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Structural and Optical Properties of Pure Iron and Iron Oxide Nanoparticles Prepared via Pulsed Nd:YAG Laser Ablation in Liquid

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Abstract

Iron and Iron oxide are widely used in catalytic, magnetic and biomedical application including contrast agents, drug delivery and hyperthermia. Recently, laser ablation is a flexible and versatile technique that extensively used to synthesis of pure metal and metal oxide nanoparticles by laser target interaction in liquid carrier media. In this research pure iron and iron oxide nanoparticles were synthesized by pulsed laser ablation in deionized water and acetone. Water is the base of many chemical solution and acetone is a simple organic liquid that can employed as polymers solvent and could be mixed with many conventional organic liquids. A Q-switched Nd:YAG laser with the fundamental wavelength at 1064 nm, energy of 180 mJ/pulse and 12 ns pulse length was employed for ablation. The laser was operated at repetition rate of 10 Hz for10 min. To avoid the texturing effect and to get uniform ablation, the pure iron target was rotated manually during ablation. Particle size and morphology, crystal structure and optical properties of the nanoparticles were characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD) and UV-visible spectroscopy respectively. In both samples the papered nanoparticles were rather spherical and the aspect ratio was close to 1. In deionized water and acetone the average particles sizes are 26 nm and 14 nm respectively. The XRD results demonstrate the formation of pure iron and a mixed of iron oxides nanoparticles in acetone and deionized water respectively. For colloidal nanoparticles in acetone the UV-visible spectrum exhibited the characteristic optical extinction of iron nanoparticle. Optical absorption of nanoparticles synthesized in deionized water demonstrated an absorption edge that employed for band gap calculation. In addition mechanism of nanoparticles formation, stability and oxidation of nanoparticles are also discussed. The results provide a flexible and fast method for synthesis of pure iron and iron oxide nanoparticles especially for biological applications.

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1. Introduction

In past decade nanoparticles (NPs) of different material such as metals and ceramics have been studied because of their variable chemical and physical properties. Recently metal NPs exhibit a multiple potential application as well as interest in their fundamental properties including magnetic fluid, magnetic micro device, magnetic resonance imaging, magnetic hyperthermia, drug delivery, Iida et al. (2007), Amendola et al. (2011), Wu et al. (2008). Remarkable size-dependent optical and structural properties of colloidal iron and iron oxide NPs correspond to the quantum-size effects and electrical structure of NPs and the size and crystal structure could be affected by synthesis method, Bogaerts and Chen (2005).

A variety of technique have been successfully applied for the synthesis of nanostructures in liquid. The laser ablation of solid target in liquid media is a relatively simple and flexible technique for NPs formation without using any surfactant and counter-ion, Simakin et al. (2001). This method have many advantages that could be applicable and unlimited variety of solid and liquid material, Rosa et al. (2014), Dolgaev et al. (2002). In this study pulsed laser ablation have been used for the synthesis of iron and iron oxide nanostructures in liquids. In addition optical properties and colloidal stability of iron NPs were investigated.

2. Experimental

Iron NPs were synthesized directly by pulsed laser ablation of Iron target in deionized water and acetone. The experiments were carried out with 1064nm of Nd:YAG laser operated at 10 Hz with a pulse width of 12 ns and was focused by a set of optical components supplied at normal incident to the surface of the iron target. The maximum energy was 180 mJ/pulse and 1 mm spot size for 10 minute. A field emission scanning electron microscopy (FESEM) (MIRA3 TESCAN) was used to determine size distribution and shape of NPs. A drop of sample solution placed on microscopic slides then dried at room temperature, the diameters of 300 particles were measured. In addition absorption spectra were measured in the 190-1100 nm range using a UV-Vis (SPUV-26) spectrophotometer. X-ray diffraction (XRD) (XPert Pro MPD- PANalytical), with Cu – Kα (λ = 1.54 Å) radiation was used to investigate the crystal structure of laser-generated NPs with a grazing incidence angle (3°) in the 2θ range of 32°-80° for NPs in water and 42°-66° for NPs in acetone.

3. Results and Discussion

3.1. Shape and size distribution of NPs

Shape and size distributions of colloidal iron NPs in water and acetone were characterized by FESEM. Due to the liquid confinement, the growth time of synthesized NPs is very short, therefore, the size distribution is narrow and usually in nanometer size. The average particle size is 30 nm for synthesized iron NPs in acetone that is demonstrated in (Fig. 1a) and 27 nm for iron and iron oxide NPs prepared in water that shown in (Fig. 1b). The observed iron NPs prepared in acetone and water are rather spherical.

