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Original Article

Synthesis of Fe_3O_4 using the Co-precipitation Method with Temperature and Time Treatment as Methylene Blue Adsorbent

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Abstract— Magnetite nanoparticles (Fe_3O_4) possess unique magnetic properties and are widely applied in various fields such as biomedical technology, environmental remediation, and material separation. This study reports the synthesis of Fe_3O_4 using the co-precipitation method under varying conditions of temperature, reaction time, and atmospheric exposure (open vs. closed system). Ferric and ferrous salts were reacted with ammonium hydroxide under controlled heating at 70°C and 80°C for 60 minutes. The synthesized materials were evaluated through visual color inspection, qualitative magnetic response, yield efficiency, and magnetic load-bearing capacity. The results showed that a closed system at 80°C produced the most optimal Fe_3O_4, indicated by a deep black color, strong magnetic attraction (149.86 mN), and a yield of 92.5%. Comparatively, open systems led to partial oxidation of Fe^2, resulting in less magnetic phases like maghemite or hematite. The findings confirm that controlling synthesis parameters, especially atmospheric exposure and temperature, significantly influences the purity, particle uniformity, and magnetic strength of Fe_3O_4 nanoparticles, highlighting the importance of optimized synthesis for practical applications.

Keywords— *Co-precipitation, Magnetite (Fe_3O_4), Magnetic properties, Nanoparticles, Synthesis parameters.*

1. INTRODUCTION

Magnetic nanoparticles, particularly magnetite (Fe_3O_4), have attracted significant attention over the past few decades due to their unique magnetic properties, good chemical stability, and wide-ranging applications in biomedical, environmental, and industrial fields. These include their use as adsorbents, in material separation, biochemistry, drug delivery, magnetic resonance imaging (MRI), and cancer therapy [1]. These black-colored particles with a cubic crystal structure exhibit ferromagnetic behavior, with a maximum magnetization value of 92 emu/g [2]. Fe_3O_4 is one of the most thermodynamically stable forms of iron oxide and exhibits superparamagnetic properties at the nanoscale [1]. The physical and chemical characteristics of Fe_3O_4 are strongly influenced by synthesis conditions, including the method used, solution pH, temperature, and reaction time [3]. Magnetite (Fe_3O_4) can be synthesized through various methods, including co-precipitation, electrochemical synthesis, hydrothermal methods, sol-gel processes, thermal decomposition, and microemulsion techniques [4]. Among these, the co-precipitation method is the most commonly used due to its simplicity, cost-effectiveness, and ability to produce large quantities of magnetite without the need for specialized equipment. This process involves the mixing of Fe^{2+} and Fe^{3+} ions in an alkaline medium under specific conditions to form Fe_3O_4 precipitates [3]. However, the characteristics of the resulting product are

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highly influenced by the synthesis conditions, such as the type and concentration of the base, reaction temperature, and reaction time. Variations in these parameters can affect the particle size, degree of crystallinity, phase homogeneity, and magnetic properties of the material [5].

A number of previous studies have reported the effects of individual synthesis parameters; however, few have systematically investigated the combined influence of base treatment, temperature, and reaction time within a single synthesis sequence. Additionally, significant variation still exists in the results, particularly in terms of phase purity, particle size distribution, and stability of the final product. This indicates a research gap in optimizing the co-precipitation method to produce Fe_3O_4 with more controlled characteristics. Therefore, this study systematically examines the effects of three parameters—base treatment, reaction temperature, and reaction time—on the crystal structure and morphology of synthesized Fe_3O_4, with the aim of determining optimal synthesis conditions for generating products with stable and controlled properties, suitable for further applications.

2. EXPERIMENTAL SECTION

2.1. Materials and Instrumentations

The chemicals used (99% purity by Merck) in this study including $FeCl_3 \cdot 6H_2O$, $FeSO_4 \cdot 7H_2O$, 25% NH_4OH, distilled water (aquadest), methylene blue, and aluminum foil. The equipment used includes a hotplate, oven, overhead stirrer, DC power supply, retort stand, clamps, beakers, shaker, thermometer, sudip, watch glass, measuring cylinder, magnetic stir bar, and vial bottles, cuvette, and UV-Vis spectrophotometer (Shimadzu UV-1800).

