Magnetism from Fe₂O₃ nanoparticles embedded in amorphous SiO₂ matrix

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Abstract

Fe₂O₃ nanoparticles are embedded in amorphous SiO₂ matrix by coprecipitation method with varying concentrations. Conditions are optimized to get almost monodispersed Fe₂O₃ nanoparticles with high chemical stability. Microstructure of synthesized nanoparticles is well characterized and found that Fe₂O₃ is in nanocrystalline form and embedded uniformly in amorphous SiO₂ matrix. Enhanced surface reactivity is found for nanoparticles which influences physical properties of the SiO₂ supported Fe₂O₃ system due to adsorption. In oxide nanoparticles, significant number of defect sites at the surface is expected but when supported medium such as SiO₂ it reduces this defect concentration. Field- and temperature-dependent magnetisation studies on these samples show superparamagnetic behaviour. Superparamagnetic behaviour is seen in all the concentration systems but the coercivity observed in the lower concentration systems is found to be anomalous compared to that of higher concentrations. The observed magnetic behaviour comes from either unsaturated bond existing due to the absence of anions at the surface of nanoparticles or reconstruction of atomic orbitals taking place at interface of Fe₂O₃–SiO₂ system.

Keywords Nanoparticles · Superparamagnetism · Electron diffraction · Maghemite

Introduction

Magnetic nanoparticles are very interesting due to applications in numerous areas, such as data storage devices, magnetic sensors, magnetic refrigeration and magnetic fluids besides rich physics involved in their behaviour. In each and every application, where the magnetic nanoparticles are used, reliability on the chemical stability of the particles is highly dependent on methods of synthesis and processing conditions (Ang et al. 2013). Nanoparticles have a large surface area compared with that of bulk materials; therefore, nanoparticles are much more reactive compared to their bulk counterparts (Aslam et al. 2005). The origin of magnetism in bulk ionic compounds or oxides is due to

spatially localized valence electrons. These electrons give rise to localized moments on each magnetic ion in accordance with the Hund's rules (Kroll et al. 1996; Vollath et al. 1995; Kodama 1999). Reduced coordination at the surfaces of the nanoparticles alters the exchange interactions between electrons, and results in a disordered spin configuration near the surface which reduces the magnitude of moment in nanomaterials compared to that of bulk compounds (Blanco-Esqueda et al. 2015). The magnetic ground state of nanoparticles is strongly influenced by their finite size and microstructure. When size of the particles is smaller than a certain critical length (material specific), then the magnetic properties of nanoparticles become different from its bulk counterpart. Most of the ferromagnetic materials in bulk form, when transformed into magnetic single domain state, behave as superparamagnetic (Teleki et al. 2009). The above features make superparamagnetic nanoparticles very much attractive especially for biomedical applications (Wu et al. 2002).

The unavoidable problem in nanoparticles is their tending to form agglomerate to reduce the energy associated with the large surface area to volume ratio. When nanoparticles agglomerate into micron size, most of their unique physical

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properties either get modified. Much attention is required to maximize individual particles' natural behaviour and many researchers are trying to find a solution by developing processing conditions to prevent agglomeration (Santra et al. 2001). Understanding the magnetic properties of nanometer scale particles is still in infancy state. Iron oxide or iron-containing oxide is widely studied due to their numerous applications in high density recording media, magneto-optical devices, magnetic refrigeration and many more. Magnetic properties of nanocrystalline maghemite (Fe_2O_3) depend strongly on the particle size and shape (Lu et al. 2007). Fe_2O_3 exists in four polymorphs, namely, alpha, beta, gamma and epsilon (Yuan et al. 2010). The most available polymorph is $\alpha\text{-Fe}_2\text{O}_3$, known as hematite, whose structure is rhombohedral–hexagonal and it shows antiferromagnetism below 260 K, known as Morin transition, and above this temperature to 950 K, it behaves as a weak ferromagnet. Another phase $\gamma\text{-Fe}_2\text{O}_3$, which is ferrimagnet, is found in metastable form converted from $\alpha\text{-Fe}_2\text{O}_3$ at high temperatures (Moreno et al. 2002). The structurally body centred cubic β -phase is also a metastable state which is converted from α -phase at above 500 °C (Drbohlavova et al. 2009). Many methods are employed by researchers for synthesizing nanoparticles such as co-precipitation, sol–gel preparation, mechanical crushing of powder and using water-in-oil microemulsion techniques (Woodward et al. 2000; Joy and Date 2000). The agglomeration and aggregation still comes, when the above mentioned methods are followed. Protection strategies are required to keep chemically stable magnetic nanoparticles against degradation during and after the synthesis. When a surfactant is used then the removal of residual surfactant is a tedious procedure (Joy and Date 2000). We followed a single step procedure to get maghemite nanoparticles in silica matrix. This paper reports the synthesis and investigation of the magnetic properties of Fe_2O_3 nanoparticles embedded in amorphous SiO_2 matrix.

