Nanostructured \( \alpha-\text{Fe}_2\text{O}_3 \): Solvothermal Synthesis, Characterization, and Effect of Synthesis Parameters on Structural Properties

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\begin{abstract}
\( \alpha-\text{Fe}_2\text{O}_3 \) is a stable, cheap, and non-toxic metal oxide with many advantages and different fields of application. Many attempts have been devoted to the synthesis of \( \alpha-\text{Fe}_2\text{O}_3 \) with different crystal structures and morphologies to obtain the desired properties. In this research, nanostructured \( \alpha-\text{Fe}_2\text{O}_3 \) were synthesized by a facile solvothermal route. The as-obtained samples are characterized by XRD, FESEM, EDS, FTIR, and BET surface area analysis. The results showed that the as-synthesized hematite consists of nanostructures with the morphology of distorted microspheres with an average diameter in the range of 1 to 1.5 \( \mu \)m each composed of self-assembled nanoparticles with an average size in the range of 10 to 30 nm. The results showed that the hematite nanostructures had a specific surface area of 41.86 m\(^2\) g\(^{-1}\). The influence of temperature and duration of the solvothermal process as well as, calcination on the structural properties of the \( \alpha-\text{Fe}_2\text{O}_3 \) samples was investigated. The results reveal that the crystallite size of the samples increases with increasing the temperature and duration of solvothermal treatment. Moreover, calcination leads to an increase in the crystallite size of the samples. The \( \alpha-\text{Fe}_2\text{O}_3 \) nanostructures with a minimum crystallite size of 13.6 nm were synthesized at 150 \(^\circ\)C for 4 h while the largest crystallite size of 75.4 nm was obtained at 180 \(^\circ\)C and 8 h with subsequent calcination of the sample at 500 \(^\circ\)C for 1 h. The results of the present study can be useful to enhance the properties of \( \alpha-\text{Fe}_2\text{O}_3 \) nanostructures in various fields of application.

\end{abstract}

\begin{keywords}
Hematite (\( \alpha-\text{Fe}_2\text{O}_3 \))
Nanostructures
Solvothermal Process
Calcination
Crystallite Size
\end{keywords}

\begin{1. INTRODUCTION}
Hematite (\( \alpha-\text{Fe}_2\text{O}_3 \)), the most stable form of iron oxides has drawn much attention due to its advantages and variety of applications [1, 2]. Various synthesis techniques including hydrolysis, chemical solution, electrospinning, molecular layer deposition, and solvothermal have been applied to prepare \( \alpha-\text{Fe}_2\text{O}_3 \) nanostructures [3]. Among these, the hydro/solvothermal process has drawn much attention, as it is a one-step process with the possibility of controlling crystal structure and morphology [4-6]. The specific properties of nanostructures are widely affected by their structures and morphologies [7-9]. Therefore, various morphologies of \( \alpha-\text{Fe}_2\text{O}_3 \) such as hexagonal plates [10], rod and ellipsoidal particles [11], flower-like [12], and nanoparticles [13] have been synthesized.

In the solvothermal process, the properties of products are widely affected by the type of precursors, type of solvent, reaction temperature, and duration of the reaction. However, regarding the environmental aspects, it is often preferred to carry out the process using non-toxic and low-cost materials at a low level of energy consumption [14].

Ma et al. [15] synthesized \( \alpha-\text{Fe}_2\text{O}_3 \) nanostructures with different sizes and shapes by changing reaction time and solvent via a simple hydrothermal process. \( \alpha-\text{Fe}_2\text{O}_3 \) nanopolyhydra, nanoparticles and microcubes were obtained by different solvents and reaction times. Zhang et al. synthesized two different morphologies of \( \alpha-\text{Fe}_2\text{O}_3 \) using two different solvents in a solvothermal process.

