

POTENTIAL OF MAGNETITE (Fe₃O₄) SYNTHESIZED FROM ZIRCON SAND WASTE AS METHYLENE BLUE DYE ADSORBENT

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Abstract

Zircon sand mining is a major industry in Indonesia due to the country's vast zircon reserves. However, this industry also generates hazardous waste that can pollute the environment. This study aims to investigate the potential of magnetite (Fe₃O₄) synthesized from magnetic waste generated during zircon sand mining as an adsorbent for methylene blue dye removal. Magnetite was successfully synthesized via the co-precipitation method and characterized using XRD and FTIR. The synthesized magnetite demonstrated the ability to adsorb methylene blue from aqueous solutions with an adsorption efficiency of 68.52% after 80 minutes of contact time. The adsorption kinetics followed the pseudo second order model, evidenced by R² value of 0.9918, and the adsorption isotherm fitted the Langmuir model, indicating adsorption energy of 24.51 kJ/mol and adsorption capacity of 69.678 mg/g. These findings demonstrate the effectiveness of natural magnetite (Fe₃O₄) derived from zircon sand waste as an adsorbent for methylene blue dye removal. This research not only addresses the environmental issue caused by zircon sand mining waste but also develops a cost-effective and sustainable solution for methylene blue dye removal from wastewater.

Keywords: co-precipitation, magnetite (Fe₃O₄), methylene blue adsorption, Zircon sand waste

1. PENDAHULUAN

The textile industry, a cornerstone of Indonesia's economic landscape, plays a pivotal role in national growth, employment generation, and the provision of essential textile products. However, the industry's expansion has been accompanied by a surge in waste production, posing significant environmental challenges. Textile waste, generated from various production stages, including dyeing, washing, and finishing processes, comprises a complex mixture of dyes, chemicals, and suspended solids (Ghaly et al., 2013; Thakur & Chauhan, 2018). Dyes, a major component of textile waste, pose a substantial threat due to their carcinogenic, mutagenic, and toxic properties, potentially causing severe environmental degradation and health hazards. Moreover, dyes contribute to water pollution, leading to water discoloration and disruption of aquatic ecosystems (Berradi et al., 2019; Mehra et al., 2021)

Methylene blue, a heterocyclic aromatic compound with the molecular formula C₃₇H₂₇N₃Na₂O₉S₃, is derived from three interconnected benzene rings. This dark blue triarylmethane dye exhibits solubility in both water and alcohol. Methylene blue finds diverse applications in medicine, industry, and

research. In the industrial realm, methylene blue serves as a colorant for fabrics, paper, and plastics. It also finds applications in food and beverage coloring. However, methylene blue poses environmental hazards when released into the ecosystem. Methylene blue waste can contaminate water, causing discoloration, reduced dissolved oxygen levels, and ultimately, the death of aquatic organisms. The permissible threshold value for methylene blue concentration in waterways ranges from 5 to 10 mg/L (Hadayani et al., 2015). To mitigate the detrimental impact of dye waste generated by the textile industry, effective waste treatment strategies are essential. Various methods, including oxidation, reduction, and adsorption, can be employed for dye waste treatment (Natarajan et al., 2018; Sanghi & Bhattacharya, 2002).

Zircon sand production in Indonesia, particularly in Central Kalimantan, is on the rise, owing to its diverse applications in various industries. Central Kalimantan, a region rich in zircon sand deposits, holds significant potential for zircon sand mining. According to the Ministry of Energy and Mineral Resources, zircon sand production in Indonesia is expected to reach 2.2 million tonnes in 2022, a 10% increase from the previous year. Geological

Agency data indicates that Central Kalimantan alone possesses approximately 33.5 million tonnes of zircon sand deposits (ESDM, 2023). Kapuas, Gunung Mas, and Palangka Raya are among the districts in Central Kalimantan with substantial zircon sand deposits, offering promising opportunities for further exploration and mining (BPS Palangka Raya, 2023). Zircon sand is often found in association with other valuable minerals such as ilmenite, monazite, and rutile, further enhancing the region's mineral resource potential (Pownceby et al., 2015).

