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Synthesis of γ–Fe_2O_3 nanoparticles with crystallographic and magnetic texture

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Abstract

Maghemite (γ-Fe_2O_3) nanoparticles are synthesized by chemical co-precipitation technique in AOT-microemulsion with a view to have possible application for biotagging. The investigations by means of X-ray diffraction, isothermal magnetization M (H) and ^{57}Fe Mössbauer Spectroscopy show that the particles are nonspherical, mostly of rod shape. The inter-particle interaction is so large that even a powder sample of thickness about 40 mg/cm^2 shows preferential orientation of magnetic moments in the plane of the sample. The saturation magnetization is much lower than the expected values for maghemite.

Keywords: Maghemite, Magnetic nanoparticles, Magnetic Ordering, Mössbauer spectroscopy.

1. Introduction

Magnetic nanoparticles have attracted intense experimental activities during the past decade among the researchers working in the field of nanoscience and nanotechnology because of their potential applications in a number of industrially important areas such as recording and storage of information (Ensling *et al.*, 1998), magneto-optical devices (Zayat *et al.*, 2003), magnetocaloric refrigerators (McMichael *et al.*, 1992), magnetic fluids (Depeyrot *et al.*, 2000), gas sensors (Tao *et al*, 1999) and also in biomedical and bioengineering (Pankhurst, 2003; Gupta and Gupta, 2005; Tartaj *et al.*, 2003). Their utility in industry is enhanced due to the ease of control which can be exercised with external magnetic fields. While nanosize effects such as superparamagnetism, collective magnetic excitation and spin canting have been reasonably well understood, inter-particle interaction and effect of texture in magnetic nanoparticles is relatively lesser exposed area.

Maghemite (γ-Fe_2O_3) has been especially interesting due to its non-toxicity, thermal and chemical stability and favorable hysteric properties and is being seen as a valuable material for mollifying biologically active compounds, bio-tagging of drug molecules, hysteretic heating of maligned cells etc. It has got a chemically active surface where a variety of bonds can be formed allowing for coating to which a variety of bio-active molecules may be attached. Maghemite nanoparticles have been made using a variety of techniques such as laser pyrolysis (Bomatí–Miquel *et al.*, 2002), co-precipitation (Jeong *et al*, 2004), sol-gel (Zhang *et al.*, 1997), microemulsion (Tueros *et al*, 2004), ball-milling (Randrianantoandro *et al.*, 2001), and sonochemistry (Shafi *et al*, 2002). Tuček *et al.*, 2006 have given excellent review on the synthesis and properties of γ-Fe_2O_3 nanoparticles with special emphasis on the Mössbauer spectroscopic studies.

The synthesis process and the process parameters involved control the properties of the resulting nanoparticles sensitively and yield particles of same composition and crystalline structure but with different magnetic and chemical properties. Single domain nanoparticles, in general, have a non-spherical shape and the resulting shape anisotropy gives rise to an enhanced coercive field. Any variation in synthesis route and parameters can change the shape anisotropy and the magnetic properties are likewise affected. Microemulsion has been used by many researchers to form nanomaterials in which water and surfactant form uniformly sized water pools in which nanomaterial is formed. For any bioconjugation experiment it is important to have water dispersible nano crystals. In most of the reported methods, initially the particles have been synthesized in a non polar media and thereafter either by surfactant exchange or by polymer coating they have been transferred into aqueous media. In our method we have reported 'one pot' synthesis of maghemite particles which can directly be suspended in water.

A variation in the process at the stage of forming microemulsion and chemical precipitation, as described in next section, resulting in nonspherical, rod-shaped nanoparticles of γ-Fe_2O_3 showing preferential magnetic alignment in powder sample, form the subject material of this communication.

