talenta PUBLISHER

Journal of Technomaterial Physics
Journal homepage: https://talenta.usu.ac.id/jotp

Fabrication and Characterization of SiO_2-Fe_3O_4 Using Co-Precipitation Method

Timbangen Sembiring * and Ema Susantika Perangin-angin
Department of Physics, Faculty of Mathematics and Natural Sciences, Universitas Sumatera Utara, Medan, 20155, North Sumatra, Indonesia
*Corresponding Author: timbangen@usu.ac.id

ARTICLE INFO

Article history:
Received 04 February 2023
Revised 17 February 2023
Accepted 24 February 2023
Available online 28 February 2023

E-ISSN: 2656-0755
P-ISSN: 2656-0747

How to cite:
T. Sembiring and E. S. Perangin-angin, "Making and Characterization of SiO_2-Fe_3O_4 Using Method CO-Precipitation," Journal of Technomaterial Physics, vol. 05, no. 01, Feb. 2023, doi:10.32734/jotp.v5i1.9887.

ABSTRACT

The fabrication of SiO_2-Fe_3O_4 has been successfully carried out using the co-precipitation method. The main materials used are silicon dioxide (SiO_2) and natural sand (Fe_3O_4). The calcination was conducted at 180℃ for 2 hours. The morphology, crystal structure, and magnetic properties were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), and vibrating sample magnetometer (VSM). The XRD pattern shows the effect of the material composition on the crystal structure of SiO_2-Fe_3O_4. The grain size of SiO_2-Fe_3O_4 crystals decreases with the increasing composition of silicon. Results of SEM-EDX show that the composition on morphology SiO_2-Fe_3O_4 is a silicon composition. The VSM result shows that sample 3 (composition 7:3) yielded a coercivity value of 114.60 Oe, remanence of 38.62 emu/g, and magnetization saturation of 76.26 emu/g. The distribution of particle size is 70 nm.

Keyword: SiO_2-Fe_3O_4, Co-precipitation Method, Morphology, Structure Crystal, Magnetic Characteristic.

ABSTRAK

Pembuatan SiO_2-Fe_3O_4 telah berhasil dilakukan dengan menggunakan metode kopresipitasi. Bahan utama yang digunakan adalah silikon dioksida (SiO_2) dan pasir alam (Fe_3O_4). Kalsinasi dilakukan pada suhu 180℃ selama 2 jam. Morfologi, struktur kristal, dan sifat kemagnetan dikarakterisasi menggunakan *X-ray diffraction* (XRD), *scanning electron microscopy* (SEM), dan *vibrating sample magnetometer* (VSM). Pola XRD menunjukkan pengaruh komposisi material terhadap struktur kristal SiO_2-Fe_3O_4. Ukuran butir kristal SiO_2-Fe_3O_4 mengecil dengan bertambahnya komposisi silikon. Hasil SEM-EDX menunjukkan bahwa komposisi pada morfologi SiO_2-Fe_3O_4 merupakan komposisi silikon. Hasil VSM menunjukkan sampel 3 (komposisi 7:3) menghasilkan nilai koersivitas 114,60 Oe, remanensi 38,62 emu/g, dan saturasi magnetisasi 76,26 emu/g. Distribusi ukuran partikel adalah 70 nm.

Kata Kunci: SiO_2-Fe_3O_4, Metode Kopresipitasi, Morfologi, Kristal Struktur, Magnet Karakteristik.

1. Introduction

Magnetic nanoparticles can be developed due to their nature and great potential in various fields of application. In addition, magnetic nanoparticles are used to remove toxic elements, while the nanomagnetic film is used in electrical and electronic devices, sensors, storage pan digital density tall, and shield electromagnetic [1] – [3].

Iron sand is a natural resource that is abundant in Indonesia. Iron sand is widespread along the south and north coasts of Java. Until now, sand iron has been mined as an ingredient raw and for sale as ingredients raw [4]. Silica has a characteristic that is more stable in acidic conditions and has a hydroxyl group of Fe_3O_4. Also, SiO_2 particles are non-toxic and biocompatible; they are used as components of supplement vitamins and ingredients in addition to food. Nanoparticles SiO_2 also own the characteristic of being hydrophilic due to a silanol group on the surface [5].

Based on the above explanation, this research aimed to fabricate and characterize SiO_2-Fe_3O_4 using the co-precipitation method. The characterization was conducted using X-ray diffraction (XRD), scanning electron microscopy (SEM), and a vibrating sample magnetometer (VSM).

