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Research Note

Greener synthesis of magnetic nanoparticles in an aqueous deep eutectic solvent

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Abstract. The simple method of fabrication of crystalline superparamagnetic nanoferrite (Fe_3O_4) particles using oxidative hydrolysis of Fe (II) salt in a deep eutectic solvent-water mixture has been reported. The spectral properties of Fe_3O_4 nanoparticles were characterized by Fourier Transform Infrared (FTIR) spectroscopy; X-Ray Diffraction (XRD) as well as Scanning Electron Microscopy (SEM) techniques were used to estimate the crystalline structure and particle size. The results of the studies revealed that this technique could be adopted to synthesize agglomerate-free superparamagnetic Fe_3O_4 nanoparticles in a simple manner in deep eutectic solvent which may find potential application in the biosensor and corrosion protective coatings.

1. Introduction

In recent years, utilization of green solvents, such as Room Temperature Ionic Liquids (RTILs) in the fields of catalysis, material chemistry, organic synthesis, and drug industry, has received great attention due to their unusual properties compared with traditional molecular solvents [1-5]. Owing to their undetectable vapor pressure, wide liquid temperature range, special solubility for many organic or inorganic compounds, and favorable environments RTILs were qualified as advanced green solvents [6-8]. Abbot and coworkers [9] developed inexpensive, synthetically easy, nontoxic and biodegradable alternatives to RTILs with similar physical properties and phase behaivior, known as room temperature Deep Eutectic Solvents (DESs) [10-16].

Irion oxide magnetic nanoparticles play an important role not only in many areas of chemistry, but also in physics and material science. During the last decade [17-19], considerable efforts have been dedicated to the potential applications of nano-sized Fe_3O_4 (as magnetite) in many industrial and biological fields [20,21], such as mineral separation [22], heat transfer applications [23], magnetic resonance imaging contrast enhancement [24], tissue repair [25], immunoassay [26], detoxification of biological fluid [27], hyperthermia [28], targeted drug delivery [29], and cell separation [30].

Furthermore, magnetic nanoparticles offer many advantages over non-magnetic nanoparticles because they can be easily separated from solvent using an external magnetic field [31]. The High Gradient Magnetic Separation (HGMS) of coated magnetic nanoparticles' core with bio-functionalized surface has been used in many magnetic separations, especially in biological and biomedical applications [32-35]. The iron oxide nanoparticles have high saturation magnetization values, low toxicity, low price, and the surface chemical modification ability which makes them suitable absorbents for heavy metal separation during the water purification process [36,37].

Additionally, due to the unique combination of high magnetization and paramagnetic behaviour functionalized by biologically active compound, the modified nanoparticles have been successfully applied to

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controlled drug delivery systems [38], magnetic resonance imaging [39], and in magnetic-induced tumour treatment [33,34].

Several methods for the synthesis of iron oxide colloids and nanoparticles have been elaborated in the literature, such as the sol-gel, microemulsion, sonochemical, ultrasonic spray pyrolysis, and microwave plasma [40-43]. Each preparation method has its advantages and disadvantages mainly related to morphology, particles size distribution, production scale, and cost and type of application. Among various available methods, thermal decomposition seems to give the best control of nanoparticles' size and morphology. The obtained magnetic nanoparticles depending on the synthesis conditions, such as starting material, concentration, and pH of the solution, have different physicochemical properties [44,45]. So, the vital issue to prevent the undesired product formation is the synthesis procedure optimization.

2. Experimental

2.1. General

All chemicals, such as iron salts, choline chloride, and urea, are commercially available. Solvents were distilled before use. The X-ray powder diffraction pattern of the sample was recorded on a Bruker D8 Advance diffractometer using CuKα (1.5406 Å) radiation. The morphology of particles was observed by using Scanning Electron Microscope (SEM) Hitachi S4100 operating at an accelerating voltage of 25 kV. FT-IR spectra were recorded by using a Bruker Vector-22 infrared spectrometer.

2.2. Synthesis of the choline-based ionic liquids (deep eutectic solvent)

Choline chloride (1 mole, 139.62 g) was mixed with urea (2 mole, 120.12 g) and heated in 80°C in air for one hour with stirring until a clear colourless liquid was obtained.

