Synthesis and Characterization of Magnetite Nanoparticles (Fe_3O_4) Based on Iron Sand from Glagah Kulon Progo Yogyakarta via Coprecipitation Method with Variations in The Dissolution Duration

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Abstract. This research aims to determine the effect of heating time of iron sand extract on HCl solution on lattice parameters, crystal structure, nanoparticle size, surface morphological structure, chemical composition and magnetic properties of magnetite nanoparticles Fe_3O_4. The dissolution process at 55ºC with a dissolution time of 30 minutes, 45 minutes, 60 minutes and 75 minutes formed magnetite nanoparticles Fe_3O_4 with similar lattice parameter values and the same crystal structure. Magnetite nanoparticles Fe_3O_4 which were formed are quite homogeneous, that are marked by grain sizes that are almost uniform and the same color. Samples with a dissolution temperature of 55ºC and a dissolution time of 75 minutes formed Fe_3O_4 with an average grain size of 193.86 nm. Magnetite nanoparticles Fe_3O_4 contained the elements Fe, O, Cl, Al and Ti. Magnetite nanoparticles Fe_3O_4 with a dissolution temperature of 55ºC for 75 minutes had a saturation magnetization (M_s) value of 31.0 emu / gram, remanent magnetization (M_r) value of 9.6 emu / gram and coercivity field (H_c) of 0.02 T. This shows that magnetite nanoparticles Fe_3O_4 produced are ferrimagnetic materials. The coercivity value obtained is smaller than 0.03 T, so it can be stated that magntetite nanoparticle Fe_3O_4 samples are soft magnetic.

1. Introduction

Java, especially the Special Region of Yogyakarta (DIY) has enormous natural potential. The one of those is iron sand founded on the southern coast of DIY. Iron sand is widely used in various fields. The use of iron sand is 80% of the total weight of capable sand increase the compressive strength and tensile strength up to 80%, this is possible because in addition to the filler properties are also chemical properties of iron sand containing SiO_2 thus helping the performance of cement as a binder [1].

A further application of magnetite nanoparticles as a microwave absorbent material is as a component in the manufacture of RAM (Radar Absorbing Material). RAM is a material that can detect the position of objects using microwaves or radio waves. Until now, material in the form of iron rock is still a very good choice of material to be used as a magnetic absorbent material on microwaves [2].

Iron sand contains several mineral ingredients, one of which is magnetite. Magnetite is a mineral material containing iron (Fe) and oxygen (O_2), with the chemical formula Fe_3O_4. Magnetite has the property of being drawn by magnets (referred to as magnetic materials). Based on several studies that have been done, nano-sized magnetite particles or commonly referred to as magnetite nanoparticles can be utilized in several ways. Research conducted by Erika and Astuti informs us that magnetite nanoparticles mixed with polyaniline can be used as microwave absorbing agents. . That is because basically the material that can be used as a microwave absorber is a material that has electrical and magnetic properties [3]. Magnetite nanoparticles doped with Zn ions in Barium Hexaferrite to form Barium M-Hexaferrite can be used as magnetic material that can absorb microwaves [4]. Magnetite particles which will be applied to various things must have good quality. The quality in question can be in the form of suitable particle size, suitable magnetic properties and surface properties of the particles. Magnetite particles (Fe_3O_4) can be obtained from the purification of coastal iron sand. The technique of purifying iron sand into magnetite particles can be done by extracting iron sand using permanent magnets. This is based on the magnetic properties of the magnetite contained in iron sand so that the magnetite minerals can be extracted and separated from impurities using permanent magnets.