Magnetic waste generated from zircon sand mining comprises a heterogeneous mixture of minerals and chemical compounds, with iron oxides, zircon, monazite, and other mineral constituents constituting its primary components (Tranvik et al., 2017). This waste exhibits a low zircon content, typically ranging from 20 to 30%. Magnetic waste from zircon sand mining is predominantly composed of magnetic minerals such as ilmenite, hematite, and magnetite (Wang, 2014). Separation of magnetic waste from non-magnetic waste can be effectively achieved using magnets. The recovered magnetic waste holds potential for reuse as industrial raw materials. Leaching represents one promising approach for managing magnetic waste from zircon sand mining (Trisnawati, 2020).

Magnetite (Fe_3O_4), an iron oxide compound with unique magnetic properties, can be isolated through various chemical methods, including reduction, precipitation, and extraction techniques. Extraction of magnetite from iron sources involves the addition of concentrated hydrogen chloride followed by the introduction of a base, resulting in the formation of a black precipitate (Malega et al., 2018). Among the various magnetite synthesis methods, the coprecipitation method stands out, involving the mixing of Fe (II) and Fe (III) compounds with NH_4OH (Agnestisia, 2017). Magnetite's remarkable ability to absorb dyes via an adsorption mechanism makes it an ideal candidate for wastewater treatment applications. Dyes adsorbed onto the magnetite surface undergo physical and chemical binding, enabling their removal from the wastewater. By introducing magnetite particles into dye-contaminated wastewater, the dyes can be effectively separated using an external magnetic field. This process not only reduces effluent

contamination but also presents an environmentally friendly solution.

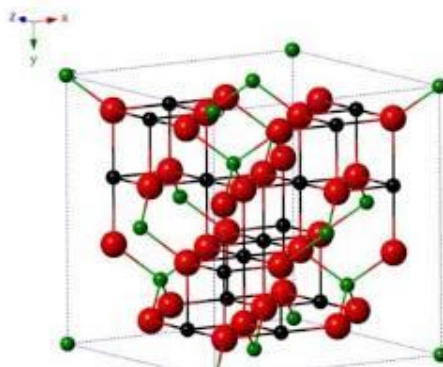


Figure 1. Magnetite Fe_3O_4 Structure
(Ganapathe et al., 2020)

Numerous studies have investigated the application of magnetic Fe_3O_4 as an effective adsorbent for various dyes. One notable example is the study by Lestari et al. (2021), which demonstrated the adsorption of Remazol Yellow dye using Fe_3O_4 modified with oil palm activated carbon. Under optimal conditions at pH 2, the adsorption efficiency reached an impressive 84.6% (Lestari et al., 2021). Further research by Sulistyaningsih et al. (2021) explored the potential of a magnetite-HA composite for Malachite Green dye removal. The study determined that the maximum adsorption capacity of Malachite Green dye using magnetite-HA was 104.15 mg/L, with optimal adsorption conditions achieved at pH 3 for a contact time of 90 minutes (Sulistyaningsih et al., 2021). Building upon these promising findings, the present study aims to evaluate the feasibility of utilizing magnetite (Fe_3O_4) waste derived from zircon sand mining in Teluk Batu sub-district, Kapuas district, as an adsorbent for methylene blue dye. The study will investigate the influence of key adsorption parameters, including pH, contact time, and methylene blue dye solution concentration, on the adsorption process.

2. RESEARCH METHODOLOGY

2.1 Materials and Instruments

The magnetic source came from zircon sand mining waste in Teluk Batu sub-district, Kapuas district. The extraction process used hydrochloric acid (Merck), precipitation using ammonia (Merck), methylene blue, distilled water, and whatman no 42. The equipment used included magnetic separator, 100-mesh sieve, mortar and pestle, pH analyzer (pH 6+ Thermo

Scientific), magnetic hotplate stirrer, orbital shaker (HSR-200 Health), centrifuge apparatus, oven (Mettler), FT-IR spectrophotometer (Shimadzu), X-ray diffractometer (XRD) (Philips PANalytical), and UV-Vis spectrophotometer (Safas).