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3D flower-like α-Fe₂O₃ nanostructures and α-Fe₂O₃ nanoparticles were obtained with isopropanol and water as the solvent, respectively [16]. Trpkov et al. [17] synthesized α-Fe₂O₃ hierarchical superstructures by glycine-free and glycine-assisted hydrothermal method. The superstructures were composed of nanoparticles as building blocks with different morphology including mushroom-like, cube-like, and sphere-like, and dimensions of 1-5 µm. Cao et al. [18] synthesized flowerlike α-Fe₂O₃ nanostructures via a solvothermal route using FeCl₃.6H₂O, urea, ethanol, and microwave irradiation as the heating source.

The effect of crystallite size on various properties of hematite has been investigated [19, 20]. Nandiyanto et al. [21] studied the correlation between crystallite size and the photocatalytic activity of WO₃ particles. The photodegradation rate of curcumin was enhanced with increasing in crystallite size of the photocatalyst.

The present study consists of two phases. In phase 1, α-Fe₂O₃ is synthesized via a solvothermal process according to the previous study to investigate the effect of the heating source on the properties of the products. In the previous study solvothermal reaction was performed under microwave irradiation while in this study heating is performed by a standard laboratory oven. In phase 2, α-Fe₂O₃ is synthesized by solvothermal treatment under different conditions of temperature and duration of solvothermal treatment. The samples are characterized by several characterization techniques. The influence of the solvothermal parameters as well as calcination on the crystal structure of the products is investigated.

2. MATERIALS AND METHODS

2.1. Materials Ferric chloride hexahydrate (99.5% FeCl₃.6H₂O, Merck), urea (99.5%, ChemLab) and ethanol (99.9% C₂H₅OH, Merck) were used to synthesize α-Fe₂O₃ nanostructures. Deionized water was used in all cases. All the materials were of analytical grade and used without further purification.

2.2. Synthesis of α-Fe₂O₃ Nanostructures In phase 1, hematite nanostructures were synthesized via a solvothermal process according to the previous study [18]. A 1.89 g (7 mmol) of FeCl₃.6H₂O and 0.63 g (10.5 mmol) of urea were dissolved in 60 ml of absolute ethanol. The mixture was magnetically stirred at room temperature for 30 minutes to obtain a clear and homogeneous solution. The obtained solution was transferred into a 100 ml teflon-lined stainless steel autoclave. The autoclave was sealed and heated in a standard laboratory oven at 150 °C for 4 h. Then, the autoclave was naturally cooled down to room temperature. The obtained precipitates were collected by centrifugation at 4000 rpm, washed with absolute ethanol 5 times, and dried in an oven at 80 °C for 4 h. In phase 2, six samples of hematite were prepared under different conditions according to Table 1. The solvothermal process was performed at temperatures of 120 °C, 150 °C, and 180 °C, each for 4 h and 8 h. The other steps of synthesis including preparation of solution, separation, washing, and drying of the precipitates were the same as phase 1. Three of the as-synthesized samples were calcined at 500 °C in a muffle furnace for 1 h. The synthesis parameters and the sample names are listed in Table 1.

2.3. Characterization The crystal structure of the as-prepared samples was determined by XRD on XRD PANalytical with Cu Kα radiation at λ = 1.5406 Å. The crystallite size of the samples was calculated by Scherrer’s equation as follows:

\[ D = \frac{0.9 \lambda}{B \cos \theta} \]  

where \( D \) is the mean crystallite diameter, \( L \) is the wavelength of the X-ray applied, \( B \) is the diffraction angle of the specified peak, and \( \theta \) is the full width at half maximum (FWHM) [22]. The morphology of the particles was investigated by FESEM with MIRA³TESCAN-XMU. The composition of the samples was determined by EDS along with FESEM. The type of bonding structure of the samples was examined by FTIR with Thermo AVATAR. The surface area of the products was studied on BELSORP MINI II by BET analysis.

