

# Study on the properties and stability of ionic liquid-based ferrofluids

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Received: 17 July 2012 / Revised: 12 August 2012 / Accepted: 12 August 2012  
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**Abstract** Ionic liquid (IL)-based ferrofluids have been prepared dispersing both bare and sterically stabilized  $\text{CoFe}_2\text{O}_4$  nanoparticles. The precipitated particles were characterized by X-ray diffraction, scanning electronic microscopy, transmission electron microscopy, Fourier transform infrared, and vibrating sample magnetometry studies. The water-absorbing property of ferrofluids at ambient temperature was estimated by weight and viscosity measurements. Colloidal dispersion stability of the ferrofluids was evaluated by particle suspension percentage. Experimental results indicate that interparticle electrostatic repulsion is not effective in stabilizing bare magnetic particles in IL. There is no significant increase on the dispersion stability when the particles were coated with a monolayer of oleic acid. The reason could be caused by the incompatibility between the nonpolar tail of surfactant and carrier liquid. When excess oleic acid was added into IL, stable magnetic colloid was achieved by a steric stabilization layer coated to be compatible with the IL.

**Keywords** Ferrofluids · Ionic liquid · Suspension stability · Nanoparticles

## Introduction

Ferrofluids (FFs) are stable colloidal dispersions of nano-sized ferro- or ferrimagnetic particles in a carrier liquid [1]. Brownian motion keeps the particles from settling under gravity, and a surfactant is placed around each particle to

provide short-range steric repulsion between particles to prevent particle agglomeration in the presence of nonuniform magnetic fields [2]. Owing to their unique physical and chemical properties, FFs have attracted wide interest since their inception in the late 1960s.

The peculiarity of FFs is the combination of normal liquid behavior with a magnetic control of their flow [3]. Thus, for the first time in history, it became possible to exert a magnetic force on a fluid to control its position. At the same time, the coexistence in such colloid systems of the magnetism and fluidity allowed them to be referred as unique physical objects [4]. Up to now, many successful applications of FFs appeared, and the three main application areas are focused on sealing, damping, and heat transfer [5].

As dispersion medium, carrier liquid is the major component of FFs. A choice of a carrier liquid, on which base a FF is prepared, is specified by a purpose and place of its application. So far, most of the FFs used in different fields of research and technology have been prepared in certain carrier liquids such as water, kerosene, or different oils [6]. However, the use of these carrier liquids may limit the potential applications of FFs in specific areas, such as ultra-high vacuum environment, for their characteristic of volatilization.

Ionic liquids (ILs), emerging in recent years as novel substances, are composed entirely of ions and liquid with melting points near room temperature (or below  $100\text{ }^\circ\text{C}$ ) [7]. It is considered to be very stable and “environmentally friendly” compounds in the chemical industry because of their negligible vapor pressure, low flammability, and liquid state in a broad temperature range. Due to their unique physicochemical properties, ILs are also prospective candidates for colloidal dispersion media for nanoparticles.

Recently, several interesting studies on FFs using ILs as carrier liquid have been reported [8–11]. Oliveira et al. [8] successfully prepared stable IL-based FF consisting of bare

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maghemite nanoparticles dispersed in 1-*n*-butyl-3-methylimidazolium tetrafluoroborate (BMIMBF<sub>4</sub>). Stable and water-tolerant IL-based FFs were reported by Nirmesh Jain et al. [9], which showed that the bare particle stability arose in several aprotic ILs due to solvation structure surrounding particles. Laura Rodríguez-Arco et al. [10, 11] studied the stability of IL-based FFs, and the results showed that stable FF could not be obtained using bare particles, while the oleate-coated magnetite nanoparticles with volume fractions of 6.5 % in IL displayed excellent stability.

For traditional FFs, the magnetic particles must be coated with at least one kind of surfactant in order to achieve long-term stability [6]. While for IL-based FFs, as mentioned above, neither has a definite answer so far. In this paper, the synthesis and characterization of IL-based FF comprised of bare and coated CoFe<sub>2</sub>O<sub>4</sub> nanoparticles dispersing in 1-ethyl-3-methylimidazolium ethylsulphate ([EMIM][EtSO<sub>4</sub>]) is studied. The samples were characterized by X-ray diffraction (XRD), scanning electronic microscopy (SEM), transmission electron microscopy (TEM), Fourier transform infrared (FT-IR), and vibrating sample magnetometry (VSM) studies. More attention was focused on the stability of the particles in IL.

