

Review Article

Review on the Synthesis and Applications of Fe_3O_4 Nanomaterials

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Recently, Fe_3O_4 nanomaterials have attracted tremendous attention because of their favorable electric and magnetic properties. Fe_3O_4 nanostructures with various morphologies have been successfully synthesized and have been used in many fields such as lithium-ion batteries (LIBs), wastewater treatment, and magnetic resonance imaging (MRI) contrast agents. In this paper, we provide an in-depth discussion of recent development of Fe_3O_4 nanomaterials, including their effective synthetic methods and potential applications.

1. Introduction

Nanomaterials have been attracting great attention owing to their excellent electrical, optical, magnetic, and catalytic properties. It is well known that the phases, sizes, and morphologies of nanomaterials have great influence on their properties and potential applications; thereby, the controlled synthesis of nanostructured materials with novel morphologies has recently received much attention [1–3]. As a kind of conventional magnetic material, Fe_3O_4 nanomaterials have been used in many fields because of their unique electric and magnetic properties [4, 5]. Several novel and effective methods have been developed to synthesize Fe_3O_4 nanomaterials with various shapes, such as nanorods, nanotubes, and hierarchical superstructures [6–10]. Fe_3O_4 nanomaterials have superior properties and great potential applications in the fields of lithium-ion batteries, wastewater treatment, and drug delivery [11–15].

Until now, several overviews of the literature on the Fe_3O_4 nanomaterials have been reported to keep the readers abreast of the rapid development. For example, a review by Yang's group has focused on the synthesis, growth mechanism, and applications of Fe_3O_4 nanomaterials [16]. Nevertheless, many successes on the synthesis, properties, and applications

of Fe_3O_4 nanomaterials have been continually reported in the last few years; thereby, it seems timely to review the development of Fe_3O_4 nanostructures.

Herein, we provide an update on currently available methods for the synthesis of Fe_3O_4 nanomaterials with various morphologies; in spite of that, some important and original findings reported earlier are also included. The unique properties, potential applications, and future prospects of Fe_3O_4 nanostructures have also been discussed.

2. Synthesis of Fe_3O_4 Nanomaterials

Generally, the intrinsic shape of nanocrystal is dominated by the crystalline structure of initial seed, and the final shape is governed by the subsequent growth stage through delicate control of external factors (e.g., kinetic energy barrier and templates) [17]. Fe_3O_4 has a cubic inverse spinel structure based on $Fd-3m$ space group [18]. The lattice constant is $a = 0.839$. In the unit cell, as shown in Figure 1, the oxygen ions form an fcc closed packing, and the iron ions occupy interstitial tetrahedral sites and octahedral sites, symbolized as $[\text{Fe}^{3+}]_A-[\text{Fe}^{2+}\text{Fe}^{3+}]_B\text{O}_4$, in which A (tetrahedral positions) is occupied by Fe^{3+} ions and B (octahedral sites) is occupied by eight Fe^{2+} ions and eight Fe^{3+} ions.

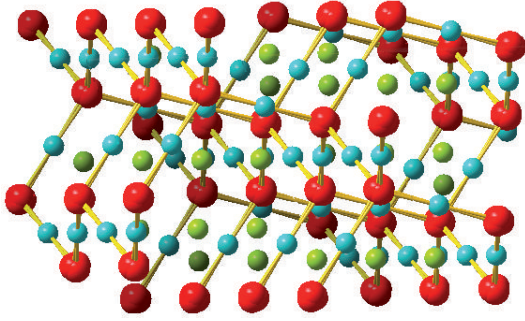


FIGURE 1: Crystal structure of cubic inverse spinel Fe_3O_4 ((green ball) Fe^{3+} (blue ball) Fe^{2+} (red ball) O).

