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Study of Temperature Dependence for Iron oxide Nanoparticles

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ABSTRACT

Iron oxide exhibits different phases such as hematite (α-Fe_2O_3), maghemite (γ-Fe_2O_3) and magnetite (Fe_3O_4). Of these, hematite is the most stable phase. Nanoparticles of hematite have versatile applications and are used as pigments, adsorbents, photo-catalysts, solar energy conversion, lithium- ion batteries and sensors. Hematite nanoparticles were synthesized by Sol- Gel method using ferric nitrate and oxalic acid as precursors. The as-prepared samples were subjected to calcination at different temperatures from 400 °C to 800 °C. These samples were characterized by X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM). The size of the particles was calculated from the XRD spectra with the help of Scherrer formula. It was found to increase from 52 nm at 400 °C to 67 nm at 800 °C. The phase of the synthesized nanoparticles was found to be α-Fe2O3 (hematite) with hexagonal rhomb-centered structure. SEM results show hexagonal morphology of the particles and confirm the increase in size with temperature. The local lattice distortion (strain) of these nanoparticles was calculated using Williamson-Hall plot and was found to be about 0.06%. These results are discussed in this paper.

Keywords: Hematite, Sol-Gel method, Williamson-Hall plot.

INTRODUCTION

In the past decade magnetic nanoparticles have been under observations of scientific and technological interests due to their strong influence on chemical and physical properties. Among them iron oxide nanostructures have gained remarkable attention due to its wide range of applications in the various fields like gas sensor, catalysis, lithium-ion batteries, magnetic recording media, pigments and water treatment [1-4]. This transition metal oxide is considered as an ideal candidate for many technological applications due to its low cost, biodegradability, high resistance to corrosion and high stability [5-7]. Iron oxide is found in different polymorphs such as α-Fe_2O_3, γ- Fe_2O_3 and Fe_3O_4. Among all α-Fe_2O_3 (hematite) is the most stable and extensively studied structure. Bulk hematite has wide band-gap energy of 2.2 eV which gets enhanced in the nanoscale [8] regime. This property is used in many applications like solar energy conversion and photo catalysis.

A variety of methods like high energy ball milling method [9], hydrothermal approach [10] and auto-combustion method [11] had been reported to synthesize α-Fe_2O_3 nanoparticles. However it is still a challenge to yield mono-size nanoparticles. In the present work we report synthesis of iron oxide nanoparticles by sol-gel technique.

METHODOLOGY

Iron oxide nanoparticles were synthesized by sol-gel method. All the AR-grade chemicals in the present work were obtained from Merck and used as received. Homogeneous solution of ferric nitrate was prepared in organic solvent under continuous stirring and used as a precursor. Small amount of oxalic acid was added drop by drop with stirring in this precursor solution as a thickening agent. This colloidal solution is heated up to 80 ^oC with continuous stirring for 15 minutes. The stirring was then stopped and heating continued till brownish thick gel was formed. It was then dried overnight in air. The dried gel was calcined from 400°C to 800°C for 2 hours each.

The as-synthesized iron oxide nanoparticles were investigated in terms of their morphological, structural and compositional properties by using various analytical techniques. The analysis of crystalline properties like crystalline nature, phase and the size of the as-prepared nanoparticles were determined by X-Ray Diffraction (XRD) using D8 Advance Diffractometer. The morphology of these particles was carried out by Scanning Electron Microscopy (SEM) using JEOL JSM 5600. The Energy Dispersive Spectroscopy (EDS) was used to estimate the atomic percentages of iron and oxygen in the samples. All these measurements were carried out at the Department of Physics, University of Pune.

RESULTS AND DISCUSSION

Structural and morphological Analysis:

The crystal phase was examined by X-ray diffraction with Cu-Kα radiation (λ = 1.54 Å) in the range of 10-80° with a scanning rate of 2°/min. Fig. 1 shows comparative XRD patterns of all the iron oxide samples calcined at various temperatures from 400 °C to 800 °C. The finite width of the diffraction peaks clearly indicates the formation of nanoparticles. All diffraction patterns match well with that of JCPDS data no. 860550 indicating the formation of α- Fe_2O_3 phase of iron oxide nanoparticles. Hexagonal – rhombo centric lattice structure is seen in these crystals of α- Fe_2O_3 nanoparticles. Moreover no extraneous peaks appear in the spectra which clearly indicate the formation of pure samples. The particle size is determined by using Debye Scherrer formula [12] and increases from 52 nm to 67 nm as the calcination temperature increases from 400 °C to 800 °C. It is usually seen that when a particle enters into a nano regime, a common phenomenon observed is lattice strain. This lattice strain can influence the properties of the nanostructures. X-ray diffraction analysis is widely used to determine the lattice strain. The average grain size of the synthesized nanoparticles could be due to finite size effect or due to a combined effect of size and strain. The Williamson- Hall plot is plotted to determine the particle size and the lattice strain. Fig. 2 presents a plot of $(\beta \cos \theta)/0.94\lambda$ versus $(4 \sin \theta)/0.94\lambda$ for all

samples. The crystalline size (d) and the local lattice distortion η can be estimated by fitting and extrapolating the linear curve. The reciprocal of the y intercept gives the particle size while the slope of the linear curve gives the value of the local lattice distortion η [13]. It is observed that the local lattice strain remains constant (0.06 %) for the α- Fe_2O_3 nanoparticles calcined at different temperatures from 400 °C to 700 °C. But for the α- Fe_2O_3 nanoparticles calcined at 800 °C the strain increases up to 0.09 %. We are finding the possible reasons behind this sudden increase. The variation of particle size and local lattice strain as a function of calcinations temperature is listed in the following table1.

