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Current Perspectives

Synthesis, characterization and studies on magneto-viscous properties of magnetite dispersed water based nanofluids

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ABSTRACT

Magnetic nanofluids, commonly known as ferrofluids, containing surfactant coated magnetite nanoparticles (having mean size ~11 nm) uniformly dispersed in water are synthesized by chemical co-precipitation method. The rheological properties of magnetic nanofluid at different concentrations of nanoparticle loading have been investigated by varying different parameters including the magnetic field strength. Shear thinning is observed in the non-Newtonian magnetic nanofluids under the application of magnetic field. The observed increase in yield stress (calculated by fitting the Herschel and Bulkley model) with the applied magnetic field and concentration of dispersed nanoparticles confirm the formation of large aggregates that restrict or prohibit the flow characteristics of the otherwise Newtonian magnetic nanofluid. The hysteresis observed during the application and withdrawal of magnetic field suggests that the chain or column like structures fail to relax within the allowed measurement time interval.

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1. Introduction

Ferrofluids or magnetic nanofluids (MNF) are stable colloidal suspensions of nanometer-sized magnetic particles dispersed in varying quantity in a carrier fluid. Due to the high magnetic moment of the nanoparticles, agglomeration is an obvious problem. This is prevented by coating the nanoparticles with a layer of surfactant or polymers to provide electrostatic or steric repulsion. Different types of magnetic nanoparticles used for synthesizing MNF include magnetite (Fe_3O_4) [1,2], hematite [3,4], manganese-zinc ferrites[5,6], cobalt zinc ferrite [7,8] and nickel ferrite [9,10]. Magnetic nanofluids are most commonly prepared by chemical co-precipitation [11,12], hydrothermal [13,14] and mechanical alloying [15] methods. Although the MNF can be prepared by various methods, chemical co-precipitation is the most commonly used synthesis procedure, both for laboratory scale and for industrial use. Since the reaction occurs at low temperatures in this technique, it can produce biocompatible materials in aqueous phase with fewer impurities. Moreover, co-precipitation technique is also very cost-effective [12].

The rheological [2,3,16–19] properties of ferrofluids can be excellently controlled and tuned by the application of external magnetic field. In the presence of magnetic field, the magnetic nanofluids may also exhibit non-Newtonian behavior. This external control of properties of the MNF by magnetic field without physical interference has many attractive applications like magneto-fluidic devices, inkjet printers, sensors, dampers, micro-pumps, etc. [20–24]. Most importantly, the biomedical applications including drug delivery, targeting, diagnostics and sensing [25–28] are the motivation for research in ferrofluids.

Modulation of the rheological properties of magnetic nanofluids with the application of magnetic field is well known as the magnetoviscous effect (MVE) [1,29]. It has been demonstrated in many theoretical and experimental works that the surfactant coated nanoparticles which otherwise remain in complete suspension in the nanofluid, are strongly attracted towards each other under the influence of magnetic field. The attraction results due to a higher magnetic interaction energy in comparison to the thermal energy which is discussed in details in an earlier work from the same group of authors [30]. The magnetic moment of the nanoparticles align along the direction of the applied field. However, the shearing action tilts the moment of the nanoparticles against the applied field direction which creates a resultant magnetic torque counteracting the viscous torque. This torque imbalance tries the realignment of nanoparticles along the direction of the applied field. As a result the free rotation of the nanoparticles are obstructed which causes the rise in viscosity of the magnetic nanofluid [22,31].

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It is interesting to note that apart from tuning the rheological properties of magnetic nanofluids by magnetic field, many factors including the effect of shear rate, time and the consecutive application and withdrawal of magnetic field considerably alter the rheological properties. In this work the effect of the above mentioned parameters on the Fe_3O_4 dispersed water based nanofluids have been investigated extensively and suitable explanations of the altered rheological properties have been provided.

2. Materials and methods

2.1. Materials

The raw materials utilized for the synthesis of magnetite nanoparticles were $FeSO_4\cdot7H_2O$, $FeCl_3\cdot6H_2O$, 25% aqueous NH_3, tetramethyl ammonium hydroxide surfactant ($C_4H_{13}NO$) (TMAH), 35% HCl, H_2SO_4, hexane and acetone procured from E-Merck. All the reagents used in this study were of analytical grade and needed no further purification. Deionized water was used for the preparation of the water dispersed magnetic nanofluid.

