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Study and Characterization of Fe_3O_4-PEG Nanoparticles Using The Co-Precipitation Method For The Production of Permanent Magnets

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ABSTRACT

Natural iron sand is one of the natural resources in Indonesia, especially in the Cianjur area, West Java which has been used optimally. This study aims to analyze Fe's content, properties, and grain size found in Cianjur, West Java. The natural iron sand samples were prepared using the calcination method, which was dried at 50°C until the samples became powder. Beach sand samples are extracted using a permanent magnet to separate magnetic and non-magnetic materials. Characterization of iron sand using XRD, SEM, VSM, and FTIR. The XRD results show that the natural iron sand sample has a single magnetite phase (Fe_3O_4). A spinel cubic crystal structure is formed with lattice parameters of 8,513 Å using Co-Precipitation. SEM results show that the sample is inhomogeneous or homogeneous, as indicated by the gap and agglomeration of particles in each sample. VSM results show that the parameter magnetic properties saturation (M_s) on average is 23.9763735 emu/g, magnetic remanence (M_r) is on average 5.14865198 emu/g, and coercivity is on average 125.139457033 emu/g. Where in sample 1, saturation is 29.7729509 emu/g, remanance is 4.0486018 emu/g, coercivity is 92.1368641 emu/g, sample 2 is saturation 21.5994425 emu/g, remanance 8.18772602 emu/g, coercivity 179.567079 emu/g and sample 3 has a saturation value of 20.55672771 emu/g, remanance 3.20962812 emu/g. Then, the FTIR results showed a shift in the vibration peak, which experienced a change in the vibrational energy of Fe-O and then showed the -OH group at an absorption of 3400/cm. The results of this study have the potential to process other magnetic materials.

Keyword: Fe_3O_4, particle clusters, particle morphology, magnetic properties, and particle structure.

ABSTRAK

Pasir besi alam merupakan salah satu sumber daya alam di Indonesia khususnya pada daerah Cianjur, Jawa Barat yang telah dimanfaatkan secara optimal. Penelitian ini bertujuan untuk menganalisis kandungan, sifat dan ukuran butir Fe yang terdapat di Cianjur, Jawa Barat. Pembuatan sampel pasir besi alam ini menggunakan metode kalsinasi yang dikeringkan pada suhu 50°C hingga sampel menjadi serbuk. Sampel pasir pantai diekstraksi menggunakan magnet permanen untuk memisahkan material magnetik dan non magnetik. Karakterisasi pasir besi menggunakan XRD, SEM, VSM, dan FTIR. Hasil XRD menunjukkan bahwa sampel pasir besi alam memiliki fasa tunggal yaitu fasa magnetite (Fe_3O_4), dan terbentuk struktur kristal kubik spinel dengan parameter kisinya 8.513 Å menggunakan Co-Presipitasi. Hasil SEM menunjukkan bahwa sampel tidak

homogen ataupun homogen yang ditunjukkan oleh adanya suatu celah dan aglomerasi partikel pada setiap sampel yang ada. Hasil VSM menunjukkan bahwa parameter sifat magnetik saturasi (M_s) rata-rata sebesar 23,9763735 emu/g, magnetik remanansi (M_r) rata- rata sebesar 5,14865198 emu/g dan koersivitas rata-rata sebesar 125,139457033 emu/g. Dimana pada sampel 1 bernilai, saturasi 29,7729509 emu/g, remanansi 4,0486018 emu/g, koersivitasi 92,1368641 emu/g, sampel 2 bernilai, saturasi 21,5994425 emu/g, remanansi 8,18772602 emu/g, koersivitas 179,567079 emu/g dan sampel 3 bernilai saturasi 20,55672771 emu/g, remanansi 3,20962812 emu/g. Kemudian, hasil FTIR menunjukkan bahwa terjadi pergeseran puncak vibrasi yang mengalami perubahan vibrasi energi Fe-O lalu menunjukkan gugus –OH pada serapan 3400/cm. Hasil studi ini berpotensi dalam pengolahan material magnetik lainnya.