2. Research methods

The research was conducted in three stages. The first is iron sand extraction from Glagah Kulon Progo beach sand. Iron sand extraction from Glagah Kulon Progo beach sand is carried out using permanent magnets. Extraction was carried out by utilizing the magnetic properties of the magnetite particles so that they could separate themselves from the impurities. The second is synthesis phase of Fe_3O_4 magnetite particles. The synthesis phase of Fe_3O_4 magnetite particles from the iron sand beach of Glagah Kulon Progo was carried out by the coprecipitation method by varying the dissolution time. The third is characterization of Fe_3O_4 magnetite particles. The synthesized magnetite particles were characterized using XRD to determine the crystal structure, lattice parameters, and crystal size. SEM and EDS characterization was also carried out to determine the surface morphological structure and chemical composition of magnetite particle material. While the magnetic nature is characterized by using VSM.

The main ingredient in the synthesis process of Fe_3O_4 magnetic nanoparticles by coprecipitation method is the iron sand beach Glagah Kulon Progo. As many 50 grams of iron sand extract is used for magnetite synthesis with 4 variations of dissolution time, which are 30 minutes, 45 minutes, 60 minutes and 75 minutes. The dissolving temperature used is fixed, which is 55 °C. The coprecipitation method consists of 2 main stages, namely the dissolution stage using HCl solution (37%) and the deposition stage using NH_4OH solution (25%).

3. Results and Discussion

3.1 Results of XRD Characterization of Fe_3O_4 Magnetic Nanoparticles

The results of XRD characterization of sample 1 (30 minutes dissolution time), sample 2 (45 minutes dissolution time), sample 3 (60 minutes dissolution time) and sample 4 (75 minutes dissolution time) are shown in Figure 1, Figure 2, Figure 3 and Figure 4. The figures are diffractograms showing the relationship between the angle of the storm (2θ) and the intensity of the X-ray scattered. Diffractogram shows diffraction peaks at certain angles. The diffractogram is then compared with the corresponding JCPDS data. So that it can be determined the value of the lattice parameters and crystal structure of the sample. Based on the XRD results, all four samples had the highest diffraction peak at 2θ of 35° until 36° which is in the plane (311), which is a typical plane of magnetite material.

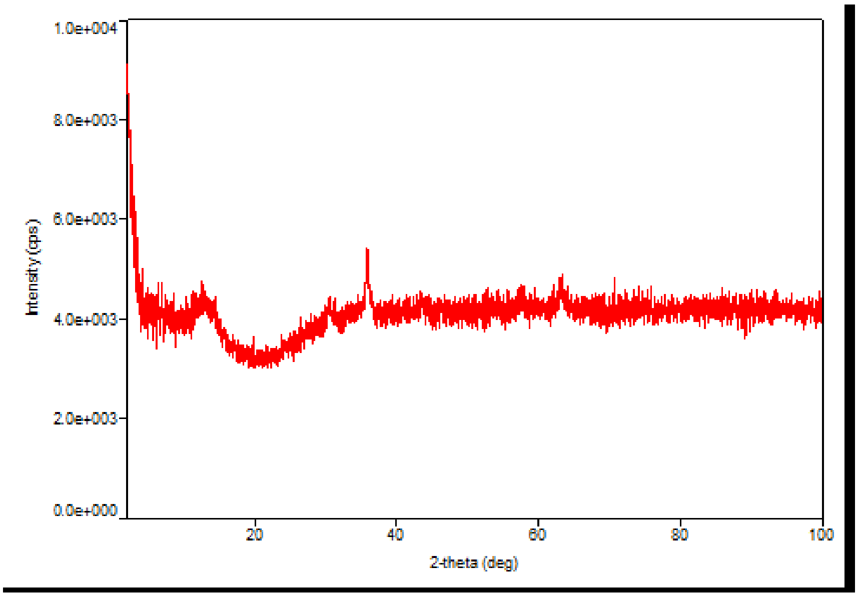

Figure 1. Diffractogram sample 1 (dissolution time is 30 minutes)