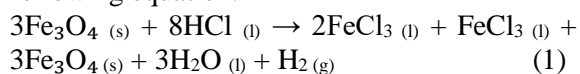
2.2 Methods

2.2.1 Magnetic Waste Material Preparation

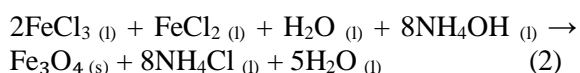
Magnetic material for adsorbent preparation was obtained from zircon sand waste through an extraction process. Waste zircon sand was subjected to magnetic separation using an external magnet to isolate the magnetic material. The extracted material was subsequently pulverized using a mortar and pestle and sieved through a 100-mesh sieve to obtain fine particles.

2.2.2 Magnetite (Fe₃O₄) Synthesis From Zircon Sand Waste

The synthesis of magnetite from zircon sand waste was based on the method described by (Budi et al., 2019), with slight modifications. The magnetic sample obtained during the preparation process was dissolved in concentrated hydrochloric acid (~12 N) at ratio of 1:10 b/v. The mixture was stirred using hot plate stirrer for 90 minutes at 80°C to promote dissolution and facilitate the subsequent reaction. The chemical reaction involved in the synthesis process can be represented by the following equation:



The solution was subjected to double filtration using filter paper. The resulting filtrate was then titrated with 2M NH₄OH solution until the pH reached 9, indicated by the formation of a black precipitate, Fe₃O₄. The reaction equation for this precipitation process is as follows:



The resulting precipitate then washed thoroughly with distilled water until the pH reached neutral. Subsequently, the precipitate was dried in an oven at 80°C for 12 hours. The obtained magnetite material, Fe₃O₄, was then characterized using Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD).

2.2.3 Determination Of Optimum pH For Methylene Blue Adsorption

A total of 20 mL of 50 ppm methylene blue solution was prepared by adjusting the pH of the solution to pH 4, 5, 6, 7, 8, 9, and 10 with the addition of 0.01 M HCl and 0.01 M NaOH solutions. Subsequently, 20 mg of magnetite Fe₃O₄ was added to each solution series. The mixture was agitated using a shaker rotator at 140 rpm under ambient light. After 1 hour, the residue was separated by centrifugation. The concentration of the solution before and after absorption was measured using a UV-Vis spectrophotometer at a wavelength of 665 nm.

2.2.4 Determination Of Optimum Contact Time For Methylene Blue Adsorption

A total of 20 mg of magnetite Fe₃O₄ were added to 20 mL of a 50 ppm methylene blue solution adjusted to the optimal pH. The mixture was agitated using a shaker rotator at 140 rpm under ambient light conditions for 20, 40, 60, 80, and 100 minutes. Subsequently, the solid residue was separated from the solution by centrifugation. The concentration of methylene blue in the solution before and after adsorption was measured using a UV-Vis spectrophotometer at a wavelength of 665 nm.

2.2.5 Determination Of Methylene Blue Adsorption Equilibrium

A total of 20 mg of magnetite Fe₃O₄ were added to 20 mL of methylene blue solutions with varying concentrations of 50, 100, 150, 200, 250, 300, and 350 ppm, all adjusted to the optimal pH. The mixtures was agitated using a shaker rotator at 140 rpm under ambient light conditions for the predetermined optimal stirring time. The solid residues were subsequently separated from the solutions by centrifugation. The concentration of methylene blue in the solutions before and after adsorption was measured using a UV-Vis spectrophotometer at a wavelength of 665 nm.