Kata Kunci: Fe_3O_4, Metode Kopresipitasi, Morfologi, Kristal Struktur, Magnet Karakteristik

1. Introduction

Fe_3O_4 nanoparticles have a great advantage in salinity technology, so this new material development is urgently needed concerning the exploration of raw materials, synthesis methods used, required calcification methods, and signal amplification [1] – [4]. In terms of basic ingredients, of course, the materials used are synthetic and alternative ones that are environmentally friendly and low in toxins. While in the main problems regarding the method, it is probable that the method used in the restoration, the method synthesis, which results in a monodisperse scalable nanoparticle product in the method used and also allows a single domain from the behavior of the particles in the method used, also requires a method that is environmentally friendly and does not use the balance of alcohols in the synthesis process.

Fe_3O_4 is a black powder material with strong magnetic properties. In addition, magnetite also exhibits super-parallel magnetic behavior for grain sizes below 10 nm. Polyethylene glycol 6000 is polyethylene glycol $H(O\text{-}CH_2\text{-}CH_2)_nOH$, and the value of n is between 158 and 204 [5]. It has the following characteristics: smooth white powder and ivory-yellow white pieces, practically unbounded in origin. Solubility is easily soluble in flow, in ethanol (95%) chloroform, and in ether. The average molecular weight is not less than 7000 and not more than 9000. Its efficacy is as an additive [6]

This research was carried out to disperse Fe_3O_4 nanoparticles with the variation of the composition of PEG-6000 using the co-precipitation method. The test used XRD, SEM, VSM, and FTIR.

2. Method

2.1 Research Procedures

In this research, the process of synthesizing natural iron palsy with polyethylene glycol (PEG) was carried out using the Co-Precipitation method. The research includes raw material preparation, precipitate drying, calcination, and calcification processes.

2.2 Preparation of Nanoparticle of Fe_3O_4/PEG

In this research, the synthesis process of Fe_3O_4/PEG nanoparticles was carried out using the co-precipitation method. According to the ratio, the material used to manufacture Fe_3O_4 nanoparticle powder is a natural iron pallant in PEG. First, mix natural iron and PEG into 50 ml of DI water, then stir using a magnetic stirrer for 1 hour at a speed of 300 rpm at a temperature of 50°C to produce a homogeneous solution. The homogeneous solution was then forged into a solution of NH_4OH in DI water with a concentration of 6.5 M. Then the mixed solution was stirred for 1 hour at a speed of 500 rpm and a temperature of 50°C. Then the result of the mixture will produce a precipitate powder. Next, the precipitate was separated from the impurities using a permanent magnet and washed with DI water; the wet powder precipitate was washed with 10 ml of ethanol and heated on a magnetic stirrer at 50°C; after the colloid was formed, it was dried in an oven for 1 hour at 50°C. After the effluent dried, the powder was ground using a mortar to produce Fe_3O_4/PEG nanoparticle powder, then characterized using XRD, SEM, VSM, and FTIR

tests until complete. There are three different samples made based on the composition of the Fe_3O_4:PEG, namely, sample 1 (Fe_3O_4: PEG = 2: 8), sample 2 (Fe_3O_4: PEG = 8: 2), and sample 3 (Fe_3O_4: PEG = 10: 0).

2.3 Characterisation of Fe_3O_4/ PEG Nanoparticle

FTIR tested the structure of chemical compounds or chemical groups of Fe_3O_4/PEG nanoparticles. To evaluate the effect of composition on structure in identifying the crystalline phase of Fe_3O_4/PEG nanoparticles using X-Ray Diffraction (XRD). The effect of magnetic properties on the molar ratio of nanoparticles Fe_3O_4/PEG was tested using a vibrating sample magnetometer (VSM). The morphological properties of the material were measured using SEM.

3. Results and Discussion

3.1 X-Ray Diffraction (XRD) Analysis

Figure 1 shows the XRD patterns of the samples. Each material shows hkl peaks (110), (200), (311), (222), (400), (422), and (511). One of the characteristics of phase Fe_3O_4 is that it has the highest intensity at 34.59 with the hkl peak point (311). The PEG phase did not appear in the XRD test, so no reaction occurred and functioned as a template. The width or height of intensity in the XRD test results also determines the size of a crystal. The higher the narrowing of a peak, the greater the crystallite size, and vice versa. If the peak narrows shorter and lower, then it can be said that the crystallite size is small [7].

