A novel rapid-combustion process for the preparation of magnetic $\alpha$-Fe$_2$O$_3$ nanoparticles

Cite as: AIP Advances 9, 125027 (2019); https://doi.org/10.1063/1.5126660
Submitted: 04 September 2019 . Accepted: 03 December 2019 . Published Online: 19 December 2019

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ABSTRACT
A novel rapid-combustion process for the preparation of magnetic α-Fe₂O₃ nanoparticles was introduced. The as-prepared α-Fe₂O₃ nanoparticles were characterized by scanning electron microscopy, the transmission electron microscope, X-ray diffraction, energy dispersive spectroscopy, the vibrating sample magnetometer, the thermogravimetric analysis, and differential scanning calorimetry. The experimental results revealed that α-Fe₂O₃ nanoparticles could form even at 400 °C, and the volume of absolute alcohol and the calcination temperature were two key parameters for the preparation of α-Fe₂O₃ nanoparticles. With the calcination temperature increasing from 400 °C to 800 °C, the average crystallite size of α-Fe₂O₃ nanoparticles with 10 ml of absolute alcohol increased from 26 nm to 45 nm and the degree of crystal defects was enlarged. When dosage of absolute alcohol increased from 5 ml to 25 ml, the average crystallite size of α-Fe₂O₃ nanoparticles calcined at 400 °C for 2 h ranged from 23 nm to 27 nm and their specific saturation magnetizations increased from 0.4 emu/g to 36.9 emu/g.

I. INTRODUCTION
Nanoscience and nanotechnology began to flourish in the past few decades. Shortly after nanomaterials came into being, extensive research of new nanomaterials and preparation methods has been conducted. With the increase in the knowledge for nanomaterial manufacturing techniques, the nanomaterials’ properties have also been continuously enhanced. Compared with the other nanomaterials, iron oxides (α-Fe₂O₃, γ-Fe₂O₃, and Fe₃O₄) have attracted considerable attention because of their remarkable properties, including their low biological toxicity, strong environmental tolerance, high resistance to corrosion, excellent thermodynamic stability, etc. α-Fe₂O₃ (hematite) nanomaterials not only have the fundamental properties of iron oxides but also are the most stable iron oxide phase under ambient conditions. The appealing versatility of hematite has also promoted its research value, so there are wide-ranging application reports in the environmental issue, biomedicine, electronic information, engineering application, and optoelectronics. Therefore, the hematite has particularly been the focus of interest as a multifunctional and nanostructured material.

At present, hematites with various morphologies, dimensions, and sizes have been fabricated in the literature, including zero-dimensional nanoparticles, one-dimensional nanorods, two-dimensional nanofilms, and three-dimensional nanoporous scaffolds. The zero-dimensional nanoparticles have the characteristics of spatial confinement properties. Owing to their intriguing structural features and potential applications, much more attention has been directed to the controlled preparation of hematite nanoparticles. There are several methods reported to obtain hematite nanoparticles, for instance, the sol-gel method, the coprecipitation method, the thermal transformation method, and the...
Different approaches for the preparation of nanoparticles will affect the crystal structure, the crystallinity, and the surface area of hematite. The preparation method of hematite nanoparticles with the controllable properties remains a huge challenge. Therefore, in order to provide a straightforward and robust process to produce hematite nanoparticles, we have developed a novel solution-based method involving a facile combustion route.

In this project, we prepared magnetic α-Fe$_2$O$_3$ nanoparticles via a rapid-combustion process, and the key factors for the calcination temperature and the absolute alcohol volume on the crystal size and the crystalline were investigated.

