

## A Review: Optimum Conditions for Magnetite Synthesis ( $\text{Fe}_3\text{O}_4$ )

Dominikus Niholan Tukan<sup>1\*</sup>, Lilis Rosmainar Tambunan<sup>1</sup>, Kustomo<sup>2</sup>, Rasidah<sup>1</sup>

<sup>1</sup>Chemistry Department, Faculty of Mathematics and Natural Sciences, Palangka Raya University, Kampus UPR Tunjung Nyaho, Palangka Raya, 73111, Indonesia

<sup>2</sup>Department of Chemistry, Faculty of Science and Technology, Universitas Islam Negeri Walisongo Semarang, Semarang, Central Java 50185, Indonesia

Email: apdominikus@gmail.com

### ABSTRACT

Magnetite, chemically represented as  $\text{Fe}_3\text{O}_4$ , is an opaque substance characterized by its black coloration. It possesses a significant saturation magnetization value, denoting a substantial magnetic strength compared to alternative materials. Magnetite ( $\text{Fe}_3\text{O}_4$ ) has significant use across several domains, including its deployment as a catalyst and sensor in the medical sector, such as drug delivery, hyperthermia therapy, and magnetic resonance imaging (MRI). The synthesis of magnetite ( $\text{Fe}_3\text{O}_4$ ) can be achieved using several methodologies, such as solvothermal, sol-gel, solid state, reverse micelle, microwave plasma synthesis, freeze drying, ultrasound irradiation, hydrothermal, and coprecipitation techniques. The co-preparation technique is extensively employed for magnetite synthesis due to its simplicity, ease of usage, and ability to generate magnetic particles at the nanoscale scale without elevated temperatures. The outcome of the coprecipitation process is influenced by various factors such as the nature of the salt, molar ratio, pH level, stirring rate, and reaction temperature. This article aims to examine the impact of many factors, including the salt type, molar ratio, pH level, stirring rate, and reaction temperature, on the production of Magnetite ( $\text{Fe}_3\text{O}_4$ ) through coprecipitation. It was found that Magnetite ( $\text{Fe}_3\text{O}_4$ ) synthesis can produce nanoparticle size material with great magnetic power, namely at pH 8, temperature  $70^\circ\text{C}$ , and stirring rate of 10,000 rpm by using  $\text{FeCl}_3$  and  $\text{FeCl}_2$  as precursors  $\text{Fe}^{3+}$  and  $\text{Fe}^{2+}$  with a ratio of 1.5:1 and using precipitating base  $\text{NH}_4\text{OH}$ .

**Keywords:** Magnetite,  $\text{Fe}_3\text{O}_4$ , Coprecipitation.

### ABSTRAK

Magnetit ( $\text{Fe}_3\text{O}_4$ ) adalah salah satu material berwarna hitam dengan nilai magnetisasi saturasi yang sangat besar yang menandakan daya magnetik yang sangat besar jika di bandingkan dengan material lainnya. Magnetit ( $\text{Fe}_3\text{O}_4$ ) memiliki pengaplikasian yang sangat besar dalam berbagai bidang seperti katalis, sensor, dalam bidang medis seperti drug delivery, terapi hyperthermia, dan magnetic resonance imaging (MRI). Sintesis Magnetit ( $\text{Fe}_3\text{O}_4$ ) dapat dilakukan dengan beberapa metode diantaranya metode solvothermal, sol gel, solid state, reverse micelle, sintesis mikrowave plasma, freeze drying, ultrasound irradiation, hidrotermal, dan kopresipitasi. Kopretisipasi merupakan metode yang paling banyak digunakan dalam sintesis magnetite karena sederhana, mudah dan tidak membutuhkan temperatur yang tinggi namun dapat menghasilkan partikel magnetik dalam ukuran nanometer. Dalam proses kopresipitasi, jenis garam, rasio molar, pH, laju pengadukan dan suhu reaksi akan berpengaruh terhadap jenis produk yang dihasilkan. Artikel ini akan membahas tentang pengaruh dari jenis garam, rasio molar, pH, laju pengadukan dan suhu reaksi dari sintesis kopresipitasi Magnetite ( $\text{Fe}_3\text{O}_4$ ). Didapatkan sintesis Magnetit ( $\text{Fe}_3\text{O}_4$ ) dapat menghasilkan material ukuran nanopartikel dengan daya magnetik yang besar yaitu pada kondisi pH 8, suhu  $70^\circ\text{C}$ , laju pengadukan 10.000 rpm dengan penggunaan  $\text{FeCl}_3$  dan  $\text{FeCl}_2$  sebagai prekursor  $\text{Fe}^{3+}$  dan  $\text{Fe}^{2+}$  dengan perbandingan 1,5:1 dan menggunakan basa pengendap  $\text{NH}_4\text{OH}$ .