2. Method

The main raw materials used in this research are silicone dioxide (SiO_2) and sand iron (Fe_3O_4). Firstly, SiO_2 was crushed using a mortar cup, sieved with sift 120 mesh, and weighed according to the weight listed in grams. In addition, NaOH (1M) is also required as a precipitant during the synthesis, and DI water as a solvent. Table 1 shows the ratio of the mass of raw materials to be used.

Table 1. The Ratio of SiO_2:Fe_3O_4

Sample Code	The ratio of SiO_2:Fe_3O_4 (g)
1	3:7
2	5:5
3	7:3

The synthesis was conducted by mixing 3 grams of Fe_3O_4 and 50 mL HCL (37%) and stirring for 30 minutes. Next, the solution was filtered through paper strain, mixed with 7 grams of SiO2, and stirred for 30 minutes. The resulted solution was then added to 150 mL of NaOH (1M) drop by drop while stirring at 700 rpm and at 180°C for 2 hours. Next, the sample was washed using DI water and ethanol to neutralize the pH and remove salt which formed during the reaction. Next, the solution was precipitated with a centrifuge. The same procedure was conducted for different ratios listed in Table 1.

The drying process was conducted using an oven at 200 °C for 2 hours and calcination at 180°C for 2 hours. The resulted powders were characterized using X-Ray diffraction (XRD), scanning electron microscopy (SEM), and vibrating sample magnetometer (VSM).

3. Results and Discussion

3.1 XRD Analysis

X-Ray Diffraction (XRD) measurement was used to determine the diffraction patterns of the samples. This test is carried out to analyze the phase, the height of peaks, and the crystal structure from the samples. Figure 1 depicts the XRD patterns of the samples.

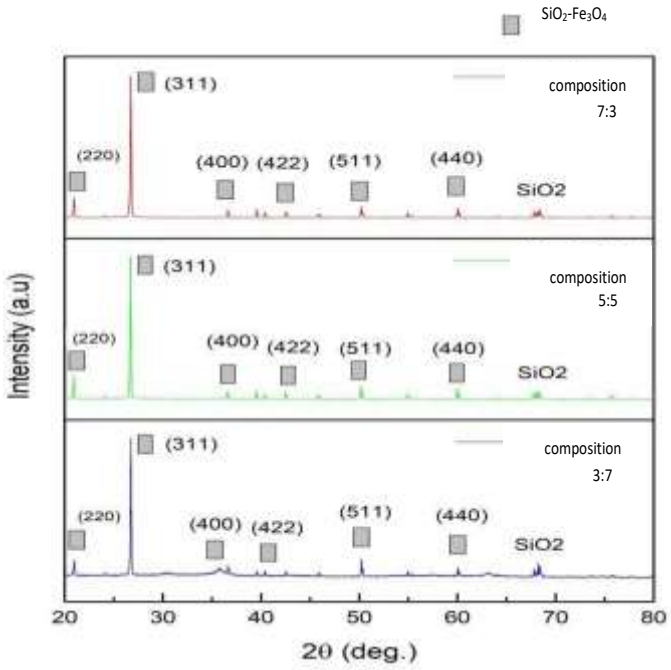


Figure 1. XRD pattern of SiO_2-Fe_3O_4 with different ratios

From Figure 1, the Bragg peaks are indexed as magnetite based on JCPDS no. 190629. The Miller Indices (hkl), which identified the SiO_2-Fe_3O_4 peaks, is (220), (311), (400), (422), (511), and (440). Based on the Scherrer equation (1), the average crystal size is around 60 nm [6].

$$D = \frac{k}{\beta \text{Cos } \theta} \quad \ldots\ldots(1)$$

Table 2. The calculated analysis of the (311) peak

Sample	Sample Composition	2θ (deg)	Intensity (a.u.)	FWHM (deg)	θ (rad)	Crystal Parameter (nm)	Crystal Size(nm)
1	3:7	26.73	7796.29	0.125	0.23	62.77	62.87
2	5:5	26.68	47927.46	0.123	0.23	62.88	64.04
3	7:3	26.73	49262.99	0.130	0.23	62.77	60.45

The obtained crystal grains size is shown in Table 2; The crystal sizes of samples 1, 2, and 3 are 62.87 nm, 64.04 nm, and 60.45 nm, respectively. The crystal structure is found to be the cubic crystal structure. The highest peak for samples 1, 2, and 3 is 26.73°, 26.68°, and 26.73°, respectively. In addition, the peaks in the range of 2θ of 20° - 30° are widened, implying the existence of more Fe particles in the silica; this result is strengthened by SEM-EDX analysis. Besides, no agglomeration occurs in the samples [7].