Experimental details

The mixed solution of 15 ml of ethanol with 6 ml of double distilled water in a beaker is stirred for 1 h. Then, 0.05 molar concentration of ferric nitrate solution is added slowly and stirring continued for another 1 h, and then 2 ml of tetra ethyl ortho silicate (TEOS) is added slowly and allowed to stir for another half an hour. The pH is adjusted to in between 9 and 10 using ammonia solution. Then the stirring (reaction) is continued for 24 h and the solution is centrifuged with 10,000 rpm for half an hour. To wash the compound distilled water is used during centrifuge for four times and then finally ethanol is used. The final powder is collected and dried in hot air oven at 100 °C for 12 h. Several different molar concentrations of ferric nitrate solution are used

between 0.05 and 0.3. Few batches of prepared samples are identified to investigate their microstructure and magnetic properties. The above synthesis procedure is followed from many authors (Ang et al. 2013; Blanco-Esqueda et al. 2015; Teleki et al. 2009; Wu et al. 2002; Santra et al. 2001). This paper reports the results of various measurements on nanoparticles of $\text{Fe}_2\text{O}_3\text{-SiO}_2$ matrix prepared using four different molar concentrations of ferric nitrate solution.

Results and discussion

Structural and micro-structural studies

Figure 1a, b, c and d shows microstructure of each sample viewed using a High Resolution Transmission Electron Microscope (HR-TEM, Tecnai). Figure 1a displays HR-TEM images and selected area diffraction pattern (SAED) patterns of SFO 1 and Fig. 1b, c and d shows HR-TEM images and SAED patterns of SFO 2, SFO 3 and SFO 4, respectively. Size distribution of the nanoparticles is found to be 4–6 nm. The Selected Area Electron Diffraction (SAED) pattern shows a diffused concentric ring structure-like pattern and left a halo around the bright spot in the centre indicating that the electrons are randomly scattered from $\text{Fe}_2\text{O}_3\text{-SiO}_2$ matrix which is observed in all the samples. Bright spot at the centre is observed in SAED pattern. The lattice d-spacing of SFO 1 is around 0.251 nm which belongs to (110) of $\alpha\text{-Fe}_2\text{O}_3$ is given in Fig. 2 and elemental analysis show Si, Fe and O in the system is given in Fig. 3 for SFO 1. Powder XRD is recorded using Bruker D 8 for all the systems is given in Fig. 4 show large broadening due to amorphous SiO_2 and very small nanocrystals of Fe_2O_3 . After structural and microstructural studies, four different concentrations of $\text{SiO}_2\text{-Fe}_2\text{O}_3$ samples are chosen for magnetization studies, such as 0.05, 0.1, 0.2 and 0.3 molar concentrations are named as SFO 1, SFO 2, SFO 3 and SFO 4.

Magnetization studies

Magnetic properties of $\text{Fe}_2\text{O}_3\text{-SiO}_2$ samples are characterized using a PPMS-VSM (QD). Magnetization versus temperature measurement for each sample is carried out with the applied magnetic field of 100 Oe in zero field cooled (ZFC) and field cooled (FC) conditions from 3 to 320 K. Results are shown in Fig. 5. Magnetization versus field (M–H) is carried out at 5, 10, 20, 30, 50, 100, 200 300 and 320 K for SFO 1 which are shown in Fig. 6, and at selected temperature for other systems are shown in Figs. 7, 8 and 9.

The following features are observed for **SFO 1** system and some of them are unusual.

- (a) M(T) with 100 Oe data measured in ZFC and FC modes overlap each other.

