Preparation and characterization of $\alpha$-Fe$_2$O$_3$/Fe$_3$O$_4$ heteroplasmon nanoparticles via the hydrolysis-combustion-calcination process of iron nitrate

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Abstract

In this work, a feasible and facile hydrolysis-combustion-calcination process of ferric nitrate for the preparation of magnetic $\alpha$-Fe$_2$O$_3$/Fe$_3$O$_4$ heteroplasmon nanoparticles was represented. The influences of hydrolysis time, hydrolysis temperature, Fe$^{3+}$ concentration, anhydrous ethanol volume, calcination time, and calcination temperature on the properties of $\alpha$-Fe$_2$O$_3$/Fe$_3$O$_4$ heteroplasmon nanoparticles were investigated. According to a series of characterization analysis, the optimal preparation conditions were confirmed: 0.05 M Fe(NO$_3$)$_3$·9H$_2$O was hydrolyzed at 90 °C for 8 h, and then the precursor was calcined at 200 °C for 2 h with 20 ml anhydrous ethanol. While, the morphology of $\alpha$-Fe$_2$O$_3$/Fe$_3$O$_4$ heteroplasmon were spherical structures with the average particle size of about 46 nm, and their saturation magnetization was 54 emu g$^{-1}$. The $\alpha$-Fe$_2$O$_3$/Fe$_3$O$_4$ heteroplasmon nanoparticles possessed controllable magnetic properties and a more stable state, which suggested promising applications.

1. Introduction

With the advancement and development of science and technology, nanomaterials have gradually entered people’s daily lives [1–3]. Nanomaterial refers to materials that have at least one dimension in the nanometer size in a three-dimensional space or are composed of them as basic units [4, 5]. When the size of materials reaches the nanometer level, nanomaterials will show novel characteristics different from traditional materials, including quantum tunnel [6], surface [7], dielectric confinement, quantum size, and volume effect [8]. Magnetic nanoparticles are a kind of nanomaterials. It is widely used in the fields of biology, medicine, and chemical engineering [9–13], on account of its unique nano-size effect and magnetic properties. For example, Chen et al [14] proposed Ti$_3$O$_5$ nanoparticles as a pure Ti$^{3+}$ system for the electrosynthesis of NH$_3$ and the yield of NH$_3$ was significantly better than other titanium-based adsorbents. Wang et al [15] applied an electrochemical activation method to change the surface structure of nickel-cobalt oxide nanosheets, and improved their energy storage capacity and cycling performance. At present, the most familiar nanoparticles are composed of ferric oxides, such as magnetite (Fe$_3$O$_4$) and hematite (Fe$_2$O$_3$). Fe$_2$O$_3$ is low cost, simple to produce, environment friendly, good catalytic properties, and its chemical properties are very stable [16–18]. However, the saturation magnetization of hematite is very small, resulting that it was difficult for recovery, separation, and so on, which limits its applications at many areas. Fe$_3$O$_4$ is an important magnetic material with a cubic opposite spinel structure. It has a wide range of applications in biological sensitive materials, magnetic resonance imaging clinical diagnosis, and the bioengineering and pharmaceutical fields [19, 20] due to its good compatibility with biological tissue, electrical and magnetic properties corresponding to its size and morphology. However, the saturation magnetization of Fe$_3$O$_4$ is large, resulting that Fe$_3$O$_4$ nanomaterials tend to agglomerate, which also limits the applications of Fe$_3$O$_4$ nanomaterials in many fields.
Combining the merit and demerit of the $\alpha$-Fe$_2$O$_3$ and Fe$_3$O$_4$, a new type of Fe$_2$O$_3$/Fe$_3$O$_4$ heteroplasmon nanomaterials cause expert attention. Wang et al [21] synthesized Fe$_2$O$_3$/Fe$_3$O$_4$ composite hollow spheres by simply aging Fe$_3$O$_4$ nanospheres in a FeCl$_3$ solution, but its experimental period was length. Menezes et al [22] prepared Fe$_2$O$_3$/Fe$_3$O$_4$ microtubes via the thermal oxidation process of a high-purity metallic iron microwire. However, the temperature of the experimental process was too high to apply widely, and Fe$_2$O$_3$/Fe$_3$O$_4$ microtubes had a larger diameter and uncontrollable magnetism. Therefore, there is a need to investigate methods for the preparation of Fe$_2$O$_3$/Fe$_3$O$_4$ composites with short times, low reaction temperatures, controlled magnetic properties, and easy preparation.

