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by M Munasir

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Composites of Fe_3O_4/SiO_2 from Natural Material Synthesized by Co-Precipitation Method

Munasir[1], A S Dewanto[1], A Yulianingsih[1], I K F Saadah[1], Z A I Supardi[1], A Mufid[2] and A Taufiq[3]

[1]Department of Physics, Faculty of Mathematics and Sciences, Universitas Negeri Surabaya (UNESA), Jl. Ketintang, Surabaya 60231, Indonesia.
[2]Department of Physics, Faculty of Mathematics and Sciences, Institut Teknologi Sepuluh Nopember (ITS), Jl. Arif Rahman Hakim, Surabaya 60111, Indonesia.
[3]Department of Physics, Faculty of Mathematics and Natural Sciences, Universitas Negeri Malang (UM), Jl. Semarang 5, Malang 65145, Indonesia.

Email: munasir_physics@unesa.ac.id

Abstract. Synthesis of Fe_3O_4/a-SiO_2 nanoparticle has been conducted based on natural materials by using the co-precipitation method. The used silica was a-SiO_2 collected from silica sand from Bancar Tuban, Indonesia. The Fe_3O_4 collected from iron sand's Lumajang, Indonesia. The samples was set for Fe_3O_4 : a-SiO_2: PEG composition of 1 : 2 : 1. The process was maintained by melting the PEG 4000 and following by adding a-SiO_2 and Fe_3O_4. The samples were characterized by using X-Ray Diffractometry (XRD), Transmission Electron Microscopy (TEM), and Fourier Transform Infra-Red (FTIR) spectroscopy. The analysis of the XRD data showed that additional silica into Fe_3O_4 tended to change the diffraction pattern that represents the existent of both Fe_3O_4 and silica. The SEM images presented that the samples formed in nanometric size with core-shell structure. Furthermore, the FTIR spectra showed that Si-O-Fe bound appeared at 555-568 cm^{-1}. Thus, this work was able to produce the Fe_3O_4/a-SiO_2 nanocomposites by using co-precipitation method in a core-shell structure.

Keyword: Composite, nanoparticle, Fe_3O_4, SiO_2, coprecipitation

1. Introduction

Indonesia is a country with abundant natural resources. Some of the natural resources extracted from minerals are quartz sand and iron sand. Most of the content of quartz sand is silica (SiO_2) with the purity of 70%. Quartz sand has a characteristic of its yellowish white color (Bancar area, Tuban-East Java, Indonesia) [1], its density of 2.50–2.70 g/cm^3, and having melting point of around 1715 °C [2]. Based on the previous research, the iron sand from Lumajang had a noticeably high level of Ferrous that could easily produce magnetic nanoparticles with high purity [3,4]. The latest reports show that Indonesia has a large number of iron minerals that consist of the main iron ore (17%), iron ore (8%) and laterite iron ore (75%) [4].

There are many applications of nanocomposites in industrial and health sectors especially the use of this material for small particles from micro to nanometric size. Thus, we need to advanced and the right technology to process it. It needs a special treatment in the process of obtaining the nanometric materials. A lot of methods were used to get the nanocomposites with the best result including the co-precipitation method, sol-gel, etc. [2,5–8]. Nanocomposite material is a combination of two or more composing materials with a different characteristic material (matrix and filler). In this research, the magnetic particle (Fe_3O_4) was combined with silica because it has multiple advantages including the ability of its pure materials to synthesize in large scale and the large capacity of cation exchange. Silica comes from a natural or synthetic material and usually is in the form of crystalline or amorphous solid [1,6,9]. Silica is a metal oxide compound that is widely available in nature, but its existence is not in the free state. It is bounded to another compound physically and chemically. This material is used in many industries and medical field because of its unique nature and morphology such as the large surface area and pore volume, and the ability to absorb various substances like water, oil, and radioactive material. In general, Silica has hydrophobic or hydrophilic properties based on its structure and morphology [10].