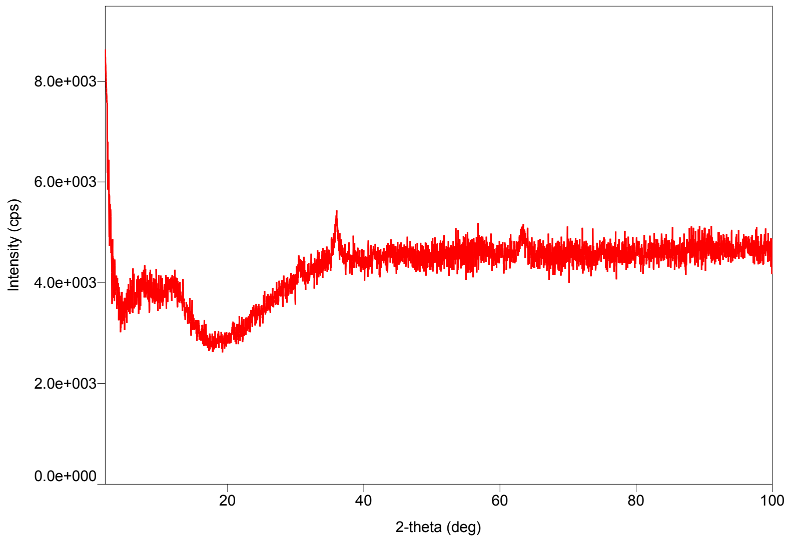

Figure 2. Diffractogram sample 2 (dissolution time is 45 minutes)

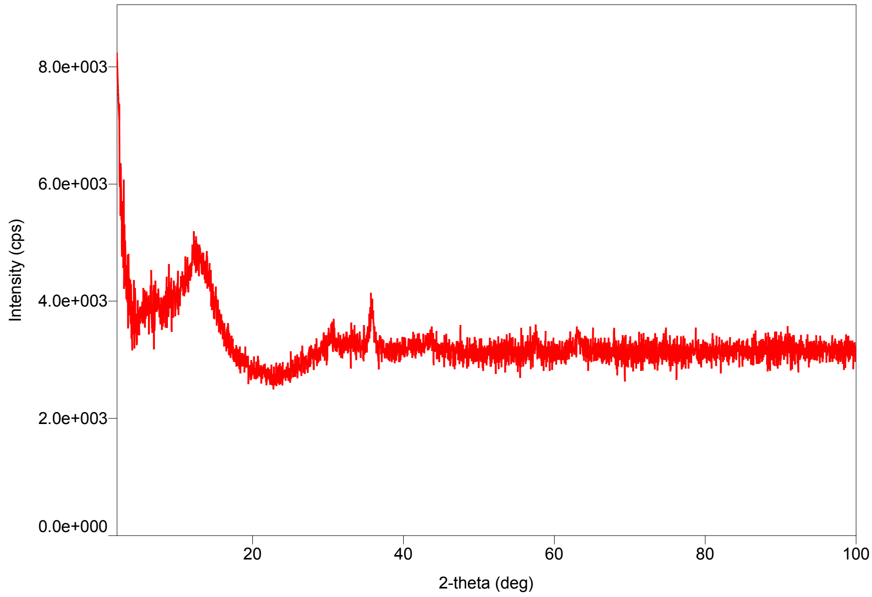

Figure 3. Diffractogram sample 3 (dissolution time is 60 minutes)