3. RESULTS AND DISCUSSION

3.1 Separation of Magnetic Materials From Zircon Sand Waste

The initial substance for the synthesis of magnetite was sourced from the waste of zircon sand mining in the Teluk Batu sub-district. The method employed was Dry Magnetic Separation using a Cross-belt separator. This apparatus comprises a magnet affixed above a moving belt that transports magnetic material.

The magnet elevates the magnetic substances from the zircon sand and positions them across the magnetic field, relegating the gangue to the tailings. The magnetic and non-magnetic minerals present in the waste were segregated based on their distinct reactions to the imposed magnetic field (Sajima et al., 2020).

3.2 Synthesis And Characterization of Magnetite (Fe_3O_4) Derived From Magnetic Waste

The obtained magnetic waste was subsequently processed to the next stage to obtain magnetite material. Magnetite (Fe_3O_4) particles were successfully synthesized via the co-precipitation method. Hydrochloric acid was added to bind and dissolve iron from the magnetic waste, followed by the addition of NH_4OH solution to induce the precipitation of Fe_3O_4 under alkaline conditions (Andimutiafitri, 2018). The resulting precipitate was washed with distilled water to remove dissolved impurities and then heated to remove

residual water content. The synthesized magnetite (Fe_3O_4) exhibited a black color and strong magnetic properties, as demonstrated by its attraction to a magnetic bar (Figure 2).



Figure 2. Magnetite synthesis results (Fe_3O_4) and testing using a magnetic bar

The synthesized magnetite was characterized using qualitative XRD and FTIR analyses. The synthesis process is illustrated in Figures 3 and 4 as follows:

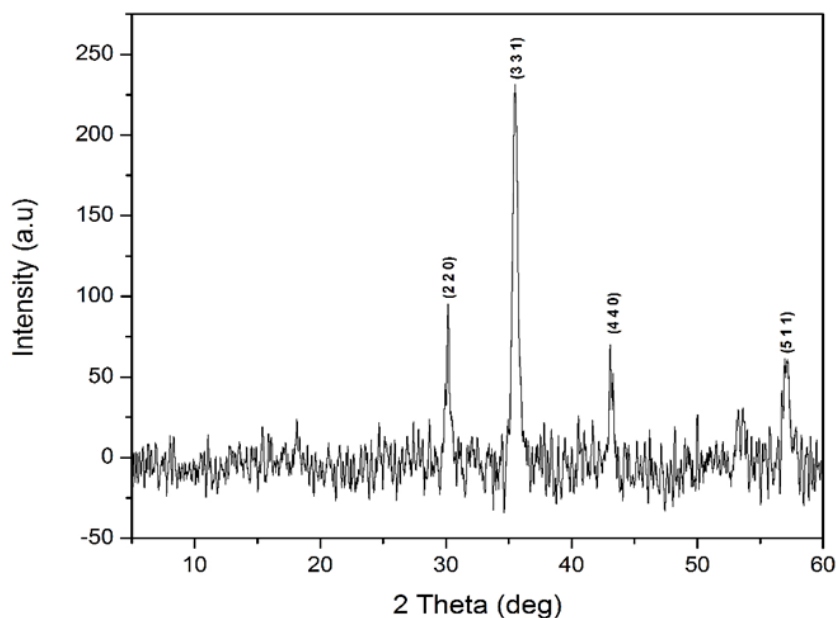


Figure 3. The X-ray diffraction (XRD) pattern of magnetite (Fe_3O_4)

The XRD pattern of the synthesized magnetite (Fe_3O_4) is presented in Figure 3. The highest diffraction peaks were observed at 2θ angles of 30.12° , 35.51° , 43.15° , and 57.06° , indicating the formation of magnetite (Fe_3O_4) as the dominant phase in the prepared material. These diffraction peaks are consistent with those reported in the literature (Budi et al., 2019) and correspond to the magnetite ICCD

single-phase structure with reference No. 00-075-0449, where the characteristic peaks of the synthesized magnetite are identical to those of standard magnetite with miller index values hkl (220), (331), (440), and (511). The sharp and intense peaks in the diffractogram suggest that the magnetite synthesized from zircon sand mining waste exhibits high crystallinity.