**Keywords:** Magnetit,  $\text{Fe}_3\text{O}_4$ , Kopresipitasi

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## 1. INTRODUCTION

Magnetite ( $\text{Fe}_3\text{O}_4$ ) is a type of black iron oxide with the highest magnetization value compared to other iron oxides at 92 emu/g (Agnestisia, 2017). Magnetite ( $\text{Fe}_3\text{O}_4$ ) exhibits a distinctive crystal structure compared to other iron oxides, characterized by the coexistence of two iron cations,  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$ , occupying tetrahedral and octahedral sites inside an inverted spinel group arrangement. This particular configuration imparts paramagnetic properties to magnetite ( $\text{Fe}_3\text{O}_4$ ) (X. Wang *et al.*, 2018). Changes in the particle size of magnetite ( $\text{Fe}_3\text{O}_4$ ) will affect its properties (Prasetyowati *et al.*, 2021).

Magnetite ( $\text{Fe}_3\text{O}_4$ ) has received significant attention in recent years due to its wide-ranging potential applications across diverse disciplines. Magnetite ( $\text{Fe}_3\text{O}_4$ ) finds extensive use in several industrial sectors, serving as a catalyst, sensor, and medium for data storage in hard drives and contributing to the production of color pigments. Magnetite, chemically represented as  $\text{Fe}_3\text{O}_4$ , finds extensive applications within the medical domain, particularly in drug delivery, hyperthermia therapy, and Magnetic Resonance Imaging (MRI). In the environmental field, magnetite ( $\text{Fe}_3\text{O}_4$ ) is extensively used as an adsorbent due to its high absorption capacity and ease of separation (Agnestisia, 2017).

Researchers have developed numerous  $\text{Fe}_3\text{O}_4$  materials for diverse applications. Maylani *et al.* (2016) utilized  $\text{Fe}_3\text{O}_4$  as an adsorbent for cadmium metal ions with an adsorption capacity of 16.0302 mg/g. Damasceno *et al.* (2020) utilized superparamagnetic magnetite nanoparticles ( $\text{Fe}_3\text{O}_4$ ) with an adsorption capacity of 17.45 mg/g. Zhang *et al.* (2020) utilized  $\text{Fe}_3\text{O}_4$  for the adsorption of Cr(VI) and Cu(II) metals with adsorption capacities of 8.67 mg/g and 18.61 mg/g. Hariani *et al.* (2013) used  $\text{Fe}_3\text{O}_4$  nanoparticles for the adsorption of Procion dye with an adsorption capacity of 30.503 mg/g. Agnestisia (2017) utilized magnetite ( $\text{Fe}_3\text{O}_4$ ) for the adsorption of methylene blue with an adsorption capacity of 43.86 mg/g, and Ngatijo *et al.* (2021) utilized magnetic nanoparticles coated with humic acid as methylene blue adsorption with an adsorption capacity of 56.96 mg/g.