2.2. Preparation of magnetite nanofluid

The Fe_3O_4 dispersed water based nanofluids were prepared following the one step chemical co-precipitation technique [2,12,32–35]. Fig. 1a shows the schematic representation of the preparation procedure of Fe_3O_4 dispersed water based magnetic nanofluid. In this technique iron salt solutions with 1 M $FeCl_3\cdot6H_2O$ and 1 M $FeSO_4\cdot7H_2O$ were freshly prepared in acidic medium of 4 M HCl and 2 M H_2SO_4, respectively. The ratio of the ionic strength of Fe^{3+}:Fe^{2+} is maintained at 2:1. The salt solutions were mixed at 70 °C while stirring at 1000 rpm. At this condition the 25% aqueous NH_3 was instantaneously added to the mixture of the salt solutions when the precipitation of magnetite nanoparticles was ensured by the black color of the solution. The pH was measured to be ~10. The mixture was stirred under the same condition for another 30 minutes to allow the completion of the reaction. After this the precipitated magnetite particles were thoroughly washed with water for several times to remove any impurities present in the particles or solvent and to reduce the pH to ~7. A small amount of the precipitated particles were collected and dried to carry out the characterization analyses. The remaining quantity of particles was coated with TMAH surfactant at higher pH (~10) by stirring continuously for 30 minutes. Subsequently the temperature was increased by 10 °C in order to eliminate the excess ammonia from the solution. Following this the coated particles were allowed to settle down with the help of magnetic attraction and cooled to the ambient temperature. The top layer of the solvent was discarded to remove the excess salts and impurities, if any. The surfactant coated particles were washed with deionized water for several times until the pH was reduced to ~7. The magnetite nanoparticles were suitably dispersed in deionized water to prepare stable Fe_3O_4 nanofluid. The fluid was diluted carefully to prepare magnetic nanofluids of four different concentrations with the same particle distribution. The behavior of the magnetic property of the nanofluid was tested with the help of a permanent magnet (Fig. 1b). The magnetic nanofluid strongly responded in presence of the magnet and could be moved defying laws of gravity. But as soon as the magnet was withdrawn, the nanofluid was observed to regain its fluidic properties and the nanoparticles formed a stable dispersion in the base fluid without any agglomeration.

2.3. Characterization of nanoparticles and ferrofluids

The synthesized nanoparticles and nanofluids were characterized using X-ray diffraction (XRD), scanning electron microscope (SEM), transmission electron microscopy (TEM), dynamic light scattering (DLS), energy dispersive spectroscopy (EDS) and vibrating sample magnetometry (VSM) to identify the shape, size, purity and magnetic properties of the samples. After the precipiation of Fe_3O_4, before coating with the surfactant, a small amount of the sample was taken out of the bulk solution, centrifuged at 10,000 rpm and dried suitably. The identity, phase and crystallite size of the resultant powder sample was determined by XRD using a BRUKER diffractometer with a scan speed of 0.05° per second in the range 20–100°.

The particle size of the synthesized nano-magnetite dispersed water based nanofluids was determined by dynamic light scattering (DLS) method using the Zetasizer Nano (Malvern) instrument. DLS measures the Brownian motion and relates this to the size of the particle which is calculated using the Stokes–Einstein equation: $d=kT/3\pi\eta D$ where d is the hydrodynamic diameter of the nanoparticles, k is Boltzmann's constant, T is the absolute temperature, η is the viscosity and D is the translational diffusion coefficient. The designated cuvette of the Zetasizer instrument was filled with the required volume of nanofluid and the tests were carried out at 25 °C. The concentration of the nanofluid was diluted by several times by deionized water for carrying out the size measurements. The hydrodynamic particle size of sample was measured four times to check the reproducibility of experimental

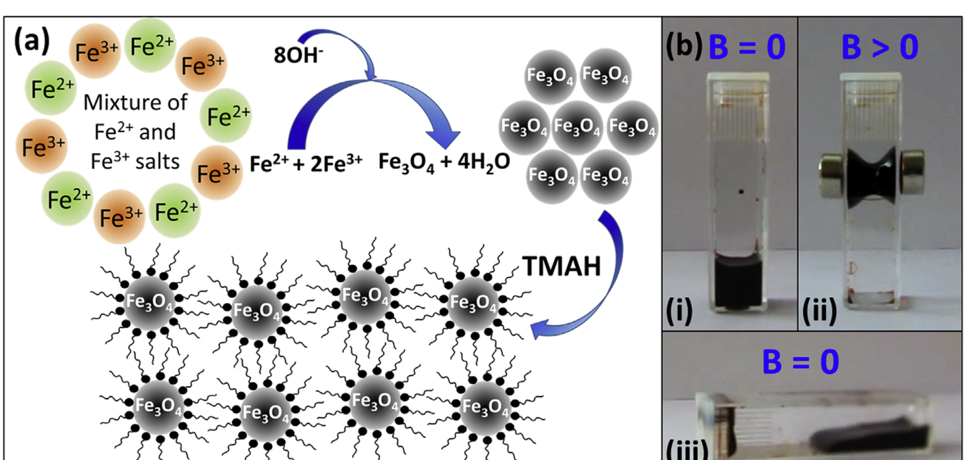

Fig. 1. (a) Schematic representation of the procedure of synthesis for Fe_3O_4 dispersed water based magnetic nanofluid by chemical co-precipitation method and (b) Images showing the response of magnetic nanofluid in the (i) absence of ($B=0$) (ii) presence of ($B>0$) and (iii) after the removal of magnetic field ($B=0$).

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