Table 1: Variation of particle size and lattice strain with temperature

Temperature (°C)	Particle Size (nm)	Lattice Strain (ηin %)
400	52.55	0.062
500	58.09	0.061
600	61.69	0.060
700	63.72	0.062
800	67.03	0.090

The morphology evolution of the α- Fe_2O_3 nanoparticles is studied by SEM. The SEM image of the as-prepared α- Fe_2O_3 nanoparticles calcined at 800 °C is shown in Fig. 3.

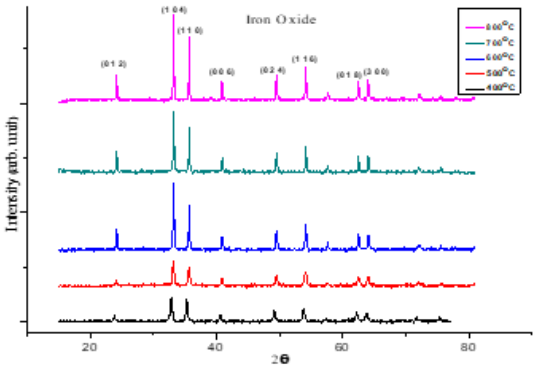

Fig. 1: XRD Spectra of Iron oxide Nanoparticles

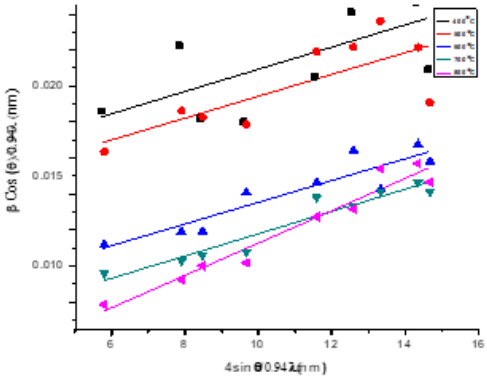

Fig. 2: W-H Plot for Iron oxide Nanoparticles

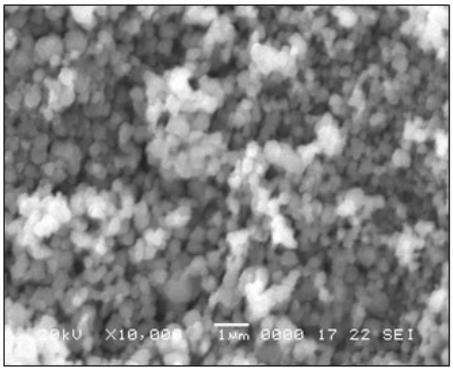

Fig. 3: SEM Image of Iron oxide Nanoparticle

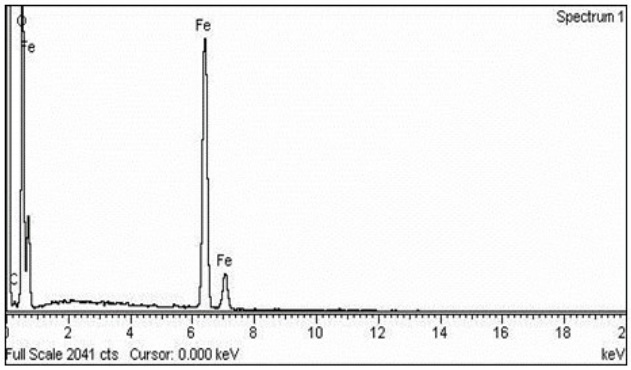

Fig. 4: EDS Spectra for Iron oxide Nanoparticles

The SEM image shows formation of well separated and uniformly distributed nano-particles of α- Fe_2O_3. These nanoparticles exhibit hexagonal symmetry. The Energy-dispersive spectroscopy (EDS) is a useful tool for confirming the atomic percentages of the constituent elements in the prepared sample. Fig. 4 presents the EDS results of the as-prepared α- Fe_2O_3 nanoparticles calcined at 800 °C. It contains the peaks corresponding to only iron and oxygen thereby confirming the purity of the sample.

CONCLUSION

In the present work α-Fe2O3 nanoparticles of various sizes have been successfully synthesized by simple Sol-Gel method. The as-prepared iron oxide nanoparticles show α-Fe2O3 phase and exhibit hexagonal symmetry. The dependence of size on temperature and the corresponding local lattice distortions have been determined.

Conflicts of interest: The authors stated that no conflicts of interest.

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