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EFFECT OF VARIATIONS MASS OF LAURIC ACID ON THE SIZE OF MAGNETIC NANOPARTICLES FROM IRON SANDS ESTABLISH BATANG MASANG PASAMAN

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ABSTRACT
Synthesis of magnetite nanoparticles has been carried out using iron sand from Muara Batang Masang Gadang Pasaman. This research is aim to investigate of lauric acid effect for measurement of crystal size in magnetite nanoparticle of iron sands. The research on the synthesis of magnetite nanoparticles used a 12 M HCl solution to dissolve iron sand, then the filtrate obtained was precipitated using the precipitation method, namely the coprecipitation method using NH4OH solution, but with the coprecipitation method it is difficult to get a uniform size, so it is necessary to add lauric acid to coat the particles in order to no agglomeration occurs. Crystal size obtained using XRD instrument. The results showed that lauric acid variations gave the same crystal structure, namely cubic, but different crystal sizes. The optimum size was obtained with the addition of 1.25 grams of lauric acid with the highest peak of 2θ = 35.54, with an intensity of 491.9154, so that the crystal size obtained by the Scherre Debye equation was 20.2 nm. The expected crystal size is below 20 nm because it can be used in the biomedical field. The results showed that the more lauric acid was added, the smaller the crystal size obtained, but there was a limit to the addition of lauric acid, because with excess lauric acid agglomeration would occur and the size obtained would increase.

Keywords: Iron Sand, Magnetite Nanoparticles and Lauric Acid.

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1. INTRODUCTION

The distribution of iron sand minerals in Indonesia is very wide, usually found in estuaries because iron sand is formed from the washing of volcanic rocks by rain, resulting in accumulation. Indonesia is one of the regions that has large waters so that it has an iron content

of 77 [1-2]. One of the areas that have iron sand deposits in West Sumatra include West Pasaman, Pariaman, Sijunjung and Solok. Iron (Fe) content in Pariaman (53.997%) and Solok (66.475%) areas lower than Pasaman (69.548%) and Sijunjung (76.365 %) because the iron sands in Pariaman and Solok areas have impurities in the form of calcium (Ca) and silica (Si) higher. The main mineral content of this iron sand is hematite (α - Fe_2O_3), magnetite (Fe_3O_4), maghemite (γ-Fe_2O_3) and impurity compounds in the form of Ti, Si, Mn, Mg, Ca and V with blackish gray color [3-4].

Iron sand is widely used as a cement mixture for the manufacture of concrete, so this utilization is not optimal because iron sand contains high iron oxide, which can be processed into various products with high selling value. The high magnetic mineral content of iron sand can be used as an industrial commodity with high economic value [5-6]. So that iron sand can be used for the synthesis of magnetite nanoparticles [7-9].

Several researches that have been done in the manufacture of magnetic nanoparticles (Fe_3O_4) generally use synthetic materials to increase the economic value of iron sand. Magnetite nanoparticles have many benefits, namely: biomedical field as antibacterial, antifungal, hyperthermic, anticancer [10], while in industrial sector as a separation technology, catalyst and one of the basic ingredients for making dyes [11-12].

Many methods have been used in magnetite synthesis such as coprecipitation method, sol-gel method, thermal decomposition [13-15] and many other methods. The coprecipitation method is easier to use because the preparation process is simple, requires low energy and near pure products are easily obtained [16-17]. So by using the coprecipitation method, the crystal structure and magnetic properties of the prepared particles can be optimized based on the following factors: calcination temperature, calcination time [18]. Stirring time, solution pH, stirring speed, metal salt concentration and concentration surfactant [19] .

Synthesis of magnetite nanoparticles requires uniform crystal size using the coprecipitation method [20]. However, with the coprecipitation method, it is difficult to obtain uniform magnetite particles [21]. So it is necessary to add surfactant to control the crystal size. The surfactant commonly used to obtain a uniform magnetite crystal size is lauric acid [22]. Based on Kurniawan at.all., (2017) research by varying the mass of lauric acid the results obtained the more addition of lauric acid the smaller the crystal size obtained, with the addition of excess lauric acid will experience agglomeration and large crystal size obtained.

