Chemistry and Materials Research
ISSN 2224- 3224 (Print) ISSN 2225- 0956 (Online)
Vol.7 No.7, 2015

Synthesis Of Fe_3O_4 Nanoparticles Of Iron Sand Coprecipitation Method With Polyethylene Glycol 6000

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ABSTRACT

This study aimed to obtain a particle size-based nano Fe_3O_4 iron sand, determine the effect of Polyethylene Glycol (PEG) -6000 to be produced from iron sand in synthetic by using coprecipitation method and the nature of magnetization. Synthetic done by mixing natural sand with HCl as solvent and NH_4OH as a precipitant, as templates are added PEG-6000 .. Then, characterized by using X-Ray Diffractometer (XRD). Vibrating Sample Magnetometer (VSM) and Fourier Transform Infra Red (FTIR).
From the test results (XRD) shows the crystal size Preparation Fe3O4 nanoparticles with PEG 6000 (1: 3), (1: 4), (1: 5) in a sequence that is 14.90 nm, 22:16 nm, 33.11 nm while without PEG 6000 29.08 nm ,
Vibrating Sample Magnetometer measurement results (VSM) shows that the value of Ms saturation field for without PEG-6000 Fe3O4, Fe_3O_4 with PEG-6000 (1: 3), (1: 4), (1: 5) respectively 27.9 emu / g, 47.4 emu / g, 35.3 emu / g, 51.7 emu / g and field coercivity (Hc), each for 0,013 Tesla, 0.022 Tesla, Tesla 0.14, 0.009 Tesla.
FTIR results showed that there was a shift peaks indicating increased number of PEG. From each sample came the peak of Fe-O owned by Fe_3O_4 where the wavelength of Fe-O at sample PEG has a lower value than the wavelength Fe_3O_4 caused the PEG
Key words: Nanoparticles Fe_3O_4, PEG 600, particle size, magnetic properties

1. INTRODUCTION .

Nanoparticles are particles with a size of nanometers, which is approximately 1-100 nm. Nanoparticles into a very interesting study, because of the material that is in nano size particles typically have chemical or physical properties that are superior to large-sized material (bulk). In this case these properties can be altered by controlling the size of the material, setting the chemical composition, surface modification and control of the interactions between the particles.

The magnetic nanoparticles has been developed recently is Fe_3O_4, is one phase of the iron oxide is amphoteric and has a high absorption, (Abdillah, G., 2013) . Compounds Fe_3O_4 ($FeO.Fe_2O_3$), black with spinel inversion shaped structure and containing Fe^{2+} and Fe^{3+}, (Gubin, S. F., 2007).

Fe_3O_4 last few years has been widely used in various applications, such as a store of information with a high density, magnetic resonance image formation, delivery systems for medicines, cosmetics, dyes, inks as well as play a role in various separation processes, including adsorption (Y. Wei et al,2011).

The research nanoscale magnetic particles have been carried out, among others, Malik, A., Baqiya, et al , (2007), with PEG-400 and without PEG, (Perdana, F.A. et al , 2013), synthetic Fe_3O_4 with template PEG-1000, using coprecipitation method. (Putri, S.T., 2011), (Nuzzuly, S,et al , 2013), template PEG-4000,(Astuti Geby ,et al ,2013),iron stone Fe_3O_4 using templates PEG-4000.

Fe_3O_4 nanoparticles usually can with some chemical synthesis method, such as, reverse micelle method, microwave plasma synthesis technique sol - gel, freeze drying, irradiaton ultrasound, hydrothermal method, technique of laser pyrolysis, coprecipitation method, and others .
In this study the method to be used is the method of coprecipitation. This method is considered more suitable because it is easier to be done, the materials and procedures used is also much simpler. The advantage of this method is the process uses low temperatures and is easy to control the particle size so that it takes a relatively short time. adapaun purpose of this study to determine the particle size of nano Fe_3O_4 made from iron sand, determine the effect of Polyethylene Glycol (PEG) 6000 and magnetic properties.

