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Effect of Heating Temperature on Magnetic Properties of Fe_3O_4 Synthesized by Coprecipitation Method

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ABSTRACT

The synthesis of Fe_3O_4 nanoparticles with iron rock raw materials was carried out using a coprecipitation method. Iron rocks were taken from the Surian village, South Solok of West Sumatera. This research was conducted to utilize local materials and produce low cost, varied magnetic materials to be applied to the electric and electronics industries. Iron sand as a base material was obtained by processing the iron rocks through destruction and extraction. Iron sand that has been extracted is reacted with HCL and NH_4OH. Furthermore, the PEG-2000 were added as a template to homogenize and inhibit the growth of particles. Heating temperature variation performed to see the effect of temperature on the magnetic properties of the particles. Heating temperature variations were used at 500℃, 600℃, and 700℃. Phase composition of the samples were confirm using X-ray diffraction method. Characterization of magnetic properties carried out using Vibrating Sample Magnetometer (VSM). The results of magnetic properties show that the saturation magnetization decreases with increasing heating temperature in the range of 32.6883 emu/g, 20,1632 emu/g, and 10.4734 emu/g respectively. The value of coercive force, H_C obtained in the range of 13,840 A/m – 19,120 A/m. The results show that Fe_3O_4 can be used as a magnetic recording material.

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1. INTRODUCTION

Magnetic materials are used to operate electrical motors, generators, and transformer, to store and retrieve information on magnetic tape or in computer, to serve as actuators and sensors, to assist in medical diagnostic devices and for a host of other applications. Magnetic behavior is determined primarily by the electronic structure of material, which provides magnetic dipoles. Interactions between these dipoles determine the type of magnetic behavior that observed. Magnetic behavior can be modified by composition, microstructure, and processing of these basic materials (Askeland, 1994). One of the magnetic materials is magnetite (Fe_3O_4) that has a cubic inverse spinel structure as having been generated by the stacking of close-packed plane of O^{2-} ions. The formula for Fe_3O_4 may be written as $Fe^{2+}O^{2-}$ - $(Fe^{3+})_2\,(O^{2-})_3$ in which the Fe ions exist in both +2 and +3 valence states. There are two types of position that may be occupied by the iron cations. For the first one, the coordination number is 4 (tetrahedral coordination); that is each Fe ion surrounded by four oxygen nearest neighbors. For the others, the coordination number is 6 (octahedral coordination) (Callister, 2001). Magnetite nanoparticles have been interested studied due to wide applications in various fields such as magnetic resonance imaging (MRI), drug delivery (Gupta & Wells, 2004; Majeed et al., 2013) and hyperthermia applications (Lao & Ramanujan, 2004). Magnetite can be produced from magnetic raw

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materials called iron oxide contained in the iron rocks through a certain process. The magnetic raw materials as iron oxide are quite a lot in Indonesia, one of deposits area of iron rock is in Surian village, South Solok of West Sumatera.

Various methods are available for preparing magnetite (Fe_3O_4) nanoparticles, such as microemulsion (Gupta & Wells, 2004), sol-gel (Xu et al., 2006), thermal decomposition (Wang & Jiang, 2009) and coprecipitation method (Pati et al., 2012; Tajabadi & Khosroshashi, 2012; Mascolo et al., 2013). The significant challenges in synthesis procedure of nanoparticle arise from the point that most production method contain environment-unfriendly ingredients, and because of usage of different kind of raw materials, these methods are complicated; these factors increase production cost. Among various synthesis method to produce magnetite nanoparticles, coprecipitation method is simple, popular, versatile, and convenient because the reaction occurs at low temperatures (< 100 C) and technique is capable of producing biocompatible materials in aqueous phase with less impurity and cost-effective (Pati et al., 2012; Tajabadi & Khosroshashi, 2012).

In the present article, magnetite Fe_3O_4 nanoparticle were prepared by coprecipitation method in HCl solution and using NH_4OH as precipitation agent. The PEG-2000 were added as a template to homogenize and inhibit the growth of particles. Furthermore, the heating at temperatures 500℃, 600℃, and 700℃ carried out to see the effect of temperature changes on the magnetic properties of magnetite nanoparticles.

