# Hematite (α-Fe<sub>2</sub>O<sub>3</sub>) Nanoparticles: Synthesis, Characterization and Optical Properties

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# Abstract

In this paper, we report a simple and convenient method for the synthesis of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticles via hydrothermal process followed by thermal decomposition using the new iron precursor, which was obtained by mixing of benzoic acid (BA) and Fe(NO<sub>3</sub>)<sub>3</sub>·3H<sub>2</sub>O in water as solvent. Two products with almost similar morphologies and sizes were obtained by changing the calcination temperature (500 and 600°C) for 2 h in the air atmosphere. They were named as Fe-500 and Fe-600, respectively, and characterized by Fourier transform infrared (FT-IR) and ultraviolet-visible (UV-Vis) spectroscopy, X-ray powder diffraction (XRD), Energy dispersive spectroscopy (EDS) and transmission electron microscopy (TEM). FT-IR, UV-Vis, XRD and EDS results confirm the formation of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> phase. Also, TEM images confirm that the size of the products is less than 100 nm.

Keywords: α-Fe<sub>2</sub>O<sub>3</sub> nanoparticles; Hydrothermal; Morphologies; X-ray diffraction.

## Introduction

The preparation of iron(III) oxide nanoparticles, such as  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>, have attracted considerable attention in recent years due to its properties and application in various fields of technology [1-8]. Hematite ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>) is one of the most stable iron oxides and have good electrochemical [9], and photocatalytic properties [10,11] and is a good candidate for applications such as degradation of organic pollutants [10.11], gas sensing [12] and as anode in Li-ion batteries [9]. This iron oxide is a low-cost and eco-friendly n-type semiconductor with narrow band gap (2.0-2.2 eV) prepared via various techniques like hydrothermal [13], solvothermal [12], co-precipitation [14], direct calcination of ferric salt in

air atmosphere [9], liquid phase-based ultrasonication [15], chemical bath deposition [16] and sol-gel [17]. The size and morphology of the  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticles may be altered by changing the raw materials and also concentration, time and temperature in the thermal routes [12,13]. Kusior et al. [10] reported different shapes of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> through ion-mediated hydrothermal technique. Li et al. [13] prepared different morphologies of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> via hydrothermal method by changes in the time and temperature. Uniform nanodisks of hematite  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> catalysts are prepared via a simple hydrothermal route by chen et al [18]. Rhombohedron and plate-like hematite ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>) as potential biomedical applications for MRI has been prepared by Tadic et al [19]. Umar et al prepared cubic shaped

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Scheme 1. Schematic representation for the preparation of iron precursor and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticles

hematite ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>) micro-structures for rapid ethanol sensor application [20]. Until now,  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> with several types of shapes including nanosheet [12], hexagonal [15,21], hollow polyhedral [11], discs [10], quasi-cluster [9], plate-like [19], cubic [20], spherical shape [22.23], has been successfully fabricated.

Herein, as a part of our ongoing works dealing with the transition metal oxide nanoparticles [24-26], we synthesized  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticles (Scheme 1) using hydrothermal process followed by thermal decomposition at 500 and 600°C. The products were characterized by FT-IR, UV-Vis, XRD, EDS and TEM.

## **Materials and Methods**

#### Materials and characterization

All chemical used in this paper were purchased from Merck company and were used without any further purification. Fourier transform infrared (FT-IR) spectra were recorded as a KBr disk on an FT-IR Perkin-Elmer spectrophotometer. Optical absorption measurements were done using a UV-Vis spectrophotometer in the wavelength ranges of 200-800 nm at room temperature. XRD patterns of the complexes were obtained on Empyrean powder diffractometer of PANalytical in Bragg-Brentano configuration equipped with a flat sample holder and PIXCel3D detector (Cu Ka radiation,  $\lambda = 1.5418$  Å). EDS data were measured with Electron Probe Microanalyzer JEOL JXA-8230 equipped with energy dispersive spectrometer (EDS) Bruker QUANTAX 200 with data processing software Esprit 2.2. Samples were applied to carbon tape and coated with a thin carbon layer to prevent the sample from charging. Average chemical composition of a large number of particles was measured. Standardless quantitative method ZAF was used due to the powder sample. The TEM images were recorded with a transmission electron microscope (TEM) FEI Tecnai G<sup>2</sup>