II. EXPERIMENTAL DETAILS

A. Preparation of magnetic α-Fe$_2$O$_3$ nanoparticles

Magnetic α-Fe$_2$O$_3$ nanomaterials were prepared via the rapid-combustion process. All chemicals were in analytical purity, 0.02 mol of ferric nitrate [(Fe(NO$_3$)$_3$)$_3$·9H$_2$O] was added into required amounts of absolute alcohol, and the ratio of ferric nitrate to absolute alcohol volume could be controlled at a mass-volume concentration of 4%-22%. The solution concentration of ferric nitrate could be controlled by the volume of absolute alcohol. Then, the mixed solution was vigorously stirred at room temperature to form homogeneous solution, and the solution was poured into a crucible and ignited. When the fire was out, the intermediate was placed in series experimental resistance furnace with the heat rate of 3 °C/min at the designed calcination temperature for 2 h. After natural cooling, the obtained dark precipitates were ground and the product was obtained. The effects of the calcination temperature and the absolute alcohol volume on the average particle size and magnetic properties were investigated.

B. Characteristics of magnetic α-Fe$_2$O$_3$ nanoparticles

The photomicrography of α-Fe$_2$O$_3$ nanoparticles was obtained by the scanning electron microscopy (SEM, JEOL JSM-7800F, Tokyo, Japan). The field emission scanning electron microscopy coupled with an energy-dispersive X-ray spectrometer (EDS) was employed to analyze the chemical composition of the products. The sample was dispersed in absolute alcohol by using ultrasonic radiation, and a drop of suspension was placed onto the copper-coated grids using transmission electronic microscopy (TEM, JEOL HR-2100, Tokyo, Japan) to obtain the detailed morphology of the α-Fe$_2$O$_3$ nanomaterial. The X-ray diffraction (XRD) analysis was applied to identify crystallographic structures, and the XRD patterns of powder samples were performed by an X-ray diffraction system (Rigaku D/max 2500PC) with Cu-Kα radiation (λ = 1.542 Å) as an X-ray source at 60 kV, 300 mA, and 2θ scan range from 10° to 80° with a scanning speed of 5°/min. The magnetic properties of the as-prepared nanomaterials were examined at room temperature using a vibrating sample magnetometer (VSM, ADE DMS-HF-4).

III. RESULTS AND DISCUSSION

A. Characteristics of α-Fe$_2$O$_3$ nanoparticles

The characterization of α-Fe$_2$O$_3$ nanoparticles calcined at 400 °C for 2 h with 10 ml of absolute alcohol is shown in Fig. 1. The particle size and the morphology were examined by SEM. As
evinced in Fig. 1(a), the \( \alpha \)-Fe\(_2\)O\(_3\) nanoparticles assembled compactly and arranged regularly, the average diameter of \( \alpha \)-Fe\(_2\)O\(_3\) nanoparticles was about 25 nm. The TEM image [shown in Fig. 1(b)] also showed that a large number of regular nanoparticles were placed close together, and the high uniformity of \( \alpha \)-Fe\(_2\)O\(_3\) nanocrystals consisted of such aggregates was well maintained. The XRD of the product was used to determine the crystalline phase characteristics and the composition of the products. The XRD pattern of \( \alpha \)-Fe\(_2\)O\(_3\) is shown in Fig. 1(c), which exhibited diffraction peaks of (012), (104), (110), (024), (116), (214), (300), (1010), and (220). The patterns matched well the characteristic diffraction (012), (104), (110), (113), (024), (116), (122), (214), (300), (1010), and (220). The patterns matched well the characteristic diffraction peaks of standard \( \alpha \)-Fe\(_2\)O\(_3\) PDF card (JCPDS No. 89-0596), which suggested that the obtained product was \( \alpha \)-Fe\(_2\)O\(_3\) nanoparticles. The observation of the EDS energy spectrum [Fig. 1(d)] indicated that the principal atomic percentages of Fe and O in the nanoparticles were 41.57% and 58.43%, respectively, which were basically consistent with the elemental composition of \( \alpha \)-Fe\(_2\)O\(_3\). All the above data implied that pure \( \alpha \)-Fe\(_2\)O\(_3\) nanoparticles had been successfully prepared via the rapid-combustion process. The whole process was feasible and highly reproducible.

