Microwave-Assisted Fabrication of $\gamma$-Fe$_2$O$_3$ Nanoparticles from Tris (acetylacetonato) Iron (III)

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Maghemite ($\gamma$-Fe$_2$O$_3$) nanoparticles which have a wide range of applications were fabricated by microwave heating of acetylacetonato iron (III) precursor. The precursor is very easy to make, and on irradiation by microwave decomposes to maghemite nanoparticles. The as-prepared $\gamma$-Fe$_2$O$_3$ nanostructured has been characterized by X-ray powder diffraction, energy dispersive X-ray spectroscopy (EDX), scanning electron microscope (SEM) and transmission electron microscopy (TEM) techniques. The maghemite nanoparticles were obtained in high yield and purity and the average size of the particles was estimated to be about 13 nm.

Keywords: Microwave-assisted, Nanoparticles, $\gamma$-Fe$_2$O$_3$, SEM, TEM, Maghemite.

Introduction

The development of reliable synthesis protocols for nanostructured materials over a range of chemical compositions, shapes and sizes is an important area of research in nanotechnology. Over the past few years, controlling the size and shape of inorganic crystals have attracted significant interest due to the fact that the shape and size of materials have much influence on their chemical and physical properties [1-6]. Among the various nano-sized inorganic materials, the preparation of magnetic nanomaterials is of a great importance for their potential applications in information storage [7], color imaging [8], magnetic refrigeration [9], bioprocessing [10], gas sensors [11,12], ferrofluids [13,14] and so on. The properties and applications of magnetic iron oxide nanoparticles have been recently reviewed by Jeja and Koh [15].

Maghemite ($\gamma$-Fe$_2$O$_3$), is one of the magnetic materials which has been widely used for the longest period of time due to its excellent properties. $\gamma$-Fe$_2$O$_3$ nanoparticles are ferromagnetic and characterized by the superparamagnetic relaxation phenomenon which is strongly affected by particle size, shape and various surface effects [16]. The interesting magnetic properties of nanostructured maghemite are due to finite size effects and/or high surface/volume ratios. Therefore, the study of the interplay between microstructure and magnetism of this iron oxide is becoming immensely interesting. Although there are many kinds of interesting magnetic nanoparticles such as iron, cobalt or ferrites, most attention in the field of biomedical applications is focused on iron oxide particles-magnetite (Fe$_3$O$_4$) and its oxidized form maghemite ($\gamma$-Fe$_2$O$_3$), because of their chemical stability and biological compatibility. These facts make this kind of nanoparticles one of the most studied materials for biomedical applications. Some examples of biomedical applications in which magnetic nanoparticles with special characteristics are needed include magnetic separation [17], drug delivery [18,19], hyperthermia and MRI contrast enhancement [20,21]. Advancement in the use of magnetic particles for biomedical and other applications depends on the new synthetic methods.
with better control of the size distribution, magnetic properties and the particle surface characteristics.

Recently, several methods for the preparation of \(\gamma\)-Fe\(_2\)O\(_3\) nanoparticles have been developed. Chemical precipitation is one of the oldest and simple techniques for the synthesis of nanoparticles [22,23]. In precipitation processes, the metal precursors (e.g. FeCl\(_3\) or FeCl\(_2\)) are dissolved in a solvent and a precipitating agent (e.g. NH\(_4\)OH) is added to form nanoparticles.

The co-precipitation technique is probably the simplest and most efficient chemical pathway to obtain magnetic particles. The main advantage of the co-precipitation process is that a large amount of nanoparticles can be synthesized [24]. However, this process is difficult to control and yields nanoparticles with a broad size distribution and irregular morphology. Thermolysis in the presence of capping agents, on the other hand, offers monodisperse nanoparticles with good crystallinity, but these methods operate at higher temperature and require toxic and expensive precursors [25,26]. Other methods for fabrication of \(\gamma\)-Fe\(_2\)O\(_3\) nanoparticles, e.g. microemulsions [27], sol–gel synthesis [28], sonochemical reactions [29], etc. offer no better job and the preparation process in most of them is cumbersome.

The microwave-assisted route is yet another method for the synthesis of metal oxides and has been gaining significance in the synthesis of oxide nanomaterials. Comparing with conventional process, one can find that microwave irradiation synthesis is generally quite faster, simpler and more energy efficient. Recently, microwave has been introduced in the preparation of nanoparticles [30-33]. Under microwave irradiation, reactants can be uniformly heated in short time, and precipitates are generated simultaneously and uniformly dispersed throughout the solution, which results in obtaining homogeneous nanoparticles. Hence, in the current work we adapted microwave-assisted route to prepare \(\gamma\)-Fe\(_2\)O\(_3\) nanoparticles using tris (acetylacetonato) ferrate (III) as precursor. The as-synthesized maghemite nanoparticles were characterized by employing X-ray powder diffraction (XRD), energy dispersive X-ray spectroscopy (EDX), scanning electron microscope (SEM) and transmission electron microscopy (TEM) techniques.