2. METHODS

The iron rocks were crushed into small parts to make it easier in the grinding process. The grinding process carried out using the grinding machine (Los Angeles-LA) and then rotated as much as 300 times. The iron rocks that has become powder and then separated by permanent magnet because magnetic powder attracted to magnet. The magnetite powder was then sieved using a 270 mesh sieve size to obtain finer magnetite (Fe_3O_4) particles.

2.1 Synthesis of Fe_3O_4 Nanoparticles.

The procedure of synthesizing magnetite Fe_3O_4 nanoparticles described as follows: 10 grams of powder Fe_3O_4 dissolved in 20 ml HCl (12M) with vigorous stirring for 60 minutes at a temperature 90 ˚C. The reaction of solution magnetite is complete as follow:

$$3Fe_3O_4(s) + 8HCl(l) \rightarrow 2FeCl_3(l) + FeCl_2(l) + 3Fe_2O_3(s) + 3H_2O(l) + H_2(g) \quad (1)$$

The results of solution was added 25 ml NH_4OH to precipitate the filtrate and aged 30 minutes to obtain a precipitate. The precipitate formed Fe_3O_4 black color is separated from the solution are then rinsed with distilled water three times. Precipitation of magnetite is completed as follow:

$$2FeCl_3\,(l) + FeCl_2\,(l) + H_2O\,(l) + 8NH_4OH\,(l) \rightarrow Fe_3O_4\,(s) + 8NH_4Cl\,(l) + 5H_2O\,(l) \quad (2)$$

PEG-2000 in the form of solids are heated and melted at a temperature of 100 ° C. PEG-2000 which has been melted and then added to precipitate Fe_3O_4 that has been cleaned with distilled water. Furthermore, the heating at temperatures 500℃, 600℃, and 700℃ carried out to see the effect of temperature changes on the magnetic properties of magnetite nanoparticles. The magnetic properties of the magnetite (Fe_3O_4) nanoparticles were measured using a vibrating sample magnetometer (VSM model OXFORD VSM 1.2H) at room temperature (25℃). A vibrating sample magnetometer (VSM) operates on Faraday's Law of Induction, which a changing magnetic field will produce an electric field. This electric field can be measured and give information about the changing magnetic field. A VSM is used to measure the magnetic behavior of magnetic materials.The measured magnetic properties of magnetite (Fe_3O_4) consist of saturation magnetization (M_S), remanent magnetization (M_R), and coercive force (H_C). The samples were prepared in powder form and the measurement was carried out in maximum field of 1 T. Measurement of X-ray diffraction method was carried out to confirm the content of magnetic material as magnetite (Fe_3O_4) contained in iron sand.

3. RESULTS AND DISCUSSION

Fig. 1 exhibits the x-ray diffraction pattern of magnetite bulk material. The diffraction peaks at 2θ = 30,0607° ; 35,4051° ; 43,0527° ; 54,1259° ; 56,9710° and 62,5030° can be assigned to (220), (311), (400), (422), (511), (440) planes of Fe_3O_4 (ICDD 01-071-4918), respectively. This result confirmed the presence of the characteristic planes assigned to cubic spinel of the Fe_3O_4 structure.