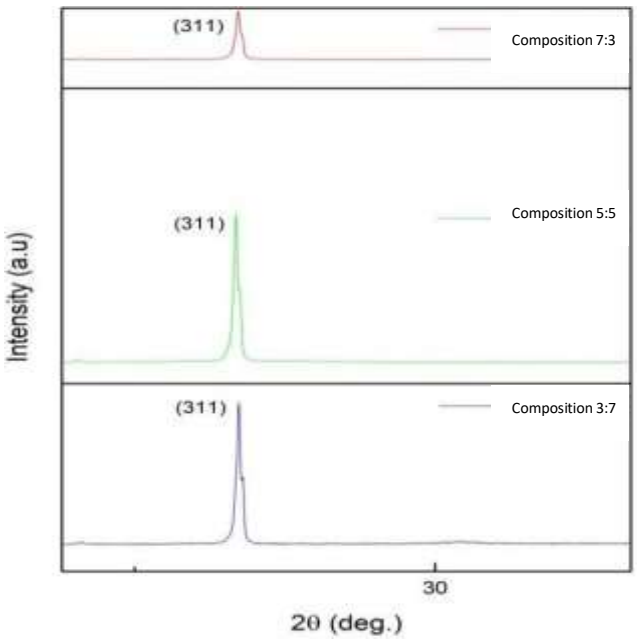


Figure 2. The enlarged peak of 311 of all compositions

Figure 2 depicts the enlarged peak of 311, which shows that the sample with the composition of 3:7 and composition of 5:5 undergoes the peak splitting from 26.88° to 26.73°. The splitting happens due to the existence of a silanol group in large quantities during the fabrication, implying that the SiO_2 does not react with the samples and only acts as a substrate [1].

3.2 SEM-EDX Analysis

SEM-EDX is conducted to investigate the morphology and elemental composition of SiO_2-Fe_3O_4. As given in Figure 3, the particles are homogenous in shape. In detail, the particles formed are rounded and homogeneous and tend to mend to each other due to the occurrence of adhesions between particles. The particle size is measured using ImageJ software.