In this project, we proposed the preparation of $\alpha$-Fe$_2$O$_3$/Fe$_3$O$_4$ heteroplasmon nanoparticles by a feasible and facile hydrolysis-combustion-calcination process of ferric nitrate, and the effects of hydrolysis time, hydrolysis temperature, Fe$^{3+}$ concentrations, anhydrous ethanol dosage, calcination temperature [23–25], and calcination time on properties of $\alpha$-Fe$_2$O$_3$/Fe$_3$O$_4$ were revealed.

2. Experimental

2.1. Preparation of $\alpha$-Fe$_2$O$_3$ and $\alpha$-Fe$_2$O$_3$/Fe$_3$O$_4$ heteroplasmon nanoparticles

$\alpha$-Fe$_2$O$_3$ nanoparticles were prepared via a facile hydrolysis process. 4.10 g Fe(NO$_3$)$_3$·9H$_2$O was added into 200 ml deionized water to form homogenous solution, the solution was transferred into a round-bottom flask, and heated for hydrolysis at 90 °C for 8 h until the orange-red suspension liquid was formed. The suspension liquid was centrifuged, and the solid was washed for respective three times with water and absolute alcohol, and then dried, the $\alpha$-Fe$_2$O$_3$ nanoparticles were obtained.

Magnetic $\alpha$-Fe$_2$O$_3$/Fe$_3$O$_4$ heteroplasmon nanoparticles were prepared by the rapid combustion-calcination method. The $\alpha$-Fe$_2$O$_3$ nanoparticles were uniformly dispersed in a certain amount of ethanol solution, the suspension was transferred into a crucible and ignited. When the fire was out, the intermediate together with the crucible was placed into a programmed temperature control furnace and calcined at various temperatures for different times, the $\alpha$-Fe$_2$O$_3$/Fe$_3$O$_4$ heteroplasmon nanoparticles were obtained.

2.2. Characterization of $\alpha$-Fe$_2$O$_3$ and $\alpha$-Fe$_2$O$_3$/Fe$_3$O$_4$ heteroplasmon nanoparticles

The phase identification of the $\alpha$-Fe$_2$O$_3$/Fe$_3$O$_4$ heteroplasmon nanoparticles was characterized by Rigaku D/max 2500 PC X-ray diffraction (XRD) with Cu-Kα radiation, the morphology and composition analyses were investigated with a scanning electron microscopy (SEM) and the transmission electron microscopy (TEM), the magnetic measurement was taken on an ADE DMS-HF-4 vibrating sample magnetometer (VSM).

3. Results and discussion

3.1. Characterization of $\alpha$-Fe$_2$O$_3$ nanoparticles

The characterization of $\alpha$-Fe$_2$O$_3$ nanoparticles prepared at hydrolysis temperature of 90 °C for 8 h with Fe$^{3+}$ concentration of 0.05 M was shown in figure 1. As SEM morphology presented in figure 1(a), $\alpha$-Fe$_2$O$_3$ nanomaterials were spherical structures with good dispersion, and the diameter was about 49 nm (figure 1(c)), these suggested the formation of $\alpha$-Fe$_2$O$_3$ nanoparticles. The TEM image of $\alpha$-Fe$_2$O$_3$ was shown in figure 1(b), it could be seen that the uniform size of $\alpha$-Fe$_2$O$_3$ nanoparticles, which was consistent with the SEM research. As reactants, Fe(NO$_3$)$_3$ was hydrolyzed under heating conditions to form Fe(OH)$_3$ and HNO$_3$. Finally, Fe(OH)$_3$ was formed into $\alpha$-Fe$_2$O$_3$ nanoparticles via resolving and anhydration in weak acid conditions. However, the HNO$_3$ cannot be completely volatilized because it had the high boiling temperature, and had a certain etching effect. So, the prepared $\alpha$-Fe$_2$O$_3$ nanoparticles were spherical particles. XRD pattern (figure 1(d)) displayed that sharp diffraction peaks possessed by $\alpha$-Fe$_2$O$_3$ nanoparticles at 20 values of 24.1°, 33.2°, 35.6°, 40.9°, 49.5°, 54.1°, 62.4°, and 63.9° could be indexed to the diffraction crystal of (012), (014), (110), (113), (024), (116), (214), and (300) of hematite (ICPDS NO. 33-0664), which indicated that $\alpha$-Fe$_2$O$_3$ nanomaterials were successfully synthesized [17]. The hysteresis loop of $\alpha$-Fe$_2$O$_3$ nanoparticles was presented in figure 1(e), it could be observed that $\alpha$-Fe$_2$O$_3$ nanoparticles exhibited weakly magnetic property, and their saturation magnetization was only 0.43 emu g$^{-1}$.

