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EFFECT OF LAURIC ACID MASS VARIATIONS ON THE SIZE OF MAGNETITE NANOPARTICLES (Fe_3O_4) FROM IRON SANDS AT SUNUR PARIAMAN BEACH ESTUARY

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ABSTRACT
The purpose of this research to investigate effect of lauric acid mass on crystal size in magnetite samples using iron sand as the basis of Sunur Pariaman beach. Synthesis of magnetite nanoparticles using using iron sand from Sunur Pariaman beach. Synthesis of magnetite nanoparticles using HCl 12 M has a solvent and NH_4OH as a precipitate. The method used is the coprecipitation method with of lauric acid as a surfactant. Lauric acid serves to prevent agglomeration. Synthesis of magnetite nanoparticles characterized using XRD. The crystal size is calculated using the Scherrer- Debye equation. The result showed that lauric acid variations gave the same crystal structure namely cubic but different crystal size. Magnetite with the addition of lauric acid 0 grams, 1.25 grams, 2.5 grams, 3.75 grams and 5 grams obtaines crystal size of 23.2 nm, 28.1 nm, 25.3 nm, 20.9 nm and 23.1 nm.

Keywords: Iron sand; Magnetite nanoparticles; Lauric acid

1. INTRODUCTION

West Sumatra is a province that has a wealth of natural resources in the form of iron sand, lead, limestone (cement), galena stone, gambier, zinc, fishery products, cocoa, gold and oil palm. Iron sand can be used [1-3]. Areas in the province of West Sumatra that are rich in iron sand are Pariaman, Sijunjung, Pasaman, Solok and Pesisir Selatan [4]. Iron sand is sand with iron content that is commonly found in nature. Iron sands generally contain iron oxides such as Fe_3O_4 (magnetite), -Fe $_2O_3$ (hematite) and - Fe_2O_3 (maghemite). Iron sand also has elements of magnesium, titanium, vanadium, silicon, calcium and others. The magnetite found in iron sand has a higher interaction with magnets than the others. Iron sand is often found on the beach,

mountains and rivers [5-7]. The iron sand of the estuary of Sunur Pariaman Beach has magnetic mineral content and high susceptibility value. The iron sand in the Pariaman area has a low iron content (5 3.997 %) compared to the Solok area (6,475 %) because the iron sand in the Pariaman area has impurities in the form of SiO_2 [8].

Iron sand is used as a mixture of cement and building materials. The high iron oxide content in iron sand can be used as an industrial commodity with high economic value [9]. There are several attempts to increase the selling value of iron sand such as catalysts, ceramics, Fe_3O_4 nanoparticles, ferrofluids, basic materials for making inorganic dyes for paints, absorbents and medical diagnosis [10]. Therefore, to increase the selling value of iron sand, magnetite (Fe_3O_4) nanoparticles can be made.

Synthesis of Fe_3O_4 from iron sand generally use several methods such as coprecipitation, high energy milling, mechanical alloying, sonochemistry, hydrothermal, sol-gel and gamma irradiation [11]. The method that is often used for the synthesis of Fe_3O_4 is the coprecipitation method. The advantages of the coprecipitation method are low temperature, simple procedure, and relatively short time [12].

The coprecipitation method is the most frequently used method. This synthesis is carried out with acids as well as bases. Acid to dissolve iron sand produces a solution of Fe^{2+} or Fe^{3+} and a base to precipitate iron ions to form solid Fe_3O_4, Fe_2O_3 in certain crystals according to the reaction conditions. Synthesis of magnetite from a solution of Fe^{2+}, Fe^{3+} using the coprecipitation method can produce monodispersive nanoparticles (uniform particle shape).

Synthesis of magnetite nanoparticles from iron solution requires a surfactant which serves to coat the magnetite particles to prevent clumping or aggregation. It aims to regulate the size of the magnetite particles produced. Surfactants used such as lauric acid [15], and Polyethylene Glycol (PEG) [13]. An example of using lauric acid as a coating reagent to prevent agglomeration. Lauric acid can also control the dispersion and particle size of magnetite in order to obtain magnetite with an even and more uniform size [14].