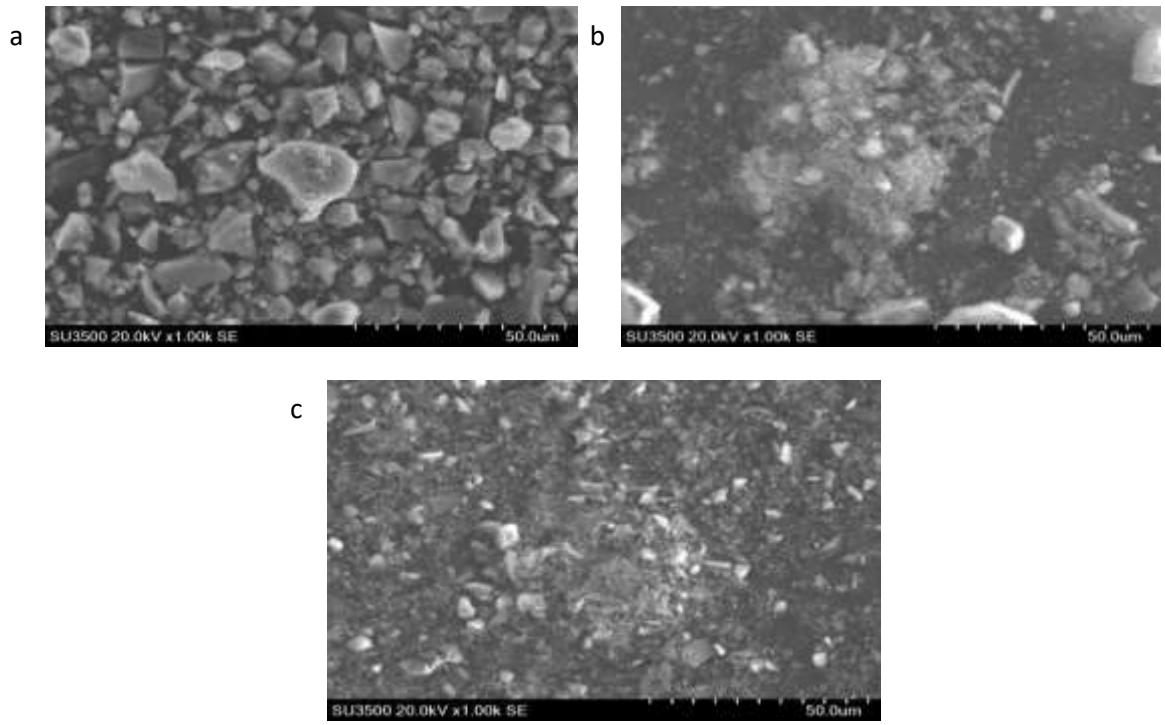


Figure 3. SEM morphology of (a) sample 1 (SiO_2-Fe_3O_4 (Composition 3:7)); (b) sample 2 (SiO_2-Fe_3O_4 (Composition 3:5)); and (c) sample 3 (SiO_2-Fe_3O_4 (Composition 7:3))

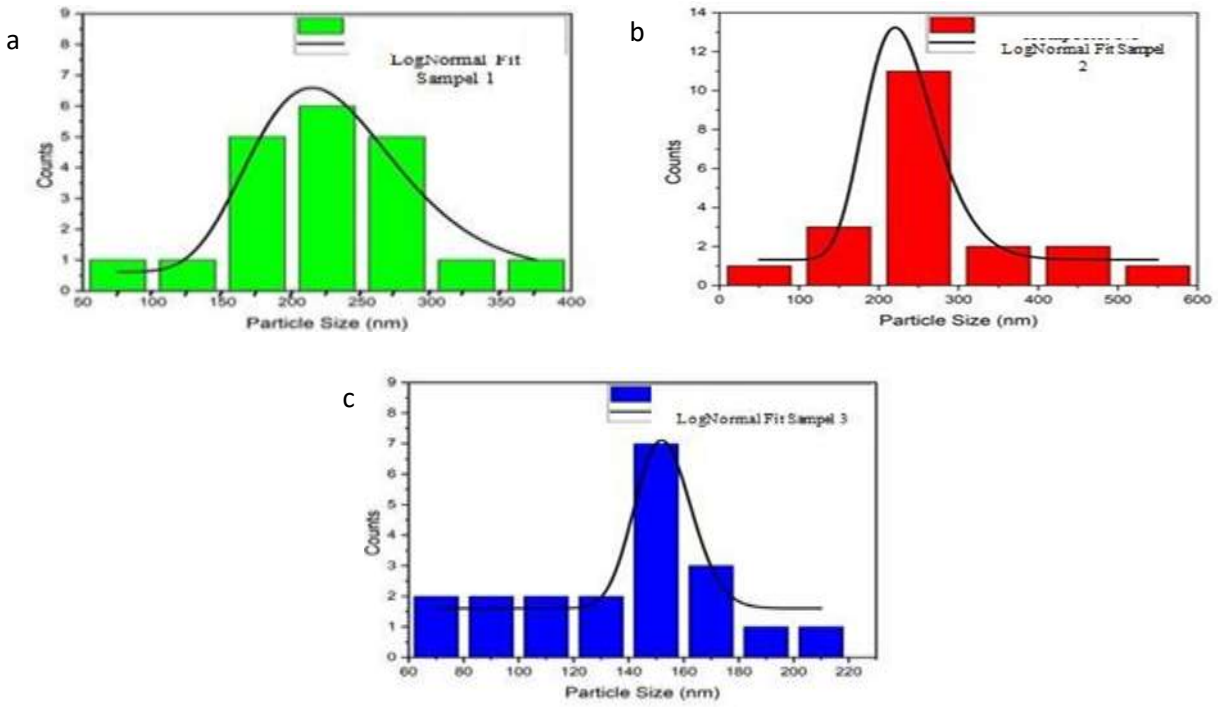
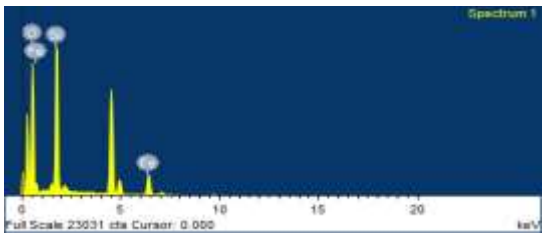


