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Nanostructured α-Fe_2O_3: Solvothermal Synthesis, Characterization, and Effect of Synthesis Parameters on Structural Properties

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ABSTRACT

α-Fe_2O_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 α-Fe_2O_3 with different crystal structures and morphologies to obtain the desired properties. In this research, nanostructured α-Fe_2O_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 µm 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^2g^{-1}. The influence of temperature and duration of the solvothermal process as well as, calcination on the structural properties of the α-Fe_2O_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 α-Fe_2O_3 nanostructures with a minimum crystallite size of 13.6 nm were synthesized at 150 °C for 4 h while the largest crystallite size of 75.4 nm was obtained at 180 °C and 8 h with subsequent calcination of the sample at 500 °C for 1 h. The results of the present study can be useful to enhance the properties of α-Fe_2O_3 nanostructures in various fields of application.

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1. INTRODUCTION

Hematite (α-Fe_2O_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 α-Fe_2O_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 α-Fe_2O_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 α-Fe_2O_3 nanostructures with different sizes and shapes by changing reaction time and solvent via a simple hydrothermal process. α-Fe_2O_3 nanopolyhydra, nanoparticles and microcubes were obtained by different solvents and reaction times. Zhang et al. synthesized two different morphologies of α-Fe_2O_3 using two different solvents in a solvothermal process.

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3D flower-like α-Fe_2O_3 nanostructures and α-Fe_2O_3 nanoparticles were obtained with isopropanol and water as the solvent, respectively [16]. Trpkov et al. [17] synthesized α-Fe_2O_3 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-Fe_2O_3 nanostructures via a solvothermal route using $FeCl_3.6H_2O$, 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 WO_3 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-Fe_2O_3 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-Fe_2O_3 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% $FeCl_3.6H_2O$, Merck), urea (99.5%, ChemLab) and ethanol (99.9% C_2H_5OH, Merck) were used to synthesize α-Fe_2O_3 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-Fe_2O_3 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 $FeCl_3.6H_2O$ 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 PANanalytical with Cu Kα radiation at λ=1.54060 Å. The crystallite size of the samples was calculated by Scherrer's equation as follows:

$$D \approx \frac{0.9\,L}{B\cos\theta} \quad (1)$$

where D is the mean crystallite diameter, L is the wavelength of the X-ray applied, θ is the diffraction angle of the specified peak, and B 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-Fe_2O_3 is shown in Figure 1. The obtained XRD pattern illustrates diffraction peaks with 2θ at 24.2°, 33.1°, 35.7°, 40.9°,

TABLE 1. Different solvothermal conditions used for the synthesis of nanostructured α-Fe_2O_3

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

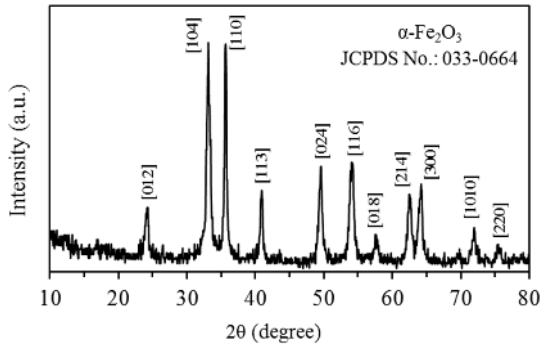

Figure 1. XRD pattern of the nanostructured α-Fe_2O_3 microparticles synthesized by solvothermal method at 150 °C for 4 h

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=13.748Å, JCPDS No.: 033-0664) [23-25]. The obtained XRD pattern shows no peaks related to impurities and confirms that pure and single phase α-Fe_2O_3 has been successfully synthesized.

The morphology of the as-synthesized α-Fe_2O_3 was characterized using the FESEM. As can be seen in Figures 2(a), 2(b), and 2(c) the pure α-Fe_2O_3 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-Fe_2O_3 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-Fe_2O_3. 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-Fe_2O_3 nanostructures.