3.2. Influences of reaction conditions on $\alpha$-Fe$_2$O$_3$ nanoparticles

3.2.1. Hydrolysis time

The XRD patterns of $\alpha$-Fe$_2$O$_3$ nanoparticles prepared via hydrolysis of 0.05 M Fe(NO$_3$)$_3$ at 90 °C for 2–16 h were shown in figure 2(a), it could be clearly shown that the prepared nanoparticles were all $\alpha$-Fe$_2$O$_3$. As the hydrolysis time equaled to 2 h, the diffraction peaks of $\alpha$-Fe$_2$O$_3$ nanoparticles were not obvious, indicating that the crystallinity degree of $\alpha$-Fe$_2$O$_3$ nanoparticles was poor. With the rise of the hydrolysis time, all the diffraction
peaks of \(\alpha\)-Fe\(_2\)O\(_3\) nanoparticles became sharper, suggesting that the crystallinity degree of \(\alpha\)-Fe\(_2\)O\(_3\) nanoparticles became better. The reason was that as the hydrolysis time was prolonged, the crystal structure of \(\alpha\)-Fe\(_2\)O\(_3\) nanoparticles became more complete, causing the crystallinity degree of \(\alpha\)-Fe\(_2\)O\(_3\) nanoparticles became better. The yield change of \(\alpha\)-Fe\(_2\)O\(_3\) nanoparticles prepared under different hydrolysis times was shown in figure 2(b). When the hydrolysis time was less than 8 h, the yield of \(\alpha\)-Fe\(_2\)O\(_3\) nanoparticles increased with the prolongation of the hydrolysis time. This revealed that the hydrolysis degree of Fe(NO\(_3\))\(_3\) was intensified with the increase of the hydrolysis time, the yield of the \(\alpha\)-Fe\(_2\)O\(_3\) nanoparticles also increased. The yield of nanoparticles rapidly decreased from 90.31% to 72.40% with the hydrolysis time increasing from 8 h to 16 h. The probable reason was that a higher hydrolysis time promoted the increase of Fe(OH)\(_3\) and HNO\(_3\) productions, which inhibited the hydrolysis of Fe(NO\(_3\))\(_3\), and the \(\alpha\)-Fe\(_2\)O\(_3\) nanoparticles were corroded by excessive nitric acid, resulting in reduced yield. As stated above, when the hydrolysis time was 8 h, it was proper for the optimal hydrolysis time due to the larger yield and the better crystallinity degree of \(\alpha\)-Fe\(_2\)O\(_3\) nanoparticles.
3.2.2. Hydrolysis temperature
The XRD patterns of α-Fe₂O₃ nanoparticles prepared at hydrolysis temperature of 60 °C–90 °C for 8 h with 0.05 M Fe(NO₃)₃ were shown in figure 3(a). When the hydrolysis temperature was lower than 80 °C, the diffraction peaks of as-prepared nanoparticles agreed with the standard card of α-FeOOH (JCPDS. NO. 29–0713), which implied that the composition of the obtained nanoparticles was α-FeOOH. Obviously, under the low temperature of 80 °C, the reaction proceeded slowly, and α-FeOOH could not dehydrate to form α-Fe₂O₃. As the hydrolysis temperature rose from 80 °C to 90 °C, the reaction gradually accelerated, resulting in α-FeOOH increased and a higher yield (figure 3(b)). When the hydrolysis temperature reached 90 °C, α-FeOOH could be further dehydrated to form, and the reaction speed would also increase, which led to the rise of yield for α-Fe₂O₃ nanoparticles [17]. Therefore, the optimal hydrolysis temperature for α-Fe₂O₃ nanoparticles was 90 °C.

