

Synthesis and Characterization of α -Fe₂O₃ Nanoparticles by Microwave Method

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Abstract:

α -Fe₂O₃ (hematite) is the most stable iron oxide under ambient conditions. This transition metal oxide has been extensively investigated because it has unique electrical and catalytic properties. In this report, a novel microwave method for preparation of α -Fe₂O₃ nanoparticles has been developed. The process contained two steps: first, precursors were obtained from a mixed solution of 50 ml of 0.3 M Fe(NO₃)₂·9H₂O and 1.2 g of urea under 540 W microwave irradiated for 6 min. Then, the precursors were calcined at 800 °C to fabricate pure α -Fe₂O₃ (hematite) nanoparticles for 4 h. Nanoparticles were characterized by Fourier transform infrared (FT-IR) spectroscopy, X-ray diffraction (XRD), scanning electron microscopy (SEM), and Energy dispersive X-ray spectrometer (EDX) technique. The results indicate that the morphologies of final products significantly depend on the reaction conditions including the reaction time, the initial concentration of precursor, reagent and calcinations temperature.

Keywords: α -Fe₂O₃, Microwave method, Metal oxides, Nanoparticles, Hematite.

1. INTRODUCTION

The fabrication of nanometer-order entities is considered to be the key for applications in data storage, functional devices, communications, and technology. The randomly distributed nanostructures, fluctuate in size, have an unchanged periodicity, and possess significant limitations to their application. Thus, the ability to systematically manipulate these nanocrystals is an important goal in modern materials science [1-4].

Over the past decade nanoscale magnetic materials have attracted intensive interest because of their potential application in high-density magnetic recording in magnetic sensors and in addressing some basic issues about magnetic phenomena in

low-dimensional systems [5, 6]. Various approaches have been developed to prepare nanoscale magnetic materials [7-10].

Besides its interesting magnetic properties, hematite, α -Fe₂O₃, has a wide field of technological applications (fabrication of ferrites, catalyses, inorganic pigments, raw material for magnetic recording media). The preparation method determines the final powder characteristics like shape, average particle size, specific surface, porosity, that are of considerable importance in the subsequent processing for specific applications.

Various α -Fe₂O₃ structures, such as 0D (particles), 1D (rods, wires, tubes, and belts), 2D/3D (disks, dendrites, flowers, and mesopores), and hybrids, have already been fabricated by a variety of methods.

In the last few decades the Microwave technique has been widely used for synthesis and growing of inorganic crystals because it is essentially less energy intensive, less polluting and leads to high homogeneity and well crystallized products, with definite composition [11-14]. Microwave method has attracted much attention because it is a much faster, simpler, and more energy-efficient technique for synthesizing new materials with improved nanostructures compared with conventional methods [15-17]. The currently available methods for the synthesis of α -Fe₂O₃ nanoparticles generally require high temperatures, tedious multi step processing, and long preparation times. The possibility of using microwave processing to synthesize the α -Fe₂O₃ nanoparticles with excellent catalytic activity was demonstrated [18,19].

A convenient technique to prepare nanocrystalline oxides in very short durations is the microwave method [17,18]. The advantages of the microwave method over the conventional method are: (a) extremely rapid kinetics of crystallization, (b) very rapid heating to reach the required temperature and (c) possible formation of new meta-stable phases [20].

In this work, we describe a rapid microwave method to synthesize α -Fe₂O₃ nanoparticles in aqueous solution, without using the organic surfactant [21].

2. EXPERIMENTAL

2.1. Instruments and reagents

All of the chemical reagents were of analytical grade and were used without further purification. Double distilled, deionized water was used as a solvent. All reagents used were bought from Merck.

The α -Fe₂O₃ (hematite) nanoparticles were characterized by using FT-IR, XRD, SEM, and EDX. FT-IR spectra was recorded (Figure 2) with a Bruker Tensor 27 spectrometer, from 400 to 4500 cm⁻¹, using the KBr wafer technique. Wafers were prepared from the mixture of 1 mg of the sample and 100 mg of KBr. Scanning electron microscopy (SEM) images of the samples were obtained on Philips XL30 equipped with an energy dispersive X-ray (EDX) microanalysis. X-ray powder

diffraction (XRD) analysis was conducted on a Philips analytical PC-APD X-ray diffractometer with graphite monochromatic Cu K_α radiation (α_1 , $\lambda_1 = 1.54056 \text{ \AA}$, α_2 , $\lambda_2 = 1.54439 \text{ \AA}$) to verify the formation of products. The X-ray diffraction pattern was indexed using Joint Committee on Powder Diffraction Standards (JCPDS) card.

