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Hydrothermal synthesis of alpha Fe_2O_3 nanoparticles capped by Tween-80

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ABSTRACT

Surface modified α-Fe_2O_3 nanoparticles capped by Tween-80 were prepared by the hydrothermal treatment of $Fe(NO_3)_2.9H_2O$ at 200 °C. The spherical nanoparticles possessed good crystallinity with an average crystallite size of 21 nm. The presence of Tween-80 on the surface of α-Fe_2O_3 was confirmed by FTIR and Mössbauer analysis. The surfactant was effective in controlling the particle shape and restricted the particle growth to a narrow range around 40–60 nm as observed by scanning electron microscopy. The α-Fe_2O_3 nanoparticles obtained without Tween-80 were irregular in shape with a wide size distribution in the range of 150–300 nm.

1. Introduction

Surface capping of inorganic nanoparticles with organic molecules has been the subject of considerable research interest over the past several years because, the application of nanoparticles largely depend on their surface properties. The physical and chemical interactions of a large variety of organic functional groups with the surface of nanoparticles often result in remarkable properties along with their stability and better dispersibility. The surface modification of iron oxide nanoparticles has recently expanded rapidly because of their increasing use in drug delivery and biological imaging [1,2]. Hematite (α-Fe_2O_3), being the most stable of all iron oxides is widely used in catalysis [3], gas sensors [4], electrode material for lithium ion batteries [5], and in water treatment [6]. A variety of surfactants and organic molecules such as SDBS [7], CTAB [8], lauric acid [9], n-decanoic acid and n-decylamine [10] have been used to control and modify the size, shape, and surface environment of α-Fe_2O_3 nanoparticles. Polyoxyethylene sorbitan monooleate, commercially known as Tween-80, is a popular non-ionic surfactant used widely in cosmetics, foods, and pharmaceutical products. Its use for the synthesis of metallic nanoparticles of silver [11] , nickel [12], copper [13] and gold [14] has already been established.

Herein, we report a single step hydrothermal synthesis of spherical α-Fe_2O_3 nanoparticles in the presence of Tween-80. For comparison, α-Fe_2O_3 nanoparticles without surfactant were also prepared under the same conditions. FTIR and Mössbauer analysis revealed the self assembly of Tween-80 on the surface of α-Fe_2O_3 nanoparticles. Scanning Electron Microscopy was used to monitor the effect of Tween-80 on the size and morphology of unmodified and surface modified α-Fe_2O_3 nanoparticles.

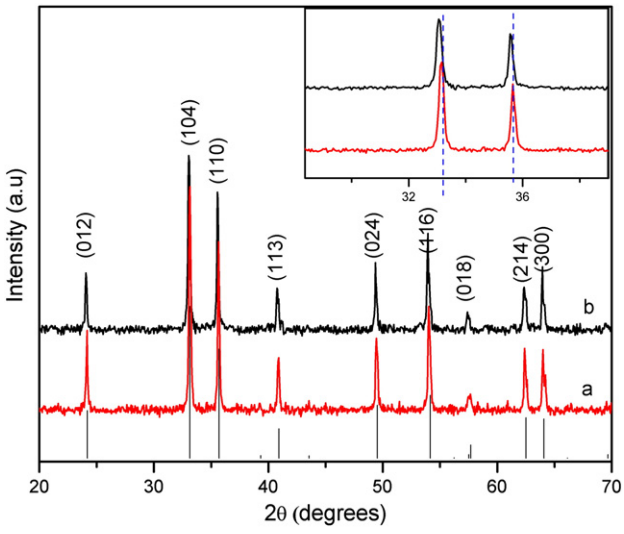

Fig. 1. XRD patterns of α-Fe_2O_3 nanoparticles synthesized (a) in the absence and (b) in the presence of Tween-80. Inset shows a magnified section of the patterns to indicate peak shift. Solid bars refer to JCPDS No. 84–0306.

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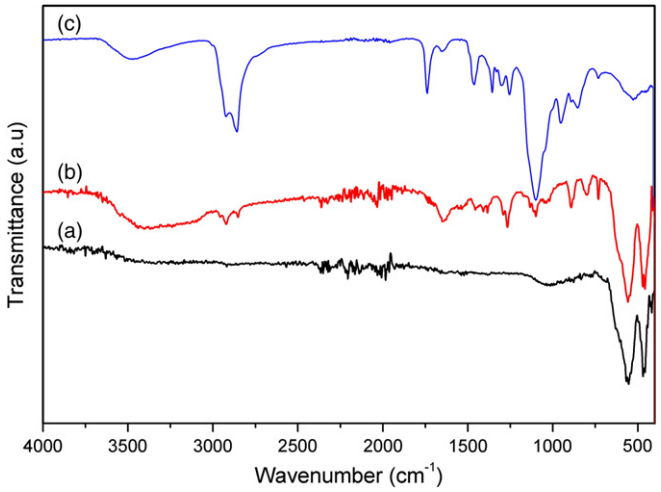

Fig. 2. FTIR spectra of α-Fe_2O_3 nanoparticles synthesized (a) in the absence and (b) in the presence and of Tween-80. FTIR spectrum of pure Tween-80 is shown at (c).

