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# Synthesis of Magnetite Nanoparticles via Co-precipitation Method

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**Abstract.** Magnetite (Fe<sub>3</sub>O<sub>4</sub>) nanoparticles becomes a new innovation that gets attention of biomedicine scientists. Magnetite can be applied to cancer treatment as a drug carrier because it's good biocompatibility and very low toxicity. The purpose of this study was to determine the effect of temperature and stirring rate on the magnetite particle characteristics prepared by co-precipitation method. Firstly, FeCl<sub>3</sub> and FeCl<sub>2</sub> with 2:1 mole ratio were reacted with 10% NH<sub>4</sub>OH at 40 - 80°C temperatures and stirring speed of 300 - 500 rpm in a beaker glass. Subsequently, the precipitate was separated using filter paper and it dried into air oven at 100°C. The properties of obtained magnetite powder were determined using XRD and SEM. From XRD pattern indicates that magnetite formed at all temperatures with crystallite diameter in the range of 10-12 nm. The SEM results indicate the agglomeration of the magnetite particles with size in the range of 2.7 to 3.3 μm. In the other hand, the higher of temperature and stirring rate of agitation will make the agglomeration of the particles become more uniform. The increasing of temperature and the stirring rate will increase the magnetite crystallinity level.

**Keywords:** drug delivery, co-precipitation, magnetite

## 1. Introduction

Nanoparticle technology become a new tren for biomedical science and used widely in various fields. One of the applications of nanoparticles in the medical field is drug delivery system that can be applied for cancer treatment. Nano drug delivery is a method of delivering drug compounds to the therapeutic target. The method coined by Paul Ehrlich, who was awarded a noble prize of medicine in 1908, principally conjugate drugs to nanoparticles as drug carriers which are injected into veins [1].

In the application, nano drug delivery is controlled with an external magnetic field to the target cell. Nano drug delivery also aims to reduce the potential for overdose, as well as to keep the drug concentration within the prescribed time frame [2].

Nanoparticles as drug delivery should have specific characteristics to achieve the desired target, in other words have a suitable combination of sizes, drug conjugation with nanoparticles, surface chemistry, hydrophobicity, surface functionalization, biodegradability, and response physical properties i.e temperature, pH, magnetism and so on. Among these factors, size is the most critical trait. Particular



particle size can only access tissues or specific cells in the body and related to clearance in the blood circulation. Long-term circulation in the blood is important in drug delivery systems and drug release. In addition, the uniform distribution of particle size is also worth noting. Particles with a wide size distribution and a non-uniform shape can cause heterogeneous pharmacological effects [3]. Therefore, nanoparticles with controlled sizes and morphology are required. Because it's become a critical factor in drug delivery systems. The size and morphology of drug delivery particles influences its accessibility and residence time in blood circulation [4].

The material currently being developed as drug delivery is magnetite [5]. Magnetite nanoparticles ( $\text{Fe}_3\text{O}_4$ ) are attractive materials and have a very wide application. In the spinel group, the magnetite material ( $\text{Fe}_3\text{O}_4$ ) is a type of iron oxide mineral phase, in addition to maghemite ( $\gamma\text{-Fe}_2\text{O}_3$ ) and hematite ( $\alpha\text{Fe}_2\text{O}_3$ ). Magnetite nanoparticles (nanomagnetite) is one of the materials that gets a lot of scientific attention because of their advantages, such as very low toxicity and good biocompatibility [6], then low coercivity, high saturation magnetization, and low Curie temperature [7].

In biomedical fields, nanomagnetite was used in applications such as magnetic resonance imaging (MRI), cell separation, heat mediators for hyperthermia, biosensing, and drug delivery for cancer therapy [8].

The synthesis of nanomagnetite ( $\text{Fe}_3\text{O}_4$ ) had been widely done using various methods, such as co-precipitation [9], hydrothermal [10], thermal decomposition [11] and the sol-gel technique [12] and in this research we will synthesis the magnetite nanoparticles via co-precipitation method. In the process of synthesis we have to know the effect of temperature and stirring rate of agitation on the magnetite particle characteristics, and the particle size of the magnetite.

## 2. Materials and methods

### 2.1. Magnetite Synthesis

Magnetite nanoparticles were synthesized via co-precipitation method. The synthesis process is carried out as follows:  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  (Merck, Germany), and  $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$  (Merck, Germany), with 2:1 mole ratio were each dissolved in 100 mL aquades, then the two solutions were mixed into a 500 mL beaker glass and then heated until various temperature were achieved for 10 min while stirring. After heating, 20 mL  $\text{NH}_4\text{OH}$  10% (Merck, Germany) was gradually added while the solution was being vigorously stirred at 300, 400, and 500 rpm using a magnetic stirrer until the pH 10. Then continuously heating until 30 minutes at temperature of 40°C, 60°C, and 80°C. Further liquid and precipitate separated using filter paper and rinsed using aquades. The precipitate was dried into air oven at 100°C through 150 minutes.

