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RESEARCH ARTICLE

STRUCTURAL, MORPHOLOGICAL, OPTICAL AND MAGNETIC PROPERTIES OF Fe_3O_4 NANOPARTICLES PREPARED BY MICROWAVE COMBUSTION METHOD

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ABSTRACT

Fe_3O_4 nanoparticles were synthesized by microwave combustion method by using thiourea as stabilizer. The synthesized Fe_3O_4 nanoparticles were characterized by powder XRD, HR-SEM, HR-TEM, EDX, PL and VSM studies. XRD results showed the well-crystalline cubic spinel structure of synthesized Fe_3O_4 nanoparticles with size of 49 nm. The morphology of Fe_3O_4 nanoparticles was confirmed by HR-SEM and HR-TEM images. In room temperature PL studies exhibited a strong UV emission and a suppressed green emission indicated the good crystalline properties for Fe_3O_4 nanoparticles. The magnetic behaviors reveal that the modification can decrease the saturation magnetization of Fe_3O_4 nanoparticles due to the surface effect.

INTRODUCTION

Compared to atomic or bulky counterparts, nano-sized materials owe superior physical and chemical properties due to their mesoscopic effect, small object effect, quantum size effect and surface effect. Recently, Fe_3O_4 nanoparticles (NPs) have been intensively investigated because of their super paramagnety, high coercivity and low Curie temperature (Kim and Kim, 2003; Raj and Moskowitz, 2002; Beydoun *et al*., 2000; McMichael *et al*., 1992). Fe_3O_4 NPs are used as sensors, antibacterial agents and many magnetic applications. In addition to these characters, Fe_3O_4 NPs are also non-toxic and biocompatible. Therefore, Fe_3O_4 NPs have brought out some new kinds of biomedical applications such as dynamic sealing (Shen *et al*., 1999), biosensors (Jordan *et al*., 1999), contrasting agent in magnetic resonance (MR) imaging (Cao *et al*., 2004), localizer in therapeutic hyperthermia (Jordan *et al*., 1999) and magnetic targeted-drug delivery system (Li *et al*., 2005), etc. It is well known that it is very important to ensure the narrow size distribution, good dispersion and high magnetic response of Fe_3O_4 NPs in tissue fluid for applications. Several techniques have been used to prepare the Fe_3O_4 NPs which comprise chemical precipitation, sol-gel, hydrothermal, surfactant mediated, precipitation, microwave-assisted and hydrothermal technique. Compared with other methods, the microwave combustion method is a valuable technique and has several advantages for the preparation of Fe_3O_4 NPs (Arul Mary *et al*., 2014). This method gives good homogeneity and high purity of Fe_3O_4 NPs. In this present work, we synthesized Fe_3O_4 NPs by microwave combustion method and characterized by using various analytical instruments.

MATERIALS AND METHODS

Preparation of Fe_3O_4 nanoparticles

All the reagents used were of analar grade obtained from Merck, India and were used without further purification. Fe_3O_4 NPs synthesized by rapid microwave combustion method. $Fe(NO_3)_2$ and thiourea in 1:2 ratio was slowly dissolved in 20 ml distilled water with constant stirring. The resulting suspension was placed in a domestic microwave oven and exposed to the microwave energy in a 2.45 GHz multimode cavity at 850 W for 10 min. Initially, the precursor mixture boiled and underwent evaporation followed by the decomposition with the evolution of gases. When the solution reached the point of spontaneous combustion, it vaporized and instantly became a solid. The powder obtained from the microwave oven was labeled as Fe_3O_4 NPs.

Characterization of Fe_3O_4 nanoparticles

The X-ray diffraction (XRD, Thermo ARL SCINTAG X'TRA with CuKa irradiation, $\lambda = 0.154056$ nm) was used to analyze

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the crystalline size and purity of the samples. The composition of the product was analyzed by energy dispersive X-ray detector (EDS, Thermo Noran VANTAG-ESI). The morphologies were characterized using scanning electron microscopy (SEM, Hitachi S-4700, 25 kV) and transmission electron microscopy (TEM, JEM200CX, 120 kV). The composition of the product was analyzed by energy dispersive X-ray detector (EDS, Thermo Noran VANTAG-ESI). BET surface areas were measured by nitrogen adsorption at 77K using a Micromeritics ASAP 2000 surface area analyzer. Photoluminescence spectrum of the samples was recorded on Edinburgh FL/FS TCSPC 920 fluorescence spectrometer. Magnetic measurements were carried out at room temperature using a PMC MicroMag 3900 model vibrating sample magnetometer (VSM) equipped with 1 T magnet.