The main iron mineral content in iron sands is magnetite and hematite. Mineral impurities such as biotite, quartz, pyroxene and others. Iron sand has a gray-black color, grain size 75-150 microns, density 2-5 gram/cm3, percentage of magnetism (MD) 6.40 - 27.16% and bulk density (Specific Gravity, SG) 2.99- 4.23 g/cm3. The melting point is at 2861 °C and the melting point is at 1538 °C. The darker the black color of the iron sand, the better the quality of the iron sand [15-17].

Figure 1. Iron Sand [18]

Magnetite has a chemical structure of FeO-Fe_2O_3, where FeO is wustit and Fe_2O_3 is hematite with a spinel cubic crystal structure. This structure is composed of several oxygen ions, Fe^{3+} and Fe^{2+} ions and is ferromagnetic. The structure of magnetite (Fe_3O_4) is an inverted spinel with Fe (III) ions distributed randomly between octahedral and tetrahedral, while Fe (II) ions are only present in octahedral [19-21].

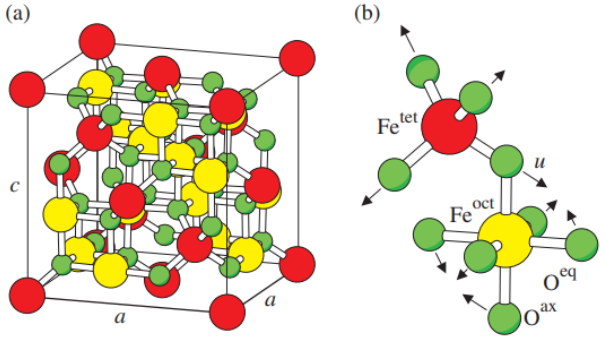

Figure.2. Crystal structure of magnetite [22]

Lauric acid (dodecanoic acid) is a saturated fatty acid consisting of 12 C atoms. The main source of this fatty acid is coconut oil, 50% contains palm oil and lauric acid. Other sources are goat's milk and cow's milk. Lauric acid has a boiling point of 225 °C and a melting point of 44 °C so that at room temperature it is a white solid, if heated it can melt. Lauric acid has the chemical formula $CH_3(CH_2)_{10}COOH$ with a molecular weight of 200.3 grams/mol, a density of 0.880 grams/cm^3. Lauric acid is soluble in polar solvents such as fats and can dissolve in water because of the carboxyl group at one end and the hydrocarbon (methyl) group at the other end [23-25].

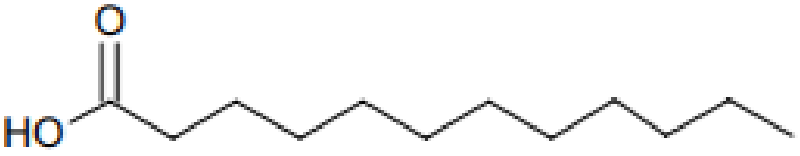

Figure 3. Lauric acid structure [26]

Magnetite synthesis using coprecipitation method can give different effects such as in acidic, neutral and alkaline conditions. In an acidic environment, the reaction for the formation

of iron oxide can only occur in a solution containing Fe^{3+} metal ions. The number of Fe^{3+} and Fe^{2+} ions in solution depends on the ability of ferrous metal ions (Fe) to react with hydroxide ions (OH^-). The higher the concentration of the solution, the larger the ions to react.

XRD is an instrumentation and characterization of elements/compounds that is used to analyze the crystalline phase of the material by determining the lattice structure, as well as to analyze the degree of crystallization of the resulting material. The form of data generated from the diffractometer can be digital/analog data.

Determination of the crystal structure formed is done by matching each peak that appears on the diffractogram at the value of d and angle of 2θ from the analysis with data from JCPDS (Joint Committee Powder Diffraction Standard) so that the crystal planes formed can be obtained. The crystal structure is in agreement if all the orientations of the crystal planes are identified. X-ray diffraction is used to determine the crystal size. To calculate the crystal size of the sample, the following Scherrer-Debye equation is used:

$$D = \frac{k\lambda}{B\cos\theta}$$

2. EXPERIMENTAL

2.1 Physical refining of iron sand

The iron sand of the estuary of Sunur Pariaman Beach is washed and dried. The dry iron sand is then separated from the impurities with a permanent magnet. Then filtered using an 80 mesh sieve.