Iron sand is a sedimentary mineral that has a grain size of 0.074-0.075 mm, where the size is coarse (5-3 mm) and fine (< 1 mm), black powder or sand. The main content of this iron sand mineral is hematite (α-Fe_2O_3), magnetite (Fe_3O_4), maghemite (γ-Fe_2O_3) and impurity compounds in the form of Ti, Si, Mn, Mg, Ca and V with blackish gray color [23].

Figure.1. Black iron sand

Iron sand is widely used in various industries such as as raw material for the steel industry, it is also used as a mixture of cement, titanium industry, while the main minerals contained in iron sand have different benefits in the industrial world, such as magnetite is used to manufacture permanent magnets, maghemite. Used as a recording material on audio cassettes and hematite as a radiation shielding material. These three minerals (magnetite, hematite, and maghemite) are also used as pigments or coloring agents in many applications [24].

Magnetite is an inorganic chemical compound that has the formula Fe_3O_4 or can also be formulated as $FeO.Fe_2O_3$. Inside magnetite there are Fe^{2+} and Fe^{3+} ions, where the Fe^{3+} ions in magnetite partially occupy the tetrahedron cavity and the other part occupies the octahedron cavity and for all Fe^{2+} ions occupy the octahedron cavity in the form of a metal crystal structure with a face-to-face cubic (fcc) arrangement [25-26].

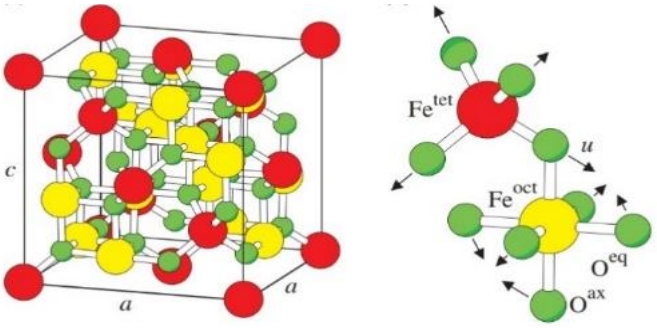

Figure 2. Crystal structure of magnetite (Fe_3O_4)

According to Ataeefard et al., (2014) the particles needed in the synthesis of magnetite are nanometer in size (smaller than 100 nm) with a uniform particle distribution. The effectiveness of particles with smaller and more uniform size distributions is very good in their use [27].

The chemical formula for lauric acid is $CH_3(CH_2)_{10}COOH$ with a molecular weight of 200.3178 $g.mol^{-1}$, a density of 0.880 g/cm^3, a boiling point of 298.9°C and a melting point of 43.2°C. Lauric acid has a concern (methyl) group at the end and a carboxyl group at the other, so that lauric acid is soluble in air and also soluble in fat which can be used in the washing industry such as shampoo [28-29].

Lauric acid can control the size and dispersion of nanoparticles because lauric acid is one of the carboxylic compounds, so that nanoparticles can agglomerate with other particles to form

bulk materials as before. Accumulation between nanoparticles can be prevented by adding lauric acid, because lauric acid can coat the particles to avoid accumulation on the material [30].

The coprecipitation method is a method of synthesizing inorganic compounds based on the deposition of more than one substance simultaneously when passing through the saturation point [31]. The coprecipitation method has a working principle, namely by converting a metal salt into a precipitate using a basic hydroxide or carbonate precipitator which is converted to its oxide form by heating.

Most researchers use the coprecipitation method to synthesize magnetite, because this method has various advantages such as a simple preparation process, requires low energy, and near pure products that are easy to obtain. In addition, the coprecipitation method is more efficient and easier because the process uses low temperatures so that the time required is relatively short [32-33]

X-ray diffraction (XRD) is a technique used to identify the crystalline phase in materials and also to determine the crystal structure and crystal size. The principle of X-ray diffraction is that when X-rays hit a solid object in the form of atoms, the X-rays will be scattered by the electrons in the atom. Constructive or destructive wave interference occurs along different directions because the scattered waves (diffraction patterns) are emitted by atoms at different positions [34-35]. Some of the advantages of X-ray diffraction (XRD) are provides unambiguous mineral determination in most cases, requires minimal sample preparation, wide availability of XRD units, data interpretation is relatively easy [36].