The synthesis of  $\text{Fe}_3\text{O}_4$  nanoparticles is commonly achieved through a range of chemical-based techniques, including solvothermal methods (Yan *et al.*, 2008), sol-gel, solid state, reverse micelle, microwave plasma synthesis, freeze drying, ultrasound irradiation, hydrothermal, and coprecipitation (He *et al.*, 2007; Yazdani & Seddigh, 2016). Among the aforementioned approaches, coprecipitation is the most effective technique for synthesizing  $\text{Fe}_3\text{O}_4$  nanoparticles. This method is favored due to its ease and simplicity, as it requires no elevated temperatures. Notably, coprecipitation has demonstrated the capability to yield  $\text{Fe}_3\text{O}_4$  particles at the nanoscale level (Nalle *et al.*, 2019). Furthermore, the success rate of this process has been reported to range from 96% to 99.9% (Hariani *et al.*, 2013).

The coprecipitation technique is a synthesis process that involves the simultaneous precipitation of cations from a particular media, resulting in the formation of carbonate, hydroxide, citrate, or oxalate compounds. Coprecipitation is a fundamental chemical synthesis technique that is extensively employed for the production of inorganic materials due to its straightforward procedure and the favorable solubility of the resulting compounds in salts. The process of coprecipitation results in the formation of a solid precipitate, which can subsequently undergo calcination at a specific temperature to yield a powdered product (Sajidah, 2017). The outcome of the coprecipitation process is influenced by various factors, including the nature of the salt, molar ratio, pH level, stirring rate, and reaction temperature. This article aims to examine the impact of many factors, including the salt type, molar ratio, pH, stirring rate, and reaction temperature, on the coprecipitation synthesis of Magnetite ( $\text{Fe}_3\text{O}_4$ ).

## COPRECIPITATION OF $\text{Fe}_3\text{O}_4$

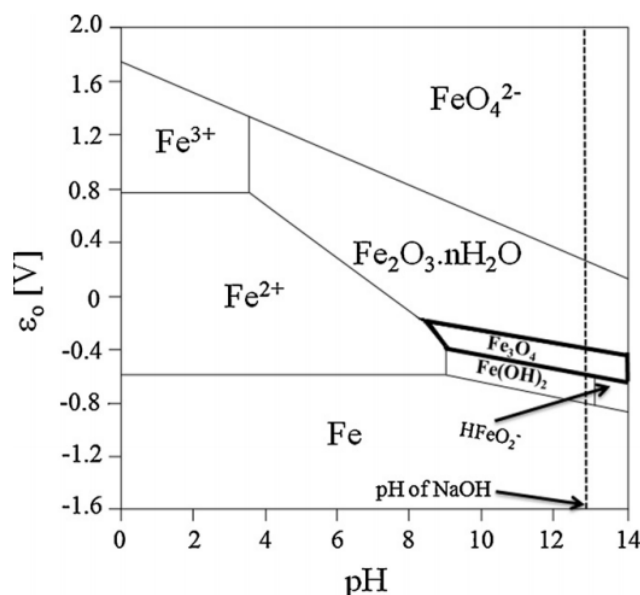
Iron oxide occurs in three forms in nature: magnetite ( $\text{Fe}_3\text{O}_4$ ), maghemite ( $-\text{Fe}_2\text{O}_3$ ), and hematite ( $-\text{Fe}_2\text{O}_3$ ). Magnetite ( $\text{Fe}_3\text{O}_4$ ) is commonly referred to as black iron oxide, magnetic iron ore, leadstone, ferrous ferrite, or Hercules stone. It exhibits the most pronounced magnetic characteristics compared to other transition metal oxides (Maylani *et al.*, 2016). Magnetite is extensively utilized in several facets of human life due to its cost-effectiveness, ease of processing, and many beneficial modification properties. The magnetic properties of magnetite

have garnered significant attention in the current study, mainly due to its superparamagnetic behavior observed when the particle size falls inside the nanometer scale, namely below 100 nm. This makes the study of magnetite synthesis to regulate the particle size obtained a very interesting study (Astuti & Hasanah, 2012).