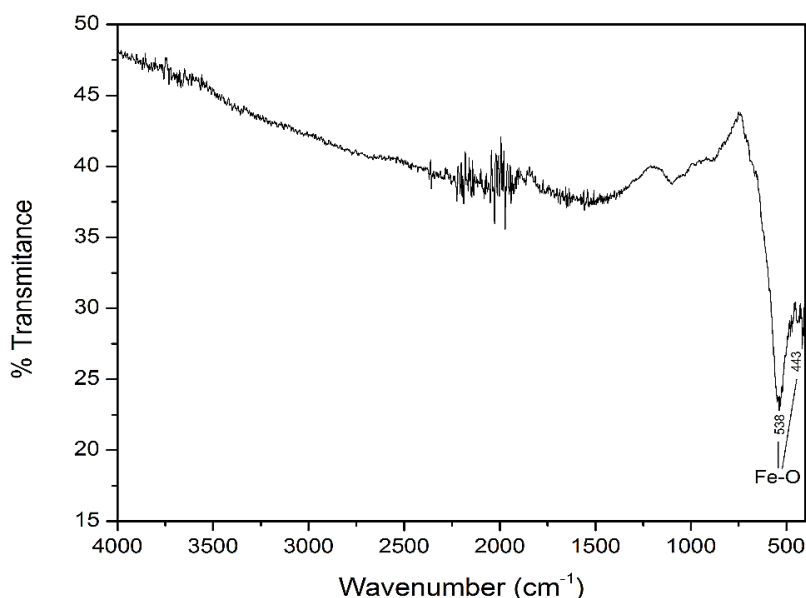


Figure 4. FTIR spectra of magnetite (Fe₃O₄)

FTIR characterization of the synthesized magnetite material was performed within the wave number range of 500 - 4000 cm⁻¹, as shown in Figure 4, to identify the functional groups present in the material. The FTIR spectra revealed distinct vibration peaks at wave numbers (cm⁻¹) of 538 and 443, corresponding to Fe-O stretching vibrations, which corroborate the magnetite formula (Fe₃O₄) and confirm the successful synthesis of magnetite (Nurhayati et al., 2021). In contrast to the FTIR spectra reported by Agnestisia (2017), which exhibited peaks at wave numbers (cm⁻¹) of 3448.72 and 1627.92 associated with O-H stretching and bending vibrations. The synthesized magnetite from zircon sand waste did not show any peaks corresponding to water molecules, indicating the absence of residual water after the drying process.

3.3 pH Optimization of Methylene Blue Adsorption

The influence of pH on the adsorption efficiency of methylene blue onto magnetite Fe₃O₄ was investigated by varying the pH from 4 to 10. pH is a crucial factor affecting adsorption as it alters the charge distribution on both the adsorbent and adsorbate due to protonation and deprotonation reactions of functional groups associated with methylene blue molecules. The results of the pH optimization study on absorption efficiency are presented in Figure 5.