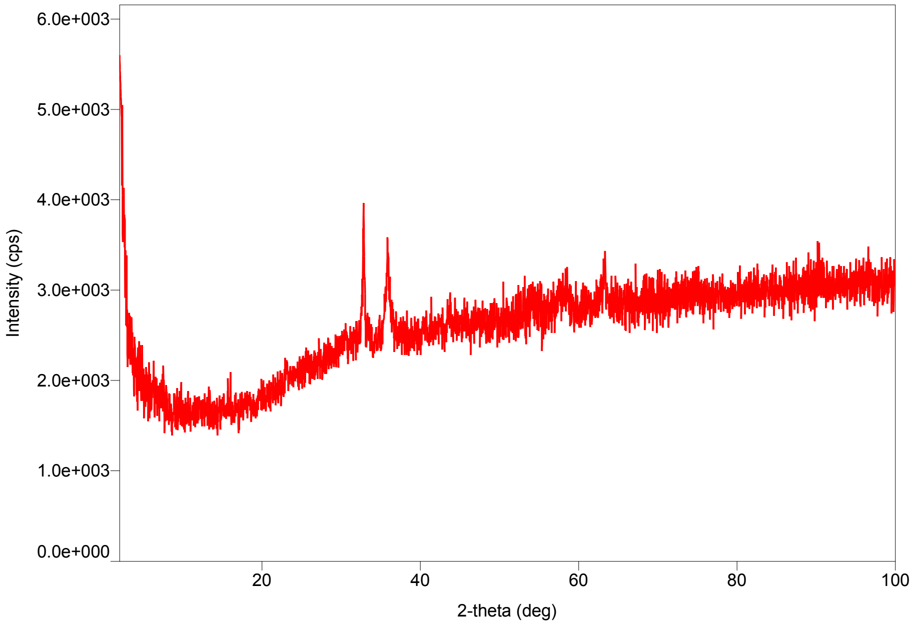

Figure 4. Diffractogram sample 4 (dissolution time is 75 minutes)

Based on the analysis of XRD results using Match obtained information on the lattice parameter values, crystal structure and crystal size. Sample 1 (Figure 1) shows that Fe_3O_4 crystal formed which has a cubic crystal structure with a lattice parameter a = 8.3053 Å. Sample 2 (Figure 2) shows that Fe_3O_4 crystals formed which have a cubic crystal structure with a lattice parameter value a = 8.3008 Å. Sample

3 (Figure 3) shows that Fe_3O_4 crystals formed which have a cubic crystal structure with a lattice parameter value a = 8.2852 Å . Sample 4 (Figure 3) shows that Fe_3O_4 crystal formed which has a cubic crystal structure with a lattice parameter a = 8.2145 Å. The results of XRD characterization of the four samples as in Table 1 shows that the length of time of dissolving iron sand extract on HCl only slightly affects the lattice parameter values and the size of Fe_3O_4 magnetic nanoparticle that are formed. While the crystal structure remains the same.

Table 1. Lattice Parameter Value, Crystal Structure and Particle Size of the Magnetic Nanoparticle Fe_3O_4 Synthesis Results with Dissolution Time

Number	Sample	Length of dissolution time (minutes)	Lattice parameter (*a*) Å	Crsytal structure	Partikel size (nm)
1	Sample 1	30	8.3053	Cubic	34.40
2	Sample 2	45	8.3008	Cubic	22.93
3	Sample 3	60	8.2852	Cubic	29.49
4	Sample 4	75	8.2145	Cubic	22.96

3.2. Results of SEM and EDS Characterization of Fe_3O_4 Magnetic Nanoparticles

SEM characterization was carried out to determine the surface morphology structure of Fe_3O_4 magnetic nanoparticle material. EDS characterization was carried out to determine the chemical composition of Fe_3O_4 magnetic nanoparticles. SEM and EDS characterization was carried out in sample 4, which is a sample that was synthesized with 75 minutes of dissolving iron sand extract on HCl. Based on the XRD results, sample 4 had the highest diffraction intensity at a particular diffraction angle, namely at 2θ = 32.809° and 2θ = 35.84°, when compared with other samples.