3.2.3. Fe³⁺ concentration
Figure 4 showed the XRD patterns and the yield of various products prepared at 90 °C for 8 h with Fe³⁺ concentrations of 0.05–0.5 M. As shown in figure 4(a), it indicated that the materials were α-Fe₂O₃, α-FeOOH, and amorphous powders with the different Fe³⁺ concentrations of 0.05 M, 0.1–0.3 M, and 0.5 M, respectively. The reason was that a large amount of nitric acid was generated with the high concentration of Fe³⁺, when the weak acid was heated at 90 °C, the dehydration reaction of α-FeOOH went in reverse, and the generate α-Fe₂O₃ was slowly converted into α-FeOOH. Because the reaction was very slow, so only a small amount of α-FeOOH was generated. The hydrolysis reaction of Fe(NO₃)₃ intensified with the concentration of Fe³⁺ increased, generated more nitric acid and the acidity of the solution also increased, resulted in α-Fe₂O₃ was greatly...
corroded by nitric acid, and the transformed $\alpha$-FeOOH decreased. Therefore, the yield decreased rapidly. When the concentration of Fe$^{3+}$ reached 0.5 M, the yield was almost 0, indicating that the reaction has not been carried out macroscopically. When the concentration of Fe$^{3+}$ was 0.05 M, the obtained nanoparticles were $\alpha$-Fe$_2$O$_3$ nanoparticles, and the yield was the highest (figure 4(b)) [17].

To sum up, it could be concluded that optimal preparation conditions for $\alpha$-Fe$_2$O$_3$ nanoparticles were hydrolysis temperature of 90 $^\circ$C, hydrolysis time of 8 h, and the Fe$^{3+}$ concentration of 0.05 M.

3.3. Characterization of $\alpha$-Fe$_2$O$_3$/Fe$_3$O$_4$ heteroplasmon nanoparticles

SEM, TEM, XRD, and VSM techniques were employed to represent the characteristic of $\alpha$-Fe$_2$O$_3$/Fe$_3$O$_4$ heteroplasmon nanoparticles calcined at 200 $^\circ$C for 2 h with absolute alcohol of 20 ml. As shown in figure 5(a), it could be seen that the prepared of $\alpha$-Fe$_2$O$_3$/Fe$_3$O$_4$ heterogeneous had a more uniform spherical structure with the average particle size of about 46 nm. Compared with $\alpha$-Fe$_2$O$_3$ nanoparticles, the average particle size of $\alpha$-Fe$_2$O$_3$/Fe$_3$O$_4$ heteroplasmon nanoparticles became smaller. The reason that the collapse of the internal pores of the nanoparticles during the calcination process, resulting that the specific surface area and surface energy of nanoparticles were decreased, they would reach a more stable condition. The pattern of $\alpha$-Fe$_2$O$_3$/Fe$_3$O$_4$ heteroplasmon was displayed in figure 5(b), indicated that the $\alpha$-Fe$_2$O$_3$/Fe$_3$O$_4$ revealed a more uniform spherical structure with the average size of about 46 nm, which was consistent with the SEM results. The HRTEM (figure 5(c)) was analyzed by means of digital micrograph software. Lattice stripes with lattice spacings of 0.29 nm and 0.18 nm could be detected in the diagram, which were attributed to the (220) crystal plane of Fe$_3$O$_4$ (JCPDS No. 19-0629) and (024) crystal plane of Fe$_2$O$_3$ (JCPDS No. 33-0664) [14], respectively. As shown in figure 5(d), the XRD pattern of the magnetic $\alpha$-Fe$_2$O$_3$/Fe$_3$O$_4$ heteroplasmon nanoparticles was similar to the Fe$_2$O$_3$ standard card (JCPDS No.33-0664), but the intensity of the diffraction peaks at 33.2°, 35.6°, 62.4°, and 63.9° were different from the standard card of hematite. This is because 35.5° and 62.7° caused by the diffraction peaks of magnetite (JCPDS No. 19-0629). Combining the conclusions of figures 5(c) and (d), the heteroplasmon nanoparticles were composed of Fe$_2$O$_3$ and Fe$_3$O$_4$ together. The hysteresis loop of the magnetic $\alpha$-Fe$_2$O$_3$/Fe$_3$O$_4$ heteroplasmon nanoparticles was illustrated in figure 5(e). It could be observed that $\alpha$-Fe$_2$O$_3$/Fe$_3$O$_4$ heteroplasmon nanoparticles exhibited soft magnetic properties, and the saturation magnetization was 54 emu g$^{-1}$.