Fig. 1 HRTEM image and selected area diffraction pattern of SFO 1 (a), SFO 2 (b), SFO 3 (c) and SFO 4 (d) systems. Fe_2O_3 nanoparticles are marked with black circles and the background is SiO_2

- (b) Change in curvature is noticed around 40 K in $M(T)$.
- (c) Magnetization curves measured in ZFC as well as FC modes are similar to paramagnetic systems and below 10 K magnetization increases sharply due to dipolar interaction. The magnetization value at 3 K is increased one order of magnitude when compared to room temperature magnetization value.
- (d) Curvature of $M(H)$ gradually changes from 5 to 100 K and at 200 K shape of $M(H)$ is different from measured at lower temperatures and 's' shape is found around near origin.
- (e) $M-H$ measured at 10 K has more change in slope when compared to 5 and 20 K data. Gradual change in slope is observed in every $M-H$ measured at different temperatures.
- (f) $M(H)$ measured at 300 K approaches to saturation and at 320 K shows saturation.

Fig. 2 HRTEM image of SFO 3 and the lattice d-spacing is found to be 0.251 nm belongs to (110) crystal face of $\alpha\text{-Fe}_2\text{O}_3$

- (g) Magnetization value measured in 90 kOe at 3 K is highest and gradually decreases towards high temperature.
- (h) Coercivity increases from 3 to 100 K and the highest value is noticed at 100 K. Values of coercivity and remanence are given in Table 1.

- (i) Remanence increases up to 50 K. Almost same value is noticed at 100 K. Afterwards it starts to decrease at higher temperatures.
- (j) Both coercivity and remanence show same feature with temperature.

Observations in other Systems SFO 2, SFO 3 and SFO 4

Magnetization versus T of all other systems SFO 2, SFO 3 and SFO 4 shows similar behaviour like the one shown by SFO 1. The value of magnetization increased when higher molar ferric nitrates used SFO systems. M versus T data taken in ZFC and FC modes overlap in the entire measured range, which is very similar to found in SFO 1. The change in slope in M–T is observed around 40 K in all the systems like observed for SFO 1. The magnetization versus magnetic field (M–H) in these systems (SFO 2, SFO 3 and SFO 4) show some similarities and differences compared to SFO 1. The coercivity increases in SFO 2 from 5 to 300 K and change in curvature in M–H loop is observed at all measured temperatures and linearity is developed at higher temperature's M–H which is shown in Fig. 5. Shape of M–H curve at 5 K looks like that observed is in a typical antiferromagnetic but does not saturate up to 90 kOe. A similar situation exists at 20 K but almost linear behaviour in M versus H is observed at fields above 50 kOe at this temperature. But at M versus H shows hysteresis nature near origin at a lower field. The coercivity at 20 K (~ 260 Oe)

Fig. 3 Elemental analyses are carried out on SFO 1 using TEM and Si, Fe and O elements are seen

Fig. 4 Powdered XRD pattern is recorded for all systems and large broadening is noticed due to amorphous SiO₂ matrix and very small nanocrystals of Fe₂O₃

Fig. 5 Magnetization against temperature of SFO 1, SFO 2, SFO 3 and SFO 4 systems with 100 Oe in zero-field cooled and field-cooled mode

Fig. 6 Magnetization against field at selected temperature of SFO 1 system

Fig. 7 Magnetization against field at selected temperature of SFO 2 system

is more than what is observed at 5 K (~ 90 Oe) and even more coercivity is observed at 300 K (~ 506 Oe). M–H measured at 300 K shows “S” shape curve near origin and linear at higher field which indicates that coexistence of superparamagnetic and paramagnetic phases. Magnetization value in SFO 3 system shows higher value compared to those found in SFO 1 and SFO 2 as expected due to higher molar concentration of ferric nitrate used. Nature of M–H curves and change in slope

in SFO 3 is very much similar to that observed in system SFO 2. The coercivity values of SFO 3 are around 5 Oe at 5, 10 and 20 K and not having much changes, but at 300 K, the coercivity increases by one order of magnitude (~ 90 Oe) compared to its value at 5 K. M–H curve at 300 K is found similar to that of a paramagnetic material, an enlarged view of the curve close to origin is shown as inset in Fig. 6. Compared to all other systems, SFO 4 shows a different behaviour where the