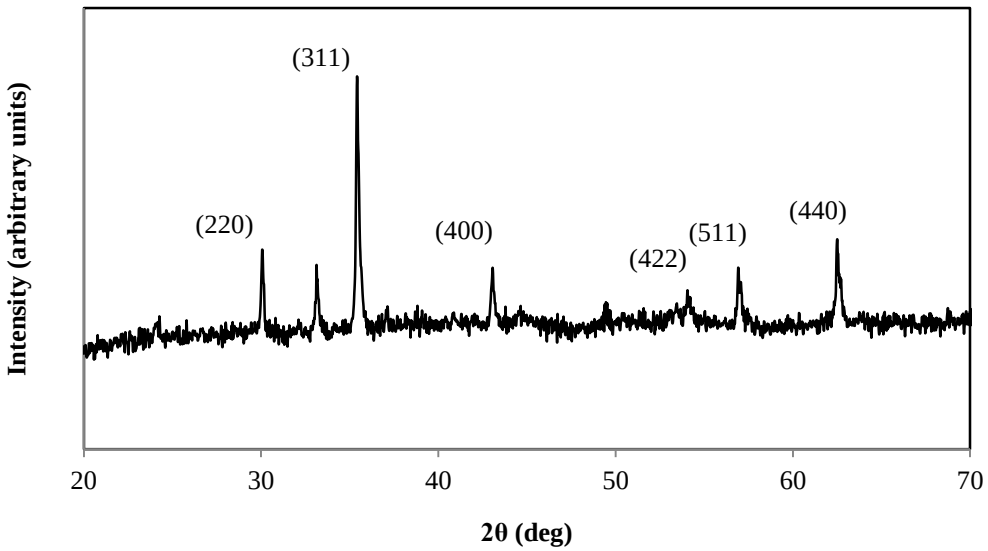

Figure 1. X-ray diffraction pattern of magnetite bulk material.

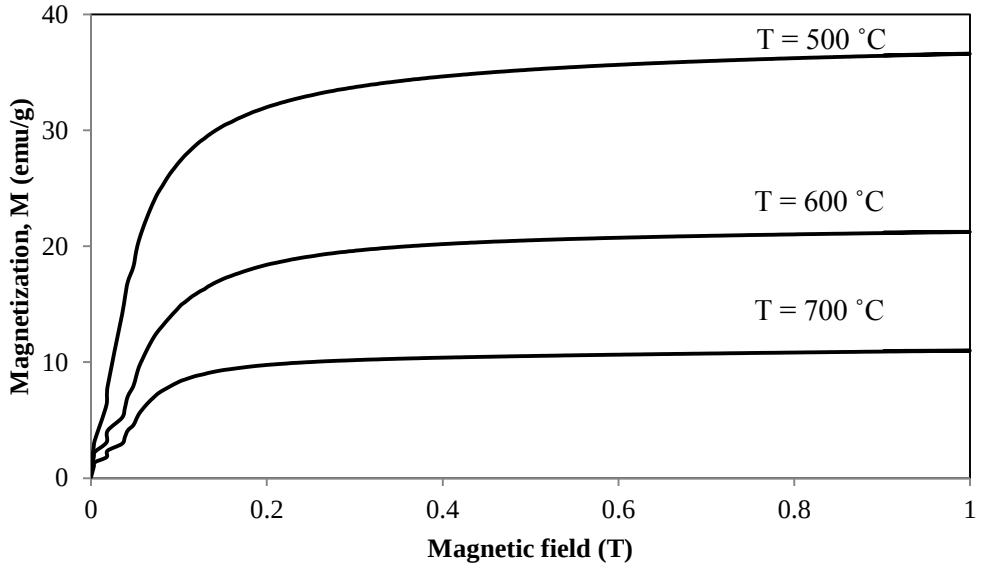

Figure 2. Initial magnetization curve for the magnetite with different temperatures

The magnetization curves for magnetite (Fe_3O_4) with different temperatures are shown in Figure 2. It can be seen that when magnetic field is first applied, the magnetization initially increases slowly, then more rapidly as the domains begin to grow through domain wall motion. Later, the magnetization is slowly increasing as the domains eventually rotate to reach saturation where the

dipoles are optimally oriented (Askeland, 1994). The saturation magnetization (M_S) decreases with increasing heating temperature from 500 °C to 700 °C with a value of 32.6883 emu/g to 10.4734 emu/g respectively. Increasing temperature of solid results in an increase in magnitude of the thermal vibrations of atoms. The atomic magnetic moments are free rotate; hence, with rising temperature, the increased thermal motion of the atoms tends to randomize the directions of any moments that may be aligned. The atomic thermal motions counteract the coupling forces between the adjacent atomic dipole moments, causing some dipole misalignment, regardless of whether an external field present (Callister, 2001). For these samples the decrease in saturation magnetization after increasing temperature is due to the weakening of coupling force between tetrahedral and octahedral site which exchange interaction because of thermal fluctuations can change the direction of magnetic moment and due to substantial conversion of cubic (γ-Fe_2O_3) to hexagonal (α-Fe_2O_3) phase (Pati et al., 2012).

The coercive force (H_C) and remanent magnetization (M_R) for all the samples were determined from magnetic hysteresis curves, shown in Fig. 3. The corresponding results from VSM for all samples are listed in Table 1. Increasing temperatures decreases the remanent of the magnetite. A small remanent magnetization indicates that magnetization diminishes when the external field is removed. These characteristics also lead to small hysteresis loop, as shown in Fig. 3, therefore minimizing energy losses during operation (Askeland, 1994; Callister, 2001).