The decomposition of \( \alpha \)-Fe\(_2\)O\(_3\) was investigated by thermogravimetric analysis (TGA)-differential scanning calorimetry (DSC) in the temperature range of 0–800°C using the heating rate of 5°C/min, as exhibited in Fig. 2. The decomposition of \( \alpha \)-Fe\(_2\)O\(_3\) started with the loss of water between 65°C and 110°C showing a corresponding endothermic heat flow. A weight loss of 4.59% occurred during this first step corresponding to the evaporation of free water. The second step between 110°C and 400°C corresponds to the degradation of the organic matter with a weight loss of 13.07% and an exothermic peak centered at 261°C. In addition, as the thermogravimetric analysis could been seen, the fact that the product was in constant weight status above 400°C suggested that the organic matter had been completely decomposed.

B. Effect of calcination temperature on \( \alpha \)-Fe\(_2\)O\(_3\) nanoparticles

The XRD patterns of the samples calcined at 400, 500, 600, 700, and 800°C with 10 ml absolute alcohol are shown in Fig. 3(a). When the calcination temperature was 400°C, the corresponding XRD peaks were bottommost compared with those peaks of the nanoparticles obtained at other calcination temperatures. The XRD patterns revealed that crystal crystallinity was the lowest at the calcination temperature of 400°C. Obviously, the XRD patterns of Fig. 3(a) showed the intensity of diffraction peaks increased with the calcination temperature ascending from 400°C to 800°C, which implied that the better crystallinity of the obtained \( \alpha \)-Fe\(_2\)O\(_3\) could be achieved at the higher temperature. Considering desirable sintering in a high temperature range, the aggregation of crystals was intense in thermal treatments.

The crystallite sizes of the prepared \( \alpha \)-Fe\(_2\)O\(_3\) were calculated using the Scherrer formula: \( D = K \lambda / \beta \cos \theta \), where \( K \) is the Scherrer constant of 0.89, \( \lambda \) is the wavelength of X-ray, and \( \beta \) is the full width at half maximum (FWHM) of the peak at the Bragg diffraction angle \( \theta \). The average crystallite sizes of \( \alpha \)-Fe\(_2\)O\(_3\) calculated from the value of the FWHM of the (104) plane are illustrated in Fig. 3(b), which could be clearly seen that the mean crystallite size at the calcination temperature, continually diminishing from 45 nm to 26 nm. The calculation data were consistent with the previous analysis from Fig. 3(a). It could be concluded that the main form of microstructural evolution was grain coarsening.

![FIG. 2. TGA/DSC of \( \alpha \)-Fe\(_2\)O\(_3\) calcined at 400°C for 2 h with 10 ml of absolute alcohol.](image-url)
The sintered strength of the nanomaterials prepared at the higher temperature was greater, which resulted in the larger crystal grain size.

The software of Jade was used to analyze the crystal lattice distortion of \( \alpha \)-Fe\(_2\)O\(_3\) grains. Here, d is the crystal plane spacing calculated from the corresponding peak of XRD patterns and a and c are lattice constants. The lattice constants of samples calcined at different temperatures are listed in Table I. As was known to all, the standard \( \alpha \)-Fe\(_2\)O\(_3\) was a hexagonal structure with unit cell parameters \( a = 5.035 \, \text{Å} \) and \( c = 13.74 \, \text{Å} \). When the calcination temperature was \( 400 \, ^\circ\text{C} \), the calculation result was in accordance with the standard value of c/a. On the one hand, accompanying the increasing tendency shown in the values for the lattice parameters of c/a, the phenomenon that higher calcination temperature led to more serious lattice distortion might be confirmed. On the other hand, the decreased lattice parameter value implied that the growth of the crystal in the direction of an axis was compressed. Additionally, it could be found that with the increase in the calcination temperature, the lattice parameter value of c/a increased gradually, which indicated that the crystal defects in the material were reduced, so the crystallinity of the crystal was better. The change of the mean crystallite size with temperature was closely related to the nanocrystalline structure. The characterization of the nanocrystals prepared at the different calcination temperatures revealed that they had some common phases and differed in crystallinity. In a summary, the crystal grain size could be controlled by simply changing temperature to calcine precursors.