2.2. Synthesis of Fe_3O_4

A solution of Fe^{2+}/Fe^{3+} was prepared by dissolving 4 g of $FeCl_3 \cdot 6H_2O$ and 2 g of $FeSO_4 \cdot 7H_2O$ in 25 mL of distilled water in an Erlenmeyer flask. The solution was gradually heated to either 70°C or 80°C in an open flask, while being stirred at 300 rpm using an overhead stirrer. Once the desired temperature was reached, 25 mL of 25% ammonia solution was rapidly added. The mixture was stirred continuously at 70°C or 80°C for 1 hour. After the reaction time, the flask was removed from the heat and allowed to cool to room temperature. The resulting black sediment was separated using a magnet and washed with distilled water. The solid was then dried in an oven at 50°C until completely dry. The experiment was repeated with the Erlenmeyer flask covered with aluminum foil while stirring under the same conditions (300 rpm).

2.3. Magnetic Property Test

A qualitative magnetic test was conducted on the synthesized Fe_3O_4 powder. Approximately 0.5 g of the dried Fe_3O_4 powder was placed into a vial. An external magnet was then brought close to the bottom of the inverted and sealed vial. The movement of the powder toward the magnet or its adherence to the magnet-facing surface was observed. The amount of powder attracted or falling due to the magnetic field was also noted. A visible attraction or adhesion of the powder indicated that the material exhibited magnetic properties.

2.4. Adsorption Ability Test

A standard solution of methylene blue with concentrations of 2, 4, 6, 8, and 10 ppm has been prepared from from a 50 ppm methylene blue stock solution. The absorbance of all standard solutions by UV-Vis spectrophotometer has been performed at a wavelength of 660 nm to obtain a standard curve. Fifty mg of synthesized Fe_3O_4 was weighed into a vial and 10 mL of 10 ppm methylene blue solution was added. The mixture was stirred using a shaker for 60 minutes, then the Fe_3O_4 was separated using an external magnet. The filtrate was analyzed using a UV-Vis spectrophotometer at a wavelength of 660 nm. The amount of methylene blue adsorbed by Fe_3O_4 was analyzed using the concentration value from the standard curve equation.

3. RESULT AND DISCUSSION

3.1. Synthesis of Fe_3O_4

Magnetite (Fe_3O_4), with a chemical structure of $FeO–Fe_2O_3$, is a mixed iron oxide produced from the reaction between iron (II) and iron (III) oxides under basic conditions. This process results in a stronger magnetic property at the nanoscale [1]. The formation reaction of Fe_3O_4 is as follows [6] equation (1).

$$Fe^{2+}(aq) + 2Fe^{3+}(aq) + 8OH^{-}(aq) \rightarrow Fe_3O_4(s) + 4H_2O(aq) \quad (1)$$

Although many types of iron oxides have been identified, the term "iron oxide" generally refers to three main types: Fe_3O_4 (magnetite), α-Fe_2O_3 (hematite), and γ-Fe_2O_3 (maghemite). Among these, Fe_3O_4 has gained the most attention due to its exceptional magnetic properties [7].

The synthesis of Fe_3O_4 (magnetite) under controlled temperature and reaction time (**Figure 1**) plays a crucial role in determining particle size, crystallinity, and product purity. To evaluate the success of Fe_3O_4 synthesis, several simple characterizations were performed, including: (1) Color test, where successful Fe_3O_4 synthesis results in a deep black color, characteristic of magnetite; (2) Qualitative magnetic attraction test, to observe whether the particles are magnetic (Fe_3O_4) particles will be easily attracted to an external magnet), and (3) yield test, to calculate the percentage of actual product compared to the theoretical yield, indicating reaction efficiency. Successful synthesis of Fe_3O_4 is generally marked by the formation of a black precipitate attracted by a magnet and a high yield (typically above 70%),

indicating the reaction proceeds effectively and the product is close to the target.

Figure 1. Mixing process of $FeCl_3.6H_2O$ and $FeSO_4.7H_2O$

3.2. Color Test Results

The effect of atmospheric conditions during Fe_3O_4 synthesis on the physical properties of the material can be clearly observed through the color of the precipitate formed. Figure 1 shows color differences in Fe_3O_4 synthesized at 70°C for 60 minutes using 25% NH_4OH as a precipitating agent in two different conditions. Under open conditions (**Figure 2a**), the precipitate appeared dark brown to dull black, while under closed conditions (**Figure 2b**), the precipitate was deep black and homogeneous. This color variation indicates differences in iron oxide phase composition due to Fe^{2+} oxidation by atmospheric oxygen.