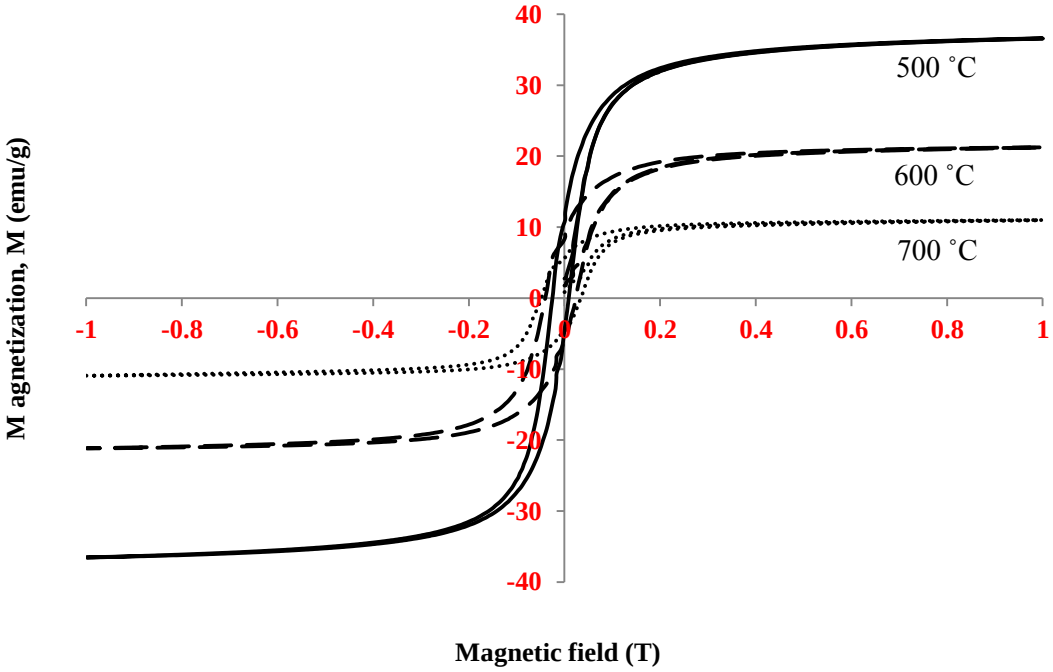

Figure 3. Hysteresis curve for the magnetite with different temperatures

The average value oh H_C is about 16.270 A/m. These value in between these two extremes of soft and hard magnets (10^3 A/m < H_C ≤ 10^5 A/m) and suitable for magnetic recording media (Askeland, 1994; O'Handly, 2000). Figure 3 exhibit small hysteresis loop produced by this material shows that minimize power losses may result from electrical currents that are induced in a magnetic material by a magnetic field. The coercive force value obtain from the hysteresis curve of Fe_3O_4 increases from 13.840 A/m to 19.120 A/m with increasing temperature in the temperature range studied from 500 °C to 700 °C. These results agree with those reported by Xu et al. (2006) on magnetite nanoparticles with synthesized by sol-gel method. The increase in the value of coercive force indicates that the increase in the power to move the domain wall to change the random magnetic domain in each direction of that moment caused by an increase in temperature, which decreases magnetization saturation (Ms) and magnetization remanent (Mr).

Table 1 Magnetization data for various samples measured at different temperatures

Heating temperature (˚C)	M_S (emu/g)	M_R (emu/g)	H_C (A/m)
500℃	32.6883	6.8734	13.840
600℃	20.1632	3.0113	15.120
700℃	10.4734	2.8402	19.120

4. CONCLUSIONS

The magnetic properties of the samples were found affected by the heating temperature. The saturation magnetization and remanent magnetization decreases with increasing temperature otherwise the coercive force value of Fe_3O_4 increases with increasing temperature. The value of saturation magnetization and the remanent magnetization in the range of 10.4734 emu/g – 32.6883 emu/g and 2.8402 emu/g – 6.8734 emu/g respectively. The value coersive force of the samples in the range of 13.840 A/m - 19.120 A/m and suitable for magnetic recording media.

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