The chemical structure of the α-Fe_2O_3 samples was studied by FTIR analysis. The FTIR spectrum was obtained in the wavenumber range from 400 to 4000cm^{-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-Fe_2O_3 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-Fe_2O_3 [26].

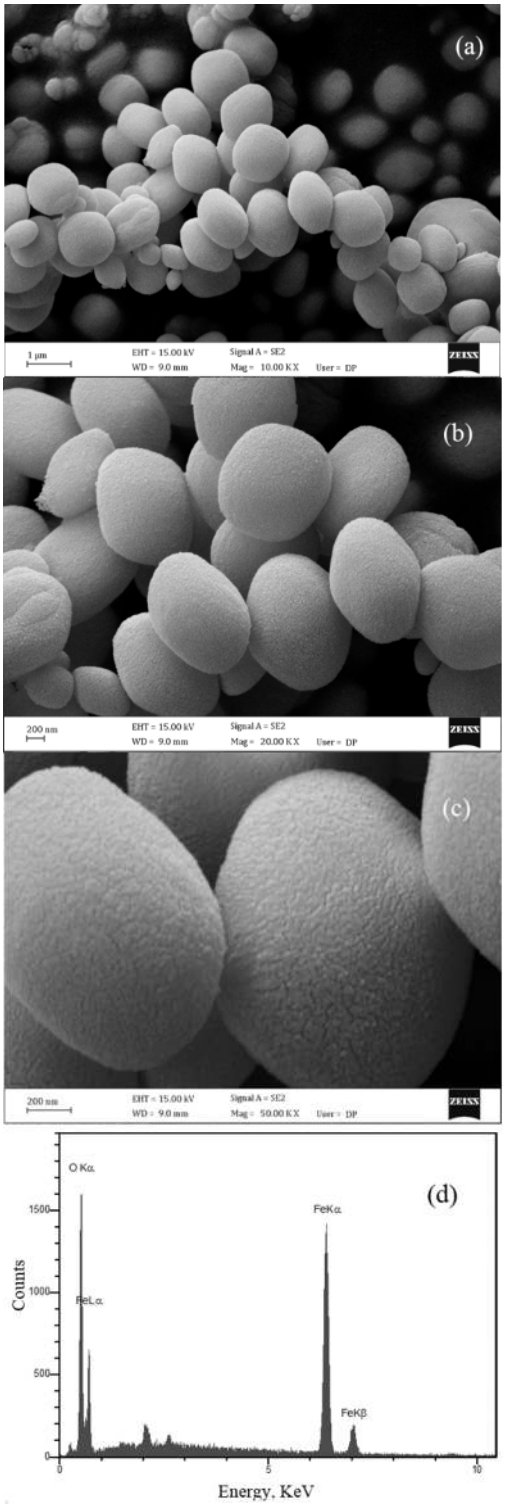

Figure 2. FESEM images (a), (b), (c), and EDS pattern (d) of the nanostructured α-Fe_2O_3 microparticles synthesized by solvothermal method at 150 °C for 4 h

Figure 4 displays the BET plot of the as-prepared α-Fe_2O_3 microparticles. The BET surface area and average pore size of the α-Fe_2O_3 microparticles are calculated to be

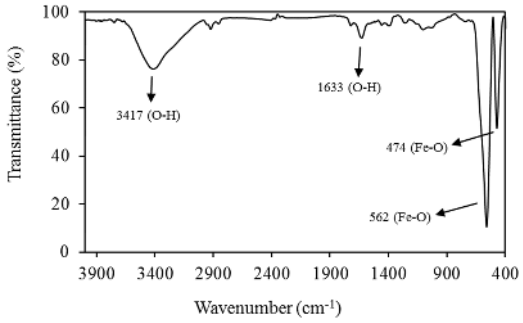

Figure 3. FTIR spectrum of the as-synthesized nanostructured α-Fe_2O_3 microparticles