2. Experimental section

Iron nitrate ($Fe(NO_3)_2.9H_2O$), sodium hydroxide (NaOH), and Tween-80 were used as the starting materials. To the 100 ml of 0.25 M aqueous solution of $Fe(NO_3)_2.9H_2O$ were added 5 ml of Tween-80 and stirred for 20 minutes. While stirring, 50 ml of 1 M NaOH were drop wise added to the mixture containing $Fe(NO_3)_2.9H_2O$ and Tween-80 to form a suspension. The suspension was stirred vigorously for another 1 h and then poured into a Teflon lined stainless steel autoclave. Hydrothermal reaction was carried out at 200 °C for 5 h. After cooling the autoclave to room temperature naturally, the precipitates were collected by centrifugation and washed with distilled water and absolute ethanol to remove the excessive Tween-80 and any residual ions left. Same procedure was used to prepare α-Fe_2O_3 under the same conditions without the use of Tween-80 for comparison. The reddish brown powders collected were dried in air at 90 °C.

Powder X-ray diffraction analysis of the samples was performed on a Rigaku diffractometer using Cu Kα radiation (λ = 1.5406 Å). FTIR transmittance spectra of the powders were collected in KBr pellets in the range of 4000–400 cm^{-1} using Nicolet 6700, FTIR. Particle morphology of the samples was characterized by Scanning Electron Microscopy (JSM-5910, JEOL). The room temperature Mössbauer data was collected in transmission geometry using a ^{57}Co (Rh-matrix) source of initially 25 mCi strength. Mössbauer spectrometer was calibrated using a thin α-Fe foil and data analysis was performed using a computer code Mos-90, assuming that all peaks were Lorentzian in shape.

3. Results and discussion

3.1. XRD analysis

The XRD patterns of α-Fe_2O_3 nanoparticles synthesized in the presence and absence of Tween-80 are shown in Fig. 1. Based on the peak positions, the diffraction peaks were indexed to the hematite phase of Fe_2O_3, consistent with JCPDS No. 84–0306. The average crystallite size calculated from the (104) and (110) reflections using Scherer Formula [15] were found to be 21 and 34 nm for the surface modified and unmodified α-Fe_2O_3 nanoparticles, respectively. A magnified section of the XRD patterns shown as inset in Fig. 1 indicates a slight shift of the peak positions toward lower 2θ values for Tween-80 capped nanoparticles. This behavior can be explained by the increase in Fe–O bond length upon coating the nanoparticles with a layer of Tween-80. For nanoparticles, it is known that there are present a number of vacancy defects and dangling bonds on the surface. During

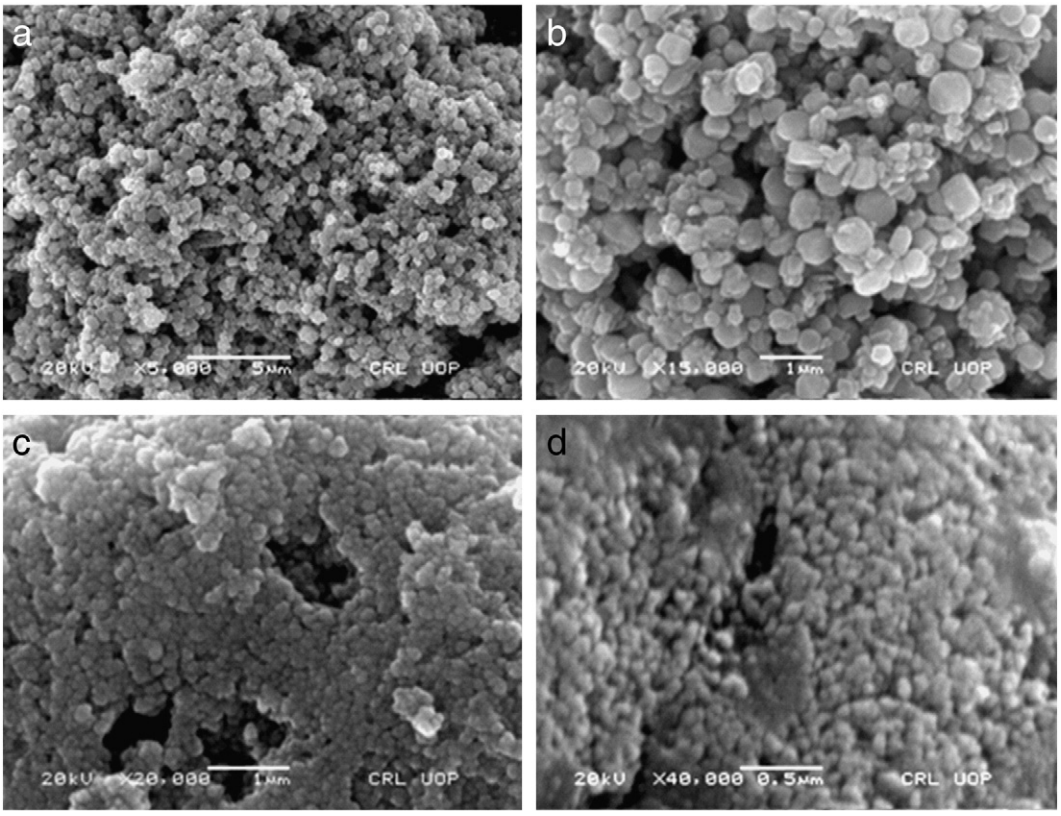

Fig. 3. Low and high magnification SEM images of α-Fe_2O_3 nanoparticles synthesized in the absence (a–b) and in the presence (c–d) of Tween-80.