In composite materials, the component did not undergo a chemical change. Fe_3O_4/SiO_2 nanocomposite is a composite material because its constituent materials have nanometric size and exist without any new compound formed. Another work reported that the Fe_3O_4/SiO_2 nanocomposites were succesfully prepared by using the co-precipitation method with $FeCl_3$ and silica solution as the basic material [11]. In this work, we were interested to develop synthesis method of Fe_3O_4/SiO_2 nanocomposites by exploring the abundant natural material via a co-precipitation method. This method can be utilized to prepare an inorganic compound based on the precipitation process of substances. The advantage of this method is having a relatively low temperature in a short process [12,13]. Interestingly, the method can prepare the Fe_3O_4/SiO_2 nanoparticle that was illustrated to form a composite formation, where the Fe_3O_4 ferromagnetic particle is covered inside the hollow of SiO_2 particle [11,14].

2. Experimental Method

2.1. Materials

The equipment used in this research were a beaker glass, measuring glass/graduated cylinder, magnetic stirrer, filter paper, digital scales, spatula, pH meter, pipette, spatula, funnel, crucible, mortal and pestle permanent magnet, cardboard and a 60-watt lamp. The material used in this study were Bancar-Tuban sand, Lumajang iron sand, NaOH, HCl 2M, NH_4OH, PEG 4000, and distilled water (DI). The manipulation variables were the a-SiO_2mass and the PEG mass. The Control Variable was the mass of Fe_3O_4. The response variable was the phase of Fe_3O_4/a-SiO_2 nanocomposites.

2.2. Experiment

The step taken to synthesize the nanocomposites of Fe_3O_4/a-SiO_2 was melting the PEG via the stirrer at a temperature of 60 °C. It was then added with a-SiO_2 powder while it was being stirred continuously until evenly combined, after that it was added with Fe_3O_4 powder and it was stirred for another 3 minutes, so that it formed precipitate. Those steps were repeated for 3 times with different compositions. The precipitate was then dried and thus formed Fe_3O_4/a-SiO_2 powder. This product was then characterized by using XRD to determine the occurrence of the composite on both $Fe_3O_4$and a-SiO_2. Furthermore, it was also characterized by using FTIR to determine the functional group possessed by the sample of Fe_3O_4/a-SiO_2. The size and distribution of the particles were characterized by using SEM/TEM.

3. Results and Discussion

3.1. XRD Characterization of Composite of Fe_3O_4/a-SiO_2

XRD Diffractogram result showed the diffraction pattern of amorphous silica and Fe_3O_4 shown in Figure 1. The curve indicates that the peaks in sample #1, and the base materials which were Fe_3O_4 and SiO_2. The XRD results proved the existence of peak particle combination of Fe_3O_4 and a-SiO_2

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particles. The diffraction peak occured at $2\theta = 30.07^o$, 35.47^o, 43.17^o, 53.33^o, 56.90^o which showed the cubic structure of the Fe_3O_4 particle, with each of crystal planes (220), (311), (400), (422), and (511). Therefore, undergoing a composite process did not change the structure of Fe_3O_4 particle . In sample #1, the wide peak on $2\theta \approx 24^o$ showed the existence of amorphous peak silica form. This peak intensity will increase along with the addition of silica, where the position of polymer form is very influential in the composite [15]. The higher the variation of silicate addition into Fe_3O_4 the more declined the diffraction peak pattern of Fe_3O_4 [11,16,17].

