Preparation of Maghemite $\gamma$-Fe$_2$O$_3$ Nanoparticles by Electrochemical Method

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Abstract

Fe$_2$O$_3$ nanoparticles were synthesized by electrochemical method using a rectangular iron plate as an anode and Graphite rod as the counter electrode cathode. The synthesized nanoparticles were examined by transmission electron microscopy (TEM), scanning electron microscopy (SEM) and X-ray diffraction (XRD). Results show that nanoparticles have tetragonal shape with average size of 17 nm. The surface of nanoparticles is smooth with good crystallinity.

Keywords: Nanoparticles, iron oxide, electrochemical method, maghemite

Introduction

Nanoparticles are zero dimensional because all the dimension are measured inside the nanoscale (under 100 nm). It shows anew and fascinating properties that rely upon the size (diminishing size from full scale and miniaturized scale to nanoscale). Nanoparticles are broadly present in the natural world as photochemical responses, because of volcanic action or delivered by plants or green growth. Nanoparticles have likewise been made, accidentally, by people as results of burning buildups and cooked nourishment or, all the more as of late, as the remaining parts of the depleted fuel of vehicles [1]. In contrast with the amount of nanoparticles delivered normally or inadvertently, nanoparticles combined for investigation or mechanical
intentions are a little minority. Press oxide nanoparticles are one of the critical oxides, which has a large portion of utilizations because of its non-poisonous quality [2-11].

**EXPERIMENTAL**

All chemicals were used without any purification. Maghemite γ-Fe₂O₃ nanoparticles were synthesized by electrolysis, using 120 ml of 0.02 M NaOH at 27 °C as electrolyte. A rectangular iron plate (4 cm x 2 cm x 0.1 cm) was used as an anode. Graphite rod (0.5 x 5 cm) was used as the counter electrode cathode. Before mounting the substrates in the cell, they were cleaned sonically using aqueous and organic cleaner Solvents (ethanol, chloroform, de-ionized water) sequentially. Each step of cleaning lasted for 10 minutes. The applied DC voltage between the electrodes was 6V under current density of 5.43 x10⁻³ mA/cm² for 1 h. A red-brown magnetic precipitate was obtained. The product was separated and washed with de-ionized water and dried over night to subsequent analysis [12].

The electro deposition reaction pathway to form γ-Fe₂O₃:-

Fe → Fe⁺³ + 3e⁻

Fe⁺³ + 3OH⁻ → Fe (OH)₃(aq)

2Fe (OH)₃ → 2 FeOOH + 2H₂O

2 FeOOH → γ-Fe₂O₃ + H₂O

(all of them at PH = 13)

**Results and discussion**

The morphological and structural of synthesized different phases iron oxides nanoparticles were examined by various techniques. The structure of the various modified samples was investigated by XRD use (Cu Kα radiation line of wavelength of 1.54 Å in 2θ range from 10° to 80°). The patterns of the phases of iron oxides nanoparticles was shown in Figure (1).
The XRD pattern shows a significant amount of broadening lines, which are characteristic of nanoparticles. The crystal size can be calculated according to Debye-Scherrer formula [13].

\[ D = \frac{k \lambda}{\beta \cos \theta} \]

Where \( k = 0.9 \), Scherrer constant, \( \lambda \) is the wavelength of the Cu-K\( \alpha \) radiations, \( \beta \) is the full width at half maximum, and \( \theta \) is the angle obtained from 20 values corresponding to maximum intensity peak in XRD pattern. The calculated mean crystal size of nanoparticles was 17 nm.

The morphology and particles size were determined by TEM. The mean particle size and distribution were determined randomly on the TEM image; fig (2). The mean particles size was 17 nm.
SEM images, Fig (3) shows the morphology and size distribution of different phases iron oxide nanoparticles. The surface of nanoparticles is smooth with good crystallinity. The average particle size and distribution were determined randomly on the SEM images. It is about 20 nm.
Conclusion
Maghemite $\gamma$-Fe$_2$O$_3$ nanoparticles was synthesized by electrolysis method. The morphological, structural and optical properties of synthesized $\gamma$-Fe$_2$O$_3$ nanoparticles was examined by X-ray diffraction (XRD), transmission electron microscope (TEM) and scanning electron microscope (SEM). The surface of nanoparticles is smooth with good crystallite.

The gamma iron oxide nanoparticles has uniform tetragonal shape with an average size of 27 nm.

References