Figure 4. Histogram of the particle size of (a) sample 1 (SiO_2-Fe_3O_4 (Composition 3:7)); (b) sample 2 (SiO_2-Fe_3O_4 (Composition 3:5)); and (c) sample 3 (SiO_2-Fe_3O_4 (Composition 7:3))

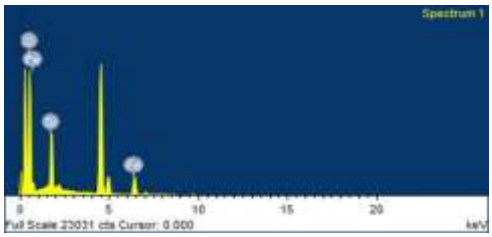
As shown in Figures 3 and 4, sample 1 has a 150 – 250 nm particle size. Sample 2 shows a surface sample with a distribution particle that is not uniform with the particle size range of 150- 280 nm. Sample 3 has more, not uniform, particles and lumps; the particle size I smaller than those of samples 1 and 2, in the range of 120 – 180 nm. These results are similar to the results from another research[8].

a



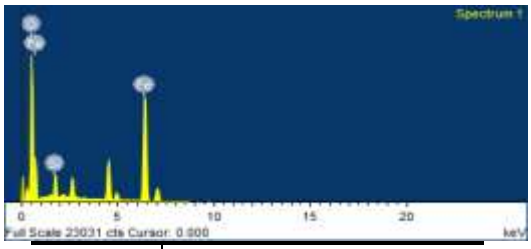
Elements	weight%	atomic%
O K	36.75	65.73
Si K	3.69	3.76
Fe K	59.56	30.51
Totals	100.00	

b



Elements	weight%	atomic%
O K	57.40	74.40
Si K	26.66	19.68
Fe K	15.94	5.92
Totals	100.00	

c



Elements	weight%	atomic%
O K	63.14	80.71
Si K	16.01	11.66
Fe K	20.84	7.63
Totals	100.00	

Figure 5. EDX Spectrum of (a) sample 1 on SiO_2-Fe_3O_4 (Composition 3:7); (b) sample 2 on SiO_2-Fe_3O_4 (Composition 3:5); (c) sample 3 on SiO_2-Fe_3O_4 (Composition 7:3)

Figure 5 depicts SEM EDX for the samples. The spectra confirm that the elemental composition for all samples are Si, Fe, and O. The result also showed that sample 3 (composition 7:3) is the most optimum condition based on atomic presentation and weight presentation. Furthermore, the atomic presentation influenced the particles' morphology and size distribution [9].

3.3 Results VSM Test

The magnetic characterization of SiO_2-Fe_3O_4 was tested using *vibrating sample Magnetometers* (VSM), as given in Figure 6. The information obtained is in the form of the magnitude of the trait magnetic field due to a changing magnetic field outside, which is depicted with curve hysteresis. Curve hysteresis could show a relationship between magnetization (M) and the magnetic field outside (H). The parameters used are magnetic saturation (M_s), field coercivity (H_c), and magnetization remanent (M_r). The saturation magnetization value, also known as saturation magnetization, indicates the ability of nanoparticles to maintain the alignment of their magnetic domains when they are subjected to an external magnetic field. The coercivity field is the magnitude of the field required to make the magnetization zero. The greater the value, the stronger the magnetic properties. Meanwhile, magnetic remanence (Mr) shows the ability of the material when given an external field [1].

