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SYNTHESIS AND CHARACTERIZATION OF α-Fe_2O_3 NANOPARTICLES AND THIN FILMS

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ABSTRACT
Nanoparticles of iron oxide (α-Fe_2O_3) were prepared by chemical co-precipitation method. X-ray diffraction data confirms the synthesis of single phase of iron oxide (α-Fe_2O_3) nanoparticles with hexagonal structure. The lattice parameters are a= 5.038 Å and c=13.754 Å. The average crystallite size is 29 nm. FTIR analysis show absorption peak at 473.62 cm^{-1} corresponding to Fe-O bond which also confirms the formation of iron oxide. Iron oxide thin films were then deposited by spin coating technique. Optical study of the film showed strong absorption below 500 nm wavelength and high transparency towards red color. The band gap of α-Fe_2O_3 is found to be 2.65 eV with direct band to band transitions.

KEY WORDS: Co-precipitation, hematite, Iron oxide, Nanoparticles.

INTRODUCTION

It is well known that α-Fe_2O_3 (hematite) nanoparticles are particularly appealing for experimental and theoretical investigations in view of their technological applications. They have been widely used as red pigments, catalysts in dehydrogenation reactions, anticorrosive agents, and starting material in the synthesis of magnetic ferrites. Their applications in nonlinear optics and gas sensors have also been investigated recently (Dong *et al.*, 2000). As a key component in modern image devices such as LCD panels and digital cameras, color filters are used to convert white light into the three basic colors to provide colorful images in conjunction with other component units (Wang *et al* 2010). For this, considerable efforts have been made in the synthesis techniques of iron oxide nanoparticles. Various processing routes such as chemical vapor deposition, sol-gel process, spray pyrolysis, chemical co-precipitation method, hydrothermal technique, forced hydrolysis, micro-emulsion technique, etc. have been developed for synthesizing iron oxide nanoparticles (Nidhin *et al.*, 2008; Drbohlavova *et al.*, 2009; Basavaraja *et al.*, 2010; Shahane *et al.*, 2010; Roshan *et al.* 2011). Among the various methods chemical co-precipitation is most suitable method as it has a better control on the size and shape of the synthesized nanoparticles (Shahane *et al.*, 2010). This paper describes the synthesis and characterization of iron oxide (α-Fe_2O_3) nanoparticles and thin films.

EXPERIMENTAL DETAILS

The nanoparticles of hematite having chemical formula Fe_2O_3 are prepared by co-precipitation method using ferric chloride ($FeCl_3$) and ammonia solution (NH_4OH) as the starting materials. Ferric chloride was dissolved in distilled water to form a clear solution. To this ammonia solution was added slowly. The pH of solution was adjusted to 8.5. The solution mixture was heated at 60°C and stirred vigorously with a magnetic stirrer for about one hour. Oleic acid was added to serve as surfactant. Oleic acid covers the NPs and prevents agglomeration. The precipitate was washed several times with distilled water to remove the salts. The precipitate was then dried by heating in air using hot plate kept at 80℃ (for 2 hours). The solid product was then grind in an agate mortar to make them powder. The powder was annealed at 500℃ for two hours to improve the crystalline properties of the material.

The crystalline phase of the prepared sample was identified by X-ray diffraction technique using RIGAKU make MINI FLEX II powder X-ray diffractometer with CuK_α radiation ($\lambda = 1.540562$ Å) operated at 30 kV and 15 mA. Scanning was performed from 20° to 80° at a step size of $5°/s$. FTIR transmission spectrum was recorded on Perkin Elmer Spectrum 65 Spectrometer from 4000 to 400 cm^{-1}. Iron oxide thin film was deposited on glass substrates by using a spin coating technique. The synthesized powder was dissolved in m-cresol and films were deposited using spin coating unit (MILMAN). The substrates were rotated at 3000 rpm for 2 minutes and then heated at 400°C for 1 minute. The UV-VIS spectrum of nanocrystalline iron oxide thin film was recorded using Shimadzu UV-VIS-NIR spectrophotometer (UV-3600) from 300 to 800 nm wavelength range.