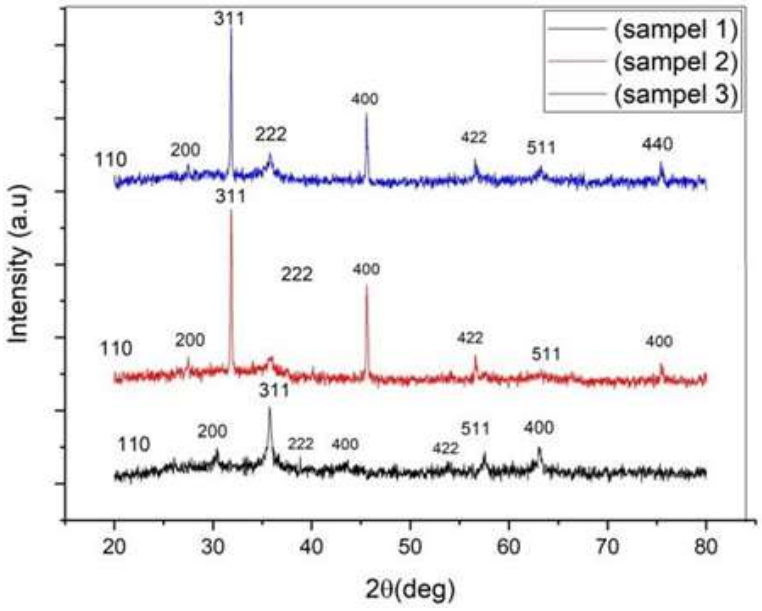

Figure 1. XRD pattern of Fe_3O_4 with PEG-6000 (JCPDS 19-0629)

Table 1. Analysis of XRD Fe_3O_4 with PEG-6000 of sample 1, 2, 3

Sample	Sample composition (Fe_3O_4: PEG)	2θ (deg)	Intensity (a.u.)	FWHM (deg)	θ (rad)	Lattice Parameter (nm)	Grain Size (nm)
1	2:8	31.84	1282.75	0.70822	0.2777	52.64	11.2330
2	8:2	30.39	1282.75	0.70822	0.2650	55.17	11.2323
3	10:0	31.85	1282.75	0.70822	0.2778	52.62	11.2322

3.2 Scanning Electron Microscope (SEM) Analysis

The SEM results are given in Figures 2, 3, and 4. Based on Figure 2, The agglomeration in sample 1 has been evenly distributed and not clustered, making it easier to identify the particles; generally, agglomeration occurs because of the high temperature, where the agglomeration is called the formation of particles. So the histogram on the particle size of the first sample is good. The particle distribution ranges from 123-138 with a frequency of 2.0, where the frequency is the number of frequencies in 1 nm.

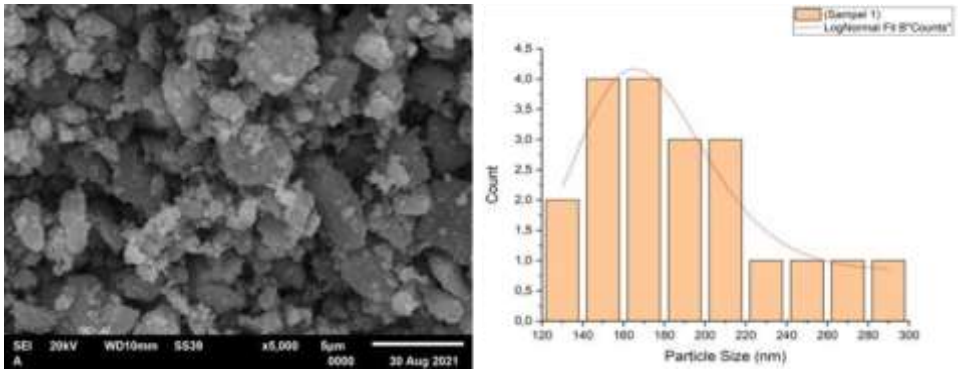

Figure 2. SEM Picture with the magnification of 5000 times and histogram with Type (Al) Fe_3O_4-PEG (sample 1)

