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Synthesis and characterization of Fe_3O_4/SiO_2 composite with in-situ method: TEOS as SiO_2 NPs precursor

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Abstract. The aim study, Fe_3O_4/SiO_2 composites have been synthesized through two stages; first, the synthesis of Fe_3O_4 particles from iron sand material (from Lumajang area, in Indonesia); second, the synthesis of Fe_3O_4/SiO_2 using the in-situ method by mixing Fe3O4 nanoparticles with ammonia, ethanol, and TEOS (as precursor SiO_2 Nanoparticles) in one pot as a chemical reaction site. The Fe_3O_4/SiO_2 composites were then characterized using XRD and FTIR, VSM and TEM. Characterization results showed that the phase structure of the Fe_3O_4/SiO_2 composite was successfully formed. The findings are supported by the functional group vibration data on FTIR, and the identified absorption pattern has Fe-O-Fe and Si-O-Si bonds which states that the surface modification of Fe_3O_4 has been successfully carried out using SiO_2. The VSM test results showed that the magnetization saturation of Fe_3O_4/SiO_2 composites was 18 emus/g and based on the TEM test results it was seen that Fe_3O_4 particles were coated by SiO_2 particles with Fe_3O_4/SiO2 particle size of ~ 100 nm.

1. Introduction

Magnetite or commonly called Fe_3O_4 is a compound in the form of iron oxide contained in iron sand minerals, in addition to other iron sand minerals such as hematite (α-Fe_3O_4) and maghemite (γ-Fe_2O_3). Magnetite Fe_3O_4 material has new characteristics such as catalytic, optical and magnetic properties. This material is one of the most commonly used types of magnetic particles. Various examples of the application of Fe_3O_4 particles include ferrofluid, energy storage, and magnetic data storage [1]. In the medical field the application of Fe_3O_4 particles can be used as a hyperthermia therapy system [2], Drug Delivery System (DDS) [3], and Magnetic Resonance Imaging (MRI) [4].

In recent years, many studies have discussed the surface modification of Fe_3O_4 particles with the aim of homogenizing particle shape and size, increasing chemical stability, and compatibility. Surface modification can be done by non-magnetic coating materials [5]. Although there are many types of materials available for particle layers of Fe_3O_4, such as metal oxides, precious metals, and polymeric materials, silica (SiO_2) is still considered the best candidate for modifying the surface of Fe_3O_4 particles. SiO_2 is a material that has a stable nature when reacted. Therefore, SiO_2 functions as a composite shell that is ideal for protecting magnetic matter [6]. Also, SiO2 layers can reduce uneven agglomeration and dispersion in Fe_3O_4 particles [1]. High-quality SiO_2 is easily obtained from ethyl silicate hydrolysis or with the common name Tetraethylorthosilicate (TEOS). TEOS will not need a

long time for the synthesis process because in the synthesis process. The silica structure obtained will also be more homogeneous. Fe_3O_4/SiO_2 composites can be synthesized using the wet mixing (*ex-situ*) method or using the *in-situ* method. The advantages of the in-situ method itself are the filler, and the matrix can be mixed evenly [7].

Zhang et al. (2016) succeeded in synthesizing Fe_3O_4/SiO_2 using a one-pot process which can be applied as MRI contrast agents [7]. Gao et al. (2011) also summarized and superparamagnetic characterization of composites of $Fe_3O_4@SiO_2$ Core-Shell nanoparticles with variations in temperature and volume of TEOS so that it has the potential to be applied as DNA purification, targeted drug delivery, and magnetic hyperthermia [8]. Besides, Farmany et al. (2016) synthesized Fe_3O_4/SiO_2 as core/shell by the ultrasound-assisted method using the same SiO_2 material in the form of TEOS which can be applied as absorption material for diazinon removal [9]. In this report, the structure and magnetic properties of Fe_3O_4/SiO_2 composites will be presented, which are synthesized using the in-situ method with silica SiO_2 from TEOS as a precursor; and magnetite particles Fe_3O_4 nanoparticles derived from iron sand using the coprecipitation method.