RESULTS AND DISCUSSION

The XRD pattern of the annealed powder is presented in Fig. 1. The pattern was compared with standard JCPD data. All peaks in the diffractogram are in good agreement with the standard data of hematite (JCPDS card no. 33-0664) which confirms the formation of single crystalline phase of hexagonal hematite (α-Fe_2O_3) with space group R3-cH. The intense peaks in the diffractogram suggest the crystalline nature of the sample.

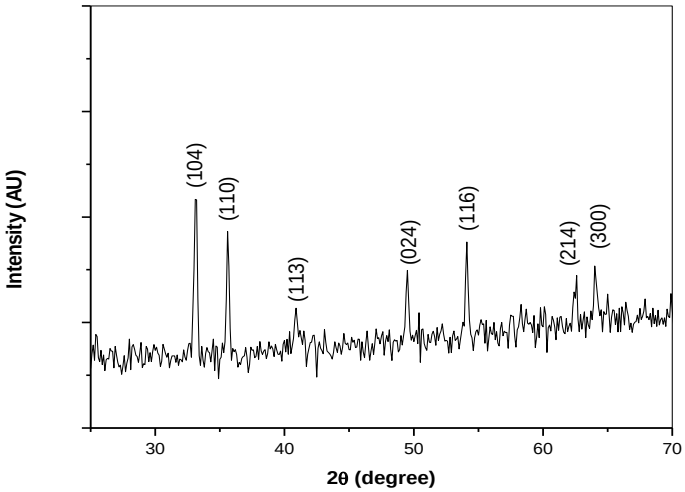

Figure 1: X-ray diffraction pattern of as-synthesized Fe_2O_3 sample

Lattice parameters 'a' and 'c' are determined and found to be 5.038 Å and 13.754 Å, respectively. These values are in good agreement with standard data (a=5.0356 Å and c=13.7489Å). The material shows negligible strain. Average particle size (D) of the synthesized powder was obtained from the main peak using Scherer's formula:

$$D = \frac{k\lambda}{\beta \cos\theta} \quad \ldots (1)$$

where, D is the particle size, k is a grain shape dependent constant (here assumed to be 0.89 for spherical particles), λ the wavelength of the X-ray used, β is Full Width at Half Maximum (in radian) for the diffraction peak under consideration and θ is diffraction angle in degree. The average crystallite size of the particle is 29 nm. Thus, nanoparticles of Fe_2O_3 with less crystallite size can be prepared by co-precipitation method. Fig. 2 shows FTIR spectra of the sample. The FTIR spectrum shows number of absorption lines.

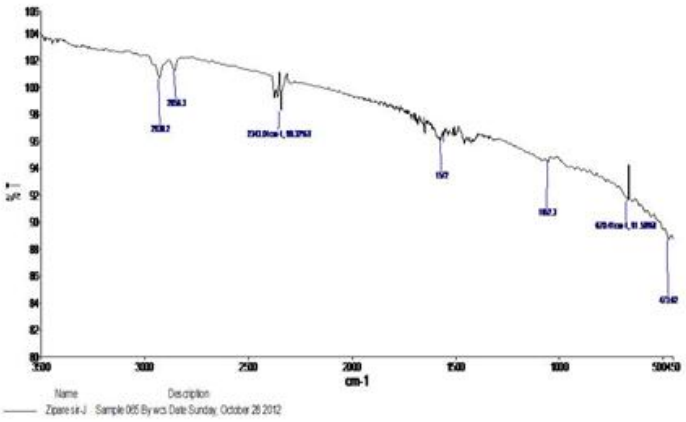

Figure 2. FTIR spectrum of as-synthesized sample

The peak appeared at 473.62 cm^{-1} is indicative of stretching and the variation modes of Fe-O which confirm the presence of crystalline Fe_2O_3. The peak appearing at 1052.3 cm^{-1} is likely related to CH-OH bonds in oleic acid. The band at 1572 cm^{-1} is assigned to the vibration of C=C bond. Bands observed at around 2930.2 cm^{-1} and 2856.3 cm^{-1} are attributed to the stretching vibration of $–CH_2$ and $–CH_3$ band in oleic acid (Zhao *et al.*, 2006).