2.3. Synthesis of magnetite (Fe_3O_4) in deep eutectic solvent-water

In a typical procedure, $FeSO_4.7H_2O$ (28 mmol, 10 g) was dissolved in 30 mL deionized water, then 30 mL DES was added, and the reaction mixture was heated to 90°C under the nitrogen atmosphere. Subsequently, a solution of 0.8 g KNO_3 and 6 g KOH in 6 ml H_2O was added drop-wise in a period of 5 minute. After 2 hours, the obtained black powder was washed with water and ethanol, separated magnetically, and dried overnight at 75°C.

3. Results and discussion

During the past decade, the green solvents, such as ILs and polyethylene glycol (PEG), have been used as reaction media, templates, and stabilizers for the synthesis of nanoparticles with controlled properties due to their low interfacial tensions, stability (non-flammable, thermally stable), and low vapour pressures. In continuation of our interest in the application of deep eutectic solvent in organic synthesis [46-51], in this paper, we present the biodegradable ionic liquid based on choline chloride as template, stabilizer, and reaction media for the synthesis of nano-Fe_3O_4 under the mild reaction condition [52]. The magnetic nanoparticles Fe_3O_4 were prepared by autoxidation of Fe^{2+} in the presence of potassium nitrate in the DES-water solutions of varying DES to water ratios. The crystal phase, purity, particle size, and morphology of the Fe_3O_4 nanoparticles were determined by powder X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), and Fourier Transform Infrared Spectroscopy (FT-IR). Wet chemical preparation of nano-Fe_3O_4 in DES-water mixture was carried out starting from the ferrous (II) sulphate with potassium nitrate as oxidation agent in DES-water mixture. Therefore, in order to investigate the effect of DES concentration on magnetite particle preparation and morphology, different volume ratios of water and DES were examined with oxidation method. The use of water is essential for solubilizing the reaction mixture in DES; in pure DES, nano Fe_3O_4 was obtained in low yields. After running several reactions in solutions with varying ratios of water and DES, it was found that the Fe_3O_4 nanoparticles, which were prepared in 1:1 volume ratios of water and DES, exhibit better dispersion and uniform size. Comparing XRD pattern of synthesized particles in DES with the pure water, the synthesized product is crystalline Fe_3O_4. The sharpness of X-ray diffraction clearly shows that the synthesized Fe_3O_4 is highly crystalline and spherical comparing with magnetite standards (Figure 1).

Crystallite size is performed by measuring the Full-Width at Half Maximum (FWHM) of the strongest reflection of the (311) peak and uses the Scherrer equation, which assumes the small crystallite size to be the cause of line broadening. The average particle size was calculated to be 26 nm using the

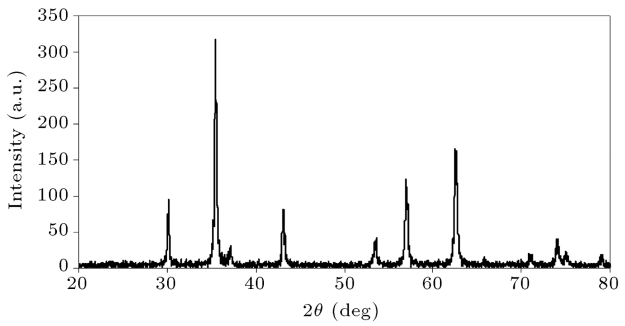

Figure 1. X-ray diffraction pattern of Fe_3O_4 nanoparticles.

Scherrer's equation (Eq. (1)).

$$D = \frac{k\lambda}{B\,Cos\theta} = \frac{0.9 \times 0.154}{0.005652 \times \cos 17.75} = 26 \text{ nm}. \quad (1)$$

Fourier Transform Infrared Spectroscopy (FTIR) spectra were performed to the dried sample of magnetite using a FTIR–Shimadzu Prestige-21 spectrophotometer in wave range of 3500-400 cm^{-1}. FTIR spectrum in Figure 2 shows that the peak at $\sim$ 3460 cm^{-1} corresponds to the hydroxyl groups attached by the hydrogen bonds in the iron oxide surface, as well as the water molecules chemically adsorbed to the magnetic particle surfaces. The intense peak at 580 cm-1 band is due to the stretching vibration mode associated with the metal-oxygen absorption band (Fe-O bonds) in the crystalline lattice of Fe_3O_4 [53].

