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Original Article

Effect of Different Temperatures in Magnetite Synthesis on Methylene Blue Adsorption

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Abstract— This study aims to synthesize magnetite (Fe_3O_4) particles using the coprecipitation method, with variations in temperature (70°C and 90°C) and reaction system (open and closed) to evaluate their effects on product quality. Characterization was conducted using FTIR, XRD, and organoleptic observation to confirm the formation of Fe_3O_4. Additional tests included magnetic attraction measurements through mass response and adsorption capacity (Q) analysis using methylene blue. FTIR analysis showed absorption bands at 3417.00 cm^{-1}, 1627 cm^{-1}, 1404 cm^{-1}, and 578 cm^{-1}, indicating the presence of O–H, C=O, and Fe–O functional groups. XRD patterns revealed diffraction peaks at 2θ values of 30.27°, 35.23°, 43.22°, 53.71°, 57.43°, and 62.11°, confirming the spinel crystal structure of Fe_3O_4. The sample synthesized at 90°C under closed conditions exhibited a darker black color and higher mass yield, suggesting improved crystallinity and phase purity. The closed system also showed higher adsorption capacities of 0.0008 $mmol \cdot g^{-1}$ at 70°C and 0.0018 $mmol \cdot g^{-1}$ at 90°C, along with stronger magnetic response. The open system produced a black precipitate with lower yield and weaker magnetic response, suggesting oxidation of Fe^{2+} to Fe^{3+} due to direct contact with oxygen, leading to the formation of compounds such as hematite or maghemite with lower magnetic properties. These results confirm that higher reaction temperatures and closed conditions optimally enhance the quality and stability of magnetite.

Keywords— *Ammonia; Close System; Coprecipitation; Magnetite; Methylene Blue; Open System.*

1. INTRODUCTION

Magnetite (Fe_3O_4) is a form of iron oxide that is included in the group of ferromagnetic minerals. This material has strong magnetic properties because its structure consists of a mixture of Fe^{2+} and Fe^{3+} ions arranged in a spinel lattice [1]. Various types of magnetic materials have been developed, including hematite (α-Fe_2O_3) [2], maghemite (γ-Fe_2O_3) [3], and ferrite ($MxFe_3{-}xO_4$, where M is a divalent metal such as Co, Mn, Zn) [4]. However, magnetite is the most widely used because of its chemical stability, strong magnetic properties at room temperature, and its ability to be synthesized in nanometer sizes with a narrow size distribution [5].

Several methods have been developed for the synthesis of magnetit including coprecipitation, sol–gel [6], hydrothermal, thermal decomposition, and microemulsion. Among these methods, the coprecipitation method is one of the most widely used because of its simple procedure, low cost, no need for organic solvents, and suitable for large-scale production [7]. This method is carried out by mixing Fe^{2+} and Fe^{3+} salt solutions in an aqueous medium, then adding a base agent to form Fe_3O_4 particle precipitates.

Basic agents play an important role in the coprecipitation process because they function to increase the pH of the solution, thereby triggering the formation of metal hydroxides which are then transformed into magnetite. Some types of bases commonly used in this method include sodium hydroxide (NaOH), potassium hydroxide (KOH), and ammonia (NH_4OH). The selection of the type of base greatly affects the reaction rate, particle size, and stability of the resulting suspension.

In this study, ammonia solution (NH_4OH) was used as a basic agent because ammonia has a lower oxidation ability towards Fe^{2+} compared to other strong bases. Therefore, ammonia produces a smaller amount of Fe^{3+}, which can affect the formation of the final phase such as maghemite. In addition to the type and concentration of base, other factors such as reaction

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temperature and stirring rate also have a significant effect on the synthesis results [8]. The reaction temperature affects the rate of nucleation and crystal growth, while the stirring rate affects the distribution of ions in the solution and the homogeneity of the particles formed.

Careful control of these parameters is essential to produce magnetite with optimal particle size, magnetic behavior, and phase purity. However, studies that directly link these synthesis variables to functional properties such as adsorption capacity remain limited. This study aimed to synthesize magnetite (Fe_3O_4) nanoparticles through the coprecipitation method using ammonia solution (NH_4OH) as the alkaline agent. It also sought to examine the influence of temperature and environmental conditions on the color and magnetic properties of the synthesized particles. Methylene blue adsorption tests were conducted to evaluate the adsorption capacity of the samples prepared at different temperatures. This study is expected to contribute to the development of an efficient and controlled synthesis method in producing high-quality Fe_3O_4 nanoparticles for various functional applications.