20 with a  $LaB_6$  cathode at acceleration voltage 200 kV. TEM is equipped with a CCD camera Olympus Veleta. Powdered samples were prepared by dispersing in water under ultrasonic treatment. A drop of the suspension was evaporated on cooper grid with carbon film.

#### Preparation of iron oxide nanoparticles

A mixture of iron nitrate (1 gr) and benzoic acid (1 gr) was dissolved in 5 mL water and stirred for 0.5 h. The solvent was evaporated upon heating at 85 °C in 4 h. The iron precursor was put into a quartz crucible and ground for about 10 min. Finally, the powder was put into a tube furnace and calcined for 2 h at 500 and 600°C with a heating rate of 20 °C/min under air atmosphere. As a results, two iron oxide samples were obtained and respectively named as Fe-2-500 and Fe-2-600.

#### **Results and Discussion**

#### FT-IR spectra

Figure 1 depicts the FT-IR spectra of the new iron precursor (Fe-2) and the as-prepared products (Fe-2-500 and Fe-2-600). In the FT-IR spectrum of Fe-2, there are some peaks corresponding to O-H (3367 cm<sup>-1</sup>), C=O (1685 cm<sup>-1</sup>), C=C (1454-1602 cm<sup>-1</sup>) and peaks between 400 - 1400 cm<sup>-1</sup> are assigned to C-O stretching of benzoic acid [27]. By calcination of the Fe-2, the number of peaks is expected to decrease. In the FT-IR spectrum of Fe-2-500, the peaks appearing at 998 cm<sup>-1</sup> correspond to =C-H bend of phenyl ring, and the peak at 1126 cm<sup>-1</sup>. The peaks at 639, 588, 538 and 469 cm<sup>-1</sup> observed in the FT-IR spectrum of Fe-2-500 could be attributed to Fe-O vibrational modes [28,29]. In this spectrum, the prominent peaks belong to C-O and C-H bonds of benzoic acid, indicating, that the calcination of Fe-2 is not completed at 500 °C. In the FT-IR spectrum of Fe-2-600, there are two prominent and sharp peaks at



Figure 1. FT-IR spectra of Fe-2, Fe-2-500 and Fe-2-600

535 and 459 cm<sup>-1</sup>, assigned to the Fe-O stretching [30.31], while there is one weak peak at 1136 related to C-O stretching of benzoic acid [27]. In the FT-IR spectra of the Fe-2-500 and Fe-2-600, there is a weak peak at about 1630 cm<sup>-1</sup> confirming the O-H bending vibrational modes relating to water molecule adsorbed on the surface of the products [27]. Also, the peaks at 3448 cm<sup>-1</sup> in Fe-2-500 and at 3400 cm<sup>-1</sup> in Fe-2-600 are attributed to the stretching vibrations of -OH [32].

## UV-Vis spectra

The UV-Vis absorption measurements of the Fe-2 and the as-prepared Fe-2-500 and Fe-2-600 products were done at room temperature and are shown in Figure 2. There are three peaks at 266, 327 and 419 nm assigned to the  $\pi \rightarrow \pi^*$  and  $n \rightarrow \pi^*$  transitions. For Fe-2-500 and Fe-2-600, the transition that was observed at wavelength around 586 nm is similar to that reported by Han et al. [33]. The as-prepared products can have potential applications in the field of photocatalytic dye degradation [34,35].