2. Materials and Methods

The maghemite (γ-Fe_2O_3) nanoparticles were synthesized by chemical coprecipitation technique of ferric and ferrous ions taken in microemulsion formed by N-ethyl hexyl sulfo succinate/dioctyl sodium sulfo succinate (AOT or aerosol-OT) /toluene, and adding NaOH dropwise. $FeCl_3$ (Merck, purity> 99.9%) and $FeCl_2$ (Sigma, purity> 99.9%) were taken in 2:1 ratio and dissolved separately in 2M HCl. This solution was mixed together in 500 ml round bottom flask for 2-3 minutes at room temperature. In this solution 1 g AOT dissolved in 20 ml of deoxygenated water was added followed by addition of 5 ml toluene into it, and stirred for 5-10 minutes. Under stirring condition chilled 5N NaOH solution was added dropwise till the pH reached 10.5. Then it was heated at 100 ^{0}C for 3 hours in a hot air oven. The dense colloidal magnetic fluid obtained was washed several times with chilled deoxygenated deionized water. It was further washed two times with acetone and dried. To obtain crystalline solid it was annealed at 400 ^{0}C for 5 hours in ambient atmosphere.

The powder sample of γ-Fe_2O_3 was subjected to X-ray diffraction (XRD) on a Seifert Iso-Debyeflex 2002 diffractometer using Cu–Kα radiation (wavelength =1.54Å) in order to check the phase purity and calculate average crystallite size. A small amount of the sample powder was dispersed in acetone and was sonicated for 15 minutes. A small drop of this solution was dropped on a Carbon coated copper grid and was used for TEM characterization using an FEI Tecnai G2 electron microscope operated at 200 kV. Room temperature isothermal magnetization (M-H) was done by using a vibrating sample magnetometer (VSM). Mössbauer absorber was prepared by sandwiching powder samples (approximately 40 mg) between two layers of a cellophane tape in a copper ring of inner diameter 13 mm. The data were recorded in a transmission geometry using a conventional ^{57}Fe constant acceleration Mössbauer spectrometer employing 25 mCi ^{57}Co (embedded in Rh matrix) source. The experimental set up gives natural iron powder spectrum with linewidth of about 0.28 mm/s and 15% absorption amplitude for the 1st and 6th lines. The spectra were analyzed using least square method assuming Lorentzian lineshapes of the spectra and also by fitting a continuous distribution of hyperfine magnetic field using a procedure described by LeCaer and Dubois (LeCaer and Dubois, 1971).

3. Results and Discussion

The sample obtained after annealing at 400^{0}C was in the form of a yellow-brown powder. This sample was characterized by X-ray diffraction (XRD) and TEM which revealed that it has the crystal structure of meghamite. The magnetic properties of these nanoparticles were characterized by a vibrating sample magnetometer (VSM), and their texture from Mössbauer spectroscopy.

3.1 X-ray diffraction

Figure-1 shows the room temperature X-ray diffraction pattern of the γ-Fe_2O_3 nanoparticles annealed at 400^{0}C. All the XRD peaks could be identified with γ-Fe_2O_3 structure. However, γ-Fe_2O_3 and Fe_3O_4 have the same spinel structure and their XRD peak positions are quite close to each other. The presence of small amount of Fe_3O_4 cannot be ruled out on the basis of XRD pattern alone. Our subsequent characterization, however, will show that the sample does not contain Fe_3O_4 in any significant amount. The crystallite size was obtained using Scherrer formula with FWHM of 311peak after correction for the instrumental broadening. The

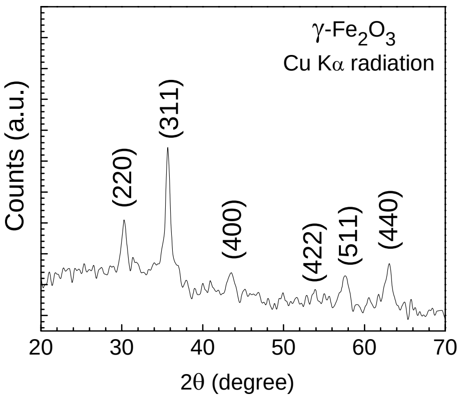

Figure 1. Room temperature X-ray diffraction pattern of γ-Fe_2O_3 nanoparticles using Cu–Kα radiation.

average crystallite size comes out to be 18 nm. There is significant background which normally results from poor crystallinity, fine size with large number of broken bonds at the surface or improper sample holding. In our case it is likely to result from nano size and distorted surface morphology.