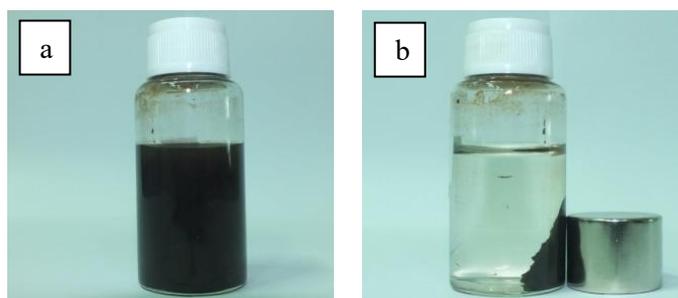
### 2.2. Characterization of Magnetite

The crystallinity of the obtained magnetite was analyzed by X-ray Diffraction (XRD) X'PERT POWDER PW 30/40. Particles morphology was examined using SEM-EDX JEOL JSM-6510 LV analysis was performed to study the elemental analysis. The particle crystal size is determined by using the Scherrer equation.

$$\beta = \frac{K\lambda}{D \cdot \cos \theta} = \frac{K\lambda}{D} \cdot \frac{1}{\cos \theta}$$

## 3. Results and discussion

The magnetite crystals ( $\text{Fe}_3\text{O}_4$ ) consist of  $\text{Fe}^{3+}$  and  $\text{Fe}^{2+}$  with a ratio 2:1. In this study  $\text{FeCl}_3$  used as  $\text{Fe}^{3+}$  and  $\text{FeCl}_2$  as  $\text{Fe}^{2+}$  source using co-precipitation method.  $\text{NH}_4\text{OH}$  10% used as the settling agent. The formation of magnetite was characterized by thick black precipitate that can interact with external magnetic fields as seen in Figure 1.



**Figure 1.** (a) Magnetite dispersed (b) Magnetite attached to the external magnet.

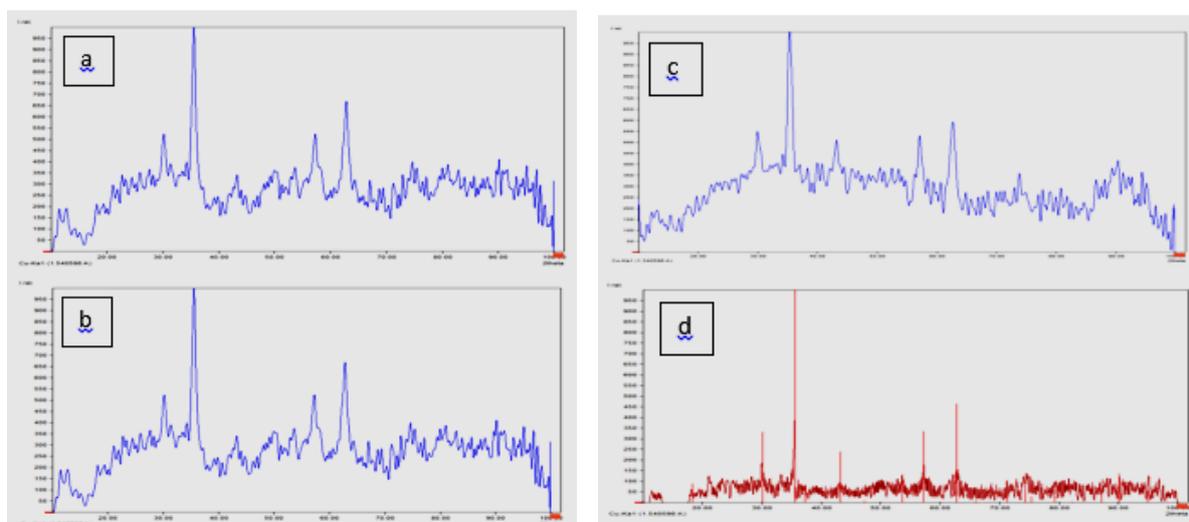
Magnetite has a black pigment [13]. So, it called as black iron oxide. But iron oxides like hematite, maghemite and geotite have red, brown and yellow pigment. Based on the synthetic solution shown in Fig. 1, it shows the characteristic of magnetite nanoparticles ( $\text{Fe}_3\text{O}_4$ ) that has black particles.