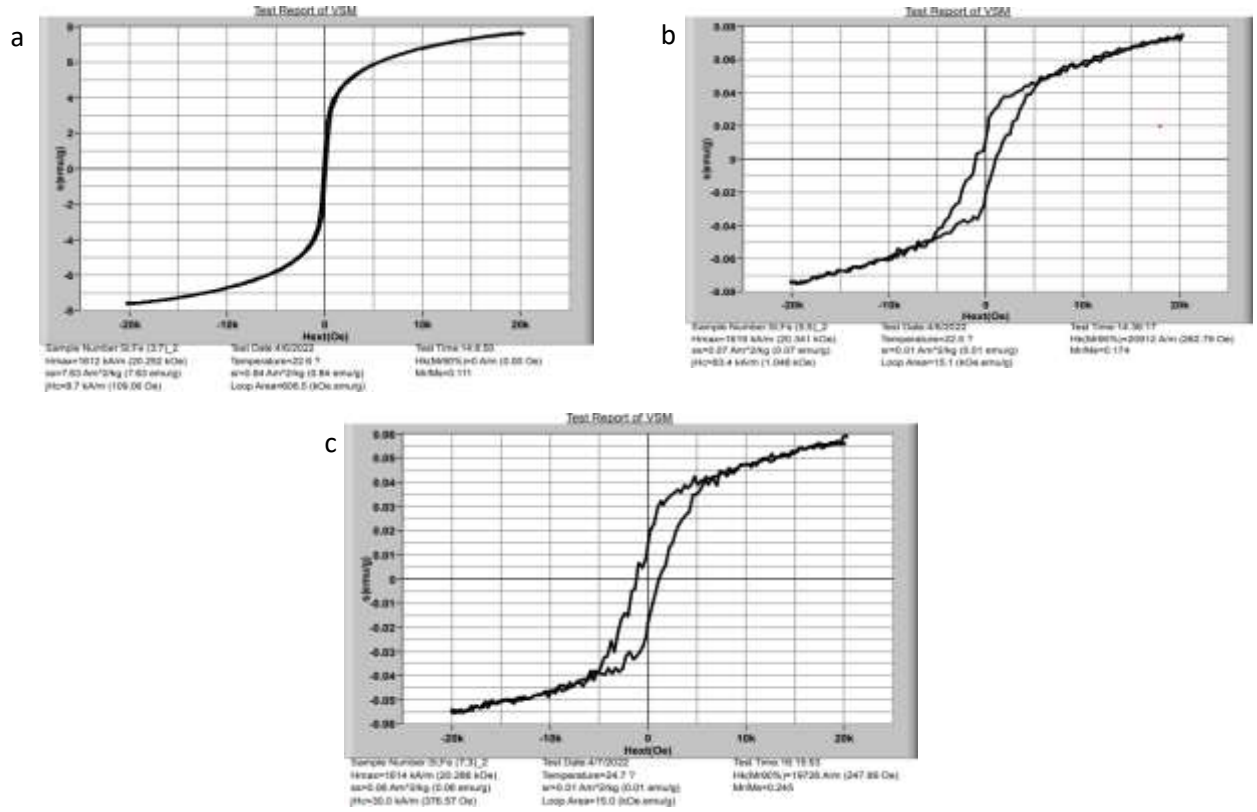


Figure 6. The hysteresis curve of (a) sample 1 on SiO_2-Fe_3O_4 (Composition 3:7); (b) sample 2 on SiO_2-Fe_3O_4 (Composition 3:5); (c) sample 3 on SiO_2-Fe_3O_4 (Composition 7:3)

Table 3. The magnetic characteristic of SiO_2-Fe_3O_4

Sample	Composition Sample (SiO_2-Fe_3O_4)	Saturation, M_s (em/g)	Remanence, M_r (em/g)	Coercivity, H_c (Oe)	H_{max} (kOe)
1	3:7	58.70	16.26	116.90	20.252
2	5:5	74.36	14.60	117.00	20.341
3	7:3	76.26	38.62	114.60	20.286

From the data presented in Table 3, each sample's saturation values were obtained: 58.70 emu/g, 74.36 emu/g, and 76.26 emu/g. Whereas for materials with a composition of 3:7 and 5:5, there is an increase in the coercivity value; this occurs due to agglomeration in the powder. Increasing SiO_2 reduces remanence and coercivity and increases saturation. Sample 2 has a maximum coercivity of 117.00 Oe, which produces a hard magnetic, while the coercivity value of sample 3 is 114.60 Oe which produces a soft magnetic, which is ferrimagnetic [10].

In sample 1, the highest silica had the smallest M_s, namely 58.70 emu/g with M_r 16.26 emu/g and H_c 116.90 Oe, while in sample 3, the least added silica decreased the M_s value to 76.26 emu/g with M_r 38.62 emu/g and H_c 114.60 Oe. This information implies that the greater the concentration of SiO_2, the greater the M_s, but the change is not always significant. The magnetic properties become weaker because the greater concentration of SiO_2 acts as a template, resulting in a smaller particle size [1].