2. EXPERIMENTAL SECTION

2.1. Materials

The chemicals used were $FeCl_3 \cdot 6H_2O$ (99% purity Merck, Germany), $FeSO_4 \cdot 7H_2O$ (99% purity Merck, Germany), NH_4OH (25 % purity Merck, Germany), and metilen blue, and aquades.

2.2. Instrumentation

This research performed Overhead magnetic stirrer, Erlenmeyer flask, goblet, watch glass, spatula, 100 mL measuring cylinder, stirring rod, spektrofotometer UV-vis, FTIR (Shimadzu Prestige 21), and XRD (BRUKER AXS D8 ADVANCE ECO).

2.3. Magnetite Synthesis

Magnetite was synthesized using the coprecipitation method A total of 4 g of $FeCl_3 \cdot 6H_2O$ and 2 g of $FeSO_4 \cdot 7H_2O$ were weighed, then put into an Erlenmeyer flask. A volume of 25 mL of distilled water was added, then the mixture was stirred using a stirrer until evenly distributed. After that, the mixture was heated to a temperature variations of 70 °C and 90 °C, and then 25 mL of 25% ammonia solution was added directly. The mixture was allowed to react for 30 minutes, and then filtered to separate the precipitate. Initial tests on magnetic materials were conducted using X-Ray Diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR) to ensure the purity and phase identity of the materials used. The precipitate obtained was subjected to physical tests (color) and magnetic tests. Methylene blue adsorption tests were conducted to evaluate the adsorption performance of the synthesized samples. A total of 50 mg of each sample was shaken for 60 minutes in a shaker with methylene blue solution at concentrations of 2, 4, 6, 8, and 10 ppm to prepare a standard calibration curve. The adsorption capacity is determined from the decrease in concentration after the sample interacts with the substance, using a calibration curve as a reference. Through Equation (1), the amount of methylene blue adsorbed can be calculated.

$$Q = \frac{(\mathrm{co} - \mathrm{ce})}{\mathrm{w}} V \quad (1)$$

3. RESULT AND DISCUSSION

3.1. Fulvic Acid Extraction

Magnetite (Fe_3O_4) was synthesized using the coprecipitation method, which involves the simultaneous precipitation of multiple soluble components in a single phase to form solid particles [9]. The one-pot coprecipitation approach offers high efficiency by reducing the number of reaction steps and enhancing the interaction between components. This allows the material formation to occur either simultaneously or sequentially without the need for intermediate product separation. The magnetite synthesis is based on the interaction between $FeCl_3 \cdot 6H_2O$, as the Fe^{3+} ion source, and $FeSO_4 \cdot 7H_2O$, as the Fe^{2+} ion source. This precursor mixture was then heated to either 70°C or 90°C. Heating was used to increase the reaction rate and accelerate the crystallization of Fe_3O_4, which exhibits strong magnetic properties [10]. Once the desired temperature was reached, 25 mL of 25% ammonia solution was added directly to trigger the precipitation of iron hydroxides. The mixture was allowed to react for 30 minutes before being filtered to separate the Fe_3O_4 precipitate. The reaction of Fe_3O_4 formation is as follows Equation (2).

$$2FeCl_3 \cdot 6H_2O(s) + FeSO_4 \cdot 7H_2O(s) + 8NH_4OH(aq) \rightarrow Fe_3O_4(s) + 6NH_4Cl(aq) + (NH_4)2SO_4(aq) + 17H_2O(l) \quad (2)$$

Initial tests on magnetic materials were conducted using X-Ray Diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR) to ensure the purity and phase identity of the materials used. The results of this initial characterization became the basis for the next step, namely varying the reaction conditions in open and closed systems. The experiment was continued at two temperature variations, namely 70°C and 90°C, to emit the effect of the reaction environment and temperature on changes in the properties of the resulting magnetite material.

Figure 1a shows the absorption spectrum of functional groups of Fe_3O4. The absorption at wave number 3417.00 cm^{-1} indicates the presence of O–H stretching, while the absorption at 1627 cm^{-1} indicates the vibration of the C=O bond. In addition, the absorption at 1404 cm^{-1} is also related to the O–H stretching vibration. The typical absorption band for the Fe–O bond is detected at wave number 578 cm^{-1}, which

indicates that the Fe ions are in tetrahedral and octahedral positions in the crystal structure of magnetite (Fe_3O_4).