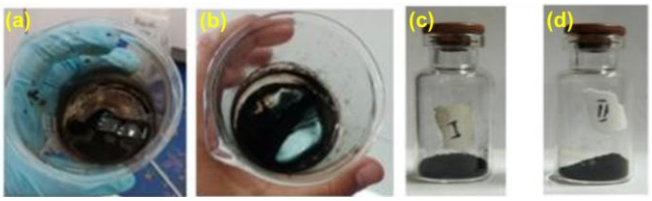

Figure 2. Color differences in magnetite synthesized at 70°C for 60 minu in open (a, c) and closed (b, d) condition

In Fe_3O_4 synthesis via the co-precipitation method, the reaction between Fe^{2+} and Fe^{3+} with OH^- ions forms a cubic spinel structure, where Fe^{3+} occupies both octahedral (B) and tetrahedral (A) sites, while Fe^{2+} only occupies octahedral (B) sites. The stability of Fe^{2+} in solution strongly depends on the system's redox conditions. In open conditions, the presence of atmospheric oxygen causes partial oxidation of Fe^{2+} to Fe^{3+} via the reaction in equation (2).

$$4Fe^{2+}(aq) + O_2\,(g) + 4H^+\,(aq) \rightarrow 4Fe^{3+}\,(aq) + 2H_2O\,(aq) \quad (2)$$

This process disturbs the required 1:2 (Fe^{2}:Fe^{3}) stoichiometric ratio for Fe_3O_4 formation and may lead to the formation of other phases like γ-Fe_2O_3 (maghemite) or α-Fe_2O_3 (hematite), depending on the pH and temperature. These phases typically appear reddish-brown or dark brown and have weaker magnetic properties due to the loss of Fe^{2+} magnetic contributions.

In contrast, under closed conditions, the reaction system is protected from atmospheric oxygen, keeping Fe^{2+} in its reduced state and allowing optimal participation in Fe_3O_4 formation. The resulting precipitate shows an intense black color typical of magnetite, indicating a homogeneous crystal structure and stable incorporation of Fe^{2+} in the spinel lattice. This deep black color is an indicator of successful Fe_3O_4 synthesis [8]. This finding aligns with Nguyen et al. (2021) [7], who reported that closed conditions produced Fe_3O_4 nanoparticles with an average size of 10–20 nm, high phase purity, and higher saturation magnetization (Ms) compared to products synthesized in open conditions.

When synthesizing Fe_3O_4 at 80°C for 60 minutes using the same base solution (25% NH_4OH) in both open and closed conditions, the open condition produced a dull black precipitate, while the closed condition yielded a deep black color. The increased synthesis temperature significantly affected the magnetite's color. Higher reaction temperatures accelerate Fe_3O_4 formation, as elevated temperatures support the endothermic citrate reduction reaction [9-11]. High temperatures (e.g., 80°C) speed up the co-precipitation process and promote the formation of purer, more crystalline Fe_3O_4 [12-13], resulting in a deep black precipitate. In contrast, at lower temperatures or non-optimal conditions, the reaction rate decreases, and Fe^{2+} stability is disrupted—especially in open systems exposed to oxygen—leading to the formation of other iron oxide phases like maghemite (γ-Fe_2O_3) or hematite (α-Fe_2O_3), which appear brown and exhibit weaker magnetic properties.

3.3. Qualitative Magnetic Attraction Test

Differences in magnetic strength during Fe_3O_4 synthesis using basic reagents such as NH_4OH (ammonium hydroxide) are greatly influenced by atmospheric conditions (open or closed systems). The study found that open conditions (**Figure 3a**) resulted in weaker magnetic properties than closed systems. This is due to several key factors, including particle size, size distribution, particle morphology, and final product purity. In open conditions, NH_3 vapor tends to diffuse into the environment, reducing its concentration in the reaction system. This limits the reduction of Fe^{3+} to Fe^{2+}, resulting in a less pure Fe_3O_4 phase and non-uniform particle sizes. Additionally, the open system allows air (containing O_2) to enter, possibly forming non-magnetic oxide phases like Fe_2O_3. In contrast, closed conditions (**Figure 3b**) preserve NH_3 concentration and limit oxygen intrusion, promoting more optimal reduction reactions. This favors the formation of purer, more homogeneous magnetite particles with smaller, narrower size distributions. All these factors contribute

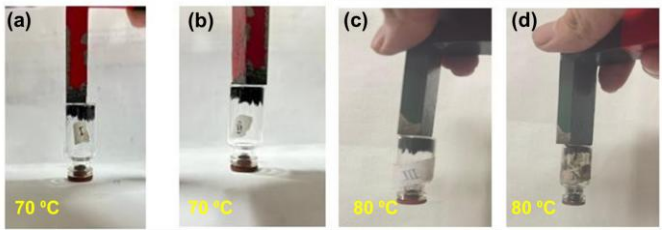

Figure 3. Magnetic strength differences at 70 and 80 °C for 60 min in open (a, c) and closed (b, d) condition

to increased magnetic moment and stronger ferromagnetic properties [14-15].