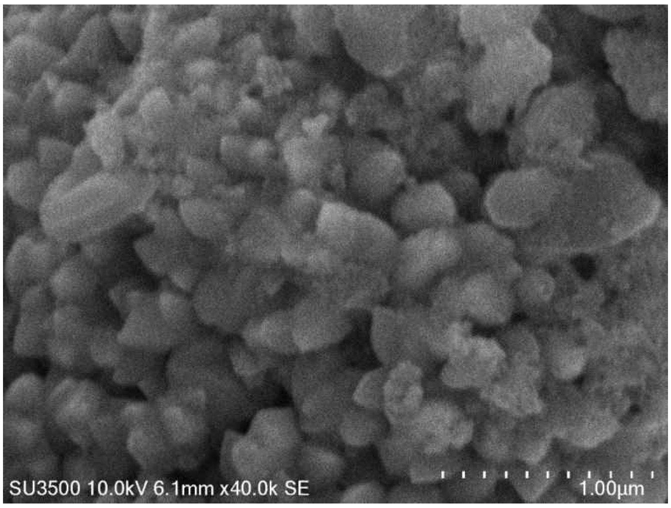

Figure 5. SEM photo surface of Fe_3O_4 magnetic material sample 4 with a magnification of 40,000 x

Based on the results of SEM characterization in Figure 5, it can be observed that the Fe_3O_4 magnetic nanoparticles formed in sample 4 are quite homogeneous, that is marked by grain sizes that are almost uniform and the same color. The SEM results can also be used to calculate the grain size of Fe_3O_4 magnetic nanoparticles formed. Fe_3O_4 magnetic nanoparticle grain size analysis was processed using paint and microsoft excel software. Based on this analysis it was found that the size of Fe_3O_4 magnetic nanoparticles varied and ranged from 96 nm to 290 nm. The average value of grain size of the Fe_3O_4 magnetite nanoparticles formed was 193.861 nm.

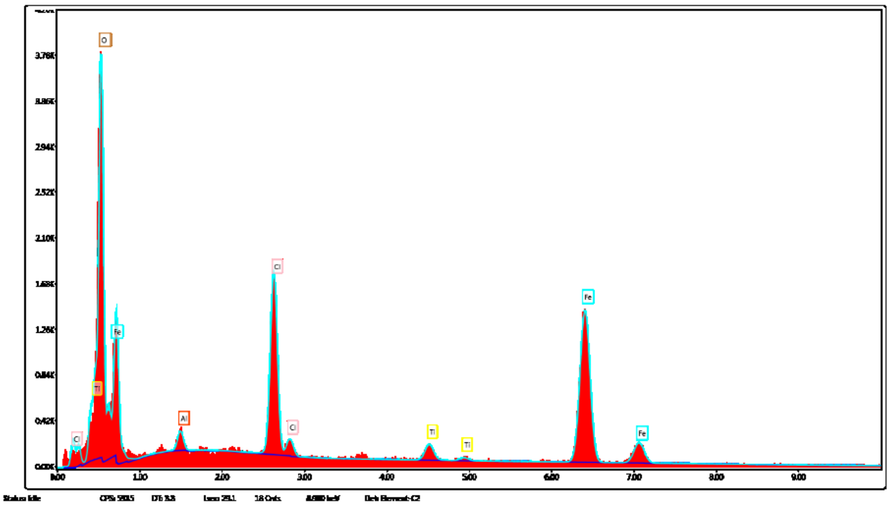

Figure 6. Graphs of EDS characterization of Fe_3O_4 magnetic material samples 4

Based on the results of the EDS characterization in Figure 10, the chemical composition of Fe_3O_4 magnetic nanoparticles can be seen. The types of chemical elements making up Fe_3O_4 magnetic nanoparticles and their numbers are shown in Table 2. The formed Fe_3O_4 magnetic nanoparticles contain the elements Fe, O, Cl, Al and Ti.

Table 2. Composition of chemicals of Fe_3O_4 samples 4 Based on EDS results

Elements	Mass (%)	Atom (%)
Fe	61.80	37.52
O	22.49	47.67
Cl	12.34	11,81
Al	1.13	1.42
Ti	2.24	1.59

3.3. Magnetic Properties of Fe_3O_4 Magnetic Nanoparticles Characterized by VSM

The magnetic properties of nanoparticles were tested using a VSM (Vibrating Sample Magnetometer) with a given external magnetic field strength reaching 1 T. The test results obtained data magnetization (M) nanoparticle samples as a variation of the external magnetic field strength (H). Figure 7 shows the results of the characterization of magnetic properties using VSM from Fe_3O_4 nanoparticles.