2.1. 0D Fe_3O_4 Nanomaterials. 0D Fe_3O_4 nanomaterials have been widely studied due to their current and promising applications. It is known that the physicochemical properties and potential applications of Fe_3O_4 nanomaterials are strongly influenced by their sizes; moreover, Fe_3O_4 nanomaterials tend to aggregate because of the strong magnetic dipole-dipole attractions between the crystals and the large surface energy [24]. Thus, many efforts have been devoted to prepare Fe_3O_4 nanomaterials with controlled size and well-defined surface property [25].

After the Sugimoto group have fabricated monodisperse Fe_3O_4 nanoparticles in 1980, various methods have been developed for the synthesis of Fe_3O_4 nanoparticles with narrow size distribution and good dispersity. Sun and Zeng have synthesized 4 nm Fe_3O_4 nanoparticles *via* the high-temperature reaction of $\text{Fe}(\text{acac})_3$ in the phenyl ether with 1,2-hexadecanediol, oleic acid, and oleylamine. “Seed-mediated” growth method is used to make larger nanoparticles, and Fe_3O_4 nanoparticles (3–20 nm) can be obtained by changing the quantity of seeds [26]. The Gao’s group have used a solvothermal method to synthesize Fe_3O_4 nanoparticles with a mean diameter of 25 nm [27]. In the synthesis, $[\text{Zn}(\text{CO}_3)_2(\text{OH})_6]$, accepting Fe^{2+} precipitates through $-\text{OH}$, can prevent the agglomeration of Fe^{2+} precipitates, and superparamagnetic Fe_3O_4 nanoparticles can be sequentially obtained. Compared to other successes [11, 28], the as-synthesized Fe_3O_4 nanoparticles with good dispersity have not been coated by other substances (e.g., silica and polymer) and can keep their naturally properties. Other routes, including coprecipitation method, reverse micelle method, and high temperature liquid phase method, have also been explored to fabricate Fe_3O_4 nanoparticles with different diameters [29–32].

Besides spherical nanoparticles, 0D Fe_3O_4 nanomaterials with other morphologies have been prepared, such as octahedron [19, 33, 34], dodecahedron [6], and cube [35–37]. Based on the literature [38], the shape of the particle is closely related to the crystallographic surfaces that enclose the particle. As to Fe_3O_4 , the relative surface energies are in the order of $\gamma_{111} < \gamma_{100} < \gamma_{110}$ owing to the distances between these three faces and coordination number with neighboring atoms [39, 40]. Therefore, the growth rate of (111) plane is quicker than that of other planes, and the octahedral shapes would

be the thermodynamically favored morphology according to the Wulff theorem. For example, Zhang et al. have presented a simple method for octahedral Fe_3O_4 nanoparticles, in which tetracosane is the reaction media, oleylamine is the surfactant and the reducing agent, and $\text{Fe}(\text{OA})_3$ is the precursor [19]. As shown in Figure 2(a), the octahedrons have a size of 21 ± 2 nm. The octahedrons can also self-assemble into oriented superstructures due to their anisotropic shapes (Figure 2(b)). As discussed above, the (110) facet has the highest surface energy, so there is much difficulty to fabricate magnetite nanocrystals enclosed by (110) plane. Nevertheless, Li and coworkers have prepared rhombic dodecahedral (RD) Fe_3O_4 nanocrystals *via* a microwave-assisted route in the presence of ionic liquid (IL) $[\text{C}_{12}\text{Py}]^+[\text{ClO}_4]^-$ [6]. In the synthesis, ILs change the surface condition of the Fe_3O_4 nanocrystals, and HMT/phenol adsorbed on (110) planes is beneficial for the crystal growth along [100] direction; thus, RD Fe_3O_4 nanocrystals enclosed by twelve (110) flakes can be obtained. Moreover, if the nuclei are bounded by (100) planes, cubic Fe_3O_4 nanomaterials can be formed [36].