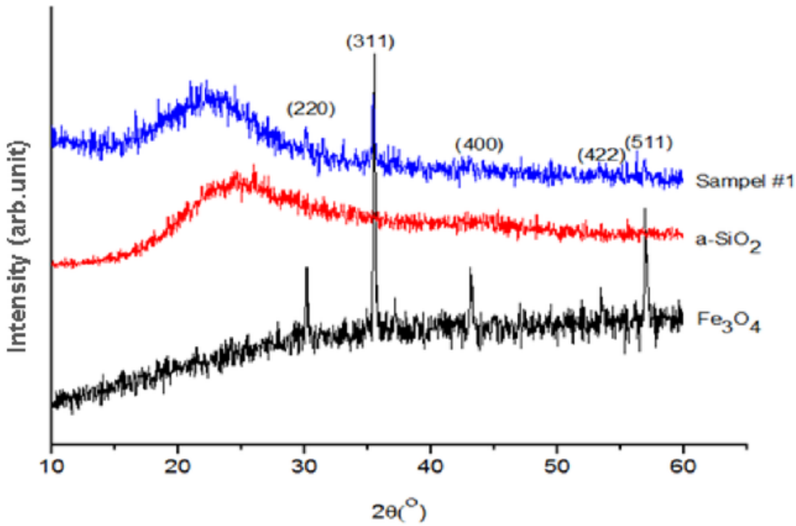

Figure 1. XRD patterns of Fe_3O_4/a-SiO_2

3.2. Data of FTIR Characterization of Fe_3O_4/a-SiO_2 Composites

After a test by using XRD, the Fe_3O_4/a-SiO_2 nanoparticles was characterized by using FTIR to determine the functional groups of the Fe_3O_4/a-SiO_2 sample. The functional groups corresponding to Fe_3O_4@SiO_2 (Sample #1) therein are depicted in Table 1.

Table 1. Functional group of the synthesized Fe_3O_4/a-SiO_2

Peak	Wavenumber (cm^{-1}) Fe_3O_4@SiO_2 (Sample #1)	Functional Group	Reference
1	468	Si-O-Si / O-Si-O	[1,7]
2	555	Si-O-Fe	[12]
3	796	Si-O-Si	[1,7,14]
4	950	Si-O	[12]
5	1093	Si-O-Si	[1,14]
6	1647	H-O-H	[12]
7	2914	H-O-H	[12]
8	3443	H-O-H	[1,14]

Based on the data, compared to the previous research, our FTIR result of Fe_3O_4/a-SiO_2 synthesis had compatibility with the previous and appropriate range of wavenumbers. In the FTIR spectra, the wavenumber of 1641-3464 cm^{-1} was a stretching vibration and bending of H-O-H bond. In 1097, the wavenumber was an asymmetric vibration of the Si-O-Si bond which was hooked by oxygen movement. There was a bending vibration of Si-O-Si and O-Si-O on the 468 cm^{-1} wavenumber. On the 796 cm^{-1} wavenumber, there was a symmetric stretch vibration of Si-O-Si in the SiO_2 [1,6,7]. In 950 cm^{-1} wavenumber, there was a symmetric functional group of Si-O and lastly, the 555-568 cm^{-1} wavenumber showed the existence of Si-O-Fe bond. Thus, the existence of Fe_3O_4 nanoparticles has been identified.