Magnetite powder that produced from the synthesis has magnetite characteristics, because when closer to the external magnet, magnetite attached to the external magnet. Figure 1.a is the condition of the magnetite solution before being brought close to the external magnet. And perfectly attracted by the magnet within 86 seconds as shown in Figure 1.b.

The magnetic properties of the precipitate appear when brought closer to the external magnet and then disappear when the magnetic field is kept away. It indicates that the magnetite sample has a paramagnetical property, i.e. a material containing a magnetic moment and can be pulled strongly by a magnet.

### 3.1. Crystalline and Size Analysis on Magnetite Synthesis Product

The analysis used to find the crystallinity level of this magnetite synthesis product is done by using XRD (X-Ray Diffraction) analysis. The XRD analysis was performed to analyze the composition or chemical compound contained in the sample and the characterization of the crystals. XRD analysis results are diffraction peak data showing the number of crystals and crystal size of the compounds contained in the sample. The higher of diffraction peak indicated the higher number of crystal compounds in the sample. While the width of the diffraction peak indicated the smaller size of the crystalline compounds in the sample.



**Figure 2.** XRD pattern of as-synthesized sample at (a) 300 rpm 80°C (b) 500 rpm 80°C (c) 500 rpm 40°C (d) ICDD (01-075-0449).

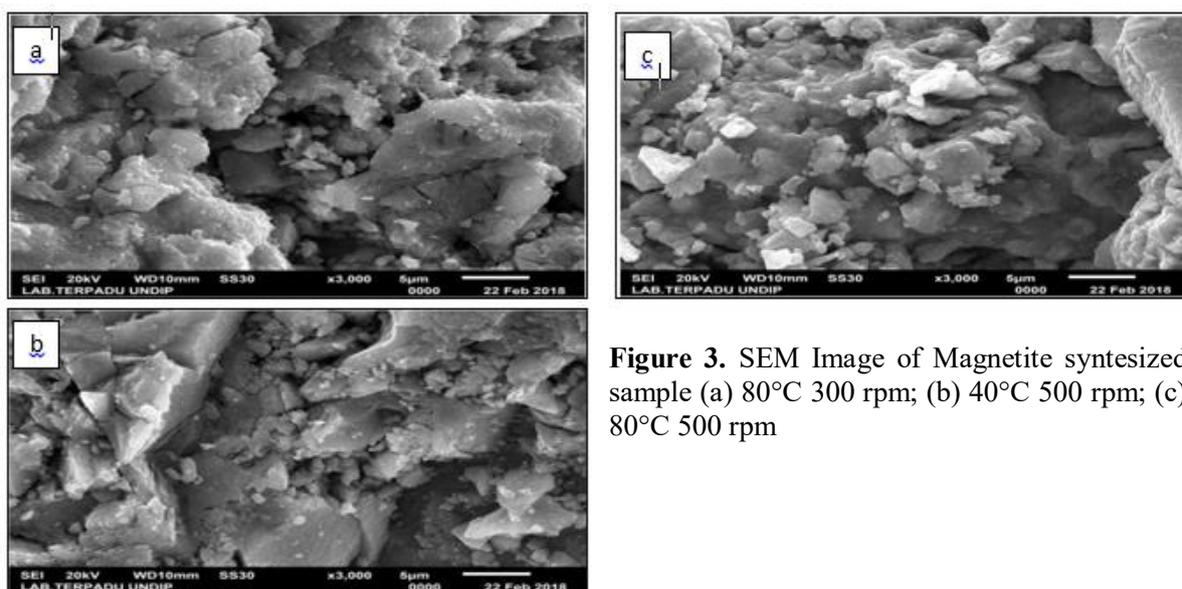
Figure 2.d shows the standard peaks of magnetite. The standard peaks of International Center for Diffraction Data (ICDD) data are (220), (311), 400, 511, 440 with angle  $2\theta$  ie 30.28; 35.67; 43.36; 57.36; and 62.99. XRD analysis can determine the effect of temperature and stirring rate on chemical compounds contained in the resulting sample.

In Figure 2. it appears that there are five typical magnetite peaks corresponding between the  $2\theta$  angles from  $30^\circ$ - $62^\circ$ . These peaks correspond to the diffractogram of the standard magnetite (01-075-0449). At 300 rpm and  $80^\circ\text{C}$  (Figure 2.a) has the highest intensity at  $2\theta$  30.19;  $35.60^\circ$  and  $63.01^\circ$ . 500 rpm  $80^\circ\text{C}$  (Figure 2.c) has an intensity at  $2\theta$  30.27;  $35.26^\circ$ ; and  $62.85^\circ$ . But at the lower temperature ( $40^\circ\text{C}$ ) has a peak at  $2\theta$  angle which is not different at  $2\theta$  30.17;  $35.62^\circ$  and  $62.85^\circ$  (Fig. 2.b). This shows that magnetite crystals have been formed on the synthesis.