Based on the loop hysteresis in Figure 7, Fe_3O_4 nanoparticles synthesized by the coprecipitation method at 55° C dissolution temperature for 75 minutes have a saturation magnetization (M_s) value of 31.0 emu / gram. The curve shows that in the external magnetic field of 1 T, the entire magnetic domain is almost completely saturated. At this time, the direction of the magnetic moment of Fe_3O_4 nanoparticles tends to follow the direction of the external magnetic field. This shows that the response of Fe_3O_4 nanoparticle samples to external magnetic fields is quite good. While the remanent magnetization value (Mr) and the coercivity field (H_c) of Fe_3O_4 nanoparticles are respectively 9.6 emu / gram and 0.02 T. This shows that the Fe_3O_4 nanoparticles produced are ferrimagnetic materials. The Fe_3O_4 nanoparticles will have a tendency to change into superparamagnetic material when it has a value of $H_c = 0$. The coercivity value obtained from the sample is smaller than 0.03 T, so it can be stated that the nanoparticle sample is soft magnetic [5].

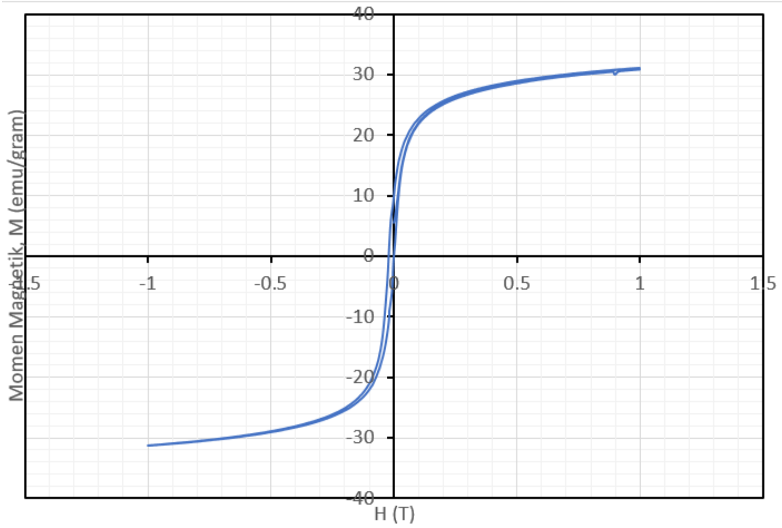

Figure 7. Hysteresis loop sample of 4 Fe3O4 nanoparticles (dissolution temperature of 55° C for 75 minutes)

4. Conclusion

Based on XRD results, Fe_3O_4 magnetite nanoparticles formed crystals in the treatment with iron sand extract dissolution temperature of 55 ºC with a dissolution time of 30 minutes, 45 minutes, 60 minutes and 75 minutes. The dissolution time did not affect the crystal structure, that is, cubic, although the lattice parameter and particle size were slightly changed. Based on the results of SEM characterization, Fe_3O_4 magnetic nanoparticles formed are quite homogeneous, that is marked by grain sizes that are almost uniform and the same color. The average grain size is 193,861 nm. Based on the results of the EDS characterization, the formed Fe_3O_4 magnetic nanoparticles contain the elements Fe, O, Cl, Al and Ti. Based on the results of the VSM test, it was found that the Fe3O4 nanoparticles with a dissolution temperature of 55° C for 75 minutes had a saturation magnetization (Ms) value of 31.0 emu / gram. Whereas remanent magnetization (M_r) and coercivity (H_c) fields were 9.6 emu / gram and 0.02 T., respectively. This shows that the Fe_3O_4 nanoparticles produced were ferrimagnetic materials. The coercivity value obtained is smaller than 0.03 T, so it can be stated that the nanoparticle sample is soft magnetic

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Acknowledgements

Researchers would like to thank DIPA UNY 2019