Study of Relation Between Particle Size And Magnetic Saturation of Synthesized Undoped Iron Oxide

***V. V. DESHMANE[1], A.V. PATIL[2]**

[1]Department of Physics, SICES Degree College, Ambarnath (W), Thane, Maharashtra, India.
[2]L. V. H. College, Panchavati, Nasik, Maharashtra, India.
***Corresponding Author E-Mail:** desh8299vv@gmail.com

Abstract

Nano sized powder of Iron Oxide was prepared using co-precipitation method. Particle size and structure was examined using XRD analysis. Saturation magnetization and other properties were studied using VSM technique. The particles of less than 30 nm size possess low saturation magnetization was confirmed by this study. Result can be attributed to magnetic unisotropy at nano-level.

Keywords: Fe_2O_3, Co-precipitation method, XRD, VSM.

Introduction

Iron Oxide has many application like pigmentation, water purification , drug delivery system etc. It has been a metal oxide of interest since very long period. It is found to work for magnetic purpose also. In the studies for ultrafine ferrite particles like CoFe2O4, MnFe2O4, it has been found that as particle size reduces below 30 nm the saturation magnetisation reduces drastically[1,2].

Particles size effect was observed in manganese ferrites study. As the particles size decreases the saturation magnetization was found to be decreasing[6]. $Ba_2Co_2Fe_{28}O_{46}$ nanocrystals synthesized by sol-gel method were found to have grain sizes ranging from 10 to 25 nm. The saturation magnetization was found lower than that of bulk $Ba_2Co_2Fe_{28}O_{46}$[7]. Fe_3O_4 nanoparticles were analyzed with average particle sizes from 5 to 150 nm. Saturation magnetization decresed as the average particle size is reduced[8]. Iron-carbon nanocomposite thin films with iron concentrations. Iron-rich nanoparticles of 2-8 nm were obtained. The saturation magnetization was found to decrease compared to the bulk value for pure α-Fe [9].Co-precipitation method was used to prepare Manganese ferrite, (MnFe2O4) fine particles in the size range 5-15 nm. The saturation magnetization again followed decreasing trend with decreasing size [10]. ZnO and ferri-magnetic γ-Fe2 O3 composites were prepared by powder pressing. The analysis of the hysteretic behavior showed that saturation magnetization of Fe2O3 is strongly dependent on particle size [11]. A non-aqueous solvo-thermal method was employed to synthesize Cobalt ferrite, $CoFe_2O_4$, nanoparticles in the size range 2-15 nm. This study also showed size dependence of saturation magnetization [12].

In our study we tried to test this. The Fe2O3 particles were prepared by co-precipitation method. The particle size was confirmed using XRD analysis. Magnetic properties of the Fe2O3 particles were studied using VSM technique. Average particle size was found to be 5nm. Saturation magnetization was very low 1 emu/gm. Results drawn for ferrites[1,2,6,7,10] and nanoparticles prepared by various method [8,9,11] were also found true for nano-Fe2O3 particles prepared by co-precipitation method.

Experimental Work

Materials and Methods

Co-precipitation method was used to synthesize Iron oxide nano-particles. All used chemicals ($FeCl_{3.}6H_2O$, NH_4OH) were of analytical grade. At room temperature 10 gm of $FeCl_3$ $6H_2O$ was dissolved in 150ml (DDW) water with continuous stirring. PH=1 was maintained during synthesis by dropwise addition of NH_4OH with continuous stirring. The dispersion was then stirred for 1 hr. and then heated at 80 °C for 2 hrs. This resulted into a brown powder. Thus prepared powder was finally calcined at 500 °C for 4 hrs. in a furnace.[3]

XRD:

The structure and size of the particles were verified using XRD analysis (at NMU Jalgaon). Cu-Kα (1.5404^{0}A) source was used for angle range20-80^0. Peaks were analyzed using X-powder software.

VSM:

Magnetic properties of the sample were studied using vibrating sample magnetometer (VSM). A Cryogenic Ltd. VSM with maximum field range of 10,000 Gauss (at SPPU, Pune) was used for the purpose

Result and Discussion

Morphology:

Figure 1 shows X-ray diffraction graph for iron oxide nano-particles prepared by co-precipitation method. As explained by M. lukouski et al.[4], the graph also confirms the major presence of α-Fe2O3 phase. Other minor peaks are attributed to underlying Fe3O4 content. This is due various oxide phases formed during oxidation process of Iron.

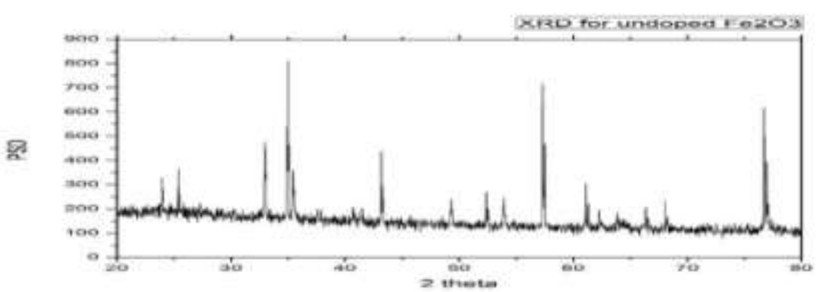

Fig.1: XRD graph of Iron Oxide powder

Table:1 shows the peak analysis data for the above XRD analysis graph. Too closed peaks cannot be analyzed by the X-powder software therefore two peaks are omitted in the tabular form.