4. Conclusion

Based on the results of research on the synthesis of SiO_2-Fe_3O_4 nanoparticles, it can be concluded that the synthesis of SiO_2-Fe_3O_4 for the manufacture of permanent magnets has been successfully carried out by the co-precipitation method. The XRD results show that the effect of composition on the diameter of SiO_2-Fe_3O_4 crystals is a decrease in the diameter of SiO_2-Fe_3O_4 crystals as the composition of SiO_2-Fe_3O_4 increases. Meanwhile, the results in SEM showed that the effect of composition on the morphology of SiO_2-Fe_3O_4 was an increase in the particle size distribution of sample 1 (Composition 3:7) = 150-250 nm, sample 2 (Composition 5:5) = 150-280 nm and sample 3 (Composition 7:3) = 120-180 nm in morphology with

increasing SiO_2-Fe_3O_4 composition. The XRD results showed that the effect of composition on the structure of the amorphous SiO_2-Fe_3O_4 phase was in the form of an amorphous SiO_2-Fe_3O_4 phase which decreased as the silicon composition increased. Then, the SEM-EDX results showed that the effect of the composition on the morphology of SiO_2-Fe_3O_4 was the increase in the silicon composition. From the VSM results, the optimum magnetic properties were found in sample 3 (7:3) with a coercivity of 114.60 Oe, a remanent magnetization of 38.62 emu/g, and a saturation magnetization of 76.26 emu/g and with a size distribution 70 nm particle, and its magnetic properties are that of a permanent magnet.

5. Acknowledgments

The authors would like to thank the National Innovation Research Agency-BRIN for laboratory equipment and testing facilities and BRIN Advanced Materials Research Center researchers for their support and assistance in conducting research.

References

[1] S. Taib and E. Suharyadi, "Magnetite (Fe_3O_4) Nanoparticle Synthesis using Silica (SiO_2) Template and Magnetic Properties Characterisation," *Indonesian Journal of Applied Physic*, vol. 5, no. 1, pp. 23-30. 2015.

[2] T. K. Indira, and P. K. Lakshmi, "Magnetic nanoparticles–a review," *International Journal of Pharmaceutical Sciences and Nanotechnology (IJPSN)*, vol. 3, no. 3, pp. 1035-1042. 2010.

[3] R. H. Kodama, "Magnetic nanoparticles," *Journal of magnetism and magnetic materials*, vol. 200 no.1-3, pp. 359-372. 1999.

[4] A. Yulianto, S. Bijaksana, and W. Loeksmanto, "Comparative Study on Magnetic Characterization of Iron sand from Several Locations in Central Java," *Indonesia Journals of Physics*, vol. 14 No 2. 2003.

[5] A. Nikmah, A. Taufiq, and A. Hidayat, "Synthesis and Characterization of Fe_3O_4 /SiO_2 nanocomposites," *IOP Conf. Series: earth and Environmental Science*, vol. 276, no. 012046. 2019.

[6] M. Pilloni, J. Nicolas, V. Marasaud, K. Bouchemal, F. Frongia, A. Scano, G. Ennas, and C. Dubernet, "PEGylation and preliminary biocompatibility review of magnetite-silica nanocomposites obtained by high energy balls milling," *International Journals of Pharmaceutics*, vol. 401, no. 1-2, pp. 103–112. 2010.

[7] Munasir, A. S. Dewanto, A. Yulianingsih, I. K. F. Saadah, Z. A. I. Supardi, A. Mufid, and A. Taufik, "Composites of Fe_3O_4/SiO_2 from natural Material Synthesized by Co Precipitation method," *IOP Conf. Series: Materials Science and Engineering*, vol. 202, no. 012057. 2017.

[8] P. E. Imoisili, K. O. Ukoba, T-C. Jen, "Synthesis and characterization of amorphous mesoporous sillica from palm kernels shell ash," *Boletin de la Sociedad Espanola de Ceramica y Vidrio*, vol. 59, no. 4, pp. 159-164. 2020.

[9] T. R. Simbolon, P. Sinuhaji, M. Hamid, T. Sembiring, M. Rianna, and M. Ginting, "Effect of Co composition in $CoFe_2O_4$ against structure micro, thermal, and characteristic magnetic," *Case Studies in Thermal Engineering*, vol. 26, no. 1. 2021.

[10] M. Karbeka, F. Veronica, and N. M. Tellu, "Characterization Characteristic magnetism Sand Iron Beach Puntaru Kabupaten ALOR-NTT," *Lantanida Journal*, vol. 8 no. 2, pp. 96-188. 2020.