3. RESULTS AND DISCUSSION

3.1. Characterization The phase and crystal structure of the products were analyzed by XRD analysis. The XRD pattern of the as-synthesized α-Fe₂O₃ is shown in Figure 1. The obtained XRD pattern illustrates diffraction peaks with 2θ at 24.2°, 33.1°, 35.7°, 40.9°, 46.4°, 57.4°, 62.7°, and 67.8°.

| Sample Name | Reaction Temperature (°C) | Reaction Time (h) | Calcination |
|-------------|---------------------------|------------------|-------------|
| F120-4      | 120                       | 4                |             |
| F120-8      | 120                       | 8                |             |
| F150-4      | 150                       | 4                |             |
| F150-4-C    | 150                       | 4                | Done        |
| F150-8      | 150                       | 8                |             |
| F180-4      | 180                       | 4                |             |
| F180-4-C    | 180                       | 4                | Done        |
| F180-8      | 180                       | 8                |             |
| F180-8-C    | 180                       | 8                | Done        |
Figure 1. XRD pattern of the nanostructured α-Fe₂O₃ microparticles synthesized by solvothermal method at 150 °C for 4 h

49.5°, 54.1°, 57.6°, 62.5°, 64.1°, 71.9° and 75.5° which can be well ascribed to the standard peaks of the hematite structure (crystal system: hexagonal, space group: R-3c, cell parameters: a=b=5.034 Å, c=13.748 Å, JCPDS No.: 033-0664) [23-25]. The obtained XRD pattern shows no peaks related to impurities and confirms that pure and single phase α-Fe₂O₃ has been successfully synthesized.

The morphology of the as-synthesized α-Fe₂O₃ was characterized using the FESEM. As can be seen in Figures 2(a), 2(b), and 2(c) the pure α-Fe₂O₃ consists of distorted microspheres with an average diameter in the range of 1 to 1.5 µm. The microspheres are composed of nanoparticles with an average size in the range of 10 to 30 nm. This morphology is different from the morphology of α-Fe₂O₃ synthesized via a solvothermal process by Cao et al. [18]. In their study, the same precursors and solvent with the microwave irradiation as the heating source were used and flowerlike nanostructures composed of nanopetals were obtained. In the present study, convection heating by a standard laboratory oven was used to carry out the reaction synthesis of α-Fe₂O₃. From the obtained results, it can be concluded that the source of energy used in the solvothermal process is a key factor, which can affect the morphology of the products.

The EDS pattern was taken to specify the composition of the sample. As shown in Figure 2(d) the EDS pattern confirms the presence of the elements Fe and O in the as-synthesized α-Fe₂O₃ nanostructures.

The chemical structure of the α-Fe₂O₃ samples was studied by FTIR analysis. The FTIR spectrum was obtained in the wavenumber range from 400 to 4000 cm⁻¹ at room temperature. As can be seen in Figure 3 two vibrational bands at 474 cm⁻¹ and 562 cm⁻¹ are related to the Fe-O stretching modes which confirm the formation of α-Fe₂O₃ crystals. The bands at 1633 cm⁻¹ and 3417 cm⁻¹ are related to the bending and stretching modes of the O-H groups respectively, that show the presence of the hydroxyl group and/or water molecules on the surface of α-Fe₂O₃ [26].

Figure 4 displays the BET plot of the as-prepared α-Fe₂O₃ microparticles. The BET surface area and average pore size of the α-Fe₂O₃ microparticles are calculated to be
3. 2. Reaction Mechanism

In the solvothermal process, the OH− group is formed by adding urea to water molecules present in the solvent of ethanol (steps 1 and 2). The brown colloidal precipitates of Fe(OH)₃ are produced by the association of OH− and Fe³⁺ (step 3). High temperature and pressure of the solvothermal process leads to dehydration of Fe(OH)₃ and finally crystallization of the amorphous iron oxide to α-Fe₂O₃ crystals (step 4). Besides, urea has the role of a capping agent in the formation of the as-synthesized α-Fe₂O₃ nanostructures [18, 27].

1) CO(NH₂)₂ + H₂O → 2NH₃ + CO₂
2) NH₃ + H₂O → NH₄⁺ + OH⁻
3) Fe³⁺ + 3OH⁻ → Fe(OH)₃
4) 2Fe(OH)₃ → α-Fe₂O₃ + 3H₂O

3. 3. Effect of Synthesis Parameters on Purity and Phase Structure

In phase 2 of this study, the α-Fe₂O₃ samples were synthesized via the solvothermal method, and the effect of synthesis parameters on the crystallite size of the products was investigated. The solvothermal reaction was performed at different reaction temperatures for different reaction times. Moreover, according to Table 1 three of the as-synthesized samples were calcined to determine the effect of calcination on the crystallite size of the samples.