3.3. Photo SEM of Fe_3O_4/a-SiO_2 Composites

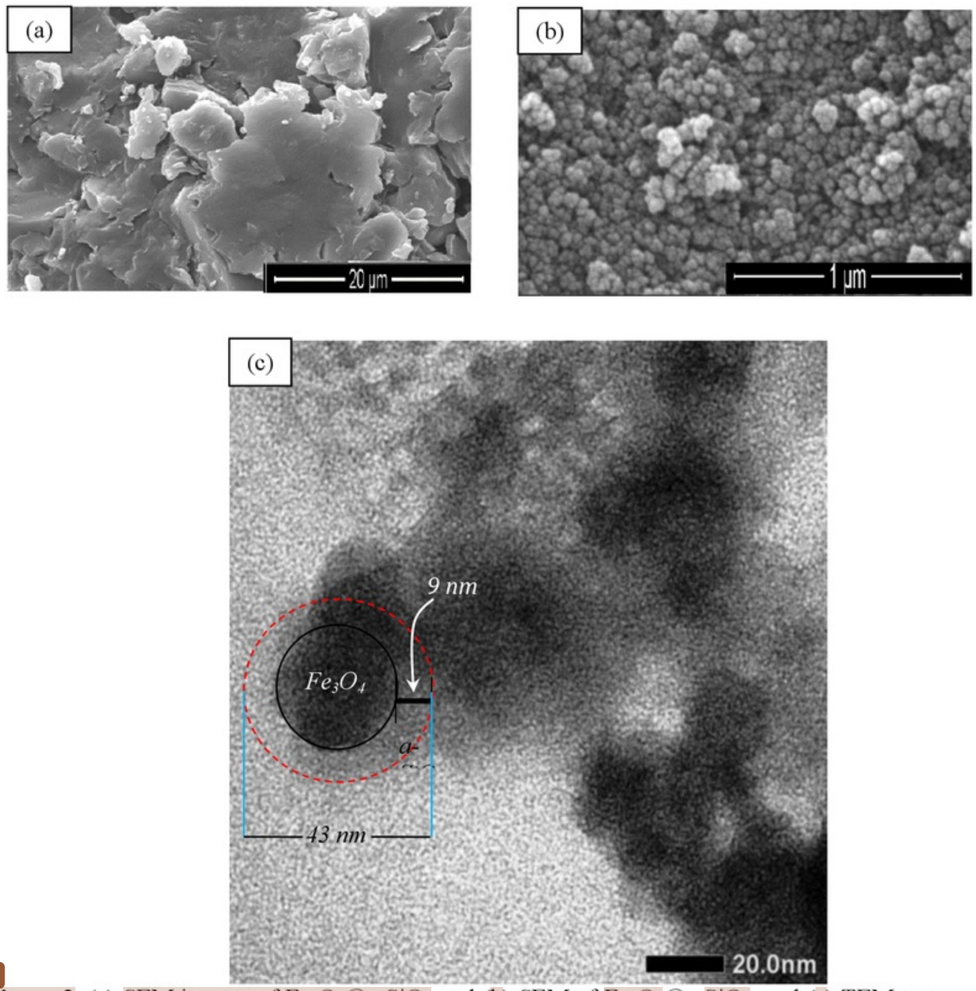

Figure 2. (a) SEM images of Fe_3O_4@a-SiO_2 and (b) SEM of Fe_3O_4@c-SiO_2, and (c) TEM test result of Fe_3O_4@SiO_2 (Sample #1)

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TEM characterization aimed for knowing the morphological structure of Fe_3O_4/a-SiO_2 nanocomposites that formed as a Fe_3O_4@a-SiO_2 core shell. Here is the result of TEM test of the Fe_3O_4@a-SiO_2 core shell (Figure 2c). It appears that the Fe_3O_4 particles are black-colored and coated with SiO_2 particle shown by a lighter cloud around it. Fe_3O_4@a-SiO_2 particle has a round-shaped property and agglomerated with other particles. The size of the Fe_3O_4@*a*-SiO2 particle was about ~25 nm and the thickness is ~9 nm. Thus, the Fe_3O_4/a-SiO_2 nanocomposites have successfully formed a Fe_3O_4@a-SiO_2 core shell [14,18]. There were significant different shape and size of the composite particles of Fe_3O_4@ a-SiO_2 amorphous (#sample 1) and Fe_3O_4@ c-SiO2 (crystal), as shown in Figure 2 (a) and Figure 2 (b); composite particle size of Fe3O4/a-SiO 2 was greater than the particle size of the Fe_3O_4@c-SiO_2 composite.

4. Conclusion

Based on the data analysis, we can conclude that Fe_3O_4/a-SiO_2 nanocompositeS can be fabricated by using PEG as the binder of Fe3O4 and a-SiO2 particleS, where Fe_3O_4, a-SiO_2 can be easily synthesized by using the co-precipitation method. The Characteristic of Fe_3O_4/a-SiO_2 based on XRD crystal structure and functional group of FTIR analysis shows that the Fe_3O_4/a-SiO_2 nanocomposites have formed a sharp peak of diffraction pattern and a distinct of functional group of Si-O-Fe on wavenumbers of 555-568 cm^{-1}.

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