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Synthesis and Magnetic Properties Investigations of Fe_3O_4 Nanoparticles*

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Abstract:
In this study, magnetic iron oxide nanoparticles (Fe_3O_4) with the size range of 20-30 nm were prepared by the modified controlled chemical co-precipitation method from the solution of ferrous/ferric mixed salt-solution in alkaline medium. In this process polyethylene glycol was used as a surfactant to prevent the solution from agglomeration. The prepared magnetic nanoparticles were characterized by X-ray diffraction (XRD) analysis, scanning electron microscopy (SEM) and vibrating-sample magnetometer (VSM). XRD image indicates the sole existence of inverse cubic spinel phase of magnetic iron oxide nanoparticles (Fe_3O_4). SEM image show that the dimension of magnetic iron oxide nanoparticles (Fe_3O_4) is about 24 nm. VSM patterns demonstrate superparamagnetic properties of magnetic nanoparticles.

Keywords: *Magnetic iron oxide nanoparticles, Polyethylene glycol, Surfactant, Superparamagnetic, Chemical co-precipitation*

1. INTRODUCTION

Nanoscience and nanotechnology have gained significant momentum in recent years. Nanotechnology involves the study and use of materials at nanoscale dimensions (nanomaterial sizes of ≤100 nm), exploiting the different physiochemical properties exhibited by nanomaterials from the same materials at a larger scale. Nanometer-sized materials have attracted substantial interest in the scientific community because of their special properties. The relatively large surface area and highly active surface sites of nanoparticles enable them to have a wide range of potential applications magnetic iron oxide nanoparticles as a new kind of nanometer-sized material, have multiple practical applications, such as physics, medicine, and biology due to their multifunctional properties such as small size, superparamagnetism and low toxicity [1-10].

2. EXPERIMENTAL

2.1. Materials

Ferric chloride hexahydrate ($FeCl_3.6H_2O$), ferrous chloride tetrahydrate ($FeCl_2.4H_2O$) and ammonium hydroxide (NH_4OH) purchased from Merck. Oleic acid and polyethylene glycol (PEG-4000) were purchased from Merck. All acids used were of the highest purity available and they were obtained from Merck**.**

2.2. Instrumentation

The size of the particles were characterized by

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X-ray diffraction (XRD, Philips model Expert) with Ni-filtered Cu Kα radiation ($CuK_{\alpha 1}$ = 0.154 nm) and Scanning electron microscopy (SEM, Philips model XL30), respectively. Magnetic properties of the particles were assessed with a vibrating-sample magnetometer (VSM, Homade 2 tesla). A magnet (Φ 17.5×20 mm, 5500 Oe) was utilized for the collection of magnetic particles.

2.3. Preparation of the polyethylene glycol-coated iron oxide nanoparticles

The magnetic nanoparticles (MNPs) were prepared according to Ref. [11,12]. Briefly, $FeCl_2.4H_2O$ and $FeCl_3.6H_2O$ (molar ratio of Fe^{3+}/Fe^{2+}=2) were dissolved in 300 mL deionized water under nitrogen atmosphere with vigorous stirring at 1000 rpm at 80 ^{0}C. Co-Precipitation occurs with the addition of ammonium hydroxide (NH_4OH) as precipitating agent to the solution and the colour of solution immediately changed to black. The black magnetic precipitate obtained was washed twice with deionized water and three times with 1-2 mL of tetramethylammonium hydroxide (25%). The chemical reaction of Fe_3O_4 precipitation can be described as follows:

$$Fe^{2+}+2Fe^{3+}+8OH^- \quad Fe_3O_4+4H_2O \qquad (1)$$

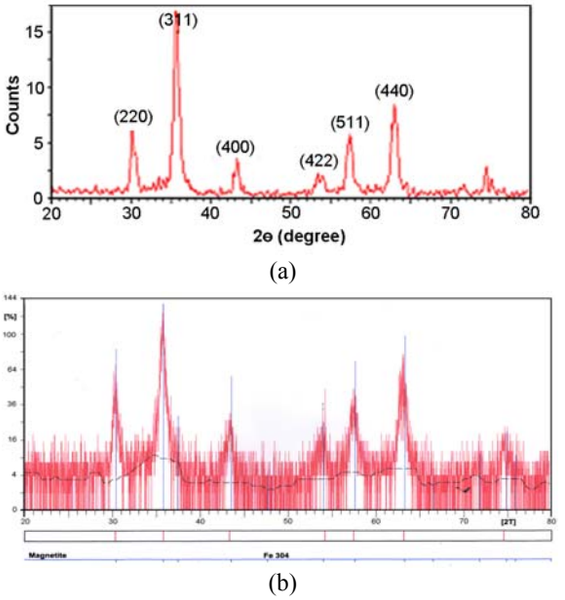

***Figure 1**: X-ray diffraction patterns for the polyethylene glycol-coated Fe_3O_4 MNPs*

The mixture of water and Fe_3O_4 nanoparticles was stirred and heated at 70 ^{0}C for 30 min under a nitrogen atmosphere, and then a solution of 25 ml oleic acid was added to the mixture with slow agitation. The suspension was then cooled slowly down to 45 ^{0}C with constant stirring. The polyethylene glycol (PEG-4000) solution was then added to the suspension. Then, the mixture was kept at 45 ^{0}C under vigorous stirring and a nitrogen atmosphere for 1 h. Then, it was cooled down to room temperature. The above solution was slowly added into deionized water for 2 days to allow the formation of hydrophilic nanoparticles and to remove organic solvents. The repulsive force between hydrophobic surfactant molecules coated on single particles can prevent them from agglomeration. Then, polyethylene glycol (PEG-4000) coated nanoparticles were separated by magnetic decantation with a permanent magnet and were dried at room temperature in air atmosphere to form Fe_3O_4 powders.

3. RESULTS AND DISCUSSION

3.1. XRD pattern

Figure 1(a) shows the X-ray diffraction patterns for polyethylene glycol (PEG) coated iron oxide (Fe_3O_4) nanopaticles powder. The XRD peaks of the Fe_3O_4 are compared with those of standard. A series of characteristic peaks at 2θ=30.29°, 35.69°, 43.30°, 53.69°, 57. 38°, and 62.98°, which corresponds to (220), (311), (400), (422), (511), and (440) Bragg reflection, respectively, in figure 1(a) agree with standard magnetite (Fe_3O_4) XRD patterns, identify that the Fe_3O_4 nanoparticles are cubic spinel structure.

The average diameter (D) of Fe_3O_4 nanoparticles was about 23 nm, which was determined according to Scherrer's equation:

$$D = k\lambda/ \beta \cos\theta \qquad (2)$$

Here the X-ray wavelength of CuKα radiation λ is 0.154 nm, k is the Scherrer constant (0.9), θ is Bragg angle and β is the full width at half maximum in radians. The reflecting peak at 2θ = 35. 69° is chosen to calculate the average diameter, the estimated

average size of the iron oxide nanoparticles (Fe_3O_4) are about 23 nm.

X-ray diffraction (XRD) was used for structural phase identification (Figure. 1(b)). The XRD measurements indicated that magnetite (Fe_3O_4) was the dominant phase for the sample.

3.2. SEM micrograph

Figure 2 shows the Scanning electron microscopy (SEM) image of the Fe_3O_4 nanoparticles coated with polyethylene glycol (PEG-4000). According to the SEM images, the normal size of Fe_3O_4 nanoparticles was about 24 nm, which matched the result the obtained from XRD analysis very well.

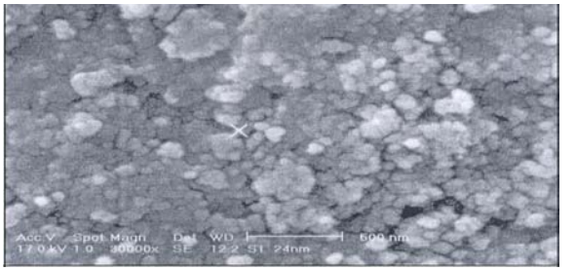

***Figure 2**: SEM image of the synthesized polyethylene glycol-coated Fe_3O_4 MNPs*

3.3. Magnetic property of Fe_3O_4 nanoparticles

The magnetic property of MNPs was measured by vibrating-sample magnetometer (VSM). The hysteresis loop of the nanoparticles, which was measured in the powder state, is shown in Figure 3 which provided evidence that all the MNPs were superparamagnetic at room temperature, with no hysteresis. The saturation magnetization M_S at 300 K which is 58.33 emu/g, is significantly less than that of the bulk magnetization, which is M_S (bulk)=92 emu/g. The decrease in saturation is ascribed to the size effect. The magnetic particle size and size distribution can also be calculated from the hysteresis curve using the following formula:

$$D_m = \left((18K_BT/\pi)(x_i/\rho M_s)\right)^{1/3} \quad (3)$$

Here, x_i is the initial magnetic susceptibility $x_i = (dM/dH)_{H \to 0}$ and ρ is the density of Fe_3O_4 (5.18 g/cm^3), and K_B Boltzmann constant. The initial slope near the origin was determined from the hysteresis plots by curve-fitting the linear portion of the data. The saturation magnetization M_s from them magnetization curve in Figure 3 for the Fe_3O_4 nanoparticles was found to be 58.33 emu/g at 300 K. Thus, the magnetic particle size D_m of sample was calculated with 6.9 nm. This value of D_m is smaller than the particle size observed from SEM measurement. The difference between D_m and D_{SEM} is most likely due to contributions of a magnetically "dead layer" reported to be present on the surface of particles.