3.2 Transmission electron microscopy (TEM)

Figure 2 shows TEM micrograph of the γ-Fe_2O_3 powder. Though the sample was ultrosonicated for 15 minutes in acetone before putting the drop on the grid, the particles seem to have large tendency for agglomeration. This could be due to enhanced magnetic interaction between the particles. Individual particles are indeed in the nanometric range 15-25 nm and are in nonspherical shape. A significant number appears to assume rod-like shape. The agglomeration tendency indicates that inter-particle interactions are quite large for this system. Also, because of the magnetic interaction between the particles, some defocusing is observed in the micrograph. Figure 2b shows SAED pattern where rings can be seen clearly confirming the polycrystalline nature of the sample but the pattern of bright dots over the rings indicates preferred crystallographic texture. The crystallographic anisotropy might have resulted from the small size and the nonspherical shapes of the particles. Indexing of all the rings could be done using the d-spacing of γ-Fe_2O_3 crystal structure. XRD and TEM experiments thus strongly suggest that the process yielded γ-Fe_2O_3 nanoparticles in pure phase, but of varying shape and susceptible for agglomeration.

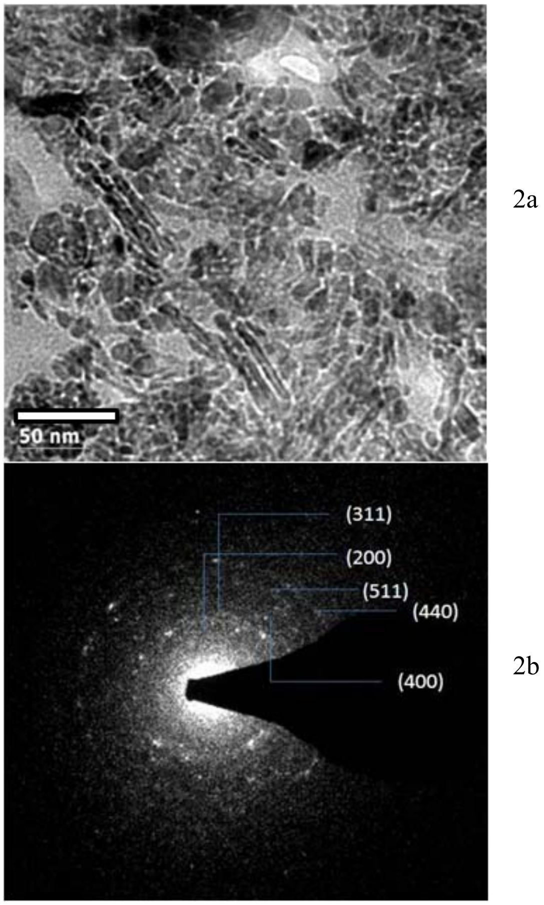

Figure 2. (a) Transmission Electron Microscopy (TEM) images of γ-Fe_2O_3 nanoparticles, (b) SAED pattern.

3.3 Magnetic properties

The M-H curve was measured for the full cycle between -1.75 T to 1.75 T at room temperature and the results are shown in Figure 3. It shows a very thin hysteresis loop with a coercive field of about 60 Oe. The magnetization becomes 13.35 emu/g at 1.75 T which is much less than the known value of 73-74 emu/g for the magnetization of bulk size sample (Bate, 1986). Spin canting at the surface is known to give reduced magnetization in nanoparticles (Coey, 1971), but this reduction is seldom more than 15-20%. At the average size of 18 nm one should not expect superparamagnetic relaxation to reduce the magnetization so much. There could be a possibility of smaller particles going superparamagnetic at the time scale of Vibrating Sample Magnetometer having a small contribution in reducing the magnetization. The coercivity of 60 Oe, though small, shows that the particles do have a ferro/ferrimagnetic ordering.