### Table I. Lattice parameters of \( \alpha \)-Fe\(_2\)O\(_3\) prepared at different calcination temperatures.

| T (°C) | d (Å)  | a (Å)  | c (Å)  |
|--------|--------|--------|--------|
| 400    | 2.7031 | 5.0469 | 13.7565|
| 500    | 2.6989 | 5.0431 | 13.7654|
| 600    | 2.6994 | 5.0355 | 13.7482|
| 700    | 2.7024 | 5.0326 | 13.7486|
| 800    | 2.6869 | 4.9980 | 13.7084|

C. Effect of absolute alcohol volume on \( \alpha \)-Fe\(_2\)O\(_3\) nanoparticles

In order to investigate the properties of the iron oxide nanoparticles, the XRD studies had been performed on nanomaterials calcined at \( 400 \, ^\circ\text{C} \) for 2 h with different volumes of absolute alcohol. Under the conditions of the absolute alcohol volume of 5 ml, 10 ml, 15 ml, 20 ml, and 25 ml, the respective XRD patterns are displayed in Fig. 4(a). The crystallinity of the crystal obtained at the lower absolute alcohol dosage was better than that with the higher dosage, which was obviously seen from the intensity of diffraction peaks. When the amount of absolute alcohol was 5 ml, the larger particles were produced due to the incomplete combustion. As depicted in Fig. 4(b), the average crystal size decreased with the arise of the solvent volume from 5 ml to 20 ml, and then it increased. Estimated by Scherrer’s equation, the average crystallite size ranged from 23 nm to 27 nm. From the above examination results, we could judge that the average crystal size and crystallinity of \( \alpha \)-Fe\(_2\)O\(_3\) were greatly affected by the consumption of absolute alcohol. The amount of absolute alcohol used changes the burning time and dispersibility of ferric nitrate, and the lengthened combustion time would make the crystal more complete. The \( \alpha \)-Fe\(_2\)O\(_3\) nanoparticles had already obtained adequate crystallization at the dosage of 20 ml. Subsequently, the increase in crystal grain size was ascribed to the directional aggregation of the crystal.

It can be analyzed from Table II that the change of \( \alpha \)-Fe\(_2\)O\(_3\) crystal lattice parameter depended on the volume of absolute alcohol. With the increase in the absolute alcohol volume, the crystal growth time would be further extended in the combustion process, the crystal elongated along the axial direction, and the c axis deviated from a and b axes. The lattice parameter value of c/a varied irregularly in a certain interval of solvent dosage. It was confirmed that more serious lattice distortion arose following with the extended combustion time, compared with the standard value of c/a. The anisotropy would affect the growth behavior of \( \alpha \)-Fe\(_2\)O\(_3\) grains in the combustion process, the lattice defects in hexagonal \( \alpha \)-Fe\(_2\)O\(_3\) crystals caused the uneven density. The absolute alcohol not only dissolved ferric nitrate very well but also played the role of the crystal, which limited the growth.\(^{31}\) Hence, the amount of absolute alcohol would affect the microstructure of nanoparticles. At the same time, the burning time disordered the growth of the crystal.

![FIG. 4. XRD patterns (a) and the average grain size (b) of magnetic \( \alpha \)-Fe\(_2\)O\(_3\) nanoparticles calcined at \( 400 \, ^\circ\text{C} \) for 2 h with different volumes of absolute alcohol.](image-url)
TABLE II. Lattice parameters of $\alpha$-Fe$_2$O$_3$ prepared at various volumes of absolute alcohol.

| V (ml) | d (Å)   | a (Å)   | c (Å)   |
|--------|---------|---------|---------|
| 5      | 2.7027  | 5.0438  | 13.7615 |
| 10     | 2.7002  | 5.0361  | 13.7541 |
| 15     | 2.7056  | 5.0544  | 13.7671 |
| 20     | 2.7014  | 5.0369  | 13.7668 |
| 25     | 2.9570  | 5.0579  | 13.7663 |