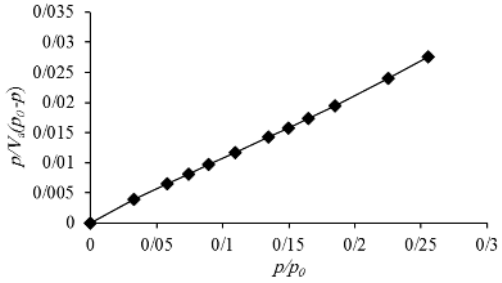

Figure 4. BET plot of the as-synthesized nanostructured α-Fe_2O_3 microparticles

41.86 m^2/g^{-1} and 3.45 nm respectively. This relatively high surface area of the samples is attributed to the nanostructured surface of the particles as confirmed by the FESEM. The large surface area is of great significance in some applications such as photocatalysis which adsorption has the main role in the process [27].

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 Fe^{+3} (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-Fe_2O_3 crystals (step 4). Besides, urea has the role of a capping agent in the formation of the as-synthesized α-Fe_2O_3 nanostructures [18, 27].

1) $CO(NH_2)_2 + H_2O \rightarrow 2NH_3 + CO_2$
2) $NH_3 + H_2O \rightarrow NH_4^+ + OH^-$
3) $Fe^{3+} + 3OH^- \rightarrow Fe(OH)_3$
4) $2Fe(OH)_3 \rightarrow \alpha\text{-}Fe_2O_3 + 3H_2O$

3. 3. Effect of Synthesis Parameters on Purity and Phase Structure In phase 2 of this study, the α-Fe_2O_3 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-Fe_2O_3 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-Fe_2O_3 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-Fe_2O_3, 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-Fe_2O_3 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-Fe_2O_3 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.

TABLE 2. Solvothermal synthesis parameters of α-Fe_2O_3 in the present and previous studies

Reference	Reaction Time (h)	Reaction Temperature (°C)	α-Fe_2O_3 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

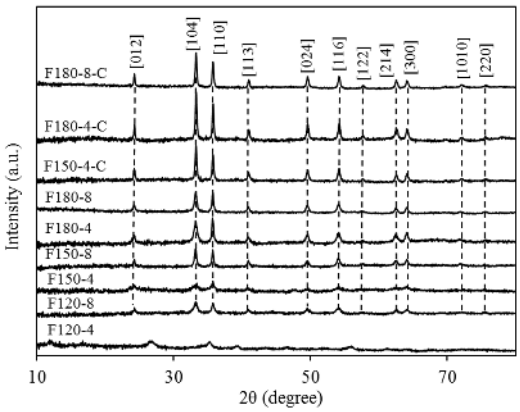

Figure 5. XRD patterns of hematite prepared by the solvothermal method at reaction temperatures of 120 °C, 150 °C, and 180 °C for reaction times of 4 h and 8 h. The samples assigned by C were calcined at 500 °C for 1 h

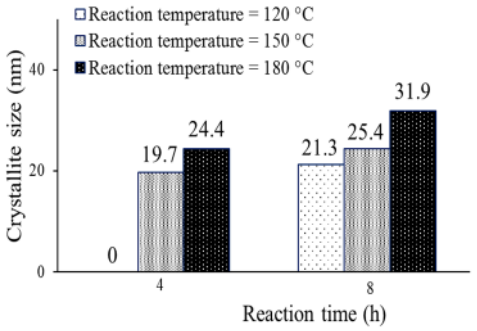

Figure 6. Effect of reaction temperature on the crystallite size of the as-synthesized α-Fe_2O_3

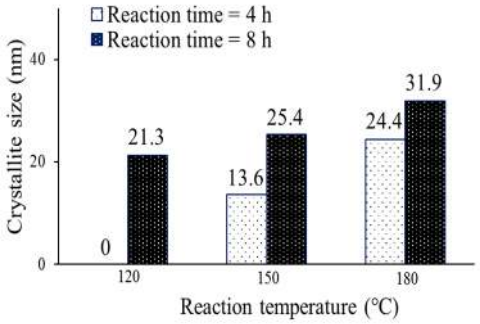

Figure 7. Effect of reaction time on the crystallite size of the as-synthesized α-Fe_2O_3