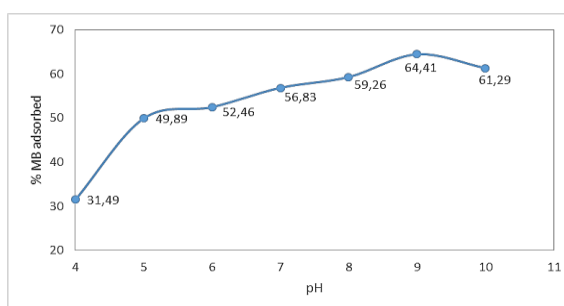


Figure 5. Effect of pH variation on methylene blue adsorption

The pH-dependent adsorption efficiency of methylene blue onto magnetite Fe₃O₄ was evaluated. The highest adsorption efficiency of 64.41% was achieved at pH 9, indicating optimal magnetite - methylene blue interactions under these conditions. The surface charge of magnetite is influenced by the solution pH, with an isoelectric point of 6.5. Below this pH, magnetite becomes positively charged, while above it, it acquires a negative charge (Agnestisia, 2017). Methylene blue, being a cationic dye, electrostatically interacts with negatively charged magnetite at pH values above 6.5. However, under excessively alkaline conditions (pH > 9), hydroxyl ions (OH⁻) compete with methylene blue for active sites on magnetite, hindering their interaction. Consequently, the adsorption capacity of magnetite for methylene blue diminishes at higher pH values.

3.4 Contact Time Optimization of Methylene Blue Adsorption

The influence of contact time on methylene blue adsorption onto magnetite was investigated by varying the contact time from 20, 40, 60, 80, to 100 minutes. Optimizing the contact time is crucial for determining the duration required for maximum methylene blue adsorption until equilibrium is established. The results of the contact time study are presented in Figure 6.

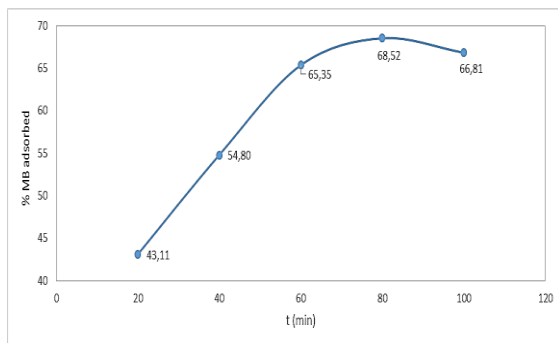


Figure 6. Effect of contact time variation on methylene blue adsorption

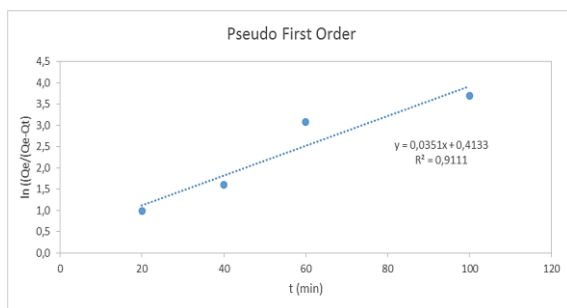
The adsorption efficiency of methylene blue onto magnetite increases with increasing contact time up to 80 minutes, as depicted in Figure 6. This extended time allows for sufficient interaction between the magnetite's active surface sites and the relatively large methylene blue molecules, facilitating the formation of a dye layer on the magnetite

surface. The maximum adsorption efficiency of 68.62% is achieved at a contact time of 80 minutes, indicating the attainment of adsorption equilibrium. Beyond this optimal contact time, methylene blue adsorption declines. This decrease can be attributed to several factors, including desorption caused by collisions between molecules during stirring, which disrupt the bonds between adsorbent and adsorbate molecules, and the reduction of available active sites on the adsorbent surface due to the formation of a complete dye layer, leading to adsorbent saturation.

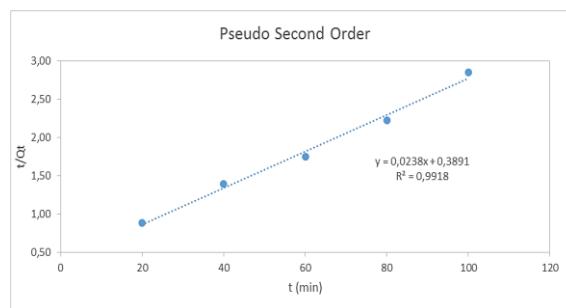


Figure 7. Adsorption of methylene blue with magnetite based on reaction time

The kinetics of methylene blue adsorption onto magnetite were investigated using the pseudo first order reaction equation of Lagergren and pseudo second order reaction equations. The adsorption process was examined by varying the contact time from 20 to 100 minutes, as shown in Figure 8.