## Experimental details

### Materials

Ferric chloride (FeCl<sub>3</sub>·6H<sub>2</sub>O), cobaltous chloride (CoCl<sub>2</sub>·6H<sub>2</sub>O), sodium hydroxide, oleic acid, and acetone were all of analytical grade. 1-Ethyl-3-methylimidazolium ethylsulphate (99.9 % purity) was chosen as carrier liquid for its wide potential application prospects. Deionized water was used throughout experiments.

### Synthesis of nanoparticles and FFs

Nanoparticles of CoFe<sub>2</sub>O<sub>4</sub> were prepared by coprecipitation method. Initial molar proportion of salts (Co<sup>2+</sup>)/(Fe<sup>3+</sup>) was 0.5, and the molarity of the coprecipitating agent (NaOH) used was 0.5 mol/l. Superfluous alkali was quickly charged into the mixed solutions of CoCl<sub>2</sub> and FeCl<sub>3</sub> in their respective stoichiometry at 75 °C. When the pH of the medium was around 10, oleic acid was added to the dispersion under vigorous stirring. (To obtain bare particles, no oleic acid was used.) As reported by various authors [12, 13], the double bond in the hydrocarbon chain of oleic acid seems to play an important role for an effective stabilization of iron oxide particles. Then, it was heated up to 90 °C to convert the hydroxides in to magnetite. Particles were collected and separated using magnetic separation at this stage. After that, they were thoroughly washed with deionized water for

several times to eliminate soluble impurities, and the final pH value is neutral. After removing the excess water by acetone washing, the bare/coated particles were placed at 50 °C under vacuum for 12 h to remove residual acetone.

Finally, 10 g of bare/coated particles were dispersed in 25 ml 1-ethyl-3-methylimidazolium ethylsulphate by ultrasonic dispersion method for 60 min to homogenize the suspension. Here, for the coated particles, additional oleic acid was added to the carrier liquid; the concrete dosage was given in Table 1.

### Characterization

An X'Pert PRO (Panalytical) X-ray diffractometer was used to analyze the phase composition and size of the prepared powder samples. The diffraction was performed with CuK<sub>α</sub> (λ=1.54060 Å), and the ray was filtered by the graphite. The particle size was calculated from XRD line broadening of the reflection peak according to the Scherrer equation [14],  $d = \frac{K\lambda}{B \cos \theta}$ , where *K* is the Scherrer constant which depends on the form of the crystallites (we use *K*=0.9), λ is the wavelength of the X-ray beam, θ is the diffraction angle, and *B* is the corrected line breadth at half-maximum in radians. In this equation, the contribution to broadening by internal strains and imperfections is not accounted. The morphology and elemental compositions of the bare/coated dry powder samples were examined with Hitachi S-3400 SEM. Average particle size of CoFe<sub>2</sub>O<sub>4</sub> was obtained using bright field FX2100 TEM, with sample prepared by evaporating dilute suspension on carbon-coated copper films. The size distributions of the particles were measured from enlarged photographs. FT-IR spectra of the particles were obtained using FT-IR NEXU670 with KBr method. LDJ9600 VSM was used to measure the magnetic properties of particles and FF at room temperature. An analytical balance and rotation viscometer were used to estimate the water-absorbing property of FF at ambient temperature and humidity.

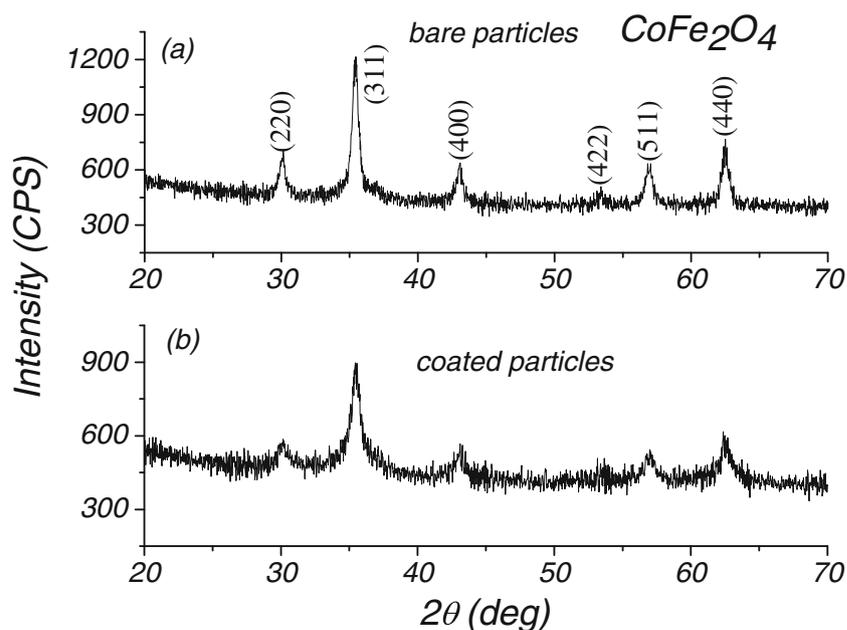
The stability is one of the most important properties for FFs, and it will strongly affect the service life. Here, the suspension stability of samples was investigated by two methods:

- (a) In plain sedimentation, samples in tubes were performed under the influence of gravity.

**Table 1** Samples with additional oleic acid

Samples	Particles	Additional oleic acid (g)
1	Bare particle	0
2	Coated particle	0
3	Coated particle	0.5
4	Coated particle	1.0
5	Coated particle	3.0

**Fig. 1** X-ray diffraction pattern of bare/coated  $\text{CoFe}_2\text{O}_4$  particles



- (b) In magnetic sedimentation, samples were placed under an external magnetic field (magnetic field gradient 15 mT/mm).

During the two processes, all the samples were redispersed by ultrasound in order to ensure equal initial conditions. Then, the density of the FFs samples ( $\rho_{\text{ini}}$ ) was measured using pycnometer. Resting all the samples in a tube for a period of time, a 0.9 volume of the upper fluid in the tube was then removed with a pipette.  $\rho_{\text{bot}}$  represents the density of the fluid left in the bottom of the tube. The smaller variation of the density ( $\rho_{\text{bot}} - \rho_{\text{ini}}$ ) is, the more stable the fluid is. The stability of the fluids was roughly estimated from the percent of particle suspending, and it was calculated as follows [15]:

$$S \% = \left( 1 - \frac{\rho_{\text{bot}} - \rho_{\text{ini}}}{\rho_{\text{ini}}} \right) \times 100 \% \quad (1)$$

where  $S\%$  expresses the suspension percentage of the magnetic particles in fluid.

## Results and discussion

The structure of the particles was analyzed using an X-ray diffractometer. The indexed X-ray diffraction pattern of bare particles is shown in Fig. 1a. The analysis confirmed the formation of single-phase fcc spinel structure for the powder samples. Lattice crystal constant of the  $\text{CoFe}_2\text{O}_4$  bare particles was calculated from the experimental diffraction peaks at 8.38 Å by using a least-squares fitting procedure. The bare particle sizes determined from X-ray line broadening analysis were 17.83–20.23 nm (see Table 2).