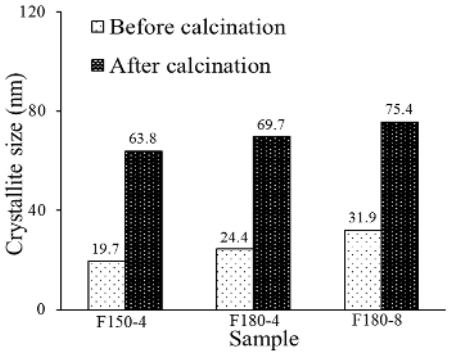

Figure 8. Effect of calcination on the crystallite size of the as-synthesized α-Fe_2O_3

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 θ=33.2 ° 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 α-Fe_2O_3 samples increases from19.7 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 8h 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 α-Fe_2O_3 crystals is desirable in some applications [34, 35].

4. CONCLUSION

The nanostructured α-Fe_2O_3 was successfully synthesized via a facile one-step solvothermal process

using $FeCl_3.6H_2O$, 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 m^2g^{-1} for the α-Fe_2O_3 nanostructures. The effect of solvothermal reaction parameters on the structural properties of the α-Fe_2O_3 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-Fe_2O_3. 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-Fe_2O_3 nanostructures with enhanced properties for different applications.

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Persian Abstract

چکیده

α-Fe_2O_3 یک اکسید فلزی پایدار، ارزان و غیر سمی با مزایای زیاد و زمینه های کاربردی گوناگون می باشد. تلاش های زیادی برای سنتز α-Fe_2O_3 با ساختار های بلوری و مورفولوژی های متفاوت برای به دست آوردن خواص مطلوب صورت گرفته است. در این پژوهش، α-Fe_2O_3 نانوساختار با یک روش ساده حلالی حرارتی سنتز می شوند. نمونه های به دست آمده با استفاده از روش های پراش اشعه X (XRD)، میکروسکوپی الکترونی روبشی نشر میدانی (FESEM)، طیف سنجی پراش انرژی اشعه X (EDS)، طیف سنجی مادون قرمز تبدیل فوریه (FTIR)، و آنالیز مساحت سطح برونر-امت-تلر (BET) مشخصه یابی می شوند. نتایج نشان می دهد هماتیت سنتزی از نانوساختار هایی با مورفولوژی میکروکره های اعوجاج یافته با میانگین قطر در محدوده ۱ تا ۱.۵ میکرومتر تشکیل شده که هر میکروکره از نانوذرات خود تشکیل با اندازه میانگین ۱۰ تا ۳۰ نانومتر تشکیل یافته است. نتایج نشان می دهد نانوساختار های هماتیت مساحت سطح ویژه ای معادل ٤١.٨٦ متر مربع بر گرم دارند. تأثیر دما و زمان فرایند حلالی حراتی و نیز اثر کلسیناسیون بر روی ساختار بلوری نمونه های سنتز شده مورد بررسی قرار می گیرد. نتایج نشان می دهد با افزایش دما و زمان واکنش حلالی حرارتی، اندازه کریستالیت در نمونه ها افزایش می یابد. به علاوه، کلسیناسیون منجر به افزایش اندازه کریستالیت نمونه ها می گردد. نانوساختار های α-Fe_2O_3 با حداقل اندازه کریستالیت ١٣.٦ نانومتر در دمای ۱۵۰ درجه سانتی گراد برای مدت ٤ ساعت سنتز شدند در حالی که بزرگ ترین اندازه کریستالیت برابر ٧٥.٤ نانومتر در دمای ۱۸۰ درجه سانتی گراد و ۸ ساعت و سپس کلسیناسیون نمونه در ۵۰۰ درجه سانتی گراد به مدت ۱ ساعت به دست آمد. نتایج این مطالعه می تواند در بهبود خواص نانوساختار های α-Fe_2O_3 در زمینه های مختلف کاربردی مفید واقع شود.