Several techniques can be employed, including microemulsion, sol-gel, sonochemical reactions, hydrothermal reactions, hydrolysis and thermolysis, flow injection synthesis, electrospray synthesis, and coprecipitation (Laurent *et al.*, 2008). The coprecipitation approach is commonly employed for magnetite synthesis due to its simplicity, ease of usage, and ability to generate magnetic particles at nanoscale scales (Astuti & Hasanah, 2012). The coprecipitation approach typically involves the utilization of a stoichiometric combination of iron and iron salts in an aqueous medium, following a formation process as described by Laurent *et al.* (2008).



The formation of various types of iron oxide in an aqueous medium can be studied from an electrochemical point of view using Pourbaix diagrams. Pourbaix diagrams show the thermodynamic stability of various iron oxide and hydroxide phases.  $\text{Fe}_3\text{O}_4$  precipitates in highly alkaline aqueous media, namely at pH 8 to 14, where the resulting nanoparticles have a negative charge on their surface (El-Kharrag *et al.*, 2012).



**Figure 1.** Pourbaix diagram for the iron-water system showing thermodynamically stable phases over various pH ranges (El-Kharrag *et al.*, 2012).

The particle size and magnetic characteristics of  $\text{Fe}_3\text{O}_4$  formed during the coprecipitation process are influenced by various factors, including the type of salt employed, molar ratio, pH level, stirring rate, and temperature.

### EFFECT OF SALT TYPE ON COPRECIPITATION OF $\text{Fe}_3\text{O}_4$

In experiments conducted by Yazdani and Seddingh (2016), which compared the salts  $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$  and  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  as precursors of  $\text{Fe}^{2+}$  and  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ,  $\text{Fe}_2(\text{SO}_4)_3$  and  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  as precursors of  $\text{Fe}^{3+}$ , a comparison of magnetic properties was obtained and particle sizes as follows:

Table 1. Comparison of salt types in producing Fe<sub>3</sub>O<sub>4</sub> (Yazdani & Seddigh, 2016).

No	Precursor	Particle Size (nm)	Magnetization Saturation (emu/g)
1	FeCl <sub>2</sub> .4H <sub>2</sub> O and FeCl <sub>3</sub> .6H <sub>2</sub> O	11.92	53.38
2	FeCl <sub>2</sub> .4H <sub>2</sub> O and Fe <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub>	11.92	35.1
3	FeCl <sub>2</sub> .4H <sub>2</sub> O and Fe(NO <sub>3</sub> ) <sub>3</sub> .9H <sub>2</sub> O	11.92	51.5
4	FeSO <sub>4</sub> .7H <sub>2</sub> O and FeCl <sub>3</sub> .6H <sub>2</sub> O	11.92	51.2
5	FeSO <sub>4</sub> .7H <sub>2</sub> O and Fe <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub>	5.12	30.5
6	FeSO <sub>4</sub> .7H <sub>2</sub> O and Fe(NO <sub>3</sub> ) <sub>3</sub> .9H <sub>2</sub> O	5.12	43.5

Based on the findings presented in Table 1, it can be inferred that the utilization of FeCl<sub>2</sub>.4H<sub>2</sub>O and FeCl<sub>3</sub>.6H<sub>2</sub>O salts yields superior magnetic characteristics in comparison to alternative salt varieties, specifically measuring at 53.38 emu/g (Yazdani & Seddigh, 2016).