The crystal structure of the nanoparticles was analyzed using X-ray diffraction (XRD) technique. The XRD patterns of both samples are shown in **Figure 1b**, which show six typical diffraction peaks of Fe_3O_4 at 2θ angles around 30.27°, 35.23°, 43.22°, 53.71°, 57.43°, and 62.11°, respectively. These peaks correspond to the (220), (311), (400), (422), (511), and (440) crystal planes based on the Miller indices. The results of the diffraction pattern analysis and lattice parameters have been confirmed with reference data from the Crystallography Open Database (COD) with entry number 9006920 for the Fe_3O_4 phase. After further synthesis with variations in temperature and system, a simple organoleptic test was carried out to ensure initial identification.

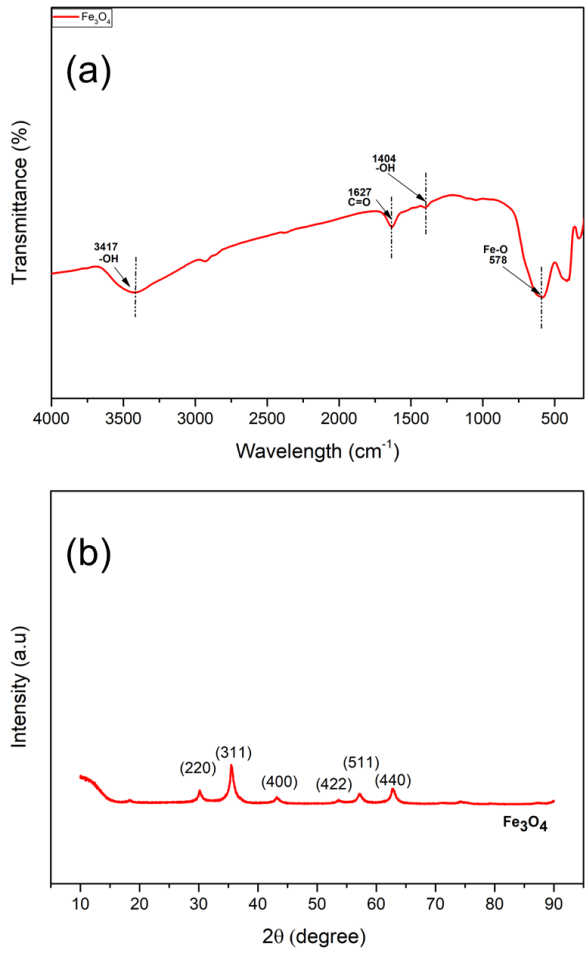

Figure 1. (a) Functional groups characterization and (b) diffractogram pattern of magnetite

The synthesized magnetite was characterized through an organoleptic test to assess its quality. According to Ngatijo [13], Fe_3O_4 is physically characterized by its distinctive black powder appearance. **Figure 2** shows a comparison of magnetite produced under four different conditions: (a) heating at 70°C in an open system, (b) heating at 70°C in a closed system, (c) heating at 90°C in an open system, and (d) heating at 90°C in a closed system.

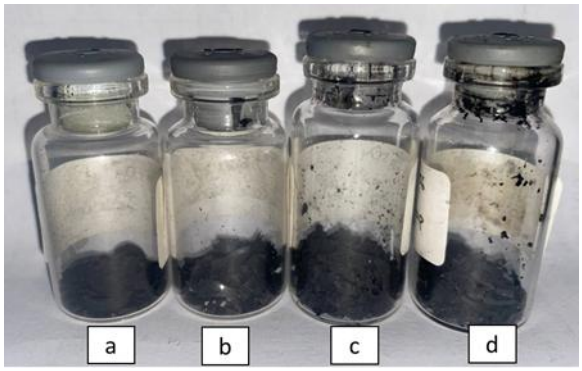

Figure 2. Visual characterization of Fe_3O_4 at (a) 70°C in an open system, (b) 70°C in a closed system, (c) 90°C in an open system, and (d) 90°C in a closed system

Observations showed that the magnetite produced at 90°C had a darker color compared to that synthesized at 70°C. This is because magnetite particles at higher temperatures have more time and energy to organize themselves into larger and more ordered crystals [11]. These more ordered crystals possess a greater number of active sites and energy pathways, allowing electrons to move more freely between the +2 and +3 oxidation states. The more electrons available to absorb photons across various wavelengths, the greater the light absorption, resulting in a deeper and darker black appearance.

In addition, the samples synthesized under closed conditions exhibited a darker color compared to those produced in open conditions. This is because, in a closed environment, there is no oxidation caused by exposure to atmospheric oxygen, resulting in a purer and more concentrated product. The denser and purer crystal structure of magnetite formed under these conditions enhances its light absorption capability, making the color appear significantly darker than when the reaction occurs in an open system with excess oxygen [12].

