

The rheological responds of the superparamagnetic fluid based on Fe_3O_4 hollow nanospheres



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ABSTRACT

In this work, a superparamagnetic fluid based on Fe_3O_4 hollow nanospheres was developed and the influence of the particle structure on the rheological properties was investigated. The Fe_3O_4 hollow nanospheres which were prepared by using the hydrothermal method presented the superparamagnetic characteristic, and the magnetic fluid thereof showed well magnetorheological (MR) effect. The stable magnetic fluid had a high yield stress even at low shear rate and its maximal yield stress was dramatically influenced by the measurement gap. In comparison to the Fe_3O_4 nanoparticles based magnetic fluid (MF), the Fe_3O_4 hollow nanospheres based MF exhibited better MR effect and higher stability since the unique hollow nanostructure. The shear stress of the hollow nanospheres is about 1.85 times larger than the nanoparticles based MF because it formed stronger chains structure under applying a magnetic field. To further investigate the enhancing mechanism, a molecule dynamic simulation was conducted to analyze the shear stress and the structure evolution of the Fe_3O_4 hollow nanospheres based MF and the simulation matched well with the experimental results.

1. Introduction

Magnetic fluids, which were composed of magnetic particles in carrier fluids, could change their viscosity in milliseconds in the presence of an external magnetic field (magnetorheological effect). After removing the magnetic field, they return to the starting state. Due to their unique semi-active characteristic, the magnetic fluids have attracted increasing attentions in aviation, apparatus, industrial and biological field, etc.[1–3]. During the past decades, many efforts have been conducted to develop high performance magnetic fluid. Various magnetic particles such as the carbonyl iron (CI), Fe, Ni, Co, Fe-alloy, Fe_3O_4 , and ferrite particles were applied in the magnetic fluids [4–6]. Since their high magnetic saturation and tunable sizes, the obtained magnetic fluids presented excellent magnetorheological effect. fabrication, low density, and multiple nanostructures. Due to their unique magnetic property, the Fe_3O_4 based magnetic fluids were widely applied in drug delivery, magnetorheological fluid, MRI imaging, separation and etc.[7,8]. As a matured magnetic material, various methods were developed for tuning the nanostructure of the Fe_3O_4 materials. Various Fe_3O_4 particles such as the nanoparticles [9–12], nanowires [13–15], nanorods [16–18], nanotubes [19,