2. Materials and Methods

2.1. Materials and Synthesis

The materials used in this study include iron sands, Ethanol, Ammonia hydroxide (NH_3•H2O) powder, Tetraethyl Orthosilicate (TEOS) precursor axles, and DI-water (aquades). And the equipment used in this research is the beaker, measuring cup, magnetic stirrer, filter paper, PH paper, digital balance sheet, spatula, pipette, stopwatch, funnel, aluminum foil, cardboard, and 60-watt lamp or oven. Fe3O4 Nanoparticles powder was prepared by co-precipitation method from iron sand taken from Lumajang area-Indonesia. The fe_{3o4}/si_{o2} composite formation was initiated by mixing Fe_3O_4 and aquades with 30 minutes of ultra-sonification. When the solution is mixed, Ethanol is added to the mixture [10]. After stirring evenly, Ammonia and TEOS are added. Then sterilize for 8 hours at room temperature. The composite results were washed with distilled water until PH 7 was then filtered. The precipitate produced is then dried at 60 ° C for 24 hours. The result of the combination of the two particles will be obtained Fe_3O_4/SiO_2 composites which are ready to be characterized.

2.2. Characterization

The Fe_3O_4/SiO_2 composite samples that have been made are then tested by the X-Ray Diffraction (Pan Analytical, Type: Expert Pro) to determine the phase and crystal structure formed. The FTIR (Nicolet iS 10 FT-IR Spectrometer) to identify the functional group; VSM (*Vibrating Sample Magnetometer*) for analysis of hysterical curves, magnetic properties. And The LR-TEM (Low-Voltage Transmission Electron Microscopy) to determine the size, shape, and morphology of particles (sample-powders).

3. Results and Discussion

3.1. X-Ray Diffraction Analysis

The XRD test results showed that the composite diffraction pattern of Fe_3O_4/SiO_2 showed a diffraction pattern of SiO_2 at position $2\theta = 21\text{-}28$ with an amorphous structure and the diffraction pattern of Fe_3O_4 corresponding to each crystal field [6, 8]. The composite diffraction peak Fe_3O_4/SiO_2 which occurs at an angle of 2θ = 30.3 °, 35.5 °, 43.2 °, 57.0 °, 62.8 ° with the respective crystal fields (220), (311), (400), (511), (440) which also shows unchanging peaks of Fe3O4 [6,8,10]. From the peak results, the samples have similarities with previous studies conducted by [8,10,11]. Based on the results obtained, it shows that the composite has been successfully formed which is characterized by the appearance of characteristic peaks of Fe_3O_4 and SiO_2.

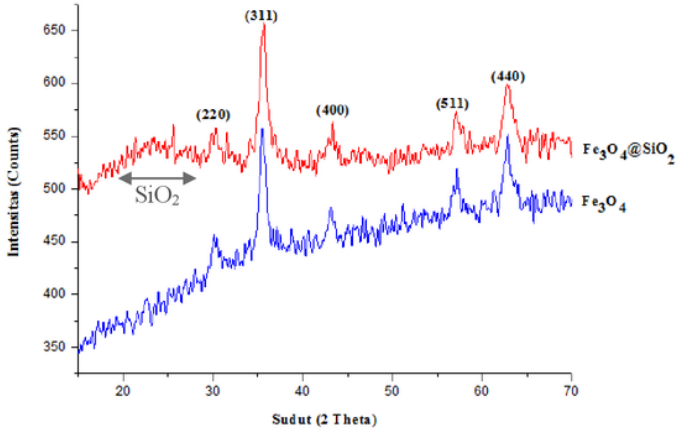

Figure 1. The result of X Ray-Diffraction pattern of samples: Fe_3O_4 NPs and Fe_3O_4/SiO_2 composite

3.2. Fourier-transform Infrared Spectroscopy Analysis

The FTIR test results showed that the synthesis of Fe_3O_4 particles and Fe_3O_4/SiO_2 composites revealed wave absorption patterns that were typical of both. The vibration of Fe-O-Fe group found at wave number 580 cm^{-1} indicates an indication of particles Fe_3O_4 [4,9] and Si-O-Si bonds at wave number 1905 cm^{-1} which states that Fe_3O_4 surface modification has been successfully carried out using silica [12,13]. The functional groups are similar to the characteristics of the Fe_3O_4/SiO_2 composite functional group obtained from the reference.