Table 1. Mass of Fe_3O_4

System	70 °C	90 °C
Opened (g)	1.633	2.066
Closed (g)	1.697	2.398

Based on theoretical calculations, the expected mass of magnetite is 1.665 g. At 70°C, the magnetite yield in the open system was 1.633 g (**Table 1**), while the closed system produced a slightly higher yield of 1.697 g. At 90°C, the yields were 2.006 g in the open system and 2.398 g in the closed system. The yields exceeding 100% indicate that the magnetite product was not yet pure, as it still contained ammonia and other contaminants that co-precipitated with the magnetite [14]. To remove these impurities, distilled water was added and the product was repeatedly washed until the pH of the solution reached neutral. This process helped eliminate residual ammonia and other unwanted substances. Additionally, allowing the suspension to

settle for 24 hours further improved the purification by enabling the magnetite particles to settle more completely while separating out impurities. Apart from purification through sedimentation, temperature also plays a crucial role in the synthesis of magnetite [15].

Higher temperatures increase the kinetic energy of molecules in the solution. This elevated kinetic energy accelerates the collision rate between Fe^{2+} and Fe^{3+} ions and hydroxide anions (OH^-) from the ammonia solution. More frequent and energetic collisions enhance the likelihood of chemical reactions that lead to the formation of Fe_3O_4. Furthermore, at 90°C, the rate of magnetite crystal formation increases as the activation energy of the reaction is more easily overcome, allowing particles to combine into crystalline structures with higher efficiency. These factors contribute to a higher yield of magnetite, as the reaction proceeds more completely and produces a greater amount of product. While temperature plays a critical role in the reaction, the reaction environment also significantly influences the synthesis outcome. In both open and closed systems, differences in oxygen exposure affect the magnetite formation process and the quality of the final product.

In a closed system, reduced contact with oxygen prevents the unwanted oxidation of Fe^{2+} to Fe^{3+}, allowing the formation of Fe_3O_4 to proceed more effectively and resulting in purer magnetite. This stable and controlled reaction environment maintains consistent temperature and pressure, preventing evaporation and fluctuations in solution concentration that could disrupt nucleation and crystal growth. As a result, the magnetite particles formed are more uniform and of higher quality, leading to an increase in product mass and overall yield. Therefore, the closed system supports higher yields by producing magnetite with optimal purity and particle size.

Based on **Figure 3**, the synthesized Fe_3O_4 composite exhibits strong magnetic properties, confirming the presence of magnetite (Fe_3O_4) in the material. This is evident from the strong magnetic attraction observed in all samples, especially under closed reaction conditions. The rapid attachment of the magnet to the material indicates that magnetite was successfully synthesized and possesses strong magnetic characteristics [16]. However, in Figures a and c, the magnetite particles tend to move away from the magnet, suggesting weaker magnetic attraction under open conditions. This indicates that the magnetite formed in open systems may have a less pure crystal structure or smaller particle size, resulting in reduced magnetic strength compared to that synthesized under closed conditions. The qualitative magnetism test shows that the sample heated at 90°C in a closed system exhibits the strongest magnetic response, indicating a higher degree of purity and more ideal particle size [11].

The magnetic test showed that all magnets were lifted perfectly. The mass addition test was conducted to determine the strength of the magnet simply. **Figure 3** presents the maximum weight that can be held by each magnet based on its magnetic properties.

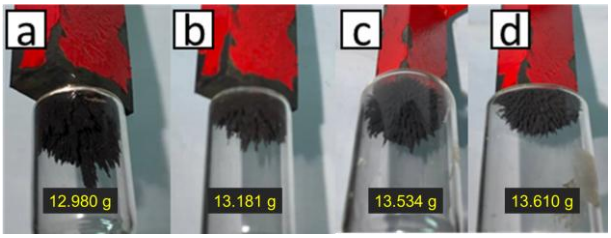

Figure 3. Magnetic attraction test of the sample using magnet

At 70°C, the maximum weight attracted to the magnet in an open system is 12,980 g, while the closed system yields a slightly higher result of 13,181 g. At 90°C, the results obtained are 13.34 g in the open system and 13,610 g in the closed system. Figures b and d show larger results because they used a closed system, while figures a and c show smaller results because they used an open system. This shows that a closed system produces stronger magnetic properties than an open system.