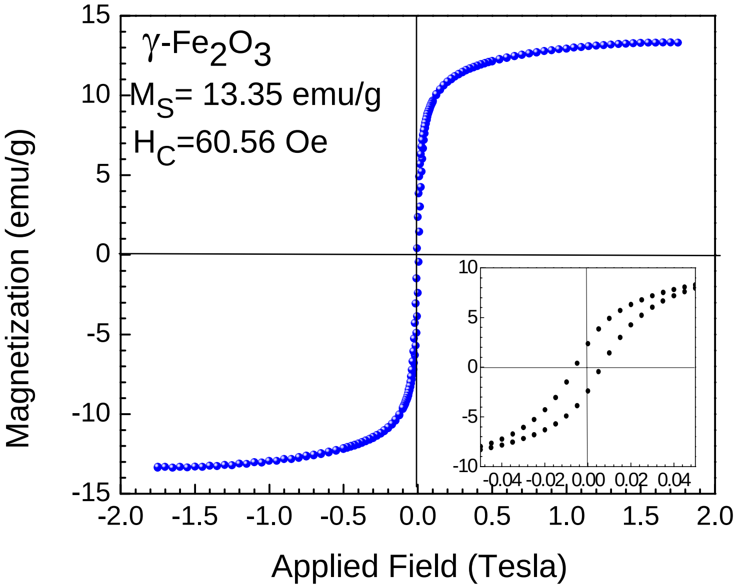

Figure 3 Room temperature M-H curve for γ-Fe_2O_3. (Small saturation magnetization and low coercivity indicates some especial magnetic ordering of these particles).

Tronc *et al.* (2005) have studied the effect of inter-particle interaction on Mössbauer spectra of nanosize maghemite. Different degree of inter-particle interaction was observed by preparing particles with surfactant coating and without surfactant coating. It was observed that temperature evolution of superparamagnetic versus magnetic component was different in the two cases and blocking temperature significantly increased when inter-particle interaction was strong.

The inter-particle interaction could be of dipole-dipole type where the dipole moments of the neighboring particles interact as a whole or it could be of exchange type where the dipole moments at the surface of the particles land up in exchange interaction. The later can change the magnetic properties much more extensively.

The TEM image shows a distribution in the shape of the particles which will give rise to different shape anisotropies. The interaction between different particles could result in some kind of freezing of magnetic moments resulting in the net lower magnetization. It is seen from Figure 3 that the magnetization does not saturate even upto 1.75 T and has a slowly increasing trend consistent with the freezing possibility.

3.4 Mössbauer spectroscopic studies

Mössbauer spectrum of the present sample at room temperature is shown in the Figure 4a. One can see quite sharp lines of the magnetically split spectra with clear kinks in the first, second, fifth and sixth lines showing that it is a combination of sextets. An unconstrained fit to two sextets with area ratio 3:2:1:1:2:3 gives B_{hf}=46.2 and 49.7 T with isomer shifts 0.32 and 0.35 mm/s. The fit is expectedly poor but gives an estimate of the parameters. This fitting is not used any more for quantitative interpretations. The spectrum is fitted to unconstrained 12 single Lorentzian lines and these are later grouped into two sextets according to the line positions. The solid line in Figure 4a corresponds to this fit. The peaks corresponding to the smaller B_{hf}, called A-site peaks and those corresponding to larger B_{hf}, called B-site peaks could be easily identified. These come from iron atoms sitting at tetrahedral voids and octahedral voids respectively in the fcc oxygen lattice.

Mössbauer Studies clearly reveal that the magnetic ordering in the sample is different from what one can expect from a normal powder sample of maghemite. γ-Fe_2O_3 has a spinel structure which can be written as $(\mathrm{Fe})[\mathrm{Fe}_{5/3}\square_{1/3}]\mathrm{O}_4$ where the parentheses denotes the tetrahedral sites (A-sites) and the square brackets octahedral sites(B-sites). The symbol $\square$ is written for vacancies. Iron ions at both sites are in Fe^{3+} states. In contrast, iron atoms in Fe_3O_4 have a mixed valency of Fe^{2+} and Fe^{3+} and gives larger isomer shifts in Mössbauer spectra. Typically the two sextet components in γ-Fe_2O_3 are barely distinguished in Mössbauer spectrum as the Mössbauer parameters are close. At room temperature, the value of B_{hf} for both A and B sites are generally in the range 50.0 to 50.5 T for the bulk size γ-Fe_2O_3, the one for A-site is slightly smaller than that for B site. The isomer shift (with respect to α-Fe) for the A site is about 0.25 mm/s and that for B site is around 0.35 mm/s. The quadruple splitting is almost zero.