2.2. 1D and 2D Fe_3O_4 Nanomaterials. Although it is difficult to prepare anisotropic Fe_3O_4 nanocrystal because of its cubic spinel structure, anisotropic nanocrystals can be obtained by using templates or surfactants to control the growth rate on different crystal planes. More recently, 1D magnetic nanomaterials, such as nanotubes, nanorods, and nanowires, have become a pressing need for their potential applications in lithium-ion batteries and field emission displays [41]. Particularly, tubular Fe_3O_4 nanostructures have stimulated extensive efforts owing to their well-defined magnetic states. It was reported that some conventional methods (i.e., template-assisted method) are not beneficial for the formation of single-crystalline nanotubes; thereby, some novel methods have been explored [9, 42–44]. Geng et al. have applied proteins from egg albumin as nanoreactors for the fabrication of single-crystalline Fe_3O_4 nanotubes. A flake structure is prepared with the assistance of egg albumin, and then Fe_3O_4 nanotubes are prepared from the flake-like precursors based on a “rolling-up” mechanism. Additionally, nanorods and nanowires have been successfully synthesized [43, 45–48]. For example, Zhang and coworkers have prepared single-crystalline Fe_3O_4 nanowires with large aspect ratio by a one-step sol-gel process; Wang et al. have reduced $\alpha\text{-Fe}_2\text{O}_3$ nanowires to Fe_3O_4 nanowires under H_2 and Ar_2 at 400–900°C *via* a V-S process; chemical vapor deposition (CVD) method has also been used to synthesize 1D Fe_3O_4 nanomaterials.

2D Fe_3O_4 nanomaterials, such as nanorings and nanoflakes, have also attracted much attention to their special properties. Jia et al. have synthesized Fe_3O_4 nanorings by the reduction of hematite in the presence of phosphate and sulfate [49]. In the synthesis, compared with (001) plane, PO_4^{3-} and SO_4^{2-} have stronger adhesion to the (110) and (100) planes; therefore, the capsule crystals have a tendency to grow along the [001] direction and the following dissolution process also takes place along the [001] direction. Finally, Fe_3O_4 nanorings can be formed. Zhu group have prepared Fe_3O_4 nanosheets

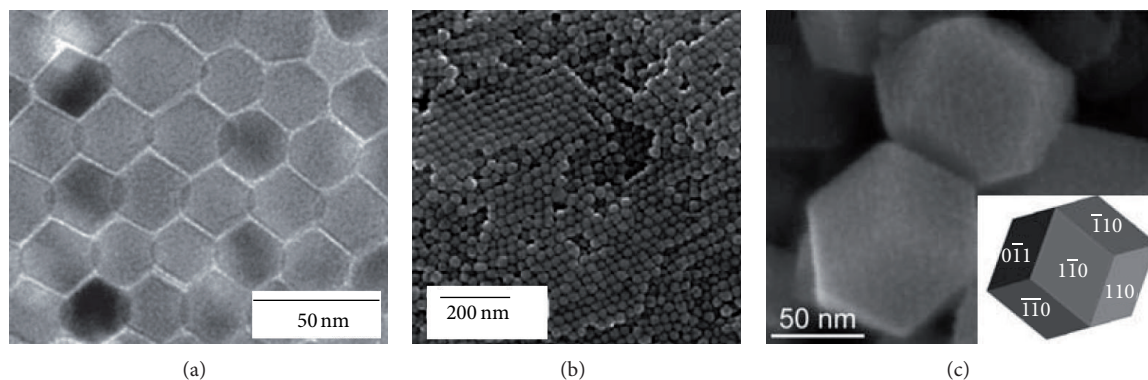


FIGURE 2: (a) TEM image of self-assembled monolayer Fe_3O_4 nanocrystals; (b) SEM image of assembled multilayer Fe_3O_4 nanocrystals; (c) SEM image of RD Fe_3O_4 nanocrystals; the inset is model of RD enclosed by twelve (110) planes [6, 19].