3.2. Structural analysis of NPs

To identify crystal structure of the sample, powder XRD has been used. (Fig. 2a) and (Fig. 2b) display typical XRD pattern of iron and iron oxide NPs respectively. The XRD pattern of iron and iron oxide (FeO) NPs prepared in water is shown in (Fig. 2a) with cubic crystal system and lattice constant of a = b = c = 2.86 Å for iron NPs and cubic crystal system and lattice parameters of a = b = c = 4.29 Å for iron oxide NPs. These diffraction peaks are indexed to (110) and (200) planes for iron NPs (code of: 01-087-0722) and (111), (200), (220), (311), (222) planes

for iron oxide NPs (code of: 01-075-1550). In (Fig. 2a) the strong diffraction peak at around 44.9 ◦ is related to (110) plane of Fe crystal.

The XRD pattern of iron NPs prepared in acetone is shown in (Fig. 2b) that demonstrates cubic crystal system with lattice constants of a = b = c = 4.29 Å . These diffraction peaks are indexed to (110), (200) planes (code of: 01-075-1550). In addition in (Fig. 2b) strong diffraction peak at around 44.28 ◦ according to (110) plane of iron.

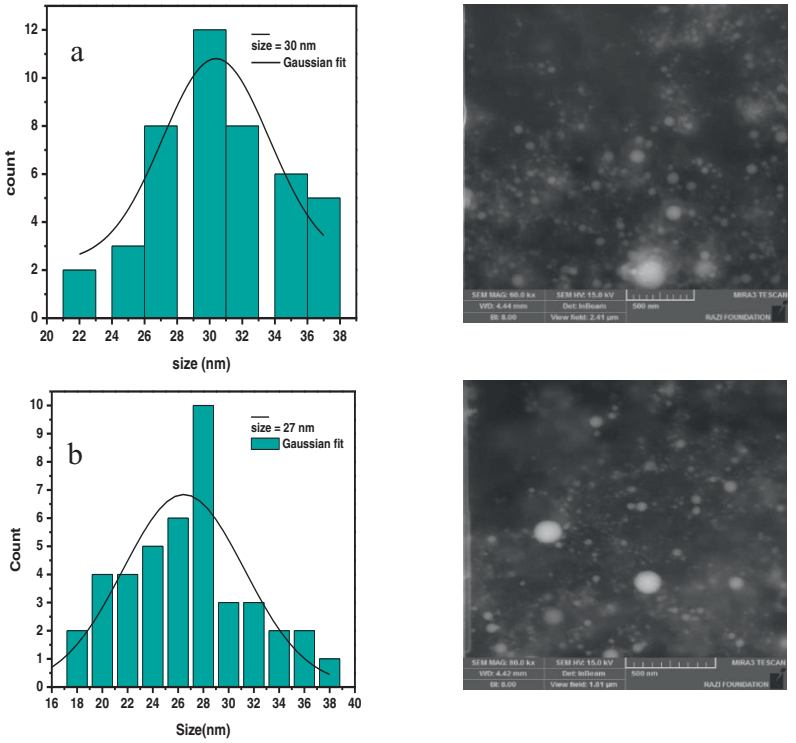

Fig. 1. SEM image of iron and iron oxide NPs prepared by laser ablation (a) in acetone; (b) in water.

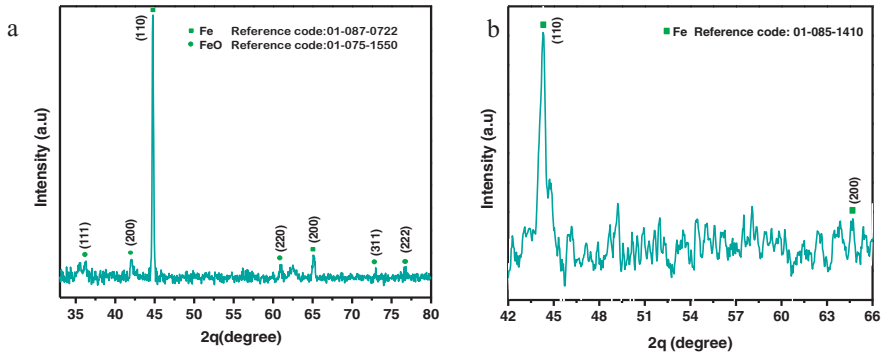

Fig. 2. XRD pattern of iron and iron oxide NPs prepared (a) in water; (b) in acetone.

