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# Application of Sol-gel Method and Co-Precipitation in the Material Synthesis Process of Magnetite Fe3O4 Nanoparticles

**Maulinda[1*], TM Zulfikar[2], Ismet[3], Vera Viena [4], Elvitriana[5]**

[1,3,4]Department of Environmental Engineering, Universitas Serambi Mekkah, Aceh, Indonesia
[2]Department of Industry Engineering, Universitas Serambi Mekkah, Aceh, Indonesia
[5]Department of Chemical Engineering, Universitas Serambi Mekkah, Aceh, Indonesia

** Corresponding E-mail:* maulinda@serambimekkah.ac.id

**Abstract.** *The development of lithium batteries is one type of research that continues to be developed. Recently, lithium batteries have become the most widely used source of electrical energy as raw materials for the manufacture of lithium batteries. This study aims to compare the synthesis with Co precipitation and sol-gel methods as electrodes in lithium batteries. Nanosized particles were characterized for characterization using (X-ray fluorescence (XRF) and scanning electron microscopy-energy dispersive X-ray (XRD) and (SEM) -EDS). Based on the XRF characterization test, the results showed that the chemical composition of Magnetite Fe3O4 contained in the iron sands of the Syiah Kuala beach showed material purities of 87.10%, 86.73%, and 81.42% (Magnetite Separation). This shows that the synthesis of Fe3O4 using the sol-gel method yields results with better material purity than the coprecipitation method. The results of the SEM-EDS characterization test observations were based on particle analysis and the distribution observed on the surface morphology of the particles, where the sol-gel method provided precise results for the synthesis of Fe3O4 magnetite nanoparticles. The results of the characterization test based on the EDS analysis based on the EDS test obtained the amount of Fe3O4 of 70.00%, this indicates that the composition of iron provides a high value compared to other components contained in it.*

**Keywords:** *magnetite Fe3O4, nanoparticles, iron sand, sol-gel method, lithium battery*

## 1. Introduction

A battery is an electrochemical cell that converts from chemical energy to electrical energy. One type of battery that is currently developing is the Lithium-Ion Battery or lithium ion battery. The main parts that make up a Lithium-Ion Battery are the negative electrode (anode), positive electrode (cathode), electrolyte and separator (Girishkumar et al., 2010). The use of Lithium Ion batteries has expanded from portable electronics to large-scale applications, particularly for electric vehicles and storage units from renewable energy sources. The use of battery-based vehicles is expected to reduce gas emissions (CO2) produced so far by oil-burning vehicles. But in addition to having many advantages, lithium batteries have disadvantages, including a relatively expensive price, relative Life Time brief, and destroyed if completely exhausted, as well as environmental problems. This weakness prompted researchers to develop an even better Lithium battery electrode material (Darvina, 2018).

One factor that can affect battery performance is the quality of the electrodes. Examples are electrode peeling which will be the main cause of battery damage, the lifetime is relatively short, and destroyed if it really runs out, and other drawbacks are the relatively expensive battery price (Zhou, 2014). Based on research that has been done previously on the use of Graphen Composite Fabrication as Lithium Ion Electricity by characterizing its Electrical Conductivity, from the results of the study obtained a high Electrical Conductivity value and has a good structure (Ahmad et al., 2019). Because the

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price of Graphen from lithium batteries is very expensive, the solution is to change the type of battery, or by making batteries at a relatively cheap price. One of the candidates to replace graphite to make Lithium battery electrodes is a material from natural materials, namely Iron Oxide (Fe3O4) nanocomposites originating from the Syiah coast of Kuala Aceh Province. Characterization of materials into nano is very necessary so that the needs of Fe3O4 in the low-cost modern technology industry can be met. Recently, the use of functional materials has been significant, as functional materials are in demand in modern technology and at the forefront of materials research (Susana & Astuti, 2016). Applications of these functional materials include electrical, dielectric, electromagnetic, optical, and magnetic applications.

*Magnetite $Fe_3O_4$*

Magnetite is one of the iron oxides that has the chemical formula Fe3O4 or often written in the form (FeO.Fe2O3). Magnetite adopts an inverted spinel build, where half the number of Fe3+ ions occupies the tetrahedron cavity and the other half occupies the octahedron cavity and all Fe2+ ions occupy the octahedron cavity of an O2- ion face-tight cube (fcc) order (Sugiyarto, 2003). Figure 1 represent the crystal shape and physical and magnetic properties of $Fe_3O_4$.

