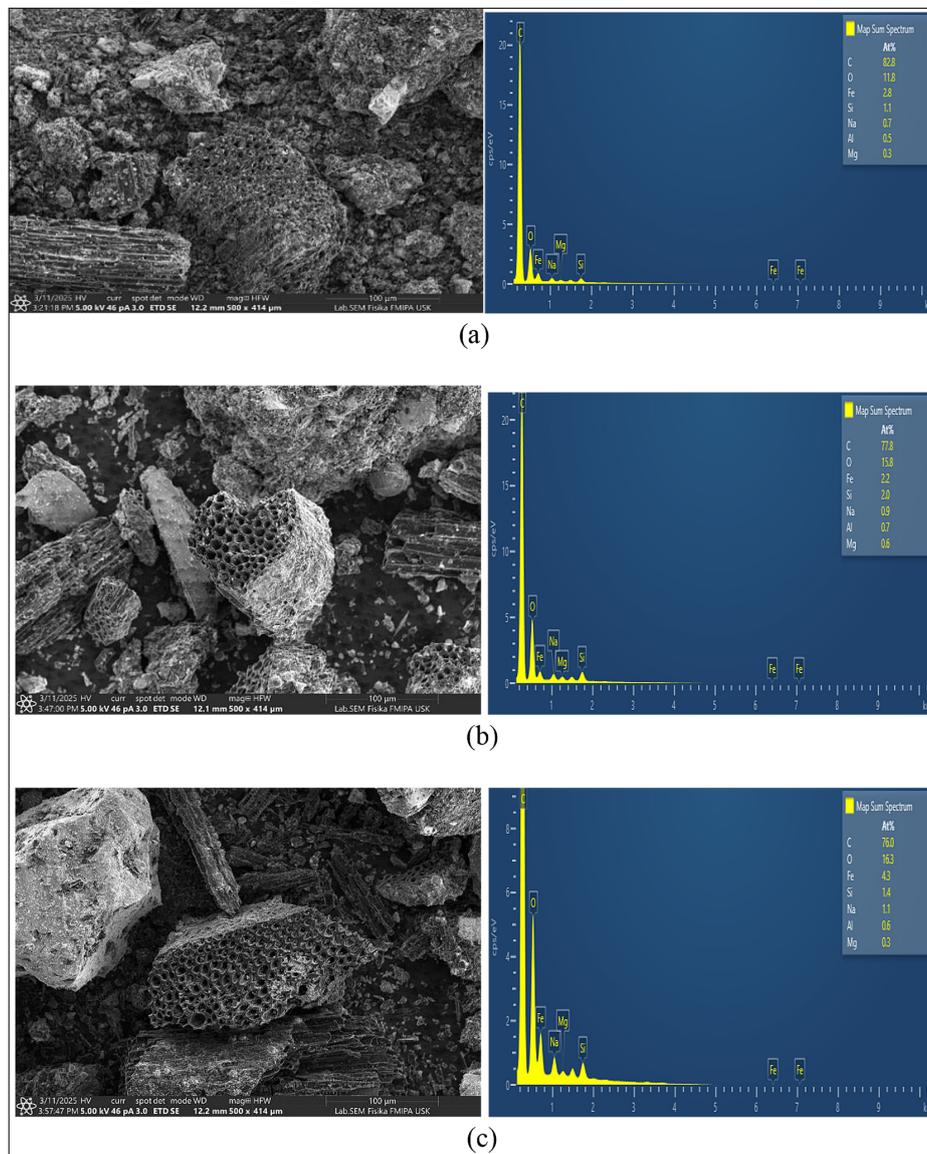

Figure 3. Morphology and chemical composition of Fe_3O_4/C form empty palm oil bunches: (a) Fe_3O_4/C 1 : 1, (b) Fe_3O_4/C 5 : 3, (c) Fe_3O_4/C 3 : 5

biocomposites from agricultural wastes, such as palm oil empty fruit bunches and sago dregs using solvothermal routes and amine functionalization, highlighting their potential as green adsorbent materials, these magnetic biocomposites contain Fe and amine at about 98.26% and 3.83 mmol.g. Kassim et al., 2023, reported that Fe_3O_4-modified biochar derived from oil palm empty fruit bunches exhibited high adsorption efficiency toward organic contaminants, particularly fluoroquinolone antibiotics such as ciprofloxacin, norfloxacin, and ofloxacin. The successful incorporation of Fe_3O_4 nanoparticles onto the biochar surface was confirmed by FTIR, SEM-EDS, and XRD analyses, resulting in removal efficiencies of up to 97% under optimal conditions and adsorption behavior following pseudo-second-order kinetics (Kassim et al., 2023). Biochar-based magnetic photocatalysts derived from oil palm EFB and coupled with $BiFeO_3$ have been reported to effectively degrade organic pollutants, such as ciprofloxacin, achieving up to 77.08% photodegradation under sunlight through enhanced catalytic activity and magnetic separability (Mohd Azan et al., 2022). Agro-based magnetic biosorbents derived from kapok fiber, EFB, and cellulose have been shown to effectively remove multiple heavy metal ions, with Pb(II) removal efficiencies up to 99.4% and exhibited improved performance compared to non-magnetic sorbents (Daneshfozoun et al., 2017). Catalysts based on oil palm EFB fibers have been successfully developed as magnetically separable heterogeneous catalysts, showing high esterification efficiency (up to 93.5%), good acidity, and stable reusability over multiple cycles (Krishnan et al., 2023).

However, these works mainly focused on material formation and functional group incorporation, with limited evaluation of adsorption performance. In contrast, the present study employed a simpler and more energy-efficient approach by utilizing activated carbon derived from oil palm empty fruit bunches and magnetite obtained from natural iron sand. The resulting Fe_3O_4/activated carbon composites exhibit a well-defined cubic magnetite structure and demonstrate excellent Cu^{2+} removal efficiency (99.8%) with a measurable adsorption capacity. This indicates that the synergistic combination of activated carbon and Fe_3O_4 not only ensures magnetic separability but also enhances practical adsorption performance, supporting the applicability of locally sourced, low-cost materials for sustainable water treatment.

CONCLUSIONS

The synthesized Fe_3O_4/C composites were successfully confirmed by XRD, FTIR, and SEM-EDS analyses, showing the presence of magnetite with a cubic structure and activated carbon functional groups. The composites exhibited irregular morphology with partial agglomeration. The AAS results revealed high adsorption performance, achieving 99.8% copper removal with an adsorption capacity of 0.377 mg/g. These findings demonstrate that oil palm empty fruit bunch activated carbon combined with natural iron sand is a highly effective adsorbent for heavy metal copper removal.

Acknowledgments

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