# XRD patterns

XRD analysis was performed in order to determine

crystal structures and phase compositions of the as-prepared products. In Figure 3, all peaks that appeared at distinct 20 values can be well assigned to the rhombohedral structure of hematite with JCPDS card number 33-0664 [36,37]. There are no peaks for impurities relating to another iron oxide in Figure 4, which confirms the high purity and single phase of the products. The peaks appearing at  $2\theta$  range of 24.15, 33.20, 35.65, 40.71, 49.49, 54.11, 57.45, 62.45, 64.05, 71.95 and 75.47 can be attributed to the 012, 104, 110, 113, 024, 116, 018, 214, 300, 1010 and 220 crystalline structures corresponding to pure hematite nanoparticles [36,37]. The lattice constants were found a=b=5.03486(19) Å and c= 13.7540(7) Å for Fe-2-500 and a=b=5.0360(2) Å and c= 13.7537(7) Å for Fe-2-600 [36,37]. The crystal sizes were calculated using fundamental parameter approach [38] integrated in Jana2006, which removes the instrumental part of the diffraction pattern by means of known geometry of the diffractometer. The average crystallite size determined for Fe-2-500 was 54.5 nm and 56.0 nm for Fe-2-600. These dis are close to the values seen in the TEM images (Fig. 4).



Figure 2. UV-Vis spectra of Fe-2, Fe-2-500 and Fe-2-600

# EDS spectra

EDS measurements detect iron, oxygen and carbon in both samples (Fig. 4). Carbon comes from the coated carbon layer or/and carbon tape. Results of quantitative analysis reveal 40.78 at.% of Fe and 59.22 at.% of O in sample Fe-2-500, and 41.81 at.% of Fe and 58.19 at.% of O in sample Fe-2-600. If we consider the lower accuracy of the standardless quantitative method, we can confirm the presence of  $Fe_2O_3$  in both samples.



Figure 3. XRD patterns of a) Fe-2-500 and b) Fe-2-600



Figure 4. EDS spectra of a) Fe-2-500 and b) Fe-2-600

## **TEM** images

Typical TEM images of the as-prepared  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> products are depicted in Figures 5 and 6, respectively, to show the particle morphology and size distribution. The

TEM images illustrate clusters of prepared nanoparticles. These particles have an irregular shape and their equivalent diameters are estimated about 50 nm for both samples. The size of the nanoparticles



Figure 5. TEM images of Fe-2-500



Figure 6. TEM images of Fe-2-600

corresponds to the size calculated from XRD patterns.

The size and morphology of synthetic nanoparticles differ from the size and morphology of hematite nanoparticles reported in similar articles. For example, the size of nanoparticles prepared by green synthesis method by Pallela et al. [39] is reported to be about 20 nanometers. Rahman et al. [16] obtained different morphologies such as nanorods for hematite by changing the initial concentration of the precursor. Spherical shapes of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> with average sizes of about 19 nm were synthesized by Taghavi Fardood et al. [40] using Arabic gum as a biotemlate source. By one step pyrolysis method, Wang et al. [41] prepared various morphologies with average particle sizes between 30-150 nm. The size and morphology of the products is analogous to nanomaterials prepared using coprecipitation method [10]. These materials are characterized by irregular shapes and sizes of 20-40 nm. Also heat treatment method [7] provides at 300 °C resulted in porous  $\Box$ -Fe<sub>2</sub>O<sub>3</sub> cubes which are about 50 nm. Nevertheless, with increasing temperature they are gradually substituted by smaller □-Fe<sub>2</sub>O<sub>3</sub> cubes which are predominant at 500 °C. Solvothermal and hydrothermal method [8, 6] produces nanomaterials of diverse morphologies depending on water bath temperature, urea concentration and hydrothermal temperature. The particle sizes ranges from tens of nm hollow-shaped particles to hundreds of nm (hollow-like rods, large agglomerated particles, and their mixtures).

#### Conclusion

From the calcination of the new iron precursor at two different temperatures, we prepared  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticles and characterized them. Results confirmed the formation of the high purity and single phase of hematite. Due to the appearance of an absorption band in the visible area, these compounds will be able act as photocatalytic for color removal. The TEM results confirm the nanostructured materials.

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