The next test involved the use of methylene blue as an adsorbate. Methylene blue was selected due to its well-defined molecular structure, strong chromophore group [17], and ability to interact with active surface sites through electrostatic and π–π interactions [18]. A total of 50 mg of each sample was shaken for 60 minutes in a shaker with methylene blue solution at concentrations of 2, 4, 6, 8, and 10 ppm to prepare a standard calibration curve. A 50 mg sample was used because it is the optimal amount to interact with the methylene blue solution without causing saturation or wastage of material. A 60 min shaking time was chosen to allow the adsorption process to reach equilibrium and produce stable data for the construction of a calibration curve.

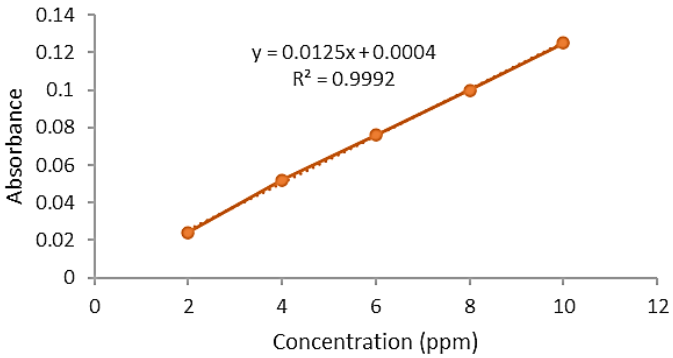

Figure 4. Standard curve of methylene blue adsorption

Based on the standard curve shown in **Figure 4**, the concentration of methylene blue after adsorption can be determined. The initial concentration of 10 ppm showed a noticeable decrease after the adsorption process. This reduction confirms the effectiveness of the magnetic material in adsorbing methylene blue. Decreasing in methylene blue concentration was observed after the addition of magnetite, indicating successful adsorption. At 70 °C, the closed system exhibited a lower final concentration (9.488 ppm) compared to the open

system (9.648 ppm), suggesting a higher adsorption efficiency. Similarly, at 90 °C, the closed system showed a more significant reduction in concentration (8.848 ppm) than the open system (9.808 ppm). These results indicate that both temperature and system conditions influence the adsorption performance, with the closed system consistently demonstrating better methylene blue removal efficiency. Using Equation (1), the amount of adsorbed methylene blue can be calculated that greater magnetic influence contributes to more efficient adsorption of methylene blue.

Table 2. Adsorption capabilities (Q)

System	70 °C	90 °C
Opened (mmol.g^{-1})	0.00055	0.0003
Closed (mmol.g^{-1})	0.0008	0.0018

4. CONCLUSION

Magnetite (Fe_3O_4) nanoparticles were successfully synthesized using the coprecipitation method with ammonia (NH_4OH) as the base agent. The study demonstrated that both temperature and environmental conditions (open vs. closed systems) significantly affect the quality, color, magnetic properties, and yield of the synthesized magnetite. Higher temperatures (90°C) enhanced particle crystallinity and increased yield due to accelerated nucleation and crystal growth. Furthermore, synthesis under closed conditions produced magnetite with a darker color, stronger magnetic response, and higher yield compared to open systems, attributed to reduced oxidation of Fe^{2+} and a more stable reaction environment. The results of the methylene blue adsorption test demonstrated that magnetite effectively reduced dye concentration, confirming its adsorption capability.The best result was obtained at 90°C in a closed system, which yielded magnetite with optimal purity, uniform particle size, and strong magnetic characteristics. The results of this study support the findings of Alibeigi and Vaezi (2008) who stated that magnetite formation is greatly influenced by the molar ratio between Fe^{2+} and Fe^{3+} ions. In open air conditions, Fe^{2+} ions are easily oxidized to Fe^{3+}, so that the ideal ratio of 1:2 is difficult to maintain and causes the magnetite phase to not form perfectly. These findings highlight the importance of temperature control and reaction environment in producing high-quality Fe_3O_4 for potential applications in magnetic materials.

SUPPROTING INFORMATION

There is no supporting information in this paper. The data supporting this research's findings are available on request from the corresponding author (R.Basuki).

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CONFLICT OF INTEREST

There was no conflict of interest in this study.

AUTHOR CONTRIBUTIONS

DDA & SN performed the conceptualization, investigation, methodology, writing original draft, review & editing. RB supervise the experiment, data calculations, and revise the manuscript. TOJT, TRY, LSA, BK, MRF, KSP, NVA, Nurwanto, and RH collaborated on writing and revising the manuscript. All authors approved the final version of the manuscript.

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