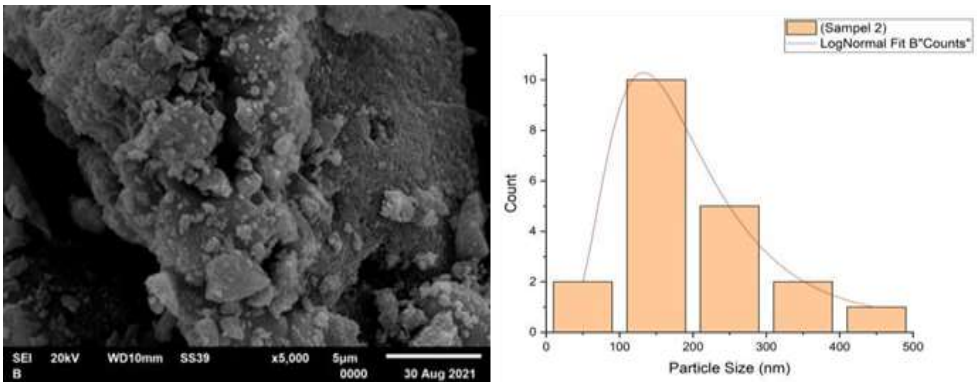

Figure 3. SEM Picture with the magnification of 5000 times and histogram with Type (A2) Fe_3O_4-PEG (sample 2)

Based on Figure 3, the agglomeration in sample 2 has also been evenly distributed and not clustered, making it easier to identify the particles, in which case the agglomeration occurs due to the high temperature where the agglomeration is called particle formation. So that the histogram on the particle size of the first sample is good because the R-Square in $X_{c,}$ which is refined, has a value; R-Square = 0.99996; X_c = 172.08928 ± 1.00923 because the acceptable range of R-Square values ranges from 0.85 – 1.0 so that it fulfills the conditions of the histogram. The particle distribution ranges from 10-80, having a frequency of 2 in terms of frequency, namely the number of frequencies in 1 nm.

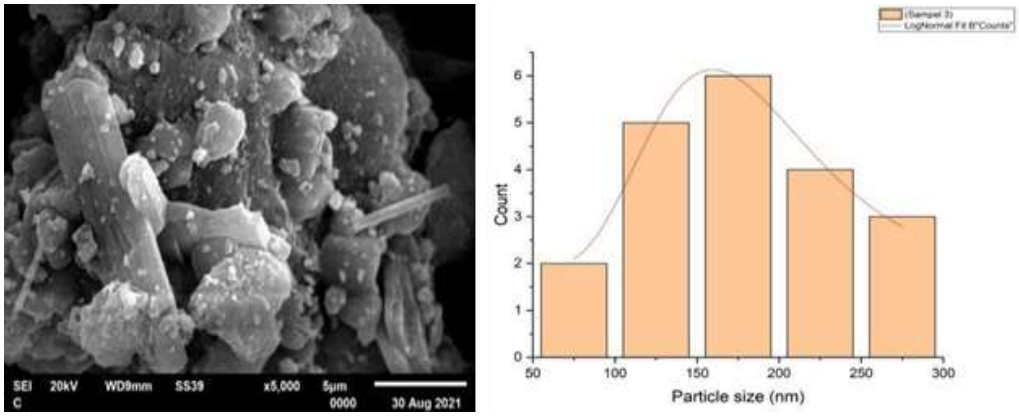

Figure 4. SEM Picture with the magnification of 5000 times and histogram with Type (A3) Fe_3O_4-PEG (sample 3)

Based on Figure 4, the agglomeration of sample 3 has also been evenly distributed even though some are clustered, but it can make it easier to identify the particles where the agglomeration occurs due to the high temperature; the agglomeration is called particle formation so that the histogram of the particle size of the first sample is acceptable. The particle distribution ranges from 60-96 with a frequency of 2.

3.3 Vibrating Sample Magnetometer (VSM) Analysis

Figure 5 depicts the hysteresis of nanoparticles Fe_3O_4-PEG. According to [8], the hysteresis curve has experienced the particle effect. Particles also tend to affect the magnitude of the curvature of hysteresis in accordance with [9], even for all particle sizes above 40 nm in the presence of permanent magnetic magnets.