RESULTS AND DISCUSSION

XRD measurement was used to identify the crystalline structure of the products. As shown in Fig. 1, the XRD peaks can match well with the characteristic peaks of inverse cubic spinel structure (JCPDS 19-0629), which indicate that the crystalline structure of Fe_3O_4 NPs can be remained after the surface modification with thiourea. The average crystallite size was calculated using the Debye–Scherrer equation $d = K\lambda/(\beta\cos\theta)$ are about 49 nm. The morphologies of the products were investigated by HR-SEM. Surface morphology of the prepared Fe_3O_4 NPs was shown in Fig. 2 and consists of nanoparticles in high quantity, homogeneousness and grains were distributed uniformly. The obtained products exhibited a cubic spinel shape randomly adsorbed on the surface of the nanoparticles. From SEM images of Fe_3O_4 NPs, we can observe the average particle sizes of Fe_3O_4 NPs was 49 nm. The morphology and distribution of these samples were further characterized using TEM as shown in Fig. 3. All the Fe_3O_4 NPs show homogeneously cubic shape with diameter about 49 nm, which is in agreement with the results of the XRD analysis. The HR-SEM and HR-TEM images further prove the synthesized Fe_3O_4 NPs have a magnetite structure. Photoluminescence spectrum is used to determine the defect-related transitions and oxygen vacancy. The density of defects and surface states may change the formation conditions, morphology, and size of the crystallites.