2. METHODS

2.1 Tools and materials

Materials; Iron sand, HCL, NH_4OH, Filter paper. PEG 6000
tools used, the 200 mesh sieve, digital magnetic balance Stirer, Vacuum pump, Oven, Shimadzu XRD 6100 X-ray diffractometer, Vibrating Sample Magnetometer (VSM), Bruker FTIR Spectrometer ALPHA

2.2. Synthesis process Fe_3O_4 Method Using coprecipitation

Sand iron (Fe_3O_4) is obtained from separation using a magnet, then sieved to 200 mesh size. then weighed 20 g and inserted with a glass beaker to be mixed with 37% HCl 40 ml. The mixture was stirred at 70 ° C for 30 minutes in a magnetic stirrer. After stirring process is completed, do the filtering with filter paper PEG-6000 melted by heating at 50 ° C for 15 minutes. PEG-6000 which has been melted added to the resulting solution was the variation Fe_3O_4 volume ratio of 1: 3; 1: 4; 1: 5 The results are mixed with a solution of PEG-6000 conducted by stirring by using (magnetic stirrer), at a temperature of 70 ° C for 40 minutes. Then 25% NH_4OH added 30 ml in Fe_3O_4 mixture while stirring and heated by using a magnetic stirrer for 40 minutes at a temperature of 70^0C.

Results precipitate formed Fe_3O_4 (solid black) separated from the solution which was then washed repeatedly using distilled water until clean of impurities then filtered .To obtain nano Fe_3O_4 powder particles, sediment is dried in an oven at a temperature of about 70 ° C for 2 hours.

2.3 Characterization XRD

Characterization X-Ray Diffractometry (XRD), which is used in room temperature by using a Shimadzu XRD 6100 X-ray diffractometer (40 kV, 30 mA), Cu Kα radiation which is used scanning rate of 2^0 / min in the range of $2\theta = 5^0 - 70^0$

2.4 Characterization Vibrating Sample Magnetometer (VSM)

By using Vibrating Sample Magnetometer (VSM) can show the relationship magnetization (M) with an external magnetic field (H) obtained from the hysteresis curve. Characterization is done at the Laboratory of Magnetic - PT BIN-BATAN using Vibrating Sample Magnetometer (VSM) Oxford types VSM 1.2H

2.5 Characterization FTIR

Characterization by using the tool Bruker FTIR Spectrometer ALPHA of spectroscopy was obtained graph of transmittance against the wavelength, the wave crests obtained from functional groups.

3 RESULTS AND DISCUSSION

3.1 Analysis XRD

XRD characterization performed to analyze the crystal structure of the sample Fe_3O_4. From the XRD data obtained, to identify peaks of XRD chart by matching the existing peak on the graph with the ICDD database. After that, the XRD data refinement using Rietveld analysis contained in Rietan program. Through the refinement, and its phase structure and lattice parameters that exist in the sample are known. Through the XRD chart, grain size of the sample can also be estimated. Grain size is calculated using the Scherrer. of the x-ray diffraction pattern.

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$$D = (\mathrm{k}\,\lambda)/(Br\ Cos\ \theta) \qquad (1)$$

With Br, K, λ and D, respectively half peak width (FWHM) in radians, Scherrer constant (0.9), the wavelength of the x-ray (1.5406 Å), and the diameter of crystallites in the size of nanometers (nm). to determine: (%) Volume fraction of magnetite (Fe_3O_4) = (intensity phase sought) / (number-intensity x-ray detectable phase) x 100% (2)

With the analysis of the data obtained as shown in Table 1

Table 1 .Analysis Data with XRD

Samples	Size (nm)	Density (g/cm^3)	Phases Fe_3O_4(%)	other Phases (%)	Cubic crystal structure with parameters a (Å)
Fe_3O_4 Non PEG 6000	29.08	5,181	38,47	61,53	8.4045
Fe_3O_4 With PEG 1:3	14.90	5,219	48,39	51,61	8.3837
Fe_3O_4 With PEG 1:4	22.16	5,272	44,41	55,59	8.3557
Fe_3O_4 With PEG 1:5	33.11	5,200	53,2	46,8	8.3557