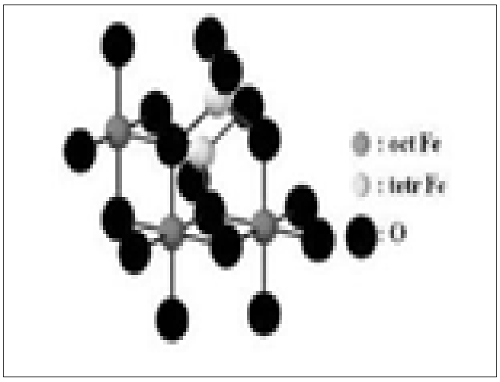


**Figure 1.** Crystal Structure $Fe_3O_4$

## 2. Method

Sample Purification Stage The iron sand samples used are samples that have been taken on the Syiah coast of Kuala Aceh Province. Samples that have been taken, purified with permanent magnets, then washed with aquabidest, mashed, filtered with a 100 mesh sieve and dried, pulled with permanent magnets 10 times, then tested using XRF, SEM and XRD.

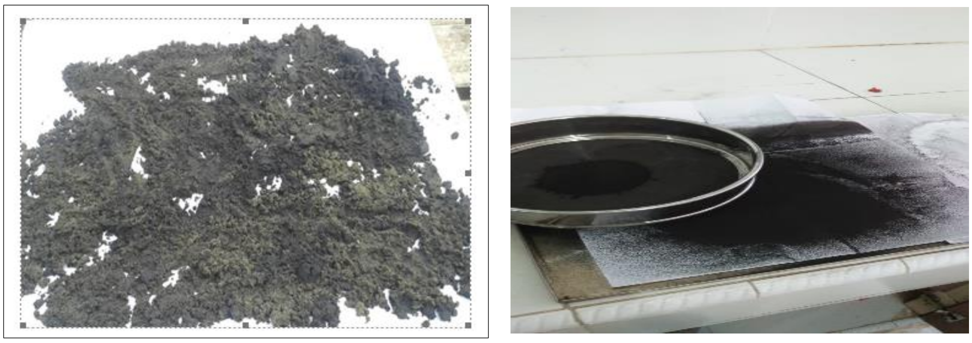

**Figure 2.** Fe3O4 material drying and drying process

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*Synthesis of Magnetite Nanoparticles (Fe3O4) by Coprecipitation Method*

The coprecipitation or precipitation method is carried out by adding a precipitating solution to the pH of the solution containing supporting material precursors and catalysts, so that metal hydroxide species are formed which will react with the surface hydroxyl group followed by the release of water molecules. This method is generally used to obtain a very uniform active phase distribution. The reaction of Fe3O4 formation is coprecipitated as follows:

$$Fe3+ + 3OH \longrightarrow Fe(OH)_3 \quad (1)$$

$$Fe(OH)_3 \longrightarrow FeOOH + H_2O \quad (2)$$

$$Fe_2 + 2OH \longrightarrow Fe(OH)_2 \quad (3)$$

$$2FeOOH + Fe(OH)_2 \longrightarrow Fe_3O_4 + 2H_2O \quad (4)$$

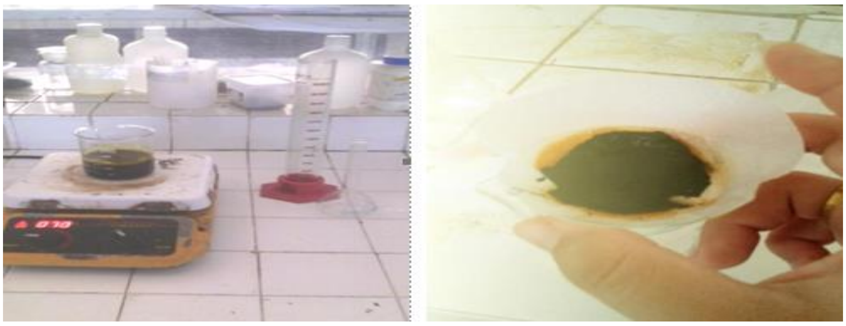

**Figure 3.** Magnetite Fe3O4 synthesis process coprecipitation method

*Synthesis of Magnetite Nanoparticles ($Fe_3O_4$ NPs) by Sol-Gel Method*

The synthesis of $Fe_3O_4$ magnetite nanoparticles is carried out by sol-gel method. Magnetite $Fe_3O_4$ and citric acid (C6H8O7) in a ratio of 1:2 dissolved in 100 mL aquadest. The mixing of these ingredients is done by dissolving completely with a magnetic stirrer at a temperature of 30-35$^0$C (room temperature) for 60 minutes. During the stirring process, a 0.5 N ammonium hydroxide ($NH_4OH$) solution is added drop by drop with the aim of adjusting the pH of the solution to neutral conditions (7.0-7.3). The reaction process is carried out for 4 hours at a temperature of 70$^0$C. Furthermore, the evaporation process is carried out until a viscous solution is obtained at a temperature of 95$^0$C which is referred to as sol. When a substance such as a sole is formed, it is poured into a porcelain dish for drying to obtain gel particles. The drying process is carried out in the Furnace at a temperature of 600$^0$C and the black product obtained is stored at room temperature. The product is washed with methanol and Aquadest.


**Figure 4.** Synthesis process of magnetite Fe3O4 nanoparticles using the sol-gel method

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## 3. Results and Discussions

*Results of X-Ray Fluorescence (XRF)*

Analysis The results of iron sand composition analysis after synthesis by sol-gel method are then characterized to determine the chemical composition of iron sand content. Characterization of the composition was performed using X-Ray Fluorescence (XRF) analysis. Based on the results of the composition characterization analysis, the content of magnetite Fe3O4 in iron sand was 87.10% (Sol Gel), 86.73% (Coprecipitation) and 81.42% (Magnetite Stirrer) The chemical composition of synthesized iron sand as shown in Table 1.

**Table 1.** Comparison of Fe3O4 synthesis

| No | Compound | Sol-Gel (%) * | Co-Precipitation** | Magnetite Separator |
|---|---|---|---|---|
| 1 | $Fe_3O_4$ | 87.10 | 86.73 | 81.42 |
| 2 | $TiO_2$ | 6.56 | 8.93 | 7.61 |
| 3 | $Al_2O_3$ | 2.01 | 0.00 | 3.00 |
| 4 | $SiO_2$ | 1.15 | 2.50 | 4.40 |
| 5 | MgO | 1.17 | 0.00 | 0.00 |
| 6 | CaO | 0.30 | 0.72 | 1.04 |
| 7 | $Cr_2O_3$ | 0.52 | 0.22 | 0.19 |
| 8 | $P_2O_5$ | 0.12 | 0.34 | 0.41 |
| 9 | $K_2O$ | 0.03 | 0.08 | 0.11 |

Fe3O4 magnetite synthesis is carried out by comparing synthesis methods, where in this study the method used is the sol-gel method, then compared with the coprecipitation and magnetite separator methods. All three methods provide the best results in the synthesis of Fe3O4 magnetite nanoparticles. But of the three methods, the solgel method is the best method to obtain Fe3O4 magnetite nanoparticles because this method provides a high yield compared to the other two methods.

*Results of X-Ray Diffractometer (XRD)*

Analysis Phase analysis using X-Ray Diffractometer (XRD) is carried out to determine what phases are contained in magnetite Fe3O4 derived from iron sand. This needs to be done considering that materials come from natural materials that are definitely still mixed or contaminated with other materials. The width of the peaks of each sample graph (Figure 5) shows how many X-rays are scattered in the same plane. So, the higher the diffraction peak, the more X-rays scattered in the same field. The width and height of the XRD graph identify the particle size of a sample (Maulinda et al., 2019).