In the study conducted by Mascolo *et al.* (2013), a comparison was made between several precipitating base solutions, namely NaOH, KOH, and (C<sub>2</sub>H<sub>5</sub>)<sub>4</sub>NOH, in the coprecipitation process of Fe<sub>3</sub>O<sub>4</sub>. The findings indicate that the lattice parameter values and crystal structure of the Fe<sub>3</sub>O<sub>4</sub> formed were not influenced by the precipitating base solution. However, the precipitating base solution affected the size of the resultant crystals, with the ratio of (C<sub>2</sub>H<sub>5</sub>)<sub>4</sub>NOH < KOH < NaOH. Nevertheless, utilizing a strong base as a precipitating agent results in diminished crystallinity or crystal content compared to employing a weaker base like NH<sub>4</sub>OH. This is because the crystal formation occurs slowly (Kustomo, 2020).

Varying the concentration of precipitating base for coprecipitation synthesis of Fe<sub>3</sub>O<sub>4</sub> was carried out by Prasetyowati *et al.* (2021) by varying NH<sub>4</sub>OH by 10%, 15%, 20%, and 25%. An increase in NH<sub>4</sub>OH concentration leads to an increase in crystal size, accompanied by a corresponding reduction in magnetic characteristics. It is advisable to employ NH<sub>4</sub>OH with a concentration of 15% due to its ability to yield a more compact crystal lattice. The chemical composition of this solution comprises 49.33% Fe, 27.90% O, and the remaining portion consists of impurities.

### EFFECT OF MOLAR RATIO ON COPRECIPITATION OF Fe<sub>3</sub>O<sub>4</sub>

The synthesis of Fe<sub>3</sub>O<sub>4</sub> must be understood in the reaction process and control several parameters needed to obtain Fe<sub>3</sub>O<sub>4</sub> with the desired size, shape, and magnetic properties. The reaction to the formation of Fe<sub>3</sub>O<sub>4</sub> can be written as follows (Rahmayanti & Santosa, 2020):



This equation shows that Fe<sub>3</sub>O<sub>4</sub> can be synthesized using a mole ratio of 2:1 at pH 9-14. The 2:1 molar ratio is difficult to maintain in an oxygen-free environment because Fe(II) is easily oxidized to Fe(III). The presence of an excessive amount of Fe(III) might lead to the generation of hematite ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>) and goethite ( $\alpha$ -FeOOH), resulting in suboptimal synthesis of Fe<sub>3</sub>O<sub>4</sub> (El-Kharrag *et al.*, 2012).

Rahmayanti (2020) compared the molar ratio of Fe<sup>3+</sup> and Fe<sup>2+</sup>, namely 2:1, 1.75:1, 1.5:1, and 1.1:1. It was found that the molar ratio of 1.5:1 with the precipitating base NH<sub>4</sub>OH, at a temperature of 60°C and a reaction time of 60 minutes produced the highest yield compared to other ratios, namely 81% and a particle size of 9 nm.

### EFFECT OF pH ON COPRECIPITATION OF Fe<sub>3</sub>O<sub>4</sub>

In their study, B. Wang *et al.* (2013) modified the coprecipitation procedure to address some limitations, including agglomeration, inadequate morphology, and uneven particle size distribution. These modifications involved utilizing an organic mixture and kerosene as a stabilizing agent. This alteration has the capability to regulate the dimensions of the resultant

particles. They found that temperature and pH affected the size of the particles produced. At low pH values, there is an increase in  $\text{Fe}^{2+}$  ions, which causes a decrease in the oxidation ratio so that magnetite crystallites with a smaller size may form and precipitate. Mascolo *et al.* (2013) carried out the synthesis of  $\text{Fe}_3\text{O}_4$  in one plot at room temperature using a ratio of three precipitating bases, namely NaOH, KOH, and  $(\text{C}_2\text{H}_5)_4\text{NOH}$ , where the particle size ratio was  $\text{Na}^+ > \text{K}^+ > \text{N}(\text{C}_2\text{H}_5)^{+4}$ .