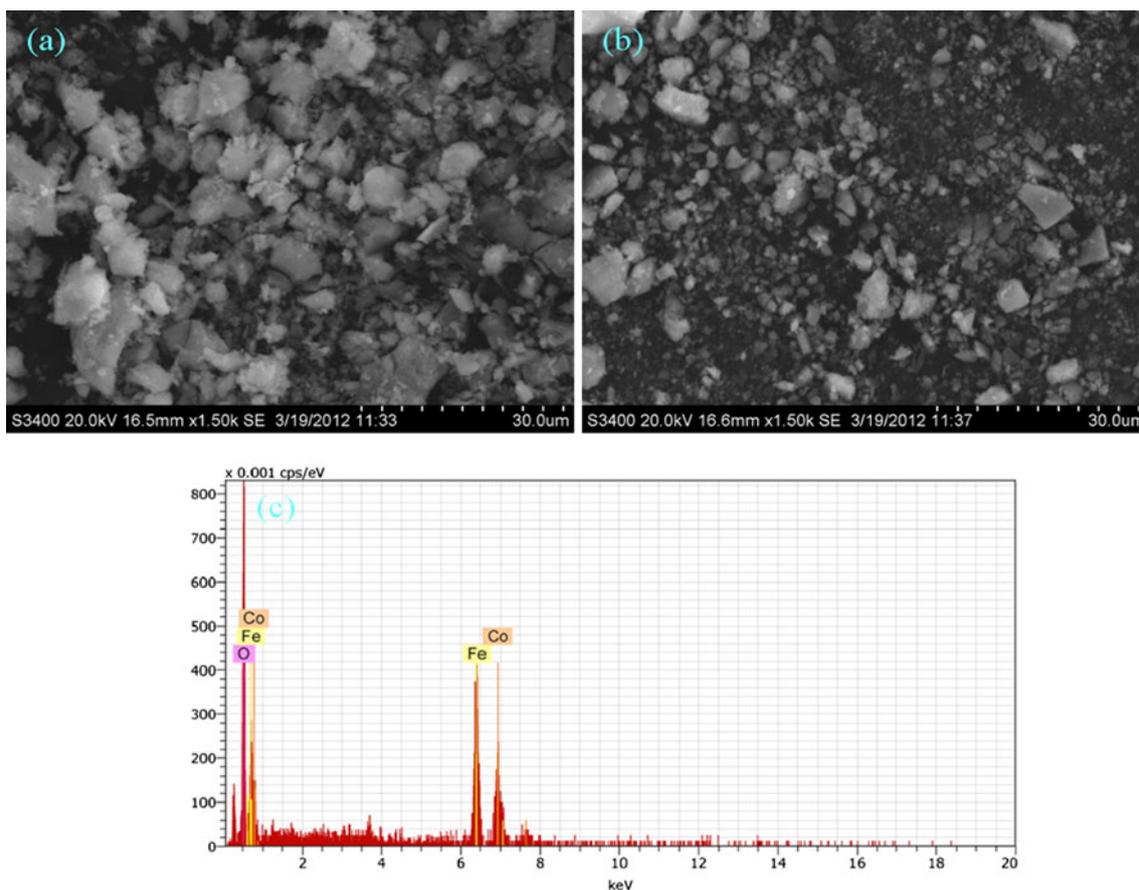
Figure 1b presents the X-ray diffraction pattern of  $\text{CoFe}_2\text{O}_4$  particles coated with oleate. It was found that particles treated with oleic acid still belonged to cubic spinel structure. Therefore, the crystalline structure of particles was not changed before and after the surface modification. The size of the coated particles is about 11.03–16.35 nm, which is smaller than those of uncoated ones. It implied that the existence of oleate coated on the particle surface could inhibit the grain growth effectively.

The SEM images of bare/coated  $\text{CoFe}_2\text{O}_4$  nanoparticles are shown in Fig. 2a, b. Because of high surface energy, the clustering phenomena took place in both bare and coated particles. By comparison, it was found that the size of particle agglomeration is reduced obviously when coated with oleate. This result was in accordance with XRD test. And three main elements, Co, Fe, and O were detected by EDS, as shown in Fig. 2c.

**Table 2** X-ray powder diffraction data of bare particle with crystallite diameter calculated from peak broadening

$2\theta$ (deg)	$hkl$	$d_{hkl}$ (Å)	$I_{\text{rel}}$ (%)	HPW (rad)	$d$ (nm)
30.10	220	2.96601	29.07	0.00796	18.06
35.50	311	2.52661	100.00	0.00816	17.83
43.05	400	2.09929	29.63	0.00830	17.95
56.96	511	1.61525	27.16	0.00779	20.23
62.48	440	1.48507	43.41	0.00887	18.28

$\theta$  diffraction angle,  $hkl$  lattice plane,  $d_{hkl}$  spacing between planes,  $I_{\text{rel}}$  relative intensity,  $HPW$  half peak width,  $d$  crystallite diameter calculated from Scherrer equation



**Fig. 2** SEM images and elements of nanoparticles. **a** Bare particles. **b** Coated particles. **c** EDS of the particles

Figure 3 shows the TEM images of the bare/coated  $\text{CoFe}_2\text{O}_4$  particles, and direct observation of the images reveals that the particles are approximately spherical in shape. As can be seen in Fig. 3a, the size distribution of the bare particles is relatively wide, and the reunion phenomenon appears especially for the large particles. However, the size of the coated particles is uniform and no obvious agglomeration happens. Compared with the bare particles, the average size of coated  $\text{CoFe}_2\text{O}_4$  particle was a slightly smaller, about  $15 \pm 2$  nm. And the size is larger than the results obtained from XRD. The difference between XRD and TEM may come from the surface coating layer.