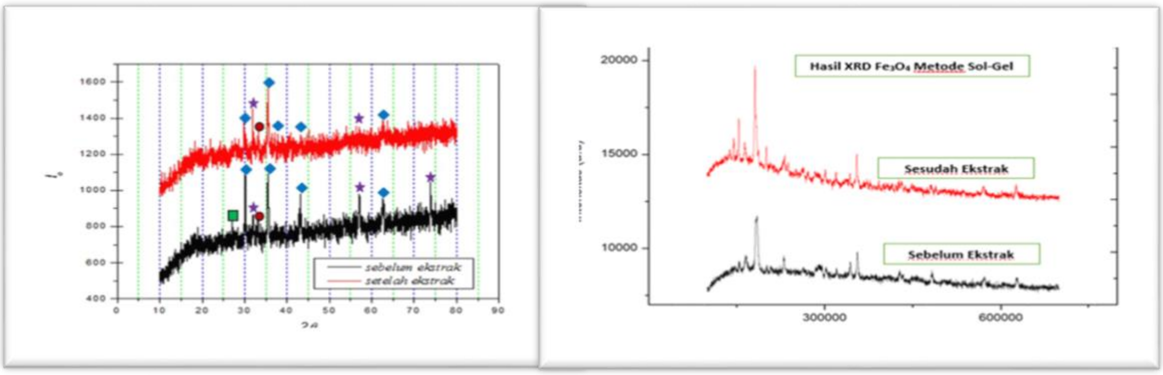


**Figure 5**. Result of a) XRD $Fe_3O_4$ (Coprecipitation); b) XRD $Fe_3O_4$ (Method Sol Gel)

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*Results of SEM EDS*

Analysis Based on the results of SEM observations in Figure 7, the morphological structure of iron sand samples from the synthesis of surface morphology still looks rough, most of the small particle clusters fuse (clump) into large particles, and the particle size is not uniform indicating that there is Non-uniformity of grain size and sample shape shows that the microstructure of iron sand samples is amorphous after synthesis. Indeed, this can be predicted by looking at the XRD results, but the sample still shows sharp or pointed peaks, one of the characteristics of amorphous materials.

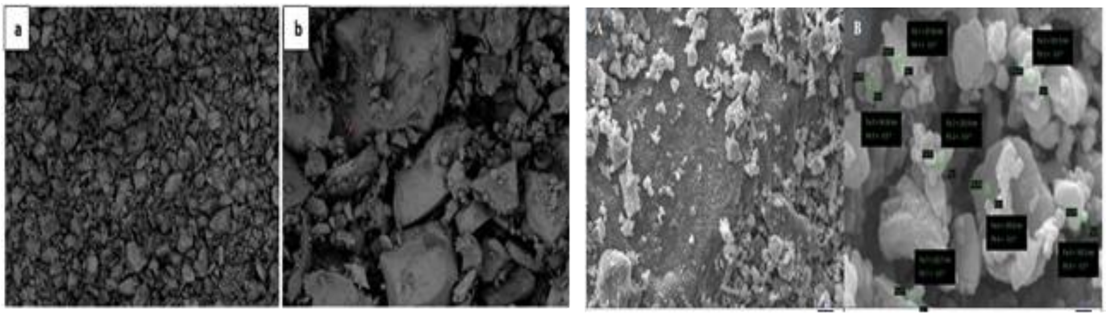

**Figure 6.** a) Result of SEM material $Fe_3O_4$ (Coprecipitation); b) Result of SEM material $Fe_3O_4$ (Sol-Gel)

From the results of SEM Analysis in the figure above shows differences in material surface morphology, where SEM using the Coprecipitation method looks coarser and clumping occurs which shows the grain size will also be large, while in the Sol-Gel method the surface morphology looks a little smooth and clumping that occurs smaller. The results of the SEM characterization test with the Sol Gel method also show that the distribution of Fe3O4 particles is even and produces nanoparticle size, when observed from the results of particle analysis shown in Figure 8B. A particle is said to be nano-sized if the particle size has a value range of 1-100nm (Hu et al., 2019). while Figure 9A shows a flat surface but there are particles scattered due to the imperfect mixing process.

## 4. Conclusions

The synthesis and characterization of magnetite Fe3O4 from iron sand taken from the beach sand of Syiah Kuala Aceh gave good results on the composition of Fe3O4 synthesized using the sol-gel method, where the chemical composition of magnetite Fe3O4 contained in the iron sand of Syiah Kuala beach iron sand showed very high numbers. high at 87.10%. The synthesis of magnetite Fe3O4 in the sol-gel method was compared to the coprecipitation method where the two methods gave very different results, namely 87.01% (sol-gel), 86.73% (coprecipitation). The observation results of the SEM-EDS characterization test based on particle analysis and distribution observed on particle surface morphology where the sol-gel method gave precise results for the synthesis of $Fe_3O_4$ magnetite nanoparticles.

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