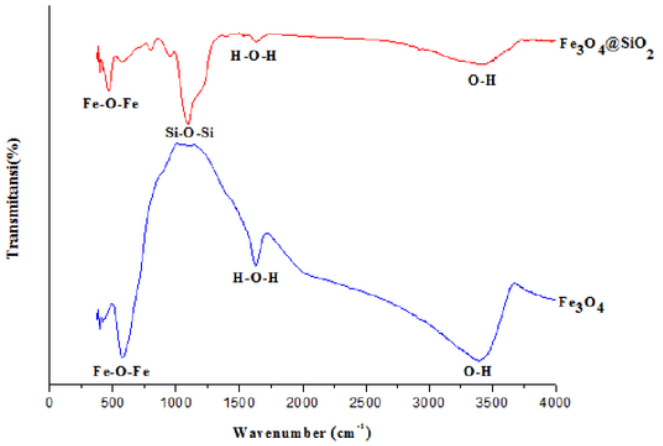

Figure 2. The result of FTIR spectroscopy of samples: Fe_3O_4 NPs and Fe_3O_4/SiO_2 composite

3.3. Vibrating Sample Magnetometer Analysis

Based on the results of the VSM (*Vibrating Sample Magnetometer*) testing, the magnetic properties of Fe_3O_4/SiO_2 composites are shown in Figure 3. The saturation of the magnetization of Fe_3O_4/SiO_2 composites was 18 emu/g by showing superparamagnetic material properties. When the strength of the

external magnetic field increases, the first magnetization increases rapidly and then reaches saturation. Then, when the external magnetic field strength decreases, the magnetization of the sample returns along the initial route, shows S-type behavior and almost no residual magnetism and stable coercivity forces, these characteristics indicate that this material can be significant for biomedical applications [1,2,8,14].

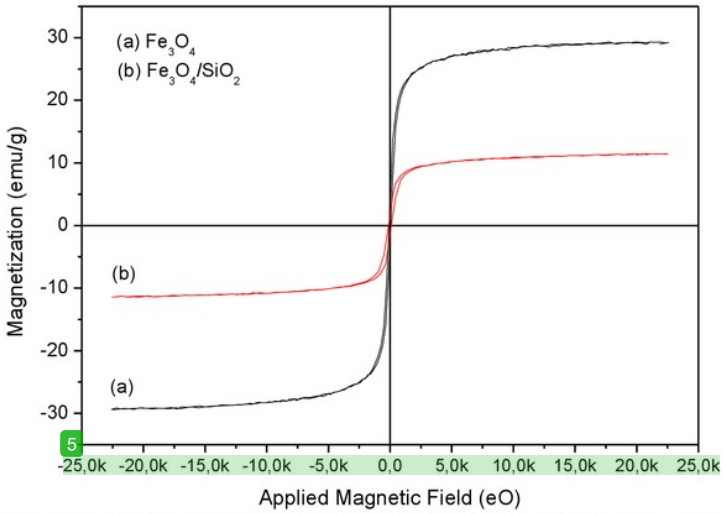

Figure 3. Result of VSM testing of samples: Fe_3O_4 NPs and Fe_3O_4/SiO_2 composite