(a)



(b)

Figure 8. Curves of pseudo first order kinetics (a) and pseudo second order kinetics (b) of methylene blue by magnetite

The regression equations for the two adsorption kinetic models are presented in Table 1. Pseudo first order kinetics were obtained from the linear relationship between $\ln(q_e - q_t)$ versus time (t), with an equation of $y = 0.0351x - 0.4133$. The correlation coefficient (R^2) for this model was 0.9111, and the rate constant (β) was $3.51 \times 10^{-2} \text{ min}^{-1}$. Pseudo

Second order kinetics were derived from the linear relationship between t/q_t versus time (t), with an equation of $y = 0.0238x + 0.3891$, yielding an R^2 value of 0.9918. Based on the linear regression equation ($y = mx + b$), the value of b in the pseudo second order kinetic model equation can be expressed as $1/(k q_e^2)$,

allowing for the calculation of the rate constant (k_2) as 1.46×10^{-3} g/mg.min.

Table 1. Adsorption kinetics models for methylene blue

Pseudo First Order		Pseudo Second Order	
β (min) ⁻¹	R ²	k_2 (g/mg.min)	R ²
$3,51 \times 10^{-2}$	0,9111	$1,46 \times 10^{-3}$	0,9918

The correlation coefficient (R) was calculated to assess the applicability of each adsorption kinetic model. Both models exhibited linear characteristics, indicating their suitability for describing the adsorption process. However, based on the correlation coefficient values, the pseudo second order kinetic model ($R^2 > 0.999$) demonstrated a better fit to the experimental data compared to the pseudo first order model ($R^2 > 0.900$). This suggests that the adsorption rate is proportional to the square of the methylene blue concentration, represented by $(q_e - q_t)^2$. This observation aligns with the chemisorption mechanism, where chemical bonding occurs between the adsorbent and adsorbate molecules.

3.5 Methylene Blue Adsorption Equilibrium

The influence of methylene blue concentration on its adsorption onto magnetite was investigated by varying the concentration from 50, 100, 150, 200, 250, 300 to 350 ppm. The adsorption experiments were conducted at pH 9 and a contact time of 80 minutes. The results are presented in Figure 9.

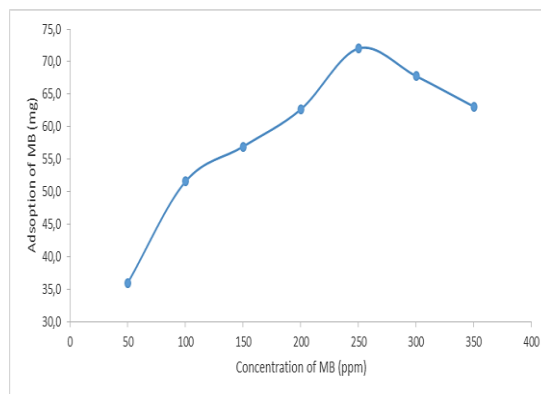
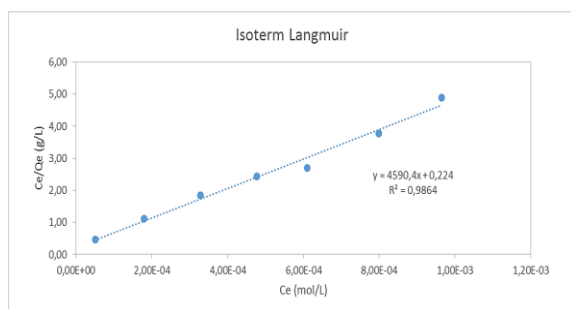
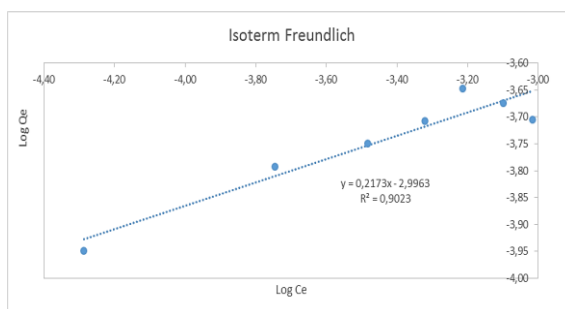