The magnetite particles produce at 500 rpm stirring rate with temperatures  $40^\circ\text{C}$  and  $80^\circ\text{C}$  were 11 nm and 10 nm with the cubic crystals that indicate characteristic of magnetite ( $\text{Fe}_3\text{O}_4$ ) [14]. However, if the stirring rate is lowered at 300 rpm, the crystal size is to 12 nm. This shows that the stirring rate and temperature affects the size of the magnetite crystals. The size of the magnetite crystals obtained is suitable as drug delivery. The magnetite crystal size which can be used as drug delivery should be 10 - 200 nm [15].

### 3.2. SEM Analysis

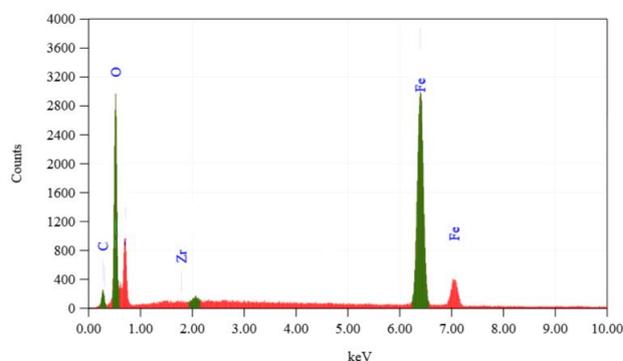
The morphological analysis of the synthesized sample is shown in Figure 3 with 3000x magnification.



**Figure 3.** SEM Image of Magnetite synthesized sample (a)  $80^\circ\text{C}$  300 rpm; (b)  $40^\circ\text{C}$  500 rpm; (c)  $80^\circ\text{C}$  500 rpm

The morphological analysis of the synthesized sample is shown in Figure 3 with 3000x magnification. The SEM results indicate there is an agglomeration the magnetite product and the uneven amount of particles. The effect of stirring rate on magnetic morphology is seen in Figure 3.a and Figure 3.c. From Figs 3.a and 3.c, the increase in stirring rate makes the clumps of magnetite particles smaller and irregularly shaped.

Increased temperatures also affect the morphology of magnetite nanoparticles. It can be seen in Figure 3.b and 3.c. Increased temperatures makes the magnetite particle size more uniform but there are still agglomerated. Measurement of particle size from SEM test image obtained magnetite particles size 2.7 to 3.3  $\mu\text{m}$ . Agglomeration in magnetite synthesis is very difficult to avoid, because of its magnetism [16]. In addition, the agglomeration between particles can be caused by incomplete sample preparation. The higher temperature and speed rate also affect for magnetite crystallinity level. This is evident from the results of SEM-EDX test which can be seen in Table 1.



**Figure 4.** Spectrum EDX of magnetite prepared at 80°C and 500 rpm.

**Table 1.** Analysis of magnetite components

Variable	Component	Composition (%)
80°C; 300 rpm	Carbon, C	10,64
	Chloride, Cl	9,23
	Magnetite	69,75
40°C; 500 rpm	Carbon, C	7,68
	Chloride, Cl	0,37
	Magnetite	81,95
80°C; 500 rpm	Carbon, C	12,81
	Chloride, Cl	1,33
	Magnetite	85,87

EDX analysis at the point on the sample only give output in the form of a percentage of the elements. The EDX analysis performed to ensure that the compound formed in the sample was  $\text{Fe}_3\text{O}_4$  based on the results of Fe and O elements in all samples. So it can be indicated that there are constituent elements of a magnetite. Figure 4 shows the EDX spectrum of the magnetite sample. The graph in the figure is the peak of 6.398 which is the Fe element, at 0.277 indicating the presence of element O and Cl element seen slightly at 2.042.

#### 4. Conclusion

This research give the conclusion that magnetite can formed via co-precipitation method. All Various temperatures make crystallite diameter in the range of 10-12 nm. The SEM results indicate the agglomeration of the magnetite particles with size in the range of 2.7 to 3.3  $\mu\text{m}$ . In the other hand, the higher of temperature and stirring rate of agitation will make the agglomeration of the particles become more uniform. The increasing of temperature and the stirring rate will increase the magnetite crystallinity level.

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