The use of NaOH, KOH, and LiOH as precipitating bases is confirmed with x-ray tests. Tests showed the presence of many nonmagnetic compounds, such as FeOOH, whereas using  $\text{NH}_4\text{OH}$  as the precipitating base, these compounds are almost non-existent (Gnanaprakash *et al.*, 2007). A pH comparison has been carried out to determine its effect on the size and magnetic properties of  $\text{Fe}_3\text{O}_4$ . Increasing pH did not have much effect on particle size, which only changed from 10 to 13 nm, accompanied by increasing pH from 8 to 12.5. Increasing the pH from 8 to 12.5 affects the magnetic properties produced, where the magnetic properties decrease, accompanied by an increase in pH from 72 emu to 44 emu (Ramadan *et al.*, 2012).

### EFFECT OF MIXING RATE ON COPRECIPITATION OF $\text{Fe}_3\text{O}_4$

In addition to factors such as concentration, the molar ratio of reactants, pH, and temperature, the stirring rate is also a crucial consideration in synthesizing  $\text{Fe}_3\text{O}_4$  to achieve the desired phase and particle size. To achieve a particle size of 10 nm, the stirring rate can be set at 10,000 rpm while maintaining a temperature of 20°C. A decrease in the stirring rate has been shown to result in the generation of bigger particle sizes. Conversely, an increase in the stirring rate up to 18,000 rpm has been found to induce the creation of goethite. According to Valenzuela *et al.* (2009), when the stirring rate was increased to 25,000 rpm, the system's temperature rose from 20°C to 37°C, forming a mixture consisting of nonmagnetic iron compounds.

### EFFECT OF TEMPERATURE ON COPRECIPITATION OF $\text{Fe}_3\text{O}_4$

The effect of temperature was observed on changes in magnetic properties, morphology, particle size, and particle distribution in the coprecipitation synthesis of  $\text{Fe}_3\text{O}_4$ . Niu & Zheng (2014) carried out a temperature comparison of particle size and magnetic properties of  $\text{Fe}_3\text{O}_4$  coprecipitation synthesis from 60, 70, 80, and 90°C. The results showed that increasing temperature caused an increase in particle size, namely from 32 nm to 60 nm. Meanwhile, the resulting magnetic properties vary. At a temperature of 60°C, the resulting magnetic properties are 45.6 emu/g. At 70°C, it has magnetic properties of 58.9 emu/g. At 80°C, it produces magnetic properties of 51.8 emu/g, and at a temperature of 90°C decreases to 50.0. The recommended temperature for the coprecipitation method is 70°C, which can produce a particle size of 47 nm and magnetic properties of 58.9 emu/g.

### CONCLUSION

The coprecipitation method is the most effective for  $\text{Fe}_3\text{O}_4$  synthesis because the process is easy and straightforward, does not require high temperatures, and can produce  $\text{Fe}_3\text{O}_4$  in nanoparticle size. Several factors influence particle size and magnetic properties in  $\text{Fe}_3\text{O}_4$  coprecipitation synthesis, such as salt type, molar ratio, pH, stirring rate, and temperature.  $\text{FeCl}_2$  as a precursor to  $\text{Fe}^{2+}$  and  $\text{FeCl}_3$  as a precursor to  $\text{Fe}^{3+}$  is believed to produce  $\text{Fe}_3\text{O}_4$  with the highest magnetic properties compared to other types of salt. A molar ratio 1.5:1 in an oxygen-free environment is recommended in the coprecipitation synthesis of  $\text{Fe}_3\text{O}_4$  because it produces the largest yield compared to other molar ratios.  $\text{NH}_4\text{OH}$  can be used in this synthesis because it can reduce the formation of nonmagnetic compounds such as FeOOH. Increasing the pH can also reduce the magnetic properties produced, so the recommended effective pH is pH 8. The rate of stirring and temperature also influence the particle size and magnetic properties produced. The recommended stirring rate to obtain a particle size of 10 nm is 10,000 rpm, while the recommended temperature is 70°C.

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