The morphology of the magnetite particles formed is examined by direct observation via high-resolution Scanning Electron Microscopy (SEM) for all the collected particles. The SEM of Fe_3O_4 is given in Figure 3. It is observed that the tested particles are homogeneous nano-powders with a narrow size distribution, and their particle sizes are in the range 20-40 nm, which is approximately the size calculated by the Debye–Scherrer formula.

Figure 4 represents the Magnetization (M) curve of Fe_3O_4 nanoparticles vs. applied field (H) at room temperature. The synthesized nanoparticles indicate superparamagnetic behavior as there is no hysteresis loop, Hc = 0 Oe. According to Figure 4, a saturation magnetization is determined to be 45 emu/g in good agreement with literature [54,55]. The magnetization value of Fe_3O_4 nanoparticles is an important factor, since it shows the nanoparticles susceptibility to an external magnetic field. Thus, with the high magnetization value, magnetic nanoparticles could be separated from the liquid and solid phases easier.

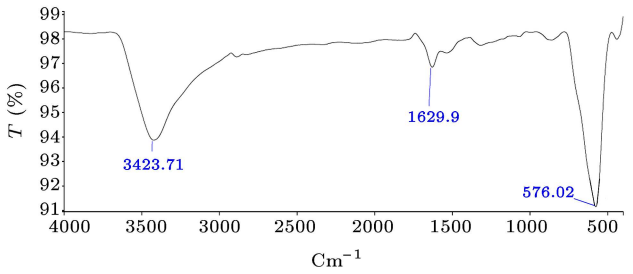

Figure 2. FT-IR spectra of (Fe_3O_4) nano-particles.

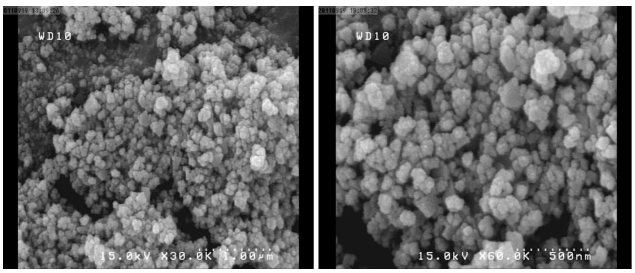

Figure 3. Scanning Electron Microscopy (SEM) images of (Fe_3O_4) nanoparticles in 1.00 μm, 300 nm, and 500 nm.

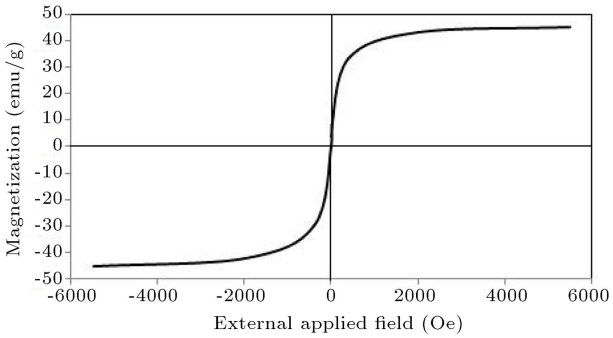

Figure 4. Magnetization curve for Fe_3O_4 nanoparticles as a function of the magnetic field (H) (Oe) measured at room temperature.

4. Conclusions

In summary, ultrafine, spherical, and uniform nano-Fe_3O_4 particles with the average diameter 26 nm have been successfully synthesized by using the biodegradable ionic liquid based on choline chloride as template, stabilizer, and reaction media. Based on the obtained results from the magnetization curve of Fe_3O_4 nanoparticles, the magnetite nano-Fe_3O_4 particles show preferable magnetic properties which could be considered as a promising way to employ Fe_3O_4 nanoparticles in nano-catalysis and bio-applications such as biosensing, Magnetic Resonance Imaging (MRI) contrast agents, and drug delivery.

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