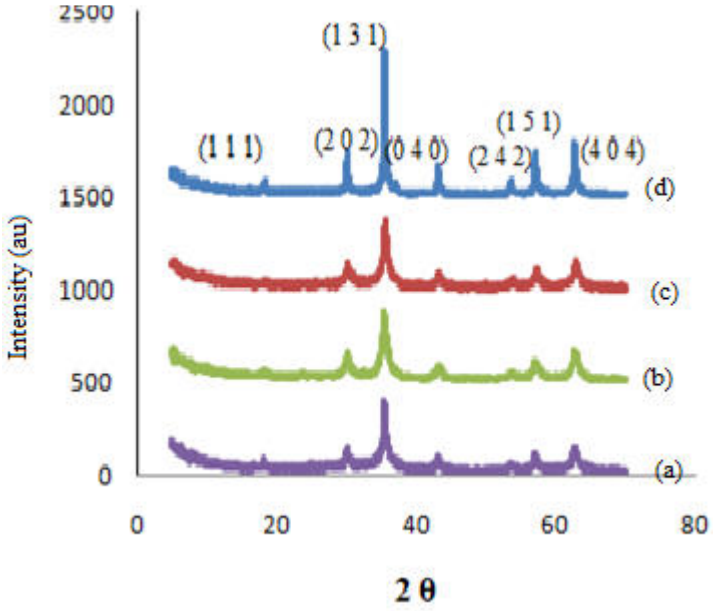

Figure. 1. Pattern XRD for Fe_3O_4 samples with multiple treatment,(a) non PEG, (b)with PEG (1:3) ,(c) with PEG (1:4), (d) with PEG (1:5)

From Figure 1 shows the X-ray diffraction pattern of the sample with a variation ratio of PEG-6000 and without PEG-6000. From the figure it can be seen that the peak formed a widening with increasing volume expansion ratio variation PEG-6000 which indicates the size of the crystals get smaller. Based on the analysis using search programs match and qualitative analysis using the method Hanawalt, that the sample containing 100% Fe_3O_4 phase. Not found also the PEG phase in the sample. This means that PEG-6000 did not come to react and simply act as a template only. as well as the results of the study (Perdana, F.A. et al , 2013).

3.2. Analysis Vibrating Sample Magnetometer (VSM) .

Magnitudes important in determining the magnetic properties by magnetic hysteresis curve is the saturation (Ms), field coercivity (Hc) and the remanent magnetization (Mr). The value of saturation magnetization, known as saturated magnetization demonstrate the ability of nanoparticles to maintain irreversibility magnetic domains when they are known outside the magnet. Coercivity field is a field magnitude needed to make the magnetization is zero. The greater the value the stronger is the nature of magnetism. While the remanent magnetic material showed ability when given external field.
In the second picture we can see the comparison curve Hysteresis curve magnetite Fe3O4 Nano Particles