3.3. Optical properties of NPs

The optical absorption of the iron NPs in the wavelength of 190-900 nm have been studied. The UV-visible spectra of iron based NPs synthesized by laser ablation in water and acetone are shown in (Fig. 3a) and (Fig. 3b) respectively. In acetone optical transmission remains unchanged for several months but in water optical transmission have some little change in spectrum. NPs in both media show a light brown color and the colloidal solutions have steady optical extinction intensity that reveals perfect colloidal stability.

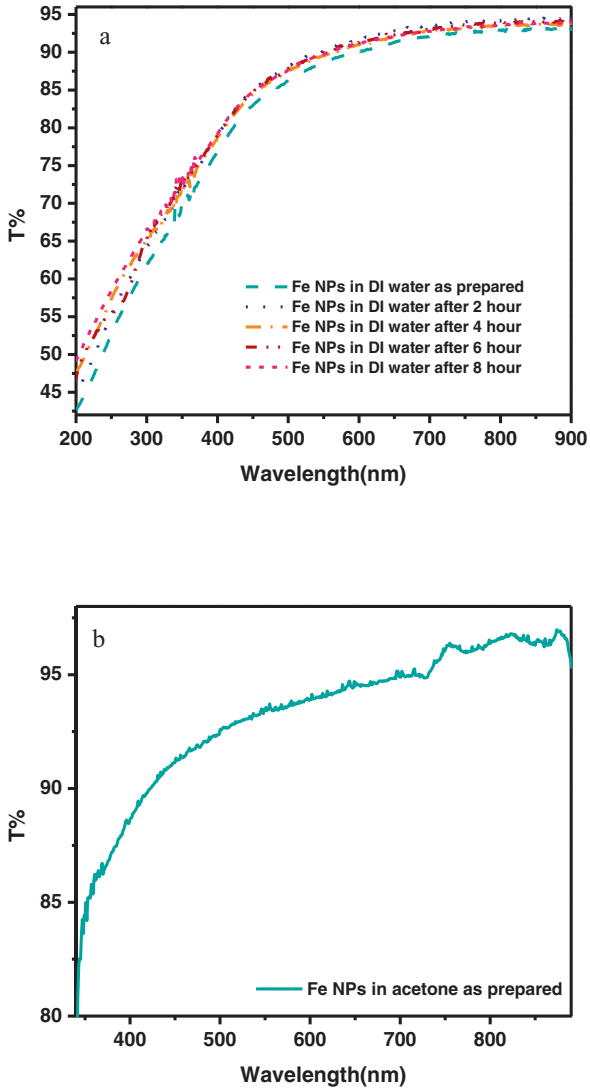

Fig. 3. UV-visible spectra of iron NPs in prepared (a) in water; (b) in acetone.

The extinction spectrum is one of the most important characteristic of metal NPs, with peaks in the extinction spectrum corresponding to plasmon resonance frequency. From the viewpoint of scattering theory the shape of the spectra and wavelength of the maximum optical extinction depends on the dielectric function of the NPs, Tilaki et al. (2006). For spherical particles a single peak of extinction spectra appear. The absorption spectra of colloidal iron NPs have been reported in the five times that is shown in (Fig. 3a) These observation suggest that the stability of colloidal solution and size distribution of NPs depend on the nature of liquid. Due to the interaction of liquid molecules and charged NPs in media, electrical double layers surround the surface of the generated NPs, Rao et al. (2004). Furthermore because of high dipole moment of molecules in acetone, sufficient electrostatic repulsive force occurs because of the overlapping of strong electrical double layer and for this reason the NPs in colloidal solution of acetone is stable without aggregation and precipitation but in water agglomeration occurs after 2 h due to the oxidation and decreasing polarity of water in comparison with acetone.

4. Conclusion

In summary, stable and pure iron NPs have been synthesized by laser ablation in acetone without the presence of any surfactant and high dispersibility, and single phase purity. The composition and structural analysis by FESEM, XRD UV-Visible photo spectroscopy confirmed the stable iron NPs synthesis. The average particle size is 30 nm for synthesized iron NPs in acetone and 27 nm for iron and iron oxide NPs synthesized in water with cubic crystal structure. The growth and size of iron NPs is controlled by nature of liquid environment and from the result changing nature of the liquid environment is an easy way to have iron and iron oxide NPs with different size distribution and stability.

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