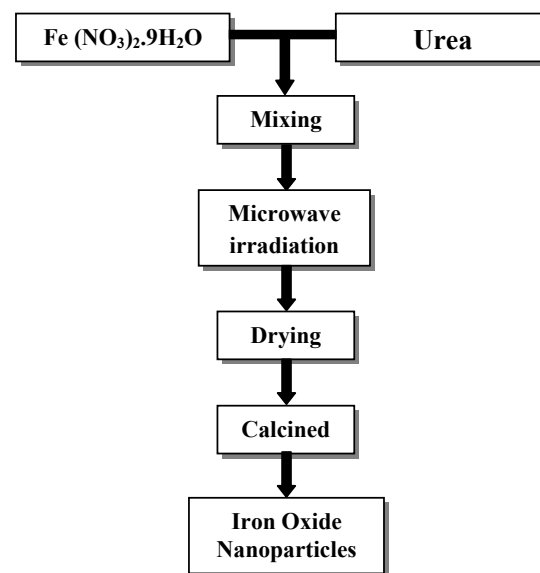


Figure 1: Flowchart of the formation of iron oxide nanoparticles via a microwave method

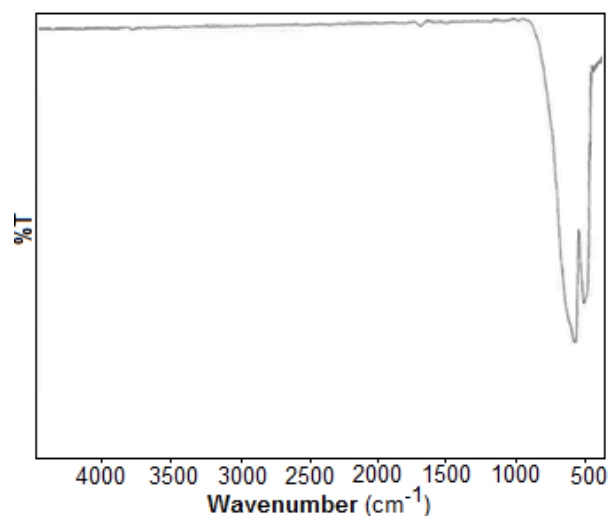


Figure 2: FT-IR spectrum of the sample prepared from microwave method

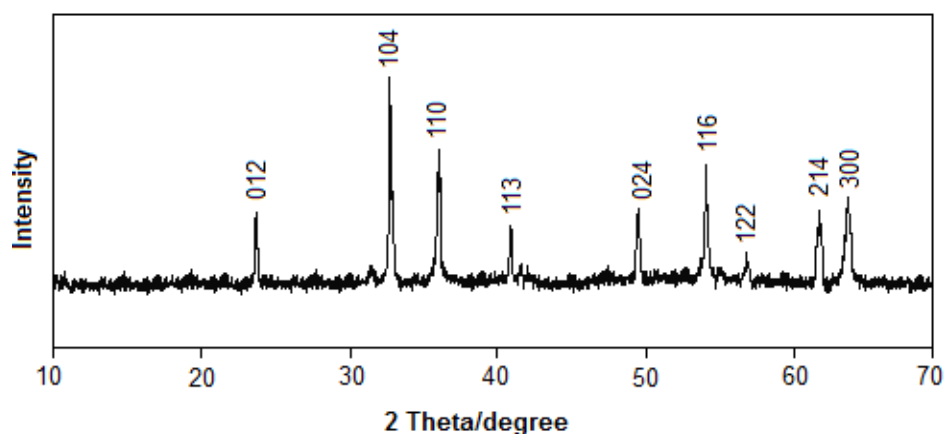


Figure 3: Representative XRD pattern of the α -Fe₂O₃ nanoparticles prepared at 800 °C for 4 h

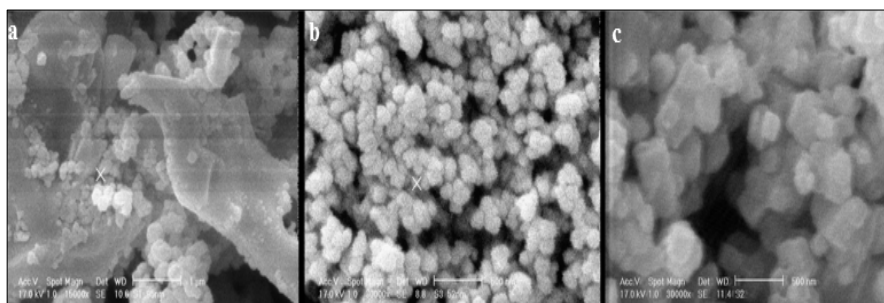


Figure 4: SEM images of α -Fe₂O₃: (a, b and c) α -Fe₂O₃ prepared by microwave method and calcined at 600, 700 and 800 °C, respectively

2.2. Synthesis of α -Fe₂O₃ nanoparticles

Figure 1 Shows a flowchart of the experimental procedure used to synthesize α -Fe₂O₃ nanoparticles in this study.

In order to prepare α -Fe₂O₃ (hematite) nanoparticles as starting materials, Fe (NO₃)₂·9H₂O and urea had been chosen. A mixed solution of 50 ml of 0.3 M Fe (NO₃)₂·9H₂O and 1.2 g of urea was irradiated under 540W microwave for 6 min. The precipitate was then filtered, washed with deionized distilled water, and dried in an oven at 100°C for 8 h to obtain the solid sample. Then, the as prepared sample was calcined at 800°C for 4 h to obtain the brown species.