The X-ray Diffraction (XRD) patterns of α-Fe₂O₃ samples were taken to determine the purity, phase, and crystal structure of the products. The XRD patterns of the samples are shown in Figure 5. For the sample F120-4, no distinctive peak of α-Fe₂O₃ can be identified in the obtained XRD pattern, which reveals that the sample prepared at the reaction temperature of 120 °C for a duration of 4 h has an amorphous phase. As can be seen in Figure 5, when reaction time increases from 4 h to 8 h and reaction temperature increases from 120 °C to 150 °C and 180 °C the diffraction peaks assigned to the α-Fe₂O₃, without any impurity peaks, appear in the obtained XRD patterns. Thus, except for the sample F120-4, the obtained XRD patterns for all other samples show diffraction peaks which can be well ascribed to the standard peaks of hematite structure with JCPDS No. 033-0664. This indicates when the reaction is performed at a low temperature of 120 °C and a low duration of 4 h, the activation energy is not enough for crystallization of the α-Fe₂O₃ nanostructures, and the sample phase remains amorphous. This is compatible with the findings in the previous studies [5].

The synthesis parameters used in this study and some previous studies are shown in Table 2. These data show that the nanostructured α-Fe₂O₃ can be synthesized at a relatively lower reaction temperature and shorter reaction time. These conditions are desirable due to the less energy and time consumption in the synthesis of nanostructured hematite.

| Reference | Reaction Time (h) | Reaction Temperature (°C) | α-Fe₂O₃ Morphology |
|-----------|------------------|---------------------------|-------------------|
| Zhang et al. [16] | 24 | 200 | 3D Flowerlike |
| Ayachi et al. [27] | 10 | 200 | Nanoplatelets |
| Liang et al. [28] | 24 | 180 | 3D Multileaf |
| Majumder et al. [29] | 8 | 180 | 3D Dendritic |
| Xiao et al. [30] | 24 | 160 | Burger-like |
| This study | 4 | 150 | Distorted microspheres |
3. 4. Effect of Synthesis Parameters on Crystallite Size

3. 4. 1. Reaction Temperature
The mean crystallite size of the samples was calculated by equation (1) based on the obtained peak at \( \theta = 33.2^\circ \) in the XRD patterns of hematite samples. The effect of the reaction temperature on the crystallite size of the samples was determined at 120 °C, 150 °C, and 180 °C. According to Figure 6, for the reaction time of 4 h the crystallite size of the \( \alpha-Fe_2O_3 \) samples increases from 19.7 nm to 24.4 nm when the reaction temperature increases from 150 °C to 180 °C. In addition, for the reaction time of 8 h, the crystallite size is 21.3, 25.4, and 31.9 nm at the temperature of 120 °C, 150 °C, and 180 °C, respectively. Thus, in the higher reaction temperature, hematite with a larger crystallite size is formed and this is in good compliance with the previous works [5, 31, 32].

3. 4. 2. Reaction Time
The effect of reaction time on the crystallite size of the samples was also investigated. The solvothermal reactions were carried out for reaction times of 4 h and 8 h and the crystallite size was calculated by Equation (1). According to Figure 7 at reaction temperatures of 120 °C, 150 °C, and 180 °C the crystallite size of the samples increases when the reaction time increases from 4 h to 8 h. By increasing the solvothermal process time, the crystal growth occurs in a longer time, thus the samples with larger crystallite size can be prepared [31].

3. 4. 3. Calcination
The effect of calcination on the crystallite size of samples was evaluated for the three samples 150-4-C, F180-4-C, and F180-8-C. According to Figure 8, the crystallite size of the samples increases by calcination at 500 °C for 1 h. The growth in the crystallite size of the samples after calcination is due to the reduction in activation energy of crystallization, which enhances the crystal growth rate in the as-prepared nanostructured hematite [32, 33]. The enhancement of crystallite size and subsequent low density of crystalline defects of the nanostructured \( \alpha-Fe_2O_3 \) crystals is desirable in some applications [34, 35].