2.2 Chemical refining of iron sand

8, 10 g, 12 g, 14 g and 16 g of iron sand were dissolved in 20 ml of 12 M HCl and then allowed to stand for one day. Then stirred using a *magnetic stirrer* at a temperature of 90°C, a speed of 350 rpm and for 60 minutes. After that, it was filtered using whatman filter paper no. 42 and the mass of dissolved iron sand was calculated to be used as material for the next magnetite nanoparticle synthesis.

2.3 Magnetite nanoparticle synthesis

The most soluble iron sand powder in dissolution with HCl was continued in the NH_4OH precipitation process. Precipitation was carried out by dripping the obtained filtrate into a container contining a mixture of 25 ml of 6.5 M NH_4OH solution and lauric acid with mass variations of 0 g, 1.25 g, 2.5 g, 3.75 g and 5 g (mixing ammonia with lauric acid is carried out at room temperature until the mixture is homogeneous). The precipitate is allowed to stand for 30 minutes then a magnetite precipitate is formed black. then washed with distilled water until pH = 7.

The precipitate was then oven-dried at 115 °C to dry to remove moisture. Then calcined at 400 °C for 1 hour. The results obtained in the form of powder. The synthesized magnetite nanoparticles were characterized using XRD. Powder with lauric acid mass variation 0g; 1.25g; 2.5g; 3.75g and 5g are labeled $MS.AL_0$; $MS.AL_{1.25}$; _ $MS.AL_{2.5}$; $MS.AL_{3.75}$ and $MS.AL_5$.

3. RESULTS AND DISCUSSION

3.1 Variation of Iron Sand Mass

Based on the results of the data obtained, the highest % yield was 81.1% at a mass of 8 grams of iron sand and the lowest % yield was 31.2% at a mass of 16 grams of iron sand. The less mass of iron sand, the higher the % yield.

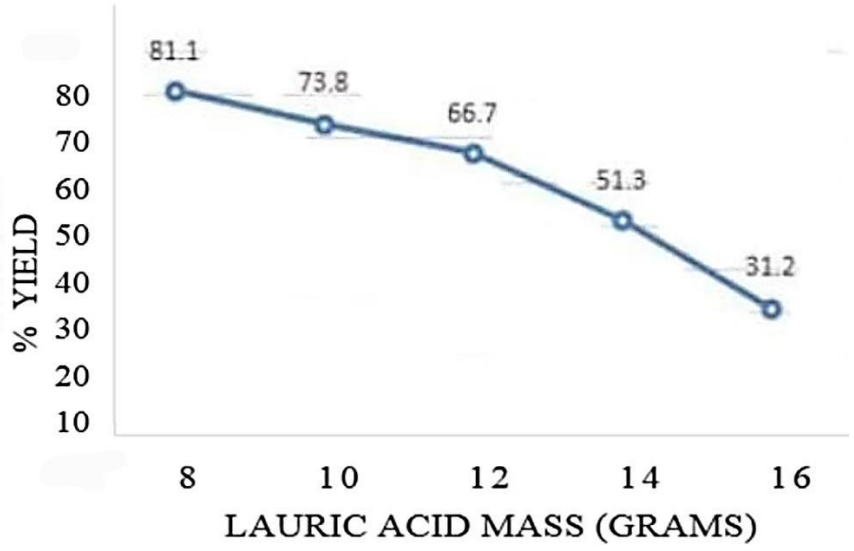

Figure 4. Yield percentage of iron sand to HCl

3.2 XRD Characterization

Based on the XRD results, it can be seen that the iron sand after physical refining obtained peaks at positions 2θ =30.223110, 32.910610, 35.568870, 57.072710, 62.625790. The resulting sample formed Fe_3O_4, Fe_2O_3 and SiO_2 phases . Si is a paramagnetic element. iron sand is attracted by a magnet, the element Si can be attracted by a magnet. Iron sand has impurities such as SiO_2. SiO_2 is insoluble in HCl solution.