D. Effect of absolute alcohol volume on magnetic property of $\alpha$-Fe$_2$O$_3$ nanoparticles

A vibrating sample magnetometer was used to characterize the magnetic properties of the prepared $\alpha$-Fe$_2$O$_3$ nanoparticles at room temperature. Figure 5 shows the hysteresis loops for the nanoparticles calcined at 400 °C with an absolute alcohol volume of 5 ml, 10 ml, 15 ml, 20 ml, and 25 ml, the inset illustrated an enlargement of the hysteresis loop near the magnetization intensity. The magnetic properties of $\alpha$-Fe$_2$O$_3$ nanoparticles prepared with different volumes of absolute alcohol were almost diversity, and the magnetization behavior of $\alpha$-Fe$_2$O$_3$ nanoparticles was changed greatly from hard magnetism to soft magnetism. The saturation magnetizations of the samples (Fig. 5) displayed a regular augment with the increase in absolute alcohol dosage. When the amount of absolute alcohol was equal to or less than 15 ml, the saturation magnetization of $\alpha$-Fe$_2$O$_3$ was not up to 3.0 emu/g. Subsequently, the saturation magnetization of the samples ascended sharply when the dosage of absolute alcohol increased to 20 ml. The high saturation magnetization of the nanoparticles was ascribed that priority orientation of the crystal growth changed to get magnetization easy axis. However, the coercivities of magnetic particles were inverse to their saturation magnetizations, and the coercivity was closely related to the micromagnetic description of magnetization reversal in small particles. The variation of coercivities for different nanostructures might result from their differences in microstructure, such as the lattice strain and the defects caused by the decrease in the crystallite sizes and the increase in surface areas. In addition, the magnetization behavior of the nanoparticles might be due to the variation of grain sizes and the magnetocrystalline anisotropy. The coercivities of nanoparticles prepared with absolute alcohol volume of 5 ml, 10 ml, 15 ml, 20 ml, and 25 ml were 1410 Oe, 1332 Oe, 407 Oe, 190 Oe, and 151 Oe, respectively. The decline of coercivity could be attributed to the occurrence of oxidation-reduction reactions. $\alpha$-Fe$_2$O$_3$ would be converted into $\gamma$-Fe$_2$O$_3$, at a relatively higher temperature, and the larger amount of absolute alcohol would reduce integral Fe$_2$O$_3$ to Fe$_3$O$_4$. Thus, the mixtures of hematite and magnetite were produced. As seen from the variation of the corresponding peak intensity [Fig. 4(a)], the $\alpha$-Fe$_2$O$_3$ nanoparticles might undergo a single magnetic phase transition. The transformation volume of magnetic produced in the present research began at 15 ml. It should be emphasized that the polymorphic phase was observed throughout the transformation. The magnetic characterizations showed that the consumption of absolute alcohol was a crucial factor to the saturation magnetization and the coercivity of iron oxides nanoparticles.

IV. CONCLUSIONS

Magnetic $\alpha$-Fe$_2$O$_3$ nanoparticles were successfully prepared by the rapid-combustion method, and they could form even at 400 °C. When dosage of absolute alcohol was 10 ml, the annealing precursor at different calcination temperatures led to the formation of monophasic $\alpha$-Fe$_2$O$_3$ in both the products. The average grain size of the obtained $\alpha$-Fe$_2$O$_3$ crystals increased from 26 nm to 45 nm with the calcination temperature increasing from 400 °C to 800 °C, and the products calcined at 800 °C had better crystallization. When 25 ml of absolute alcohol was used as a reducing agent, $\alpha$-Fe$_2$O$_3$ was transformed into Fe$_3$O$_4$ and magnetite could maintain its contribution to spontaneous magnetization. The polymorphic phases of hematite and magnetite were formed with the high saturation magnetization of 36.9 emu/g and the low coercivity of 151 Oe. The mass-volume concentration of ferric nitrate to absolute alcohol volume would determine the content of magnetite.

ACKNOWLEDGMENTS

This work was supported by the Opening Project of Key Laboratory of Green Chemistry of Sichuan Institutes of Higher Education (Grant No. LYG1910).

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