3. RESULTS AND DISCUSSION

3.1. FT-IR analysis

Two main broad metal-oxygen bands are seen in the

FT-IR spectrum α -Fe₂O₃ nanoparticles. The highest one ν_1 (Figure 2) observed at 541 cm⁻¹ corresponds to intrinsic stretching vibration of the, Fe \leftrightarrow O, where as the ν_2 -lowest band, usually observed at 471cm⁻¹, is assigned to bending vibration of the, O \leftrightarrow Fe \leftrightarrow O [22].

3.2. XRD analysis

The crystal structure confirmation analysis was carried out by the X-ray diffraction patterns. XRD patterns of the product obtained by calcination of precursor at 800 °C are shown in Figure 3. XRD peaks for the nanometer sized α -Fe₂O₃ sample are significantly broader than those for micrometer sized sample, indicating a very small particle size. All the characteristic peaks of α -Fe₂O₃ are present: 24.39° (012), 33.45° (104), 36.02° (110), 41.15° (113), 49.80° (024), 54.36° (116), 57.33° (122), 62.70° (214), 64.37° (300). These peaks are

attributed to $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles (JCPDS NO. 01-1030). At this temperature, only the peaks due to the $\alpha\text{-Fe}_2\text{O}_3$ are observed. The increase in the calcinations temperature will significantly intensify the peaks. This can be explained by the fact that the crystalline phase of $\alpha\text{-Fe}_2\text{O}_3$ becomes more precise with the calcination temperature.

From the XRD data, the crystallite size (D_c) of the as prepared $\alpha\text{-Fe}_2\text{O}_3$ particles was calculated to be 57 nm, using the Debye-Scherrer equation [23],

$$D_c = \frac{K\lambda}{\beta \cos \theta}$$

Where β is the breadth of the observed diffraction line at its half-intensity maximum, K is the so-called shape factor, which usually takes a value of about 0.9 and λ is the wavelength of the X-ray source used in XRD.

The size increased with increasing calcination temperature (see Table 1).

3.3. SEM analysis

Figure 4 depicts SEM images of $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles. Figure 4a, b and c are the images of $\alpha\text{-Fe}_2\text{O}_3$ prepared by microwave route and calcined at 600, 700 and 800 °C, respectively. $\alpha\text{-Fe}_2\text{O}_3$ prepared by microwave irradiation and calcined at 600 °C had small and uniform grain size around 35 nm. The size increased and sintered to around 54 nm particles after calcinations at 800 °C. As shown in Fig. 4a, the $\alpha\text{-Fe}_2\text{O}_3$ prepared by microwave method and calcined at 600 °C shows the different morphology of particles from others. This can be explained by the fact that the crystalline phase of $\alpha\text{-Fe}_2\text{O}_3$ becomes more precise with increasing the calcination temperature.

Table 1: The depended of average grain size on calcinate temperature.

calcinate temperature (°C)	Average grain size (nm)*
600	35
700	47
800	54

*calculated from Scherer formula

3.4. EDX analysis

The EDX analysis was performed to further confirm the composition of the obtained products. Figure 5 shows that the products calcined at 800°C for 4 h are composed of Fe. The C peak in the spectrum is attributed to the electric latex of the EDX sample holder.

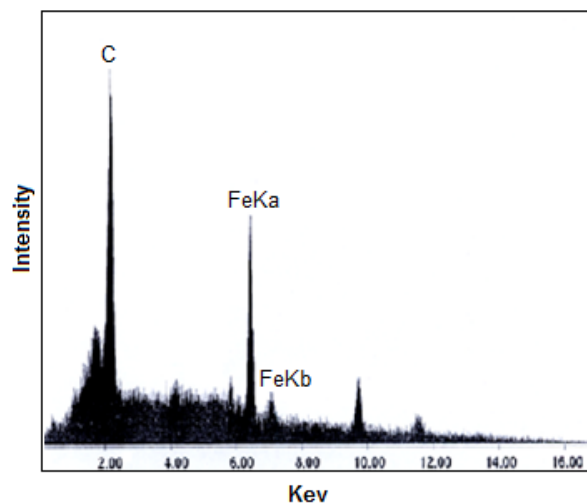


Figure 5: EDX spectrum of the obtained $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles

4. CONCLUSIONS

Nanoparticles of $\alpha\text{-Fe}_2\text{O}_3$ were successfully synthesized by a simple microwave route. A pure hematite $\alpha\text{-Fe}_2\text{O}_3$ phase was formed after heat treatment at 800 °C for only 4 h. The crystalline phase of $\alpha\text{-Fe}_2\text{O}_3$ became more precise with increasing calcination temperatures and the particle size increased with increasing calcination temperatures. Microwave irradiation, is rapid, environmental friendly and low-cost method.

5. ACKNOWLEDGMENTS

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