Table 1 Coercivity and remanence values of SFO systems

S. No.	Temp (K)	H _C (Oe)	H _R (emu/g)	S. No.	Temp (K)	H _C (Oe)	S. No.	Temp (K)	H _C (Oe)
SFO 1				SFO 2			SFO4		
1	5	50	0.00562	1	5	90	1	5	20
2	10	95	0.00527	2	20	260	2	20	20
3	20	185	0.00658	3	300	506	3	300	20
4	30	300	0.0075	SFO3					
5	50	450	0.00823						
6	100	600	0.00782						
7	200	550	0.00629						
8	300	400	0.00443						
9	320	400	0.00433	4	300	90			

highest mole concentration of ferric nitrate solution is used. M–H curve at 5 K shows an antiferromagnetic behaviour and at 20 K there is slight change in curvature above 30 kOe field and it shows linear behaviour like that of a paramagnetic material, which is shown in Fig. 7. Almost same coercivity value of 20 Oe for this system was found at all the measured temperatures even though the highest magnetization value is observed in magnetization versus T measurement. In the SFO 4 system having coercivity value of almost 20 Oe is unique feature among the synthesized Fe₂O₃–SiO₂ matrices because M–H shapes are different at each measured temperature and coercivity is almost same.

M versus T, shown in Fig. 3, follows Curie–Weiss behaviour and the data are analysed above 100 K to calculate effective magnetic moment which is found to be around 1 μ B for all the systems. Our synthesized magnetite particle size distribution is 4–6 nm and Fe₂O₃ particles of this size are expected to stay mostly as in single domain state only. At single domain state retaining, long range magnetic order is difficult to establish and most of the systems prefer to be in superparamagnetic state even though their bulk counterpart shows long range magnetic order. In single-domain particles, all moments are rigidly aligned and behave as a single giant spin. In such single-domain particles, overcoming the rotational barriers caused by magnetocrystalline, magnetoelastic and shape anisotropies is difficult, and nanoparticles can trap in any one of the metastable states, which may give rise to hysteresis. These single domain or very small magnetic individual nanoparticles having large constant magnetic moment behave like a giant paramagnetic atom and response to applied magnetic fields is rapid with negligible values of remanence and coercivity. Several authors reported ferrite nanoparticles and other core–shell type nanoparticles show high field hysteresis due to disordered surface spins. In core–shell structure, the exchange coupling between the surface and core gives rise to variety of spin distributions which causes the relaxation of magnetization due to irreversible reorientations against field. Large coercivities and loop shifts observed in single domain antiferromagnetic nanoparticles suggests that there may be

variety of internal spin distributions available and many number of weakly interacting spin sublattices allow many spin reversal paths against field (Kumar et al. 1996; Anil Kumar et al. 1999; Roy et al. 1995; Sendil Kumar et al. 2017; Kumar et al. 1998; Schuele and Deetscreek 1962; Cannas et al. 2001; Pereira et al. 2010; Lopez-Sanchez et al. 2016).

Finite size affects the magnetic order strongly in nanoparticles. In a situation of metal atoms existing at the surfaces, these have a tendency to contract bond lengths, and therefore average lattice parameter of nanoparticles is reduced. This leads to more overlap of atomic orbitals which reduce the moment per atom. Similarly, when metal nanoparticles are in supported media such as SiO₂, it also reduces the moment and it is difficult to understand intrinsic size effect or interdiffusion with the matrix. When crystal structure changes with size reduction and when more than one phase are present, a balance between surface and bulk free energies decides the physical properties which means that size effect. Our system is Fe₂O₃ nanocrystals embedded in amorphous matrix of SiO₂ which acts as a supported medium. Silica matrix can extend the bond to iron oxide and possibility of interdiffusion occurs. This could cause all anomalous magnetic behaviour in the Fe₂O₃–SiO₂ system as observed (Berkowitz et al. 1999; Kodama et al. 1997; Kodama et al. 1997; Rockenberger et al. 1999; El-Hilo et al. 1993; Ulman 1996; Iwaki et al. 2003; Cannas et al. 1998).

Conclusion

Uniform size distribution of Fe₂O₃ nanoparticles embedded in amorphous SiO₂ with varying concentration has been successfully prepared. Microstructural studies, such as electron diffraction, show Fe₂O₃ is in the nanocrystalline form. Magnetization study show superparamagnetic behaviour and M–H measurements show hysteresis behaviour. Anomalies seen in magnetic properties are attributed to size effect as well as interdiffusion or interfacial adsorption at the surface between Fe₂O₃ and SiO₂ systems.

Fig. 8 Magnetization against field at selected temperature of SFO 3 system

Fig. 9 Magnetization against field at selected temperature of SFO 4 system

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