Superlattices and Microstructures 86 (2015) 306–312

Contents lists available at ScienceDirect

Superlattices and Microstructures

journal homepage: www.elsevier.com/locate/superlattices

Sol–gel synthesis and characterization of α-Fe_2O_3 nanoparticles

K. Raja [a], M. Mary Jaculine [b], M. Jose [c], Sunil Verma [d], A.A.M. Prince [e], K. Ilangovan [a], K. Sethusankar [a], S. Jerome Das [b,*]

[a] Department of Physics, Ramakrishna Mission Vivekananda College, Chennai 600 004, India
[b] Department of Physics, Loyola College, Chennai 600 034, India
[c] Department of Physics, Sacred Heart College, Tirupattur 635 601, India
[d] Laser Materials Development and Devices Division, Raja Ramanna Centre for Advanced Technology, Indore 452 013, India
[e] Department of Chemistry, Ramakrishna Mission Vivekananda College, Chennai 600 004, India

ARTICLE INFO

Article history:
Received 30 April 2015
Received in revised form 13 July 2015
Accepted 15 July 2015
Available online 16 July 2015

Keywords:
Nanomaterials
X-ray diffraction
Optical properties
SEM analysis
Hysteresis

ABSTRACT

The α-Fe_2O_3 nanoparticles have been successfully synthesized via sol–gel technique. The lattice parameters, the lattice strain and crystallite size were investigated by X-ray diffraction (XRD) analysis. Fourier transform infrared spectroscopy (FT-IR) analysis has been carried out to confirm the presence of functional groups. Using UV–visible spectrum, the optical band gap energy is estimated as 2.55 eV. The morphology and chemical composition of the α-Fe_2O_3 nanoparticles were examined by high resolution scanning electron microscopy (HR-SEM) and energy dispersive X-ray diffraction analysis (EDX) respectively. The hysteresis loop was traced out using vibration sample magnetometer and the values of coercivity and saturation magnetization are found out to be 3891 G and 0.4193 emu/g respectively.

1. Introduction

Magnetic nanomaterials have been in the trajectory for scientific and technological interests due to their strong influence on physical and chemical properties. Iron oxide nanoparticles are widely used in data storage devices, electrodes, magnetic fluids and bio-medicines [1–7]. The iron oxide exhibits variety of crystalline structures such as hematite (α-Fe_2O_3), akaganeite, maghemite (γ-Fe_2O_3) and magnetite (Fe_3O_4) [8,9]. Among these, the eco-friendly, economically flexible, thermally stable, abundant and non-toxic α-Fe_2O_3 nanoparticles have been towed the methodical attention in recent years [10–12]. They are extensively used as red pigments [13], catalysts [14], anticorrosive agents [15], high density magnetic recording media [16], printing ink [17], magnetic resonance imaging (MRI) contrast agents [18] and nanofillers [19]. The promising applications of α-Fe_2O_3 nanoparticles are also in nonlinear optics [20], gas sensors [21,22], lithium ion batteries [23] and dye sensitized solar cells [24]. Various techniques are available to prepare α-Fe_2O_3 nanoparticles like forced hydrolysis [25], hydrothermal technique [26], micro-emulsion technique [27], sol–gel process [28], template assisted synthesis method [29], vapor–solid growth technique [30], high energy ball milling [31] and thermal decomposition technique [32]. Review of literature reveals that the size and morphology of α-Fe_2O_3 nanoparticles strongly depend on the composition of precursors, pH of the solution and rate of heating/cooling during synthesis. Thus a considerable attention must be paid on the optimization of experimental parameters to avoid the formation of undesired products [33–35]. This manuscript presents the synthesis of α-Fe_2O_3 nanoparticles using ethylene glycol with ferric nitrate crystals and their interesting structural, optical and magnetic properties.

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* Corresponding author.
E-mail address: jeromedas.s@gmail.com (S. Jerome Das).

http://dx.doi.org/10.1016/j.spmi.2015.07.044
0749-6036/

2. Experimental

A 0.15 M of ferric nitrate ($\mathrm{Fe\ (NO)_3 \cdot 9H_2O}$) was dissolved in 50 ml of ethylene glycol ($C_2H_6O_2$) at 50 °C. The solution was vigorously stirred at this temperature using magnetic stirrer for 90 min to make stable sol. The sol was further heated and maintained at 80 °C under continuous stirring until a brown semi-solid (gel) is formed. This was followed by aging about 5 days for the condensation and polymerization reactions, and then dried in a hot air oven at 100 °C for 5 h. This xerogel was annealed under air atmosphere at 800 °C for 150 min and the final product was crushed to powder form, which was reddish in color.