**Fig. 3** TEM images of the  $\text{CoFe}_2\text{O}_4$  particles. **a** Bare particles. **b** Coated particles

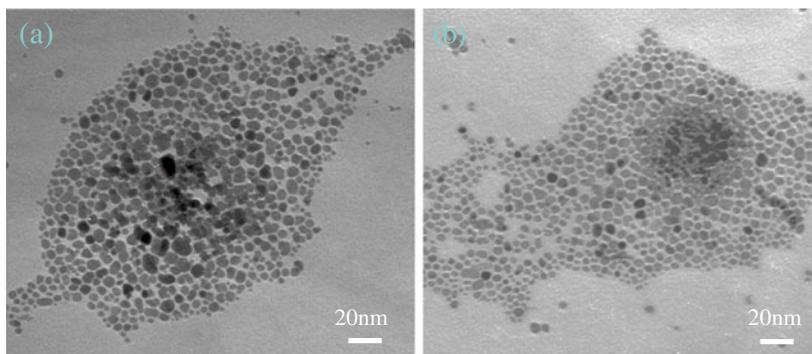
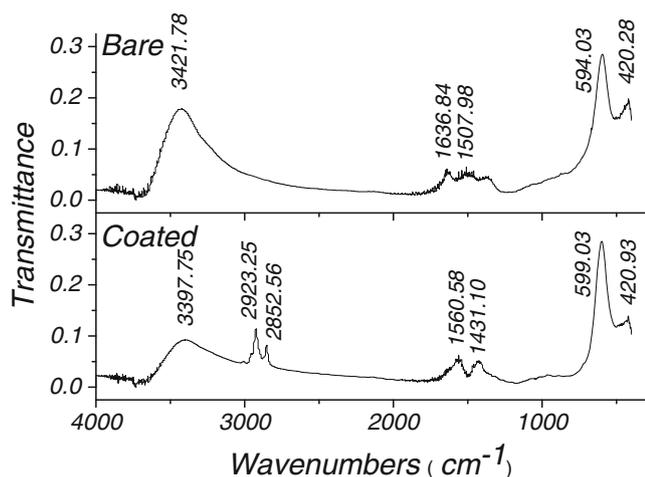
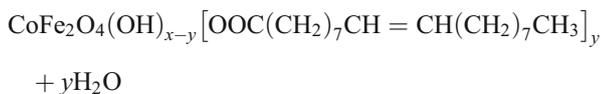


Figure 4 shows the FT-IR spectrum of bare/coated  $\text{CoFe}_2\text{O}_4$  nanoparticles. The band close to  $590 \text{ cm}^{-1}$  is ascribed to the characteristic absorption of Fe–O bond. Comparing the FT-IR spectra of bare particles, some new absorption peaks were found when the particle was treated with oleic acid. The absorption at  $2,923.25$  and  $2,852.56 \text{ cm}^{-1}$  are featuring for the stretching vibration of  $\text{CH}_2$ , and the band at  $1,560.58 \text{ cm}^{-1}$  is originated from the oleate in coating layer ( $\text{COO}^-$  stretching of oleate) [16]. The absorption at  $1,431.10 \text{ cm}^{-1}$  is due to the  $\text{COO-Fe}$  band [17]. It is believed that oleate has been grafted onto the surface of  $\text{CoFe}_2\text{O}_4$  particles though the reaction of hydroxide radical groups on



**Fig. 4** FT-IR spectra of bare/coated  $\text{CoFe}_2\text{O}_4$  nanoparticles

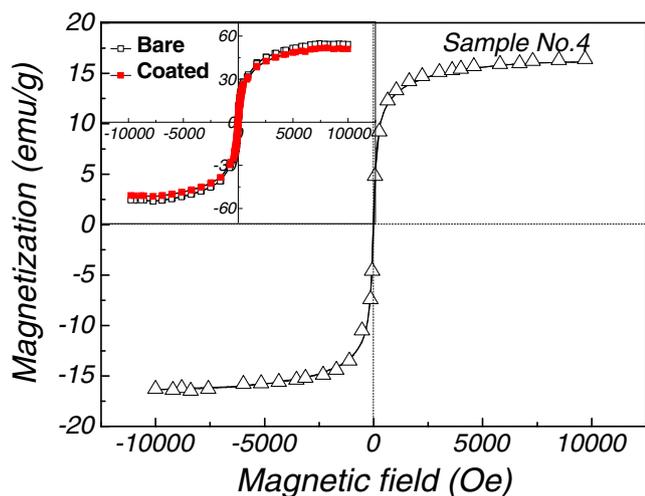
the surface of  $\text{CoFe}_2\text{O}_4$  with carboxyl groups of oleic acid. The chemisorption can be schematized as follows:



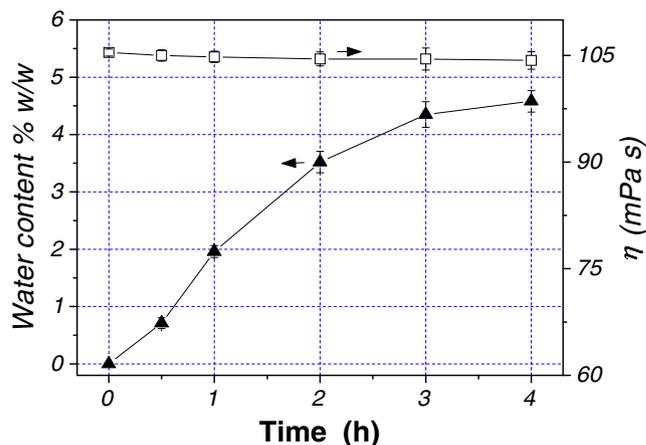
In FF, individual particles are separated by the surfactant, so that they move freely more or less, and the FF equilibrium magnetization can be accurately described by the Langevin function [18]:

$$M_0 = M_s \left[ \coth \alpha - \frac{1}{\alpha} \right], \quad \alpha = \mu_0 m H / kT \quad (2)$$

where  $M_s = M_d \Phi$  is the saturation magnetization when all magnetic dipoles with magnetic nanoparticle volume  $V_p$  and



**Fig. 5** Magnetization curve of IL-based FF (sample no. 4) fitted by the Langevin function. *Inset* Hysteresis loops of dry bare/coated  $\text{CoFe}_2\text{O}_4$  powders

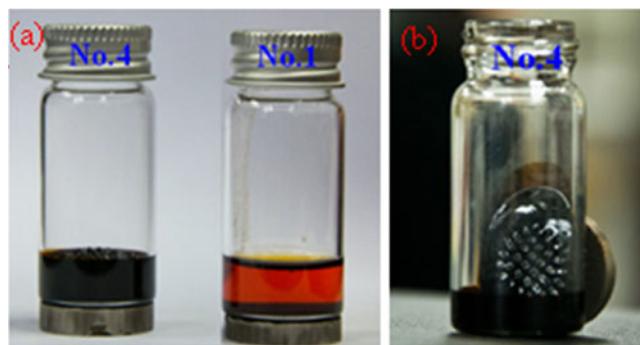


**Fig. 6** Water content and viscosity of IL-based FF (sample no. 4) as a function of exposure time

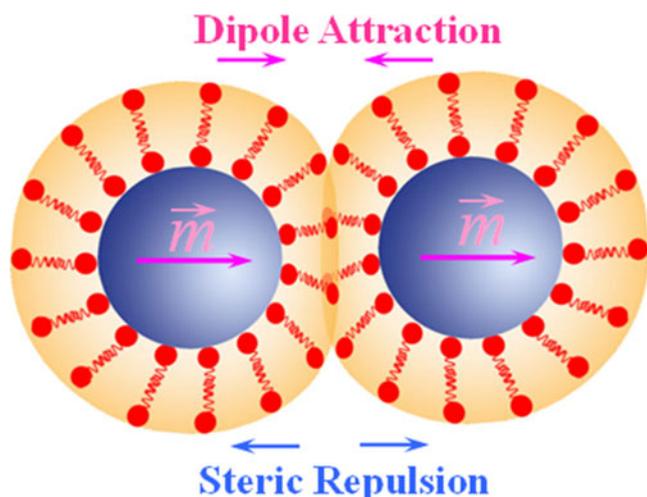
magnetization  $M_d$  having moment  $m = M_d V_p$  tend to align parallel to the magnetic field  $H$  under the action of thermal agitation  $kT$ .  $\mu_0$  is the magnetic permeability of vacuum, and  $\Phi$  is the volume fraction of magnetic nanoparticles in FF.

Figure 5 shows the magnetization curve of IL-based FF (sample no. 4) fitted by the Langevin function at room temperature. The inset in Fig. 5 gives the hysteresis loops of dry bare/coated powders. The  $\text{CoFe}_2\text{O}_4$  colloidal dispersion exhibited superparamagnetic behavior as indicated by zero coercivity and remanence on the magnetization curve. This behavior could be understood by the action of an extra relaxation process (the Brownian relaxation associated with the rotation of the magnetic aggregates in the liquid) [19]. The superparamagnetic behavior assures a reversible magnetic behavior of the suspension, which will help to preserve the colloidal stability even after a magnetic field is applied.