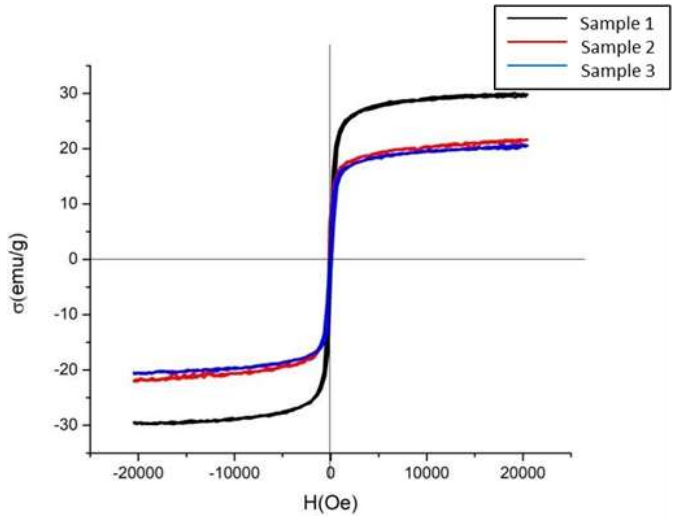

Figure 5. Hysteresis of nanoparticles Fe_3O_4-PEG

Table 2. Data of testing results from VSM

Sample	Sample Composition Fe_3O_4: PEG	Saturation, M_s (emu/g)	Remanance, M_s (emu/g)	Coercivity, M_r (emu/g)	B_h Max (kOe)
1	2: 8	21.59	8.18	179.56	20.289
2	8: 2	29.77	4.04	92.13	20.261
3	10: 0	20.55	3.20	103.71	10.00

Based on Table 2, in compositions 8:2 and 10:0, there is an increase in the value of coercivity; this occurs due to agglomeration in the powder. In the saturated value, there is a gradual decrease in remanence and coercivity that fluctuates up and down due to agglomeration and gaps between particles in the three samples.

Then there is a decrease in the value of ups and downs in the coercivity affected by the size of a particle. The smaller the particle size, the easier it is for the material to crack and release its magnetic properties when the magnetic field is recognized from the outside and vice versa [10].

3.4 Fourier Transform Infra-Red (FTIR) Analysis

FTIR is one tool widely used to determine molecular vibrations that can be used to predict the structure of chemical compounds or chemical groups. Therefore, focusing on the position of the sample and then focusing on the detector is a combination of different wavelengths. Wavelength used was 500-4000 (/cm).

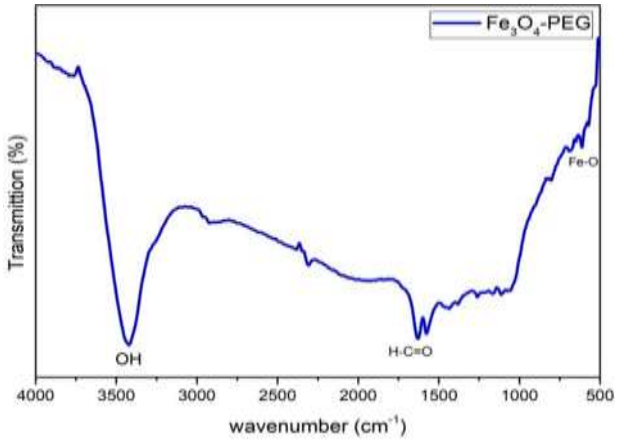

Figure 6. FTIR spectrum of Fe_3O_4 with additions of PEG

Figure 6 depicts the FTIR spectrum of Fe_3O_4 with additions of PEG. Based on Figure 6, there is a shift in peak vibration caused by changes in the energy of the Fe-O vibrations located in absorption between 500-1000 cm, allowing PEG absorption by Fe atoms to occur. It has been synthesized in the presence of ultrasonic waves. However, the FTIR spectra show that there is another absorption at 3400 cm^{-1} which is indicated to be from the –OH group of water, and absorption in the 1600 cm^{-1} area, which is considered to arise from an impurity H-C=O.

4. Conclusion

In summary, the structure of the Fe_3O_4-PEG nanoparticles produced using the Co-Precipitation method has a single phase, namely the magnetite phase (Fe_3O_4), and forms a spinel cubic crystal structure with a lattice parameter of 8.513 Å. The results of the SEM test show that the sample is not homogeneous or homogeneous, as indicated by the presence of a gap and agglomeration of particles in each sample. Magnetic properties of nanoparticles Fe_3O_4-PEG with VSM testing showed that the parameters of the magnetic properties of saturation (M_s) averaged 23.9763735 emu/g, magnetic remanence (M_r) averaged 5.14865198 emu/g with an average coercivity of 125.139457033 emu/g. The functional group of nanoparticles Fe_3O_4-PEG from the FTIR test showed a shift in the vibration peak, which experienced a change in the vibrational energy of Fe-O at an absorption of 500-1000, then showed a –OH group at an absorption of 3400/cm.

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