In open conditions, ammonia vapor (NH_3) easily evaporates, reducing base concentration in the solution. Consequently, the reduction of Fe^{3+} to Fe^{2+} is incomplete. Incoming oxygen can oxidize Fe^{2+} back to Fe^{3+}, forming non-magnetic compounds like Fe_2O_3. As a result, the synthesized material exhibits weaker magnetism, uneven particle sizes, and lower Fe_3O_4 purity. Conversely, in closed conditions, NH_3 vapor remains in the system, enabling stable and efficient reactions. Oxygen is prevented from entering, allowing purer Fe_3O_4 formation with smaller, more uniform particles. This condition supports the formation of materials with higher magnetic strength.

3.4. Yield Test Results

The mass of Fe_3O_4 obtained in the first repetition (open system) was 1.605 g with a theoretical mass of 1.665 g, resulting in a yield of 96.39% (**Figure 4**). In the second repetition (closed system), the mass was 1.457 g with a theoretical mass of 1.675 grams, yielding 86.98%. At 80°C for 60 min, the first repetition (open) yielded 1.612 g with a theoretical mass of 1.715 g (93.90% yield), while the second repetition (closed) gave 1.541 g with a theoretical mass of 1.6653 g (92.50% yield).

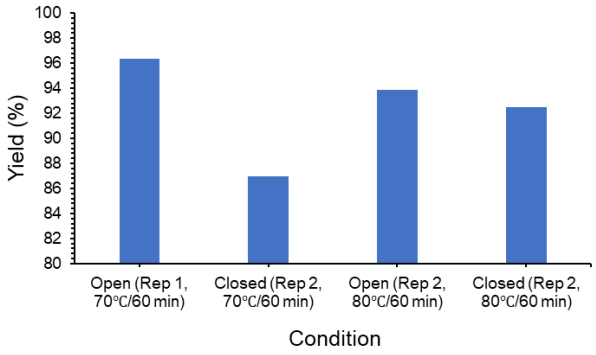

Figure 5. Yield of Fe_3O_4 under open vs closed system at different temperatures

3.5. Magnetic Load Test Results

The magnetic load test using synthesized magnetite particles aimed to determine the extent of magnetic attraction to an external magnet. In this test, as shown in **Figure 4**, magnetite particles were placed in a magnetic field and gradually loaded using paper clips with varying masses: 13.03 g (I), 14.873 g (II), 14.251 g (III), and 15.292 g (IV) until the maximum load caused the particles to detach. When the gravitational force exceeds the magnetic attraction, the particles fall off. Results showed that the higher the load mass, the greater the gravitational force the magnetic pull must

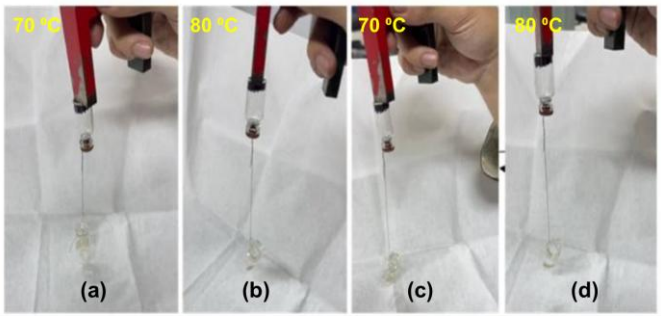

Figure 4. Difference in magnetite strength with load variation open (a,c) and close (b,d) condition at 70 and 80°C

resist. The magnetite with the highest magnetic strength was in order: IV, II, III, and I.