3.4. Effects of preparation conditions on $\alpha$-Fe$_2$O$_3$/Fe$_3$O$_4$ heteroplasmon nanoparticles

3.4.1. Absolute alcohol consumption

The effect of absolute alcohol volume on the preparation of the magnetic $\alpha$-Fe$_2$O$_3$/Fe$_3$O$_4$ heteroplasmon nanoparticles calcined at 200 $^\circ$C for 2 h was shown in figure 6(a). The XRD pattern revealed that the diffraction peak intensity at 35.6° gradually reduced with the absolute alcohol volume increasing from 20 to 100 ml.
indicating that the Fe₃O₄ content in the heteroplasmon nanoparticles decreased. At the same time, with the decrease of the Fe₃O₄ content, the saturation magnetization of the heteroplasmon nanoparticles also gradually reduced, and the saturation magnetization was the largest of 51.4 emu g⁻¹ when the volume of absolute alcohol was 20 ml (figure 6(b)). With the increase of absolute alcohol volume, the dispersion of α-Fe₂O₃ nanoparticles in absolute alcohol increased, the area in contact with the atmosphere also increased, and the oxidation enhanced during the combustion process [26]. When the volume of absolute alcohol increased, the combustion and oxidation time were belonged, which caused the decreased of Fe₃O₄ content. Therefore, the optimal volume of absolute alcohol for α-Fe₂O₃/Fe₃O₄ heteroplasmon nanoparticles was 20 ml.

3.4.2. Calcination temperature
Figure 7 exhibited the XRD patterns and hysteresis loops of the magnetic α-Fe₂O₃/Fe₃O₄ heteroplasmon nanoparticles calcined at 200 °C–400 °C for 2 h with absolute ethanol of 20 ml. As shown in figure 7(a), it can be observed that the heteroplasmon nanoparticles had better crystallinity under various calcination temperatures. With the rise of the calcination temperature, the intensity of diffraction peak gradually increased [23, 24], but the ratio of Fe₃O₄ and Fe₂O₃ was decreased at 35.6°, which signified that the content of Fe₃O₄ was also decreased. The reason was that the oxidation level of Fe₂O₃ in α-Fe₂O₃/Fe₃O₄ heteroplasmon nanoparticles rose with the increase of the calcination temperature, resulting in the increase of Fe₂O₃ content and the reduce of the diffraction peak intensity d at 35.6°. By reason of the reduction of Fe₂O₃, figure 7(b) presented that saturation magnetization of the heteroplasmon nanoparticles monotonously decreased from 53.80 emu g⁻¹ at 200 °C to
39.24 emu g\(^{-1}\) at 400 °C with the increase of the calcination temperature \([27]\), and the saturation magnetization was largest at 200 °C.