Here Scherrer equation $D = \frac{0.9\lambda}{\beta Cos\theta}$ is used for calculation of particle size.

Where, D- particle size,
λ- wavelength of X-ray source
β- full width at half maximum for the peak
θ- diffraction angle

By taking average of all peaks we got average particle size of 5 nm

Table 1: Peak analysis data for XRD peaks

Angle 2θ	**d spacing**	**Intensity**	**Relative Intensity (%)**	**FWHM (radians)**	**Scherer Neglected strain particle size (nm)**	**Corrected Instrumental Particle size(nm)**
32.328	2.767	2010	100	7.2130	8	8
34.789	2.577	1750	87.1	15.601	3	3
40.035	2.250	1413	70.3	27.363	10	10
48.617	1.871	1425	70.9	32.896	5	5
53.280	1.718	1530	76.2	32.791	4	4
61.624	1.504	1359	67.6	45.646	3	3
63.270	1.470	1309	65.1	48.078	2	2

Magnetic Properties:

Magnetic measurements for sample were carried out on a cryogenic ltd. Vibrating sample magnetometer at room temperature. Mass of the sample was 25.1 milli grams and maximum possible applicable field of VSM was 10 KGauss. Hysteresis curve was plotted for analysis. Figure:2 shows the hysteresis plot for the sample. Saeed Kamali et al. got saturation magnetization of about 6 emu/gm for nano particles synthesized by sol-gel method[5]. At applied field of 9276 Gauss we got relatively very low saturation magnetization of 1.0037 emu/gm.

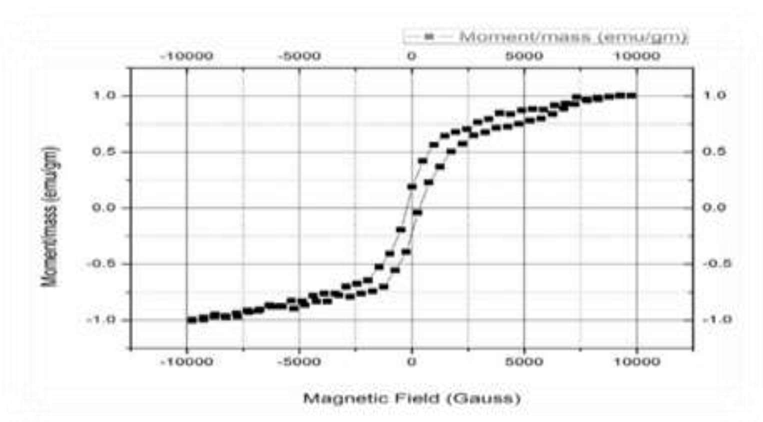

Fig. 2: M vs H curve (Hysteresis curve) for the iron oxide (majorly Fe_2O_3)

Fig. 3 Bar graph shows quadrantwise distribution of curve surface area. Areal distribution shows slight shift in fourth quadrant.This is due to absolute negative retentivity(-0.22160 emu/gm) being slightly

more than the positive retentivity(0.18403 emu/gm). Positive coercive field (334.76 Gauss)is found to be slightly larger tha absolute negative coercivity. Average coercivity
is about 284.46 Gauss.

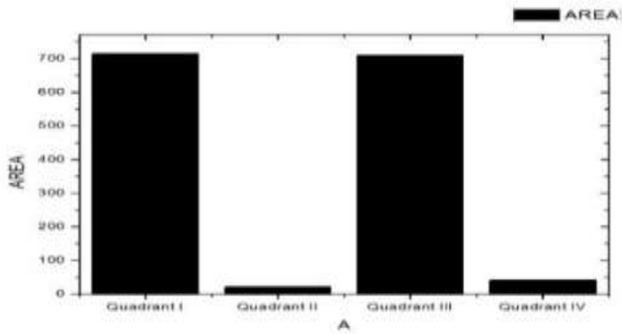

Fig. 3: Quadrantwise area distribution of M/H curve

Conclusion

Morphological and magnetic properties of Fe_2O_3 nano particles prepared by co-precipitation method were studied using XRD and VSM analysis. T. Sato et al. observed drastic reduction in saturation magnetization for particles of ferrites below 20nm size[1]. Our study confirms similar conclusion of very low saturation magnetization (1 emu/g) for Fe_2O_3 particles of 5nm size prepared by co-precipitation method. Saturation magnetization decrease can be mainly due to magnetic unisotropy[1] or can be explained by assuming the existence of a nonmagnetic layer on the surface of the particles[7,8]. SQUID study can be further made to count amount of un-isotropy.

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