3. Results and discussion

3.1. Structural analysis by X-ray diffraction

The phase formation and crystallinity of synthesized α-Fe_2O_3 nanoparticles were identified using BRUKER DRX500 X-ray diffractometer with Cu Kα radiation of wavelength 1.5406 Å through the angular range 20–70° by scanning at a step rate of 0.04° per minute. Fig. 1 shows the powder X-ray diffraction pattern of α-Fe_2O_3 nanoparticles recorded at room temperature. All diffraction peaks are in excellent agreement with JCPDS card number 86-0550. The strong peaks are indexed and the absence of additional peaks suggests the definite yield of α-Fe_2O_3 nanoparticles excluding impurities. X-ray diffraction data reveals that α-Fe_2O_3 nanoparticles are crystallized in hexagonal crystal system with rhombohedral lattice structure (space group: R $\bar{3}$ c) whose lattice parameters are $a = 5.035$ Å and $c = 13.74$ Å. The peak intensity of (104) plane is stronger than (110) plane which implies that the sample has the preferential growth along (104) plane. The average crystallite size was calculated by measuring the full width at half maximum of all the reflections based on Scherrer formula, which is close to 35 nm. The sharp peaks indicate that the synthesized sample is highly crystalline in nature. Table1 shows the variation in crystallite size of α-Fe_2O_3 nanoparticles for different 2θ values, which varies from 12 to 56 nm. The increase in crystallite size may be due to Ostwald ripening. It is a thermodynamically driven spontaneous process occurs because larger particles are more energetically stable than smaller particles [36–38]. The lattice strain and the crystallite size were calculated by Williamson–Hall (W–H) plot using the relation [39],

$$\frac{\beta \cos\theta}{\lambda} = \frac{\varepsilon \sin\theta}{\lambda} + \frac{1}{L} \tag{1}$$

The W-H plot of α-Fe_2O_3 nanoparticles is shown in Fig. 2 and the determined values of lattice strain and mean crystallite size are -0.04036 and 30 nm respectively which is very well in agreement with the XRD results. The negative slope indicates that strain broadening must be small [40–42]. The relations obtained from powder XRD profile are graphically represented in Fig. 3.

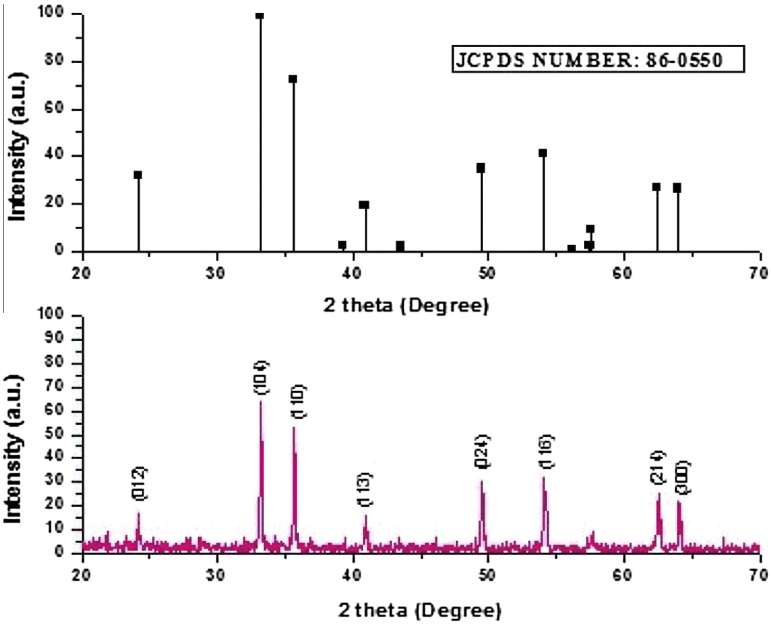

Fig. 1. Powder X-ray diffraction pattern of α-Fe_2O_3 nanoparticles.