While XRD peak positions and TEM ring radii of the sample nicely matched with the expected values of γ-Fe_2O_3 peaks, Mössbauer parameters are quite different from the typical values. The hyperfine field values are smaller than the standard values and more importantly, the sextets (assuming two) are much more separated than usual. The nano size is known to decrease the effective hyperfine field because of the fluctuation of the magnetic moment about an easy direction of magnetization and the present sample can show up nanosize effects. However, when the field is decreased, the lineshape gets distorted due to size distribution and the smaller particles becoming superparamagnetic. In our case the lineshape is almost intact. Hence the fields observed are too small to be accounted by nanosized effects. The different set of parameters indicates a different kind of magnetic coupling, probably due to the shape anisotropy and inter-particle interaction.

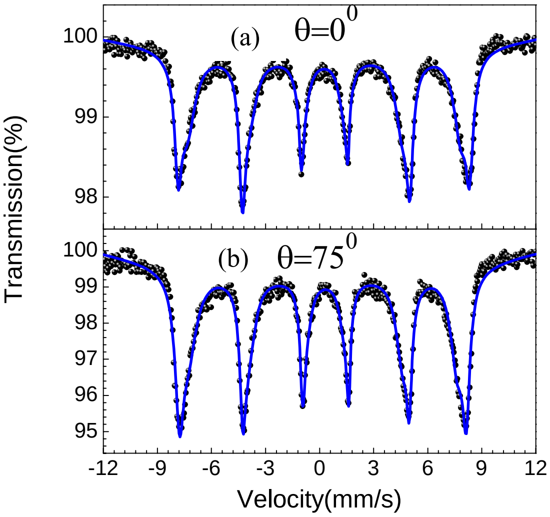

Figure 4 Room temperature Mössbauer spectra of γ-Fe_2O_3 nanoparticles at different angles between the γ-rays and normal to the sample plane a) 0^0, b) 75^0 (see text).

No evidence of Fe_3O_4 is observed in the Mössbauer spectrum which is very sensitive to distinguish Fe^{2+}/ Fe^{3+} components. Fe_3O_4 gives two sextets with different isomer shifts resulting in a good overlap of sixth lines and well separated first lines. The present spectrum has no such component.

The powder was sprinkled on a cellophane tape to form the Mössbauer sample. No external magnetic fields are applied. The thickness of the sample is 40 mg/cm^2, which would correspond to a thickness of several tens of microns. This is a large thickness for randomization of particles in three dimensional space but still thin as far as the self absorption affecting the linshape is concerned. The dimensionless effective thickness t (= σ_0f d) is still less than 1. The larger area of 2-5 lines than expected (area ratio should be 3:2:1:1:2:3 for a randomly oriented powder sample) indicates that the magnetic moments are not randomly oriented in the volume of the sample. It is possible that the rod like shape and inter-particle interaction have oriented the particles in a particular way to give this bulk anisotropy. The increase in the intensity of 2-5 line suggests that the angle of the moments with the gamma ray is closer to 90^0. This would be the case if the magnetic moments of the particles preferentially oriented in the plane of the cellophane tape.

The other possibility is the presence of more environments for iron in the sample giving rise to number of sextet components, whose superposition can give the observed overall peak area ratio. TEM results show that the shapes of the particles have a distribution and particles are agglomerated to a large degree. The iron atoms in the interparticle regions are therefore likely to have different atomic environments. A small third sextet component in the Mössbauer spectrum, whose outer lines overlap with the middle lines of the main sextets was tried. However, data analysis with three sextet fit did not give physically acceptable parameters.

To probe into the effect of magnetic texture further, Mössbauer spectra were recorded with the sample rotated about the vertical direction. If the magnetic moments are preferentially distributed in the plane of the sample, such a rotation will, to some extent, randomize the angle between the gamma ray direction and the magnetic moments, from the value 90^0 when the sample plane was perpendicular to the gamma rays. Figure 4b shows the Mössbauer spectrum with the rotation angle of 75^0. It is seen that the ratios change as the sample is rotated. The relative areas of the 2-5 lines do decrease as compared to 1-6 lines. The ratio of intensity of 2-5 lines to that of 3-4 line for B-site sextet decreases from 2.8 at 0^0 to 1.7 at 75^0. This supports the proposition of preferred orientation of magnetic moments. It may be noted that the rotation would increase the effective thickness by a factor of sec θ which would affect the intensity ratio in free Lorentzian fitting. But the effective thickness even at the maximum rotation angle of 75^0 would be less than 4 and would only moderately change the line intensity.