Synthesis of magnetite nanoparticles has been carried out using iron sand from Muara Batang Masang Gadang Pasaman. The research on the synthesis of magnetite nanoparticles used a 12 M HCl solution to dissolve iron sand, then the filtrate obtained was precipitated using the precipitation method, namely the coprecipitation method using NH4OH solution, but with the coprecipitation method it is difficult to get a uniform size, so it is necessary to add lauric acid to coat the particles in order to no agglomeration occurs. This research is aim to investigate of lauric acid effect for measurement of crystal size in magnetite nanoparticle of iron sands.

2. EXPERIMENTAL SECTION

2.1 Physical refining of iron sand

The iron sand is taken at Muara Batang Masang Gadang Pasaman, then the iron sand is washed and dried to dry. The dry iron sand is pulled with a permanent magnet, then the iron sand is ground and filtered using an 80 mesh sieve.

2.2 Chemical refining of iron sand

Dissolve 8 g, 10 g, 12 g, 14 g and 16 g of iron sand powder with 20 ml of 12 M HCl. The solution was allowed to stand for 24 hours, then the solution was stirred for 60 minutes using a magnetic stirrer at a temperature of 90°C and a speed of 350 rpm until the solution is

homogeneous. To separate the solution with impurities, the solution was filtered through Whatman filter paper no. 42 to obtain the filtrate.

2.3 Magnetite nanoparticle synthesis

Precipitation was carried out by dripping the obtained filtrate into a container containing 0g, 1.25g, 2.5g, 3.75g, and 5g lauric acid which had been dissolved with NH4OH 6.5M as much as 25 ml, mixing ammonia with lauric acid was carried out at room temperature until the mixture was homogeneous. The precipitate was allowed to stand for 30 minutes to form a black Fe_3O_4 precipitate. Separating the excess solution contained in the precipitate, then the precipitate was washed with distilled water to pH 7. The water content contained in the Fe_3O_4 precipitate can be removed by drying using an oven at a temperature of 115°C. Then the powder is calcined at a temperature of 400° C for 1 hour. Synthesis of Fe_3O_4 obtained in powder form, with a mass variation of 0g lauric acid; 1.25g; 2.5g; 3.75g and 5g were labeled $M_P.AL_0$, $M_P.AL_{1.25}$, $M_P.AL_{2.5}$, $M_P.AL_{3.75}$ and $M_P.AL_5$. Magnetite (Fe_3O_4) nanoparticle powder was characterized using XRD to see its crystal structure and size.

3. RESULTS AND DISCUSSION

3.1 Variation of iron sand mass

The following graph shows the percentage of iron sand soluble in 12 M HCl:

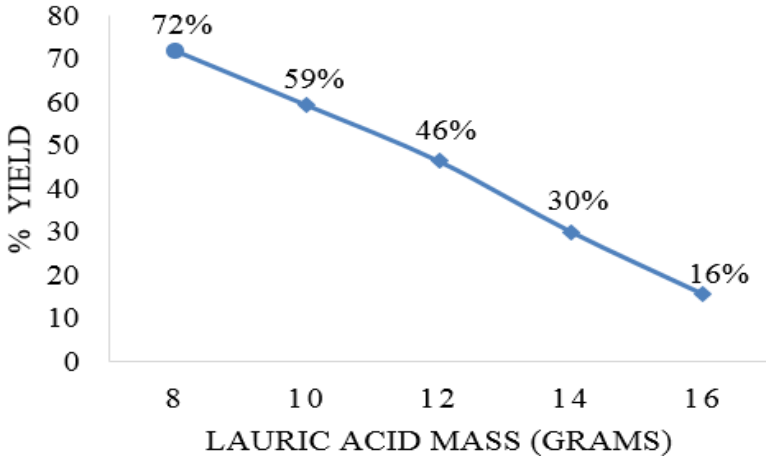

Figure 3. Mass percent dissolved iron sand

From the graph it can be seen that the percentage of iron sand mass that dissolves the most is 72% in the mass of iron sand 8 grams while the percentage of iron sand that is the least soluble in the mass of 16 grams of iron sand is 15.56%, so the less mass of iron sand that is reacted will increase percentage solubility.

3.2 Characterization Analysis with XRD

Characterization using XRD can determine the crystal structure and size of the crystals formed.