3.4. Transmission Electron Microscopy Analysis

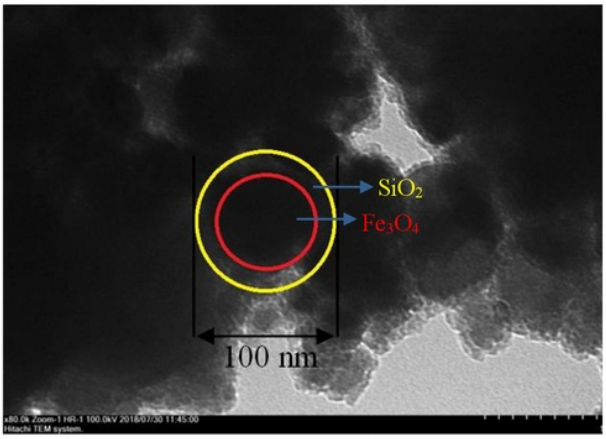

Figure 4. Result of TEM analysis of Fe_3O_4/SiO_2 composite

Based on the TEM analysis results, it was seen that the particles of black Fe_3O_4 and light gray SiO_2 particles were seen covering the entire Fe_3O_4 particles as shown in Figure 4. Also, Fe_3O_4/SiO_2 particles appeared round and agglomerated between Fe_3O_4 particles due to the composition of Fe3O4 too much [1,15,16]. The particle size of Fe_3O_4/SiO2 is around~ 100 nm with a core size (core) of ~ 67 nm, and the thickness of the shell is ~ 16 nm as shown in Figure 4.

4. Conclusion

The Fe_3O_4/SiO_2 composites have been successfully synthesized by the In-situ method, using TEOS as a precursor of SiO_2. The diffraction pattern corresponding to Fe_3O_4 magnetic and SiO_2 phase is amorphous, and absorption of infra-red waves shows the dominant functional groups of Fe-O-Fe and Si-O-Si. The Fe_3O_4/SiO_2 composite magnetization saturation was 18 emu/g which showed superparamagnetic and soft magnetic properties. And the results of morphology analysis, it was known that Fe_3O_4 particles were black (core) and SiO_2 particles with light gray covered Fe_3O_4 particles (shell); and with Fe_3O_4/ SiO_2 particle size around ~ 100 nm.

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References

[1] Rahmawati R, Taufiq A, Sunaryono S, Fuad A, and Yuliarto B 2018 *J. Mater. Environ. Sci.* **9** 155.
[2] Thiesen B and Jordan A 2008 *Int. J. Hyperthermia* **24** 467.
[3] Dobson J 2006 *Drug Dev. Res.* **67** 55.
[4] Haw CY, Mohamed F, Chia CH, Radiman S, Zakaria S, Huang NM and Lim HN 2010 *Ceram. Int.* **36** 1417.
[5] Buzea C, Pacheco II and Robbie K 2007 *Biointerphases* **2** 17.
[6] Munasir M, Dewanto AS, Yulianingsih A, Saadah IKF, Supardi ZAI, Mufid A and Taufiq A 2017 *IOP Conf. Series: Mater. Sci.Eng.* **202** 012057.
[7] Zhang L, Shao H, Zheng H, Lin T, and Guo Z 2016 *Int. J. Miner. Metal. Mater.* **23** 1112.
[8] Gao M, Li W, Dong J, Zhang Z, and Yang B 2011 *J. Con. Matter Phys.* **1** 49.
[9] Farmany A, Mortazavi SS, and Mahdavi H 2016 *J. Magnetism Magnet. Mater.* **416** 75.
[10] Munasir, Dewanto AS, Kusumawati DH, Putri NP, Yulianingsih A, and Sa'adah IKF 2018 *IOP Conf. Ser. Mater. Sci. Eng.* **367** 012010.
[11] Munasir, Triwikantoro, Zainuri M, and Darminto, 2015 *Mater. Sci.-Pol.* **33** 47.
[12] Munasir, Sulton A, Triwikantoro, Zainuri M, and Darminto 2013 *AIP Conf. Proc. AIP Publishing 2013* 28.
[13] Ubaid AH, Nurul and Munasir 2017 *IOP Conf. Ser. Mater. Sci. Eng.* **202** 012022.
[14] Sunaryono, Hidayat MF, Insjaf C, Taufiq A, Mufti N, and Munasir 2018 *IOP Conf. Ser. Mater. Sci. Eng.* **367** 012025.
[15] Larumbe S, Gomez-Polo C, Perez-Landazabal JL, and Pastor JM 2012 *J. Phys. Condens. Matter.* **24** 266007.
[16] Jian G, Liu Y, He X, Chen L, and Zhang Y 2012 *Nanoscale* **4** 6336.

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