The saturation magnetization values of the powder obtained (shown in Fig. 5 inset) are all lower than that of bulk  $\text{CoFe}_2\text{O}_4$  (65 emu/g) [20]. The reduction in magnetic property may be due to the particle size effect or the presence of superparamagnetic particles [20]. The different magnetization between bare and coated particles is mainly explained by the adsorption of oleate on the particle surface.



**Fig. 7** Samples under the external magnetic field



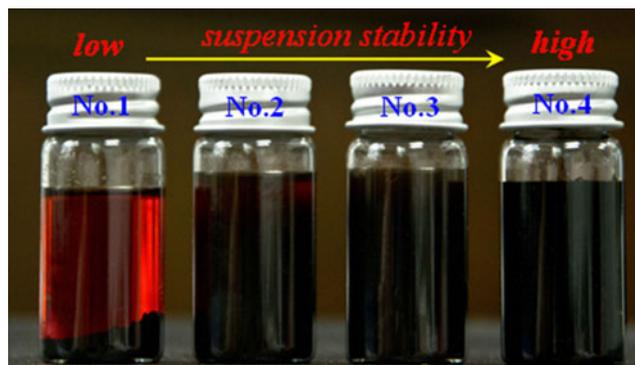
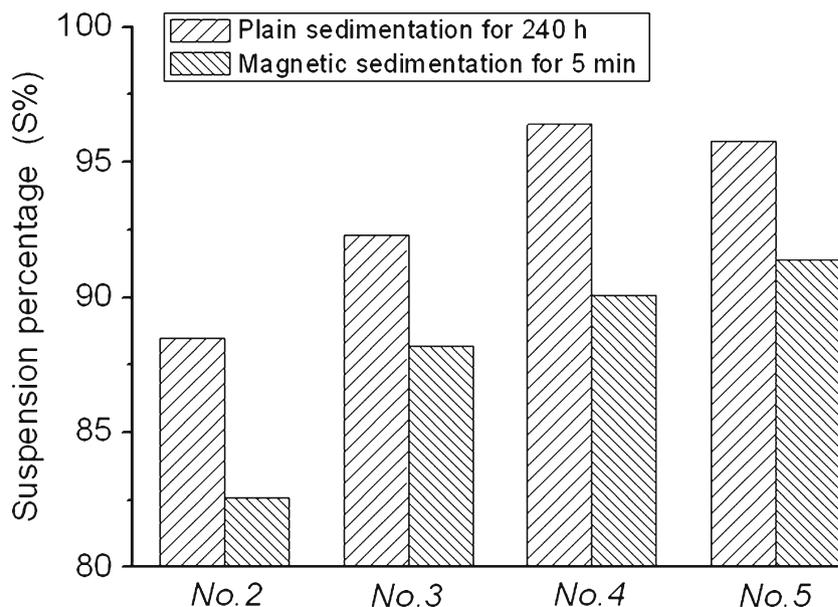
**Fig. 8** Sketch of the steric repulsion mechanism between two particles generated by the oleate-bilayer (dipole attraction was presented only)

The particle volume fraction ( $\phi$ ) in the IL-based FF (sample no. 4) was about 7.6 %, which is estimated using the measured saturation magnetization of coated particles and FF.

As mentioned by Seddon et al. [21], a number of ILs tend to absorb water from the environment, which affects their physicochemical properties. When ILs absorb water from the atmosphere, their mass and viscosity will be expected to change. Figure 6 displays the water content and viscosity of IL-based FF (sample no. 4) as a function of exposure time. The results show that the water content in FF increases over time, and it tends to be water-saturated at the end of 4 h exposure. The viscosity of FF changes slightly, which may be due to the high initial viscosity of the IL-based FF.

Figure 7a shows the picture of samples under the external magnetic field, and their responses are distinct from each other. Solid–liquid separation appeared in sample no. 1, and

**Fig. 9** Stabilities of the coated particles in IL (plain sedimentation and magnetic sedimentation)



**Fig. 10** Photographs of FF samples after plain sedimentation for 240 h

it implies that stable suspension cannot be achieved when dispersing bare  $\text{CoFe}_2\text{O}_4$  particles in  $[\text{EMIM}][\text{EtSO}_4]$ . As mentioned by Ueno et al. [22], stable colloid of bare silica particles dispersed in ILs was also not obtained even if the particles were highly charged. The interaction potentials between two silica particles in colloid were derived from simple DLVO theory, and two kinds of interactions (van der Waals potential and a repulsive electrostatic potential) were taken into account [22].