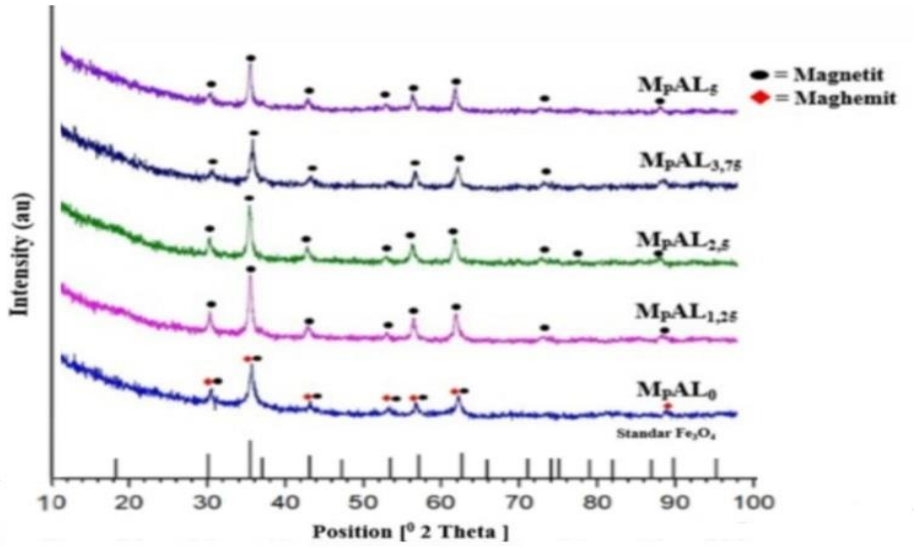

Figure 4. Magnetite nanoparticle XRD test results

Based on the diffractogram above, it is a comparison of the mass variation of the lauric acid diffractogram used. The results showed that lauric acid variations gave the same crystal structure, namely cubic, but different crystal sizes.

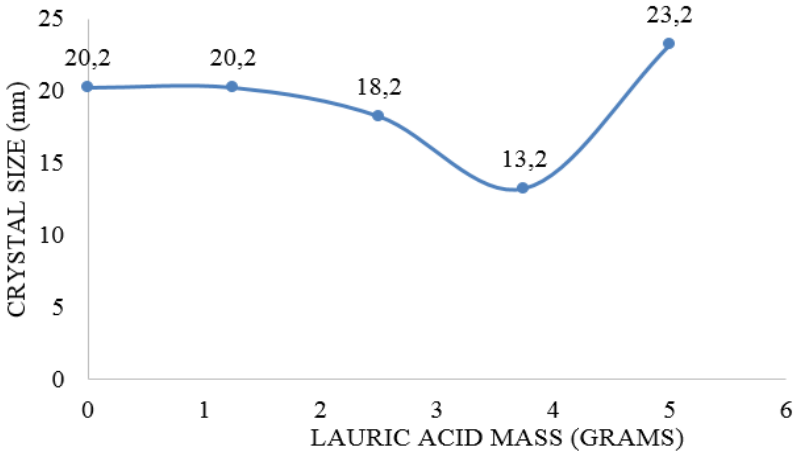

Figure 5. Crystal size with acid variation lauric

In Figure 5 the optimum size obtained with the addition of 1.25 grams of lauric acid with the highest peak of 2θ = 35.54, with an intensity of 491.9154, so that the crystal size with the Scherre Debye equation is 20.2 nm. The expected crystal size is below 20 nm . Based on research by Saragi, et al,. 2016 that sizes below 20 nm can be well utilized in the biomedical field because of their ability to affect the rate of protein relaxation in water. The results were obtained based on the research of Ataeefard et al., (2014) the more lauric acid is added, the smaller the magnetite crystal size is formed, but there is a limit to the addition of lauric acid to reduce the particle size because the addition of excess lauric acid will cause aggregation and the formation of large crystal sizes.

4. CONCLUSION

The optimum results obtained with the addition of 1.25 gram of lauric acid is the size of the crystal formed 20.2 nm.The addition of lauric acid mass does not affect the crystal structure, but it does affect the size of the Fe_3O_4 crystals formed, the more lauric acid is added, the smaller the magnetite crystal size is formed, but there is a limit to the addition of lauric acid to reduce the particle size because the addition of excess lauric acid will cause aggregation and the formation of large crystal sizes.

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