Figure 9. Effect of methylene blue concentration variation on magnetite adsorption capacity

The adsorption of methylene blue onto magnetite exhibited a concentration-dependent trend, with the adsorbent reaching maximum capacity at a concentration of 250 ppm methylene blue, suggesting a tendency towards equilibrium. Beyond this concentration, adsorption efficiency gradually declined. The equilibrium mechanism of adsorption can be further investigated through the determination of adsorption isotherms, which describe the relationship between methylene blue concentration and its distribution on the magnetite surface. Two commonly employed adsorption isotherm models are the Langmuir and Freundlich models. The Langmuir model assumes monolayer adsorption, while the Freundlich model accounts for multilayer adsorption. The suitability of each model can be assessed by converting the Langmuir and Freundlich isotherm equations into linear equilibrium curves and evaluating the coefficient of determination (R^2). A high R^2 value indicates a better fit of the model to the experimental data. The adsorption isotherm model for methylene blue adsorption onto magnetite is presented in Figure 10.



(a)



(b)

Figure 10. Curves of isotherm Langmuir (a) and isotherm Freundlich model (b) of methylene blue adsorption by magnetite

The findings of the magnetite (Fe_3O_4) adsorption isotherm analysis for methylene blue using the Langmuir and Freundlich models, presented in Table 2, demonstrate the high linearity of both isotherm types. The Langmuir isotherm exhibits a correlation coefficient (R^2) of 0.9864, while the Freundlich isotherm yields an R^2 value of 0.9023. Both models are suitable for determining the appropriate adsorption

isotherm based on the R^2 value, which is close to 1. However, a comparison of the two isotherms reveals that the Langmuir isotherm's linearity is closer to 1 than the Freundlich isotherm's. Consequently, the Langmuir isotherm is better suited for characterizing the adsorption mechanism of magnetite onto methylene blue.

Tabel 2. Equilibrium models for methylene blue

Isoterm Langmuir				Isoterm Freundlich		
B (mg/g)	K (L/mol)	E (kJ/mol)	R^2	B (mg/g)	N	R^2
69,678	20492,86	24,51	0,9864	$2,08 \times 10^{-3}$	4,602	0,9023

The Langmuir isotherm model was used to determine the adsorption capacity (B) of magnetite for methylene blue dye, which was found to be 69.678 mg/g. The Langmuir isotherm model was also used to calculate the adsorption energy using the equation $E = RT \ln K$, which was found to be 24.51 kJ/mol. This suggests that the adsorption of methylene blue onto magnetite occurs via monolayer chemisorption (Nafiah, 2016). This is likely due to electrostatic interactions between the cationic methylene blue and the anionic magnetite (Fe_3O_4).

4. CONCLUSIONS

Magnetite (Fe_3O_4) was successfully synthesized from magnetic waste of zircon sand mining which has magnetic properties. The X-ray diffractogram characteristics of magnetite have diffraction peaks that match the magnetite ICDD data No. 00-075-0449, this result is supported by FTIR characterization which shows Fe-O specific vibration peaks. The synthesized magnetite exhibited effective adsorption of methylene blue dye in solution, with an optimum adsorption efficiency at pH 9 and a contact time of 80. The adsorption kinetics followed the pseudo-second-order model, and the adsorption isotherm followed the Langmuir isotherm, suggesting monolayer chemisorption as the adsorption mechanism. The adsorption energy was calculated to be 24.51 kJ/mol and adsorption capacity yield of 69.678 mg/g, further supporting the chemisorption mechanism. These findings demonstrate the potential of synthesized magnetite for methylene blue dye removal from wastewater.

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