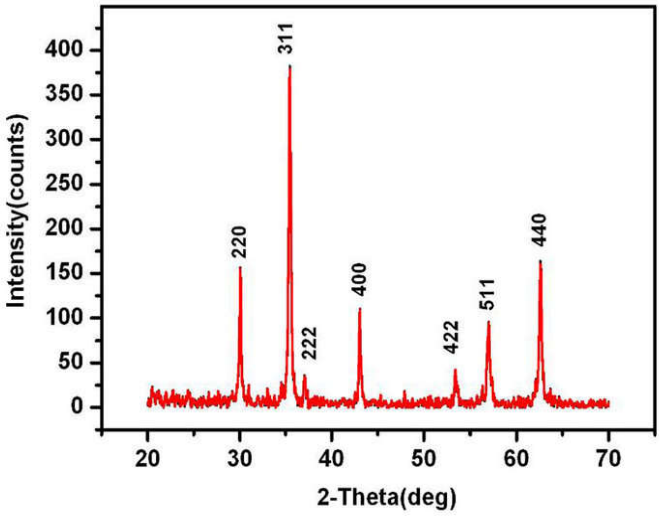

Figure. 1. XRD patterns of the Fe_3O_4 nanoparticles

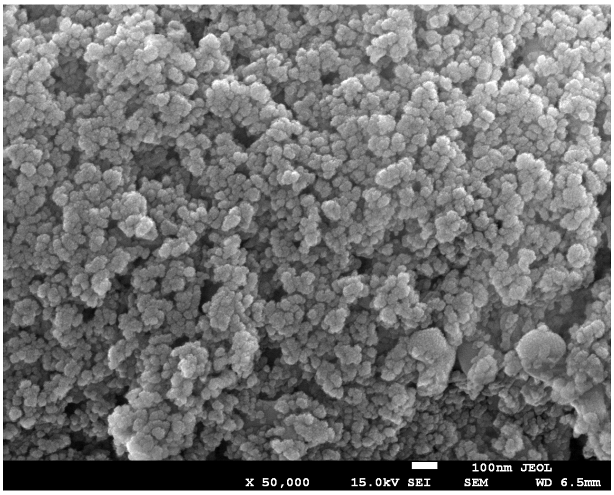

Figure 2. SEM images of the Fe_3O_4 nanoparticles

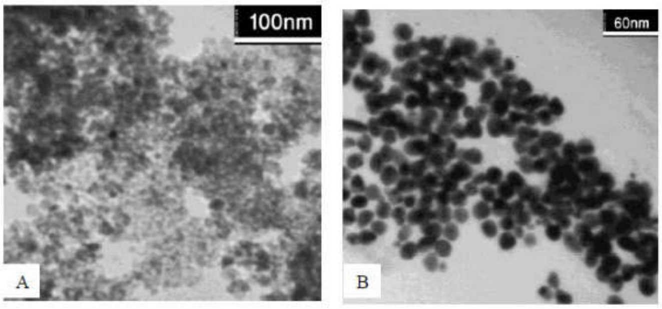

Figure 3. TEM images of the Fe_3O_4 nanoparticles

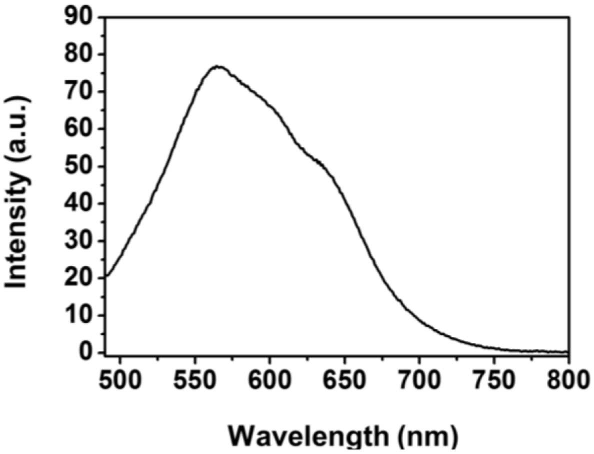

Figure 4. PL spectra of the Fe_3O_4 nanoparticles

The photoluminescence spectra were recorded for two reasons: the quantum size effect and structural defects in the crystals (Ragupathi *et al*., 2014; Van Dijken *et al*., 2000). In the present study, the PL results showed that the synthesized Fe_3O_4 nanoparticles are semiconductors with estimated oxygen vacancy. In order to study the feature of electron–hole pairs on the surface of photoluminescence spectra (PL) of as synthesized Fe_3O_4 Nps were illustrated in Fig. 4. The PL spectra in Fe_3O_4 NPs consisted of a sharp UV emission peak and one broad peak in the visible region. The sharp UV emission band generally assigned as a near band edge emission band and the position agrees well with the literature (Ragupathi *et al*., 2014; Van Dijken *et al*., 2000). The broad emission in the visible-light region is associated to defect surface detects, in which oxygen vacancies are the most accepted defects. Changes in the green emission intensity have been associated to changes in the concentration of the oxygen vacancies reported in the literature. In our study, by using Gaussian Fitting the visible emission in spectra can be fitted in to green emission and yellow emission. The great advantage of using Fe_3O_4 NPs in applications was that they can be operated by an external magnetic field. Consequently, in water decontamination, one could use the Fe_3O_4 NPs with high adsorption efficiency with the aptitude of recuperating them using magnetic separation from water as a simple, quick, and cheap method. For this purpose, the magnetic behavior of the prepared samples was studied. The lower saturation magnetization might be due to the crystal anisotropy and S-shaped anisotropy of the obtained samples. Fe_3O_4 NPs consisted of the domains with different spin orders; the magnetic field position in its direction depends on its strength. Increasing the magnetic field in case of ferri, ferromagnets (FM), leads to the saturation magnetization. In reduced dimensions (nanoscale), magnetic nanoparticles changed from multi- to single domain when they reach a crystal size less than the critical one.

Conclusion

Fe_3O_4 NPs was successfully prepared by microwave combustion method by using thiourea as stabilizer. The structure, morphology, and optical and magnetic properties of the prepared Fe_3O_4 NPs were investigated by XRD, HR-SEM, TEM, PL, and VSM respectively. The synthesized Fe_3O_4 NPs had size about 49 nm with well-crystalline cubic spinel structure. Room temperature PL studies exhibited a strong UV emission and a suppressed green emission indicated the good crystalline properties for Fe_3O_4 nanoparticles. The magnetic behaviors reveal that the modification can decrease the saturation magnetization of Fe_3O_4 nanoparticles due to the surface effect.

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