by oxidizing Fe substrates in acidic solution in a hot plate at 70°C [50]. Fe_3O_4 nanoplates have been synthesized by reducing $\gamma\text{-Fe}_2\text{O}_3$ nanoplates in the presence of PVP. In the experiment, PVP can selectively coordinate with (111) facet of $\gamma\text{-Fe}_2\text{O}_3$, which reduces the growth rate along the [111] direction, resulting in nanoplates bounded by the (111) planes. Finally, $\gamma\text{-Fe}_2\text{O}_3$ nanoplates could be transformed to Fe_3O_4 with the shape and size being unchanged. Other strategies (i.e., hydrothermal and solvothermal methods) have been developed to fabricate Fe_3O_4 nanoprisms [15, 51, 52].

2.3. Fe_3O_4 Hierarchical Superstructures. Recently, many research efforts in nanoscience have been devoted to the self-assembly of nanoscale building blocks into 2D and 3D hierarchical superstructures, which could prevent the agglomeration of nanomaterials and supply more tunable and unique properties [53]. In addition, the “superparamagnetic limit,” that is, the conflict between reducing the magnetic energy barrier and decreasing the size, restrains the development of Fe_3O_4 nanomaterials [9]. To some extent, Fe_3O_4 hierarchical superstructures could overcome this limit, and some researchers concerned about the synthesis of self-assembled Fe_3O_4 superstructures.

As shown in Figure 3(a), 1D chainlike arrays of hollow Fe_3O_4 nanospheres have been prepared by aging preassembled Fe nanoparticles in aqueous solution [20]. The formation mechanism is proposed based on the nanoscale Kirkendall effect: Fe nanoparticles are firstly self-assembled into chainlike structure and solid Fe spheres are then gradually oxidized into Fe_3O_4 hollow nanospheres. Other self-assembled Fe_3O_4 chains have also been synthesized [10, 54].

The Wang group have successfully synthesized a hierarchical and porous structure of Fe_3O_4 hollow submicrospheres with Fe_3O_4 nanoparticles *via* a solvothermal method [21]. As displayed in Figure 3(b), Fe_3O_4 submicrospheres are built from Fe_3O_4 nanoparticles with diameters of 20–30 nm. The formation mechanism can be attributed to reduction and Ostwald ripening: Fe_2O_3 submicrospheres are firstly synthesized; hematite is then reduced to magnetite and an incomplete layer consisted of Fe_3O_4 nuclei is formed on the solid Fe_2O_3 surfaces; a $\text{Fe}_2\text{O}_3\text{-Fe}_3\text{O}_4$ core-shell structure is

formed in the presence of 1,6-hexanediol; finally, Fe_3O_4 hollow submicrospheres are obtained through Ostwald ripening process.

Hierarchical flower-like Fe_3O_4 superstructures have been widely researched [22, 55–57]. Zhong et al. have reported the synthesis of flower-like Fe_3O_4 superstructures by an ethylene-glycol-(EG)-mediated self-assembly process. Han and coworkers have prepared flower-like Fe_3O_4 under 80°C in the absent of any surfactant or organic solvent (Figure 3(c)). Ultrasound-assisted hydrothermal route has also been used to fabricate Fe_3O_4 hierarchical flower-like microspheres.

Many other Fe_3O_4 hierarchical superstructures with special morphologies have been synthesized. For example, Fe_3O_4 microspheres assembled by tetrahedral nanocrystals [58], porous hollow Fe_3O_4 beads constructed with rod-like nanoparticles [59], and nanoparticles-assembled Fe_3O_4 dendritic patterns [60].