4. CONCLUSION
The nanostructured \( \alpha-Fe_2O_3 \) was successfully synthesized via a facile one-step solvothermal process.
using FeCl₃·6H₂O, urea, and ethanol treated at 150 °C for 4 h. The FESEM images showed that the samples are composed of distorted microspheres with an average diameter in the range of 1 to 1.5 μm and each microsphere is built from self-assembled nanoparticles with an average size in the range of 10 to 30 nm. The BET analysis showed a relatively high surface area of 41.86 m²g⁻¹ for the α-Fe₂O₃ nanostructures. The effect of solvothermal reaction parameters on the structural properties of the α-Fe₂O₃ nanostructures was investigated. It was found that an increase in reaction temperature and reaction time in the solvothermal process, leads to an increase in crystallite size of the products. In addition, the effect of calcination on the crystallite size was studied and the results showed that calcination enhanced the crystallite size of the nanostructured α-Fe₂O₃. The sample synthesized at 150 °C for 4 h had the smallest crystallite size of 13.6 nm. The sample that was treated at 180 °C for 8 h and then calcined at 500 °C for 1 h had the largest crystallite size of 75.4 nm. The obtained results can be suitable to synthesize α-Fe₂O₃ nanostructures with enhanced properties for different applications.

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**Persian Abstract**

چکیده

یک کسی که فلزات درونی، خانه یا هر مسی، را می‌خواهد باید تهیه کنند. در نه ساله‌های پیشین، مولکولی‌هایی مانند Fe₂O₃ با میکرو‌ساختارها و ساختارهای دیگر مورد استفاده قرار گرفته‌اند. این روشهای از این روشهای برای ساختن هایی با این طول‌ریزی از شکل‌های میکرو‌ساختارها، Fe₂O₃، XRD و میکروسکوپی الکترونی روبن‌شیر می‌باشد. طیف نجوم فلزات مورد بررسی فرآیند پترن (FTIR) و آنالیز مکانی و دقت ناحیه ساختمان (BET) می‌باشد. فناوری‌های با مولکول‌هایی میکرو‌ساختارهای های اوماجه باید به اینکه فرآیندی که به میکرو‌ساختاری از مولکول‌های شکل‌نام نشان می‌دهد. ناحیه میکرو‌ساختارهای Fe₂O₃ و ناحیه‌هایی که میکرو‌ساختاری به دست آمده‌اند، آنالیز ناحیه ساختمان به دست آمده‌اند. در نهایت ناحیه میکرو‌ساختارهای Fe₂O₃ و ناحیه‌هایی که میکرو‌ساختاری به دست آمده‌اند، آنالیز ناحیه ساختمان به دست آمده‌اند. در نهایت ناحیه میکرو‌ساختارهای Fe₂O₃ و ناحیه‌هایی که میکرو‌ساختاری به دست آمده‌اند، آنالیز ناحیه ساختمان به دست آمده‌اند. در نهایت ناحیه میکرو‌ساختارهای Fe₂O₃ و ناحیه‌هایی که میکرو‌ساختاری به دست آمده‌اند، آنالیز ناحیه ساختمان به دست آمده‌اند. در نهایت ناحیه میکرو‌ساختارهای Fe₂O₃ و ناحیه‌هایی که میکرو‌ساختاری به دست آمده‌اند، آنالیز ناحیه ساختمان به دست آمده‌اند. در نهایت ناحیه میکرو‌سافتارهای Fe₂O₃ و ناحیه‌هایی که میکرو‌سافتاری به دست آمده‌اند، آنالیز ناحیه ساختمان به دست آمده‌اند. در نهایت ناحیه میکرو‌سافتارهای Fe₂O₃ و ناحیه‌هایی که میکرو‌سافتاری به دست آمده‌اند، آنالیز ناحیه ساختمان به دست آمده‌اند.

**Keywords:** Fe₂O₃, α-Fe₂O₃, XRD, BET, FTIR.