While for FF, it is necessary to consider the magnetic dipole interactions between particles, as shown in Fig. 8. Since each particle in carrier liquid is single-domain, it is spontaneously magnetized to saturation even in the absence of any external field. The magnetic dipole–dipole interaction shows at least two interesting features: (a) it is attractive or repulsive, depending on the relative orientation of the particles and their dipole moments (dipole attraction was shown in Fig. 8 for the same moment orientations); and (b) it is of long-range nature [23]. Experimental results show that the existence of magnetic interaction has not demonstrated any superiorities of colloid stability though it could

be a repulsive force (the two dipoles are in opposite direction). A spontaneous condensation phase transition of the suspended particles appeared during the plain sedimentation process. For the long-range nature, such a transition in the magnetic colloid system is more obvious when applied an external magnet.

No phase separation was found when sample no. 4 was subjected to the external magnet, indicating that particles were well stabilized and dispersed in the IL. As shown in Fig. 7b, typical spiking behavior exhibited in sample no. 4 when using a static magnetic field.

Figure 9 shows the two kinds of stability of the coated particles in IL (sample 2–5). Obviously, the stability of each sample in the manner of plain sedimentation is higher than that of magnetic sedimentation. When an external magnetic field is applied, the magnetic dipoles of the particles will attempt to align to the direction of the applied field. Magnetic particles in IL display a strong magnetic interaction between neighboring molecules, which leads to the decrease of suspension stability.

As shown in Fig. 9, sample (no. 2) without additional oleic acid exhibits lowest suspension percentage. According to the FT-IR test result, it can be deduced that oleate has been chemisorbed on the surface of  $\text{CoFe}_2\text{O}_4$  particles forming a monolayer. The surfactant molecules orient themselves so that the polar heads are adsorbed by the aqueous core while the nonpolar long chain sections are pointed radially outward. While for the high polarity of IL, there may be no compatibility between the tail and carrier liquid. Thus, unstable suspension appears in sample no. 2, even though the nanoparticles were coated.

It can be seen that the suspension percentage increased when additional oleic acid was added into carrier liquid. The increasing stability of FF may be explained by an oleate-bilayer formed on the particle surface via hydrophobic interaction. As a result, one polar tail of the bilayer extends out into solution, which can be solvated by the carrier liquid. The deep reason may be described by steric repulsion, which is efficient in nonaqueous dispersion media, while charge stabilization is less effective [24]. It is known that the effectiveness of steric stabilizers has been attributed to the thermodynamic penalty when one tries to confine polymeric chains to smaller volumes. The bilayer is most probably like a “brush” forming at the particle/IL interface, shown in Fig. 8. When the two brush layers get in contact, steric repulsion arises, which stabilizes the particles.

Figure 10 shows the picture of FF samples after plain sedimentation for 240 h. It suggests that the stability of the samples increases with the increasing dosage of oleic acid. For sterically stabilized suspensions, their behaviors depend upon the thickness of the adsorbed polymer layer [25]. When the polymer dosage is low, particle surface coverage will be incomplete, providing weak flocculation through

polymer bridging of particle [26]. An increase of the oleic acid bulk concentration seems to strengthen the steric repulsion, and the dispersion stability of the particles is heightened accordingly.

## Conclusions

In this work, a kind of IL-based FF was prepared containing both bare/coated  $\text{CoFe}_2\text{O}_4$  particles. The particle sizes were determined by XRD and TEM. The FT-IR spectrum shows that oleate has been successfully introduced onto the surface of particles. Typical superparamagnetic behavior of the prepared IL-based FF was observed from the magnetization curve. The colloid stability of magnetic nanoparticles was evaluated by particle suspension percentage. The results show that stable FF cannot be achieved when dispersing bare particles in this specific IL, and it implies that electrostatic repulsive force hardly stabilizes the bare one in the high-ionic strength fluid. The unstable FF for the coated particles is also observed which may be caused by the incompatibility between the tail of surfactant and carrier liquid. However, the stability of the magnetic colloid increased for the coated particles when excess oleic acid was added into the carrier liquid. One possible reason is that an oleate-bilayer formed in the vicinity of a solid–liquid boundary with tails coordinating with the carrier liquid. And the oleate-bilayer surrounding the particle surface contributes to the steric stabilization for obtaining stable IL-based FF.

**Acknowledgments** This work was supported by the National Natural Science Foundation of China (no. 51105199) and the Launching Foundation of NUAA (56YAH10064).

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