3. Potential Applications of Fe_3O_4 Nanomaterials

3.1. Lithium-Ion Batteries (LIBs). Lithium-ion batteries are regarded as the most promising rechargeable energy storage technology due to the increasing applications of portable electronic devices and transportations. In order to obtain high power and energy density, Fe_3O_4 nanomaterials have been extensively explored as LIB anode materials for their high theoretical capacity ($900\text{--}1000\text{ mA}\cdot\text{h}\cdot\text{g}^{-1}$), low cost, environmental benignity, and special properties [59, 61]. For example, single-crystalline mesoporous Fe_3O_4 nanorod exhibits a high reversible capacity of $843.5\text{ mA}\cdot\text{h}\cdot\text{g}^{-1}$ after 50th cycle at 0.1 C; furthermore, the nanorods have superior electron transport ability, which makes them highly attractive for the potential application as LIB anode materials [46]. However, the high surface area of nanomaterials may cause secondary reactions such as electrolyte decomposition between electrode and electrolyte and form thick solid electrolyte interphase (SEI) films on the electrode surface [62]. Fortunately, it was found that surface modifications could partly solve these problems [23]. Carbon-coated Fe_3O_4

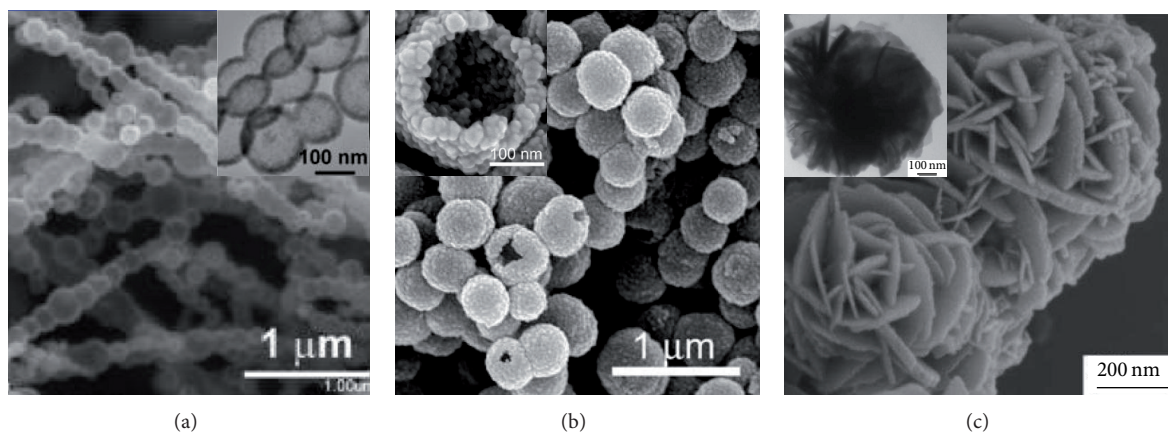


FIGURE 3: (a) TEM images of the chainlike arrays of Fe_3O_4 hollow nanospheres; (b) SEM image of the hierarchical porous Fe_3O_4 hollow submicrospheres; (c) SEM and TEM images of the flower-like Fe_3O_4 superstructures [20–22].