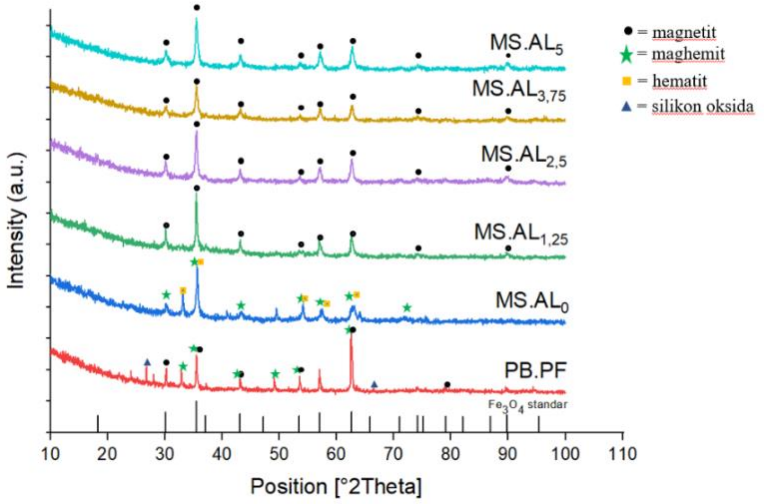

Figure 5. XRD characterization results

In the sample $MS.AL_0$ it can be seen that hematite and maghemite phases are formed and no magnetite phase is formed in the sample. This indicates the occurrence of an oxidation process and transformed into maghemite and hematite. Therefore, surfactants are needed to reduce the tendency of magnetite which is easily oxidized. The crystal structure is rhombohedral.

All samples with the addition of lauric acid formed a magnetite phase and no lauric acid phase was formed in the sample. Lauric acid does not react and only acts as a surfactant. Samples with the addition of lauric acid have a crystal structure that is *cubic.*

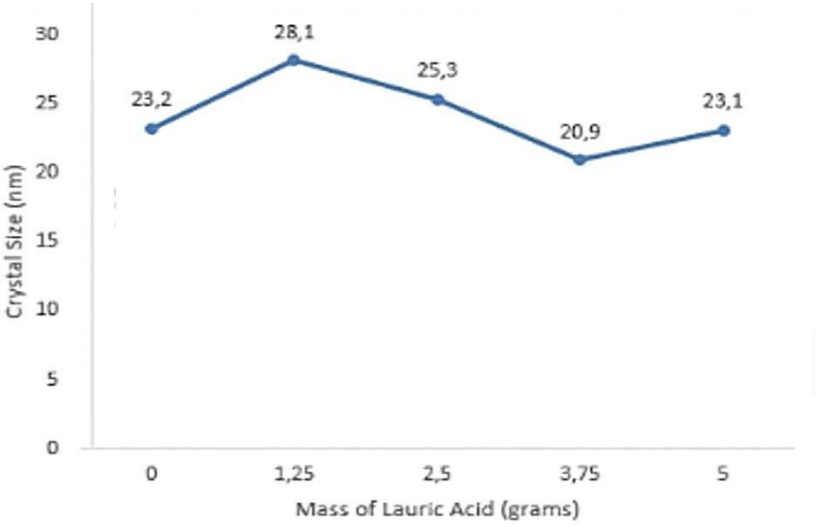

Figure 6. Mass of lauric acid with crystal size

The more lauric acid used, the smaller the Fe_3O_4 crystal size. The lauric acid surrounds the surface of the Fe_3O_4 particles so that it can inhibit the crystal growth. At the addition of 1.25 grams of lauric acid, the highest crystal size was 28.1 nm with the highest intensity peak compared to other samples. At the addition of 3.75 grams of lauric acid, the lowest crystal size

was 20.9 nm with the lowest peak intensity compared to other samples. With the addition of 5 grams of lauric acid, the crystal size increased again by 23.1 nm.

4. CONCLUSION

The difference in the mass of iron sand in HCl solution produces a solution of $FeCl_2$, $FeCl_3$ with different yields. The highest yield was in the mass of 8 grams of iron sand as much as 81.1%. The addition of lauric acid mass did not affect the structure but affected the size of the Fe_3O_4 crystals formed . The more lauric acid is added, the smaller the magnetite crystal size . There is a limit to the addition of lauric acid to reduce the crystal size because the addition of excess lauric acid will cause large crystal sizes.

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