Gravitational force is calculated using F = m × g, where m is mass and g is gravitational acceleration (≈ 9.8 m/s²). Magnetite particles remain attached as long as the magnetic force is equal to or greater than the gravitational force. For example, if magnetite holds at mass m_1 but detaches at m_2 (with $m_2 > m_1$), the magnetic adhesion limit lies between these two forces. In test IV, magnetite particles remained attached up to a 15.292 g load, detaching when it exceeded the maximum load capacity. Hence, the magnetic adhesion strength lies between the gravitational forces of the tested masses.

Using the formula in (3) and (4), The magnetic force results obtained were 127.694 mN, 145.7554 mN, 139.6598 mN, and 149.8616 mN for the synthesized magnetite. Magnetic strength is influenced by several factors, including particle size and morphology, Fe_3O_4 content, external magnetic field strength, and surface conditions.

$$F = m \times g \quad (3)$$

$$F = 13.03\ g \times 9.8\ m/s^2 = 127.694\ mN \quad (4)$$

3.6. Adsorption Ability Test Results

Based on **Figure 5**, the standard curve of methylene blue (MB) obtained shows a coefficient of determination (R^2) value of 0.9992, indicating a very good linear relationship between concentration and absorbance. This standard curve serves as the calibration reference used to convert absorbance data from the adsorption experiments into MB concentration values. Thus, the MB concentrations reported in the following dicussion, including the final concentrations measured at different temperatures and system conditions were calculated based on this standard curve.

The adsorption test results using magnetite (Fe_3O_4) show a significant effect of temperature and system

conditions on adsorption capacity. At 70 °C for 60 minutes, open conditions resulted in a final MB concentration of 9.968 ppm, while closed conditions yielded a lower final concentration of 9.568 ppm. The decrease in concentration in the closed system indicates that the limited contact with the external environment makes the diffusion of MB molecules toward the magnetite surface more effective, thereby increasing the adsorption capacity [7].

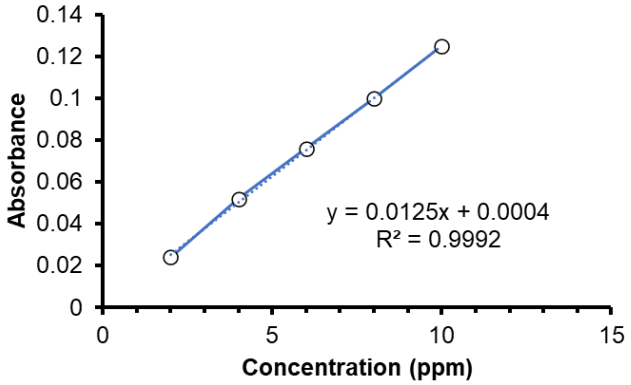

Figure 7. Methylene blue standard solution curve

At a higher temperatire of 80°C, a more pronounced decrease in MB concentration was observed. Under open conditions, the final MB concentration reached 9.728 ppm, while under closed conditions it decreased further to 8.208 ppm. The improved removal efficiency at elevated temperature is associated with the higher kinetic energy of MB molecules, which enhances their diffusion to the magnetite surface. The adsorption mechanism of MB onto Fe_3O_4 generally involves electrostatic attraction between the positively charged MB molecules and the negatively charged surface groups of magnetite, complemented by π–π interactions between the aromatic ring of B and surface hydroxyl groups. In addition, closed conditions likely reduce the escape or desorption of MB molecules due to minimal contact with ecternal air, thereby maintaining higher adsorption efficiency. Based on the interaction forces involved, the adsorption of MB into magnetite is predominantly classified as physisorption, although weak surface complexation may occur depending on pH and surface charge conditions. This behavior is consistent with adsorption thermodynamics, where increased temperature accelerates diffusion and facilitates faster approach to equilibrium [9].

4. CONCLUSION

Magnetite nanoparticles (Fe_3O_4) were successfully synthesized using the co-precipitation method by varying temperature, time, and reaction conditions. The optimal result was obtained in the 4th repetition (closed system, 80°C, 60 minutes), indicated by a deep black color, a magnetic force of 149.8616 mN, a yield of 92.5%, and has the largest adsorption capacity based on the final concentration of methylene blui, which is 8.208 ppm. This demonstrates that controlling synthesis parameters significantly affects the quality of the resulting magnetite.

SUPPROTING INFORMATION

There is no supporting information in this paper. The data that support the findings of this study 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

MFPK, APL, SN, WS, FOP, and AMK performed the conceptualization, investigation, methodology, writing original draft, review & editing. RB supervises the experiment, data calculation, and revise the manuscript. All authors approved the final version of the manuscript.

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