Figure 3. Shows the optical absorption spectrum of iron oxide thin film deposited by spin coating technique. The spectrum showed strong absorption below 500 nm wavelength and high transparency towards red color. The spectrum was studied to evaluate band gap and nature of the transition. Figure 4 shows the variation of $(\alpha h\nu)^2$ versus hν. The plot shows straight line behavior on the higher energy side that confirms direct type of transitions involved in these films. The band gap of α-Fe_2O_3 is found to be 2.65 eV. Similar results have been reported by Banerjee et al. for sol-gel synthesized Fe_2O_3 nanoparticles (Banerjee *et al.* 2011).

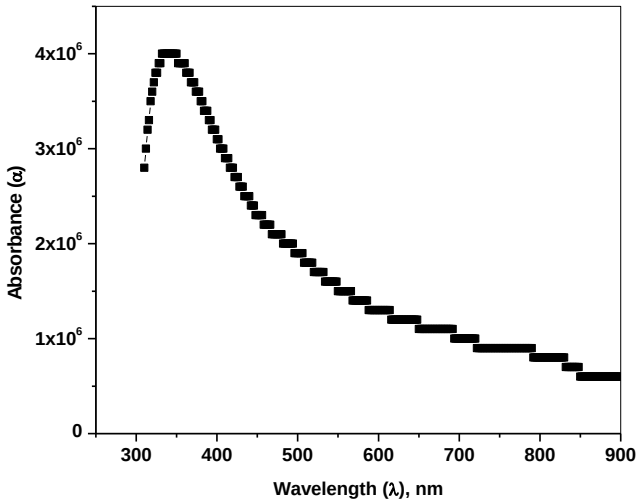

Figure 3. Optical absorption spectra of iron oxide thin film

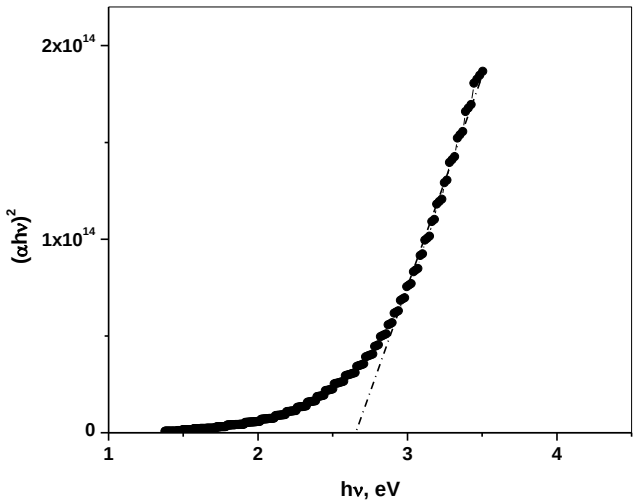

Figure 4. Variation of $(\alpha h\nu)^2$ vs hν for the determination of band gap

CONCLUSION

Nanoparticles of iron oxide can be synthesized through chemical co-precipitation route. All peaks in the diffractogram are in good agreement with the standard data of hematite. The intense peaks in the diffractogram suggest the crystalline nature of the sample. The material shows negligible strain. The average crystallite size of the particle is 29 nm. FTIR analysis also confirms the formation of iron oxide. Optical study of the film showed strong absorption below 500 nm wavelength and high transparency towards red color. The band gap of α-Fe_2O_3 is found to be 2.65 eV with direct band to band transitions. The method is suitable for synthesis of nanocrystalline iron oxide powder with controlled size.

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