The most interesting aspects of the Mössbauer spectrum of the present sample is the area ratio of 2-5 lines versus 1-6 lines and also 3-4 lines. For a thin sample with randomly oriented magnetic moments, the area ratio is ideally 3:2:1:1:2:3. Visually the amplitudes of the 2-5 lines in Figure 4a are more than those of 1-6 lines. The sample used in our study is a powder sample.

The variation in the relative intensity on rotation of the sample shows conclusively that the sample has magnetic texture and the moments are preferentially aligned in the sample plane. The spectra were also fitted to a continuous distribution of hyperfine fields using LeCaer and Dubois algorithm (LeCaer and Dubois, 1971). This assumes standard area ratio 3:2:1:1:2:3 of the components. Figure 5 shows the distributions obtained for $\theta=0^0$ and $\theta=75^0$ cases. Clearly they are quite different. For example, there are much more intense components with $B_{hf} > 50$ T in the case of $\theta=0^0$ than in the case of $\theta=75^0$.

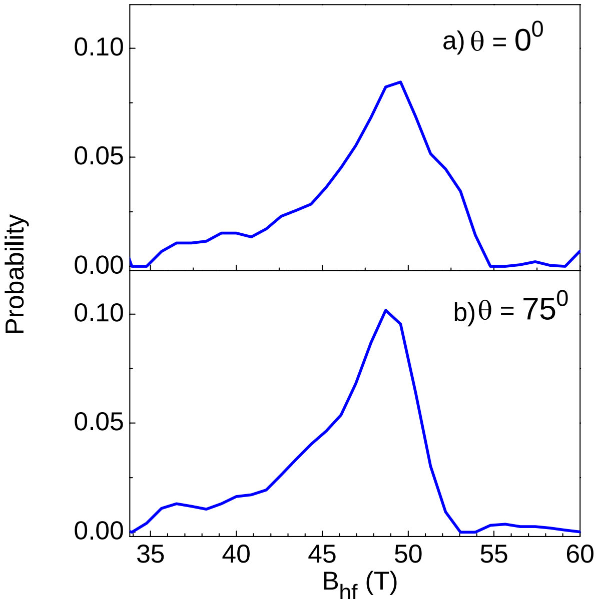

Figure 5 P-B distribution of the Mössbauer spectra of γ-Fe_2O_3 nanoparticles recorded at angles a) 0^0 and b) 75^0 between the γ-rays and normal to the sample plane (see text).

One cannot explain appearance of new sextet components in the Mössbauer spectra (meaning the creation of new iron environment) just by rotating the sample. This also shows that the observed area ratio is not coming due to superposition of several environments even if they are present due to distribution of size and shape, and a fitting based on standard intensity ratio such as LeCaer and Dubois algorithm is not suitable for the analysis.

Ngo and Pileni (2003) have reported elongated maghemite particles, together with hematite particles with large aspect ratio using chemical method. When these particles were deposited as a thin film on an oriented substrate, in presence of magnetic field, the particles oriented along the field. In absence of field during deposition the particles oriented randomly. In contrast our particles aligned in the plane of the powder sample only due to their own interactions.

4. Conclusions

1. Maghemite particles synthesized in this process have somewhat uncommon magnetic structure.
2. The interparticle interaction is strong so that even the powder sample, spread over a sheet in density of about 40 mg/cm^2, shows preferential magnetic moment alignment in the plane of the sample.
3. This magnetic structure is likely to come from the rod-like shapes of the nanoparticles which results in strong shape anisotropy.
4. The magnetization data shows a small coercivity and a small magnetization, not expected from γ-Fe_2O_3.
5. As γ-Fe_2O_3 is a potential candidate for so many biomedical applications, such a novel magnetic structure should be of great interest.

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