**Experimental**

**Materials and apparatus**

All chemicals, including FeCl\(_3\).6H\(_2\)O, acetylacetone and polyethylene glycol (PEG-200) were obtained from Merck and used as received. The XRD patterns were obtained on a Philips Analytical X-ray diffractometer, using CuK\(_\alpha\) radiation (\(l=1.54056\) Å ), with a flat sample holder mounted on a PW1830 spectrogonimeter. The EDS measurements were carried out on a LEO 1455 VP energy dispersive spectrometer. The transmission electron microscopy (TEM) analysis was performed using a LEO 906 E microscope. Scanning electron microscopy (SEM) analysis was carried out in order to investigate the microstructure and morphology of the sample, using LEO 1455 VP microscope.

**Synthesis of \(\gamma\)-Fe\(_2\)O\(_3\) nanoparticles**

The tris (acetylacetonato) ferrate (III), [Fe(acac)\(_3\)], precursor was prepared according to the procedure reported in the literature [34]. In a typical procedure, 3 mmol of the precursor complex was dissolved in 5 mL of ethanol in 100 mL glass beaker. To this solution, 10 mL of PEG-200 and 0.05 mol of NaCl (dissolved in the least amount of water) were added. The reaction vessel was placed on the spot zone in a domestic microwave oven operated at 2.45 GHz. Microwave irradiation was carried out at the electric power levels of 1000 W for 5 m. The beaker was then taken out of the microwave oven. After it was cooled to room temperature 20 mL of water was added to the produced precipitates. The precipitates were separated by centrifugation, washed with distilled water until complete removal of sodium chloride and PEG and then dried at 100 °C in an oven for 3 h to afford \(\gamma\)-Fe\(_2\)O\(_3\) in almost quantitative yield.

**Results and discussion**

Most of the acetylactonato complexes of transition metals, especially those of Fe (III), are rather labile and can be readily decomposed by thermal or microwave heating. The decomposition...
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of complex [Fe (acac)$_3$] by microwave radiation in PEG, as particles size controller, and sodium chloride, as microwave absorber, lead to the production of $\gamma$-Fe$_2$O$_3$ nanoparticles. PEG is one of the polymers with major interest in the synthesis of nanoparticles because it is low cost, nontoxic, non-inflammable and easy to handle. It has been reported that PEG with uniform and ordered chain structure is easily absorbed at the surface of metal oxide colloid [35]. When the surface of the colloid adsorbs PEG, the colloidal activities will greatly decrease and the growth rate of the colloids in some certain facet will be confined [36].

Fig. 1. XRD pattern of as-prepared $\gamma$-Fe$_2$O$_3$

Fig. 2. TEM image of as-prepared $\gamma$-Fe$_2$O$_3$

Fig. 3. SEM image of as-prepared $\gamma$-Fe$_2$O$_3$ nanoparticles

The TEM image of the as-prepared $\gamma$-Fe$_2$O is displayed in Fig. 2. As shown in Fig. 2, $\gamma$-Fe$_2$O$_3$ nanoparticles are nearly spherical and have an average diameter of about 13 nm.

Scanning electron microscopy was used for further analysis of the samples morphology and Energy Dispersive X-ray Analysis (EDS). The SEM image of as-prepared $\gamma$-Fe$_2$O$_3$ (Fig. 3) indicates the presence of agglomeration for these nanoparticles. As shown in Fig. 4, the EDS spectrum shows only iron and oxygen elements in 2:3 ratio and the sample is almost pure Fe$_2$O$_3$.

Conclusion

In summary, the procedure we introduced is an easy method for the fabrication of pure and high yield $\gamma$-Fe$_2$O$_3$ nanoparticles. The materials used in the synthesis are inexpensive and there is no need for controlling the reaction conditions and heat treatments. Since microwave irradiation was used in this method, a sample of $\gamma$-Fe$_2$O$_3$ can be prepared in only 10 m time period. The as-prepared $\gamma$-Fe$_2$O$_3$ nanoparticles have been characterized by XRD, TEM, SEM and EDX techniques.
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Fig. 4. EDS spectrum of $\gamma$-Fe$_2$O$_3$ nanoparticles

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