### 3.4.3. Calcination time

The XRD patterns and hysteresis loops of magnetic \(\alpha\)-Fe\(_{2}\)O\(_3\)/Fe\(_3\)O\(_4\) heteroplasmon calcined at 200 °C for 0.5–4 h with absolute ethanol of 20 ml. As shown in figure 8(a), the crystallinities of magnetic \(\alpha\)-Fe\(_{2}\)O\(_3\)/Fe\(_3\)O\(_4\) heteroplasmon nanoparticles were better. With the extension of the calcination time, the intensity of diffraction peak increased and then decreased. The variation trend of the saturation magnetization of nanoparticles was consistent with that of the XRD patterns (figure 8(b)). In the time range (0.5–2 h), the saturation magnetization gradually rose with the extension of the calcination time \([28]\). However, the saturation magnetization decreased from 54.34 emu g\(^{-1}\) to 49.50 emu g\(^{-1}\) as the calcination time increased from 2 to 4 h. Which indicated that magnetic \(\alpha\)-Fe\(_{2}\)O\(_3\)/Fe\(_3\)O\(_4\) heteroplasmon nanoparticles had the highest saturation magnetization at 2 h. The main reason was that the absolute ethanol was not fully burned to produce carbon in the combustion process. During the calcination period, part of the carbon was incompletely oxidized to form carbonic oxide. Both carbon and carbonic oxide would reduce \(\alpha\)-Fe\(_2\)O\(_3\) nanoparticles to form Fe\(_3\)O\(_4\). When the calcination temperature was less than 2 h, the carbon was not completely oxidized and generated less carbon monoxide. Therefore, the reduction degree of \(\alpha\)-Fe\(_2\)O\(_3\) was slightly lower and the Fe\(_3\)O\(_4\) component was few, leading to the small saturation magnetization. When the calcination time was prolonged, the crystallinity of the heteroplasmon became better. The carbon was perfectly decomposed and \(\alpha\)-Fe\(_2\)O\(_3\) could not be further reduced when the calcination time exceeded 2 h. By this time, the Fe\(_3\)O\(_4\) was re-oxidized to \(\alpha\)-Fe\(_2\)O\(_3\) because of the longer calcination time, that resulted in the decreased of saturation magnetization of \(\alpha\)-Fe\(_2\)O\(_3\)/Fe\(_3\)O\(_4\) heteroplasmon nanoparticles \([27]\). Therefore, the calcination time of 2 h was the optimal preparation condition.

To sum up, the optimal preparation conditions for the magnetic heteroplasmon nanoparticles were 20 ml anhydrous ethanol, the calcination time of 2 h, and the calcination temperature of 200 °C.

### 4. Conclusions

This paper mainly introduced the optimization on preparation conditions of \(\alpha\)-Fe\(_2\)O\(_3\) nanoparticles and magnetic \(\alpha\)-Fe\(_2\)O\(_3\)/Fe\(_3\)O\(_4\) heteroplasmon nanoparticles, and characterized the above two types of nanoparticles. The results were as followed:

1. \(\alpha\)-Fe\(_2\)O\(_3\) nanoparticles were successfully prepared by the hydrolysis method, the obtained \(\alpha\)-Fe\(_2\)O\(_3\) has better dispersed spherical particles with the average particle size of around 49 nm and the saturation magnetization of 0.43 emu g\(^{-1}\) under the optimal preparation conditions: hydrolysis temperature of 90 °C, hydrolysis time of 8 h, and the Fe\(_{3+}\) concentration of 0.05 M.

2. Magnetic \(\alpha\)-Fe\(_2\)O\(_3\)/Fe\(_3\)O\(_4\) heteroplasmon nanoparticles were prepared via the rapid combustion-calcination method, and the optimal preparation conditions for the heteroplasmon nanoparticles were 20 ml anhydrous ethanol, the calcination time of 2 h, and the calcination temperature of 200 °C. The magnetic
α-Fe2O3/Fe3O4 heteroplasm nanoparticles were characteristics with uniform particle size and high dispersion, their average diameter was about 46 nm, and their saturation magnetization was 54 emu g−1 under the optimal preparation conditions. Compare with α-Fe2O3, α-Fe2O3/Fe3O4 heteroplasm nanoparticles were not only chemically stable, but also possess controllable magnetic property. Therefore, the discovery of the hydrolysis-combustion-calcination process for the preparation of magnetic α-Fe2O3/Fe3O4 heteroplasmon nanoparticles would greatly gratify their application needs [29].

Data availability statement
All data that support the findings of this study are included within the article (and any supplementary files).

Declaration of interests
The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

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