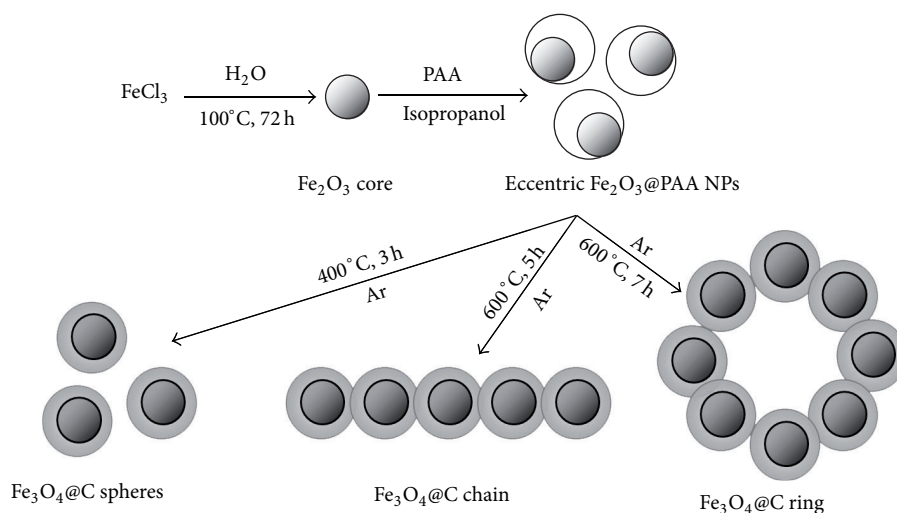


FIGURE 4: Schematic illustration of the synthesis of $\text{Fe}_3\text{O}_4/\text{C}$ core-shell spheres, chains, and rings [23].

nanospindles can increase the electronic conductivity of electrodes leading to thin and uniform SEI films, but also stabilize the obtained SEI films; thereafter, the $\text{Fe}_3\text{O}_4\text{-C}$ composites are excellent anode materials for highly efficient LIBs with high reversible capacity, high rate capability, and enhanced cycling performance. Li group have reported monodisperse $\text{Fe}_3\text{O}_4/\text{C}$ core-shell spheres, chains, and rings with tunable magnetic properties based on structural evolution from eccentric $\text{Fe}_2\text{O}_3/\text{poly}(\text{acrylic acid})$ core-shell nanoparticles [63]. The possible formation mechanism is shown in Figure 4. Compared with the $\text{Fe}_3\text{O}_4/\text{C}$ core-shell spheres, the chains and rings exhibit higher reversible capacity and better cycling stability. Several other ways have been used to form $\text{Fe}_3\text{O}_4\text{-C}$ composites [30, 64, 65]. For instance, porous carbons or mixing graphene layers are impregnated with Fe_3O_4 precursor; Fe_3O_4 NPs and carbon are simultaneously formed from a precursor with high surface area and porosity.

3.2. Wastewater Treatment. In recent years, wastewater treatment has attracted considerable attention because clean water is vital to the human and because of a variety of key industries [66]. The development of nanoscience opens a novel and effective way for the wastewater treatment. Many groups have used Fe_3O_4 nanomaterials to treat heavy metal ions and organic pollution. Nanostructured Fe_3O_4 microspheres (NFMSs) with a large specific surface area ($135.9 \text{ m}^2 \cdot \text{g}^{-1}$) can remove toxic Cr^{6+} from polluted water, and it is found that 1 g NFMSs remove 43.48 mg Cr^{6+} ions at room temperature [67]. Fe_3O_4 nanomaterials have also been successfully used as catalysts to remove organic pollutions, such as xylene orange, phenol, and aniline, from wastewater [68–70].

3.3. Other Applications. Fe_3O_4 nanomaterials have been applied in many other fields, including metal chemosensor [71], magnetorheological elastomer [72], SERS spectroscopy

[57], magnetic resonance contrast agent [73], catalyst [74, 75], drug delivery [76], and magnetic resonance imaging (MRI) contrast agents [77].

4. Conclusions

In conclusions, recent synthetic efforts have led to the formation of Fe_3O_4 nanomaterials with various morphologies. In spite of the exciting new development, the application of Fe_3O_4 nanomaterials in industry is still in its infancy. However, with the progress in the fundamental understanding of the physics and chemistry in the Fe_3O_4 nanomaterials, we foresee that novel properties and applications will be demonstrated in the not-so-distant future.

Acknowledgments

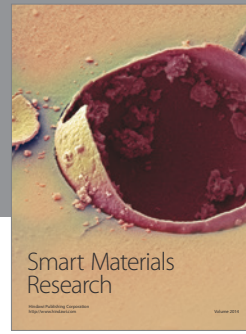
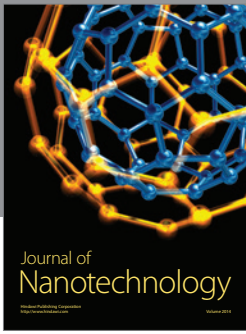
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