For superparamagnetic particles, the true magnetic moment at a particular temperature can be calculated using the Langevin function:

$$M = M_s (\coth(\mu H/K_BT) - K_BT/\mu H) \quad (4)$$

Where $\mu = M_s \pi D^3/6$ is the true magnetic moment of each particle, D the diameter of the particle, K_B the Boltzmann constant, T the absolute temperature (300K in this article) and M_s is the saturation magnetization. Figure 3 shows the best fit for the Langevin function in Eq. (4). From this data fitting, the mean-magnetic moment per particle of sample is found to be 3 μ_B. The above analysis shows that the particles have typical superparamagnetism.

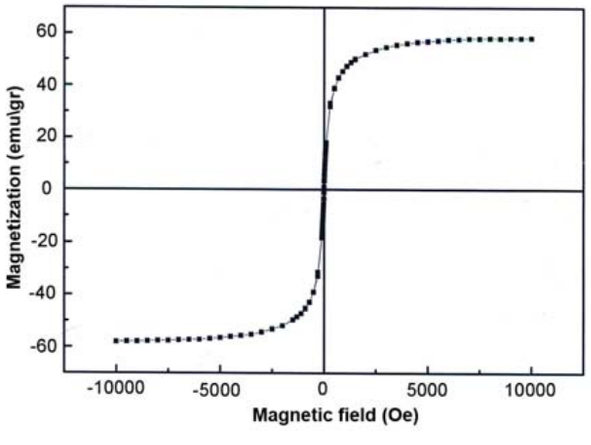

***Figure 3**: Magnetization vs. applied magnetic field for polyethylene glycol-coated Fe_3O_4 MNPs*

4. CONCLUSIONS

In this article, the superparamagnetic polyethylene glycol coated iron oxide nanoparticles (Fe_3O_4) with cubic spinel structure and an average particle size of 24 nm were successfully synthesized by the co-precipitation method. We carefully studied the magnetic properties of Fe_3O_4 nanoparticles. Through various analyses it is shown that the Fe_3O_4 particles prepared are superparamagnetism particles.

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REFERENCES

1. N. Chomchoey, D. Bhongsuwan, T. Bhongsuwan1, Kasetsart Journal. (Nat. Sci.), 44 (2010) 963 - 971.
2. C. Yue-Jian, T. juan, X. Fei, Z. Jia-Bi, G. Ning, Z. Yi-Hua, D. Ye, G. Liang, Drug Development and Industrial Pharmacy, 36 (2010) 1235–1244.
3. L. T. M. Hoa, T. T. Dung, T. M. Danh, N. H. Duc, D. M. Chien, Journal of Physics, 187 (2009) 012048.
4. H. Y.C. Acar, R. S. Garaas, F. Syud, P. Bonitatebus, A. M. Kulkarni, Journal of Magnetism and Magnetic Materials, 293 (2005) 1–7.
5. S. P. Gubin, Y. A. Koksharov, G. B. Khomutov, G. Y. Yurkov, Russian Chemical Reviews, 74 (2005) 489-520.
6. D. K. Kim, Y. Zhang, W. Voit, K.V. Rao, M. Muhammed, Journal of Magnetism and Magnetic Materials, 225 (2001) 30-36.
7. H. Y. C. Acar, R. S. Garaas, F. Syud, P. Bonitatebus, A. M. Kulkarni, Journal of Magnetism and Magnetic Materials, 293 (2005) 1–7.
8. J. Sun, S. Zhou, P. Hou, Y. Yang, J. Weng, X. Li, M. Li, Journal of Biomedical Materials Research Part A, (2006) 333-341.
9. A. Kebede, A. V. Gholap, A. K. Rai, World Journal of Nano Science and Engineering, 1 (2011) 89-92.
10. G. Utkan, Synthesis, Journal of Colloid and Interface Science, 353 (2011) 372-379.
11. R. Y. Hong, S. Z. Zhang, Y. P. Han, H. Z. Li, J. Ding, Y. Zheng, Powder Technology, 170 (2006) 1-11.
12. T. K. Indira, P.K. Lakshmi, International Journal of Pharmaceutical Sciences and Nanotechnology, 3 (2010) 1036-1042.