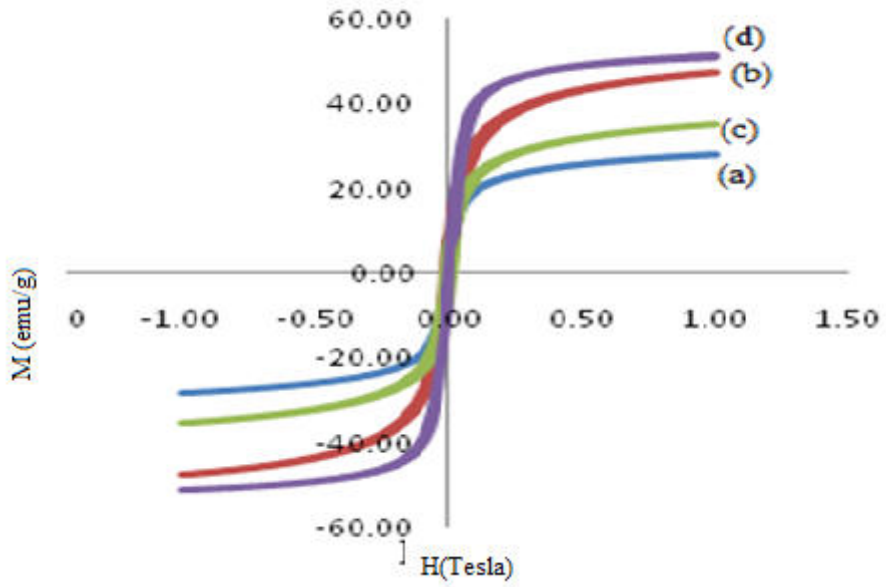

Figure 2 VSM images of Fe_3O_4,wit Diffrent Ratios of Fe_3O_4 and PEG 6000
(a) Without PEG 6000 , (b) 1:3 ,(c) 1:4 ,and (d) 1:5

For more details, the value of saturation magnetization, coercivity field and remanent magnetization of each sample are shown in Table 4.2.

Table 2. The Data Analysis Results VSM

Samples	Size (nm)	Ms (emu/g)	Hc (Tesla)	Mr (emu/g)
Fe_3O_4 non PEG	29.08	27,9	0,013	10.3
Fe_3O_4 with PEG 1:3	14.90	47,4	0,022	11,7
Fe_3O_4 with PEG 1:4	22.16	35,3	0,14	12,3
Fe_3O_4 with PEG 1:5	33.11	51,7	0,009	18,1

From Table 2 it can be seen also that the value of Hc (coercivity field) Fe_3O_4 without PEG-6000 obtained around 0,033Tesla while Fe_3O_4 with the addition of PEG-6000 (1: 3), (1: 4), (1: 5) consecutive which is about 0,022 T, 0.14 T, 0,009 T. From Table .2 shows a strong relationship between the size of the crystals with magnetic properties.
This research is in accordance with previous studies which, saturation magnetization (Ms) for Fe_3O_4 powder with PEG-400 higher compared without PEG-400 (Malik, et al., 2007) but not in accordance with research that states the value of remanent magnetization of Fe_3O_4 without PEG-1000 higher than Fe_3O_4 with the addition of PEG-1000. In other words, the value of Hc (coercivity field) and Mr (remanent magnetization) tends to decrease with the decrease of crystal size (Perdana, et al., 2013).

From Table 2 can be seen the value of Hc is inversely proportional to the size of the crystal, or in other words, the larger the crystal size smaller then the value Hc. With a little more explanation crystal size means more boundaries between the crystal and the more barriers domain wall motion so that the resistance to demagnetization fields which means the greater the higher the value Hc. Instead the larger crystal size, the easier it is to move the domain walls so that the resistance to demagnetization magnetic field is getting smaller, which means the lower the value Hc.

It can be concluded that the samples Fe_3O_4 with Peg-6000 (1: 3), which has the smallest size and has a larger magnetic properties compared to other large particle size resulting from Fe_3O_4 with Peg-6000 (1: 4), Fe_3O_4 with Peg-6000 (1: 5) and Fe3O4 without PEG-6000.

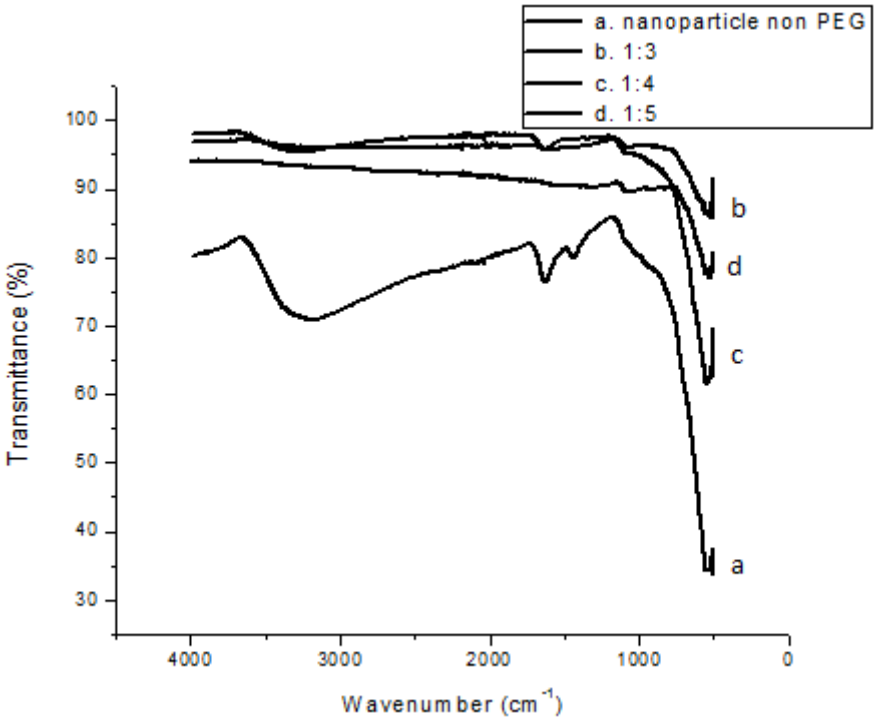

Figure 3 . The results Fe3O4 Characterization Using FTIR
(a) Without PEG 6000 , (b) 1:3 ,(c) 1:4 ,and (d) 1:5

3.3 Analysis Fourier Transform Infra Red (FTIR)

FTIR results on a sample Figure 3 showed the presence of peaks 3172.64 which showed the presence of OH groups. 540.38 peak showed the presence of clusters Fe – O
In the sample b shows the peak 3114.13 which is the OH group which indicate the presence of PEG. 1613.78 peaks showed the presence of carboxylic groups that are building blocks of PEG . Peak of 1077.53 is a hydroxyl group bonded to hydrogen in the surface of the iron oxide, as well as water molecules adsorbed on the surface of the magnetic particles 513.21 peak shows the Fe group - O .
At the peak of 3229.15 sample c shows that an OH group which indicate the presence of PEG. 1640.04 peaks showed the presence of carboxylic groups that are building blocks of PEG. peak of 529.84 showed the presence of clusters Fe - O owned by Fe_3O_4
At the peak of 3819.90 sample d appears that the OH group which indicate the presence of PEG . Peak of 1069.13 is a hydroxyl group bonded to hydrogen in the surface of the iron oxide, as well as water molecules adsorbed on the surface of the magnetic particles . 529.38 peak shows the Fe group – O. (Zhao, et al., 2010) (Lopez, et al.2010).
From the results of FTIR showed that there was a shift peaks indicating increased number of PEG. From each sample came the peak of Fe-O owned by Fe_3O_4 where the wavelength of Fe-O at sample PEG has a lower value than the wavelength Fe3O4 caused the PEG .

4. CONCLUSION

The results of testing X-Ray Diffractometer (XRD) to Fe3O4 without template PEG-6000 showed crystal size Preparation Fe3O4 nanoparticles with PEG 6000 (1: 3), (1: 4), (1: 5) which is 14.90 nm sequentially, 22:16 nm, 33.11 nm without PEG 6000 while the 29.08 nm.

Vibrating Sample Magnetometer measurement results (VSM) shows that the value of Ms saturation field for without PEG-6000 Fe_3O_4, Fe3O4 with PEG-6000 (1: 3), (1: 4), (1: 5) respectively 27.9 emu / g, 47.4 emu / g, 35.3 emu / g, 51.7 emu / g and field coercivity (Hc), each for 0,013 Tesla, Tesla 0.022, 0.14 Tesla, Tesla 0,009 FTIR results showed that there was a shift peaks indicating increased number of PEG. From each sample came the peak of Fe-O owned by Fe_3O_4 where the wavelength of Fe-O at sample PEG has a lower value than the wavelength Fe_3O_4 caused the PEG

ACKNOWLEDGMENTS

The author would like to thank the Ministry the Higher Education research and technology at the expense of fundamental research grant budget year 2015 with contract: 016A / UN33.8 / KU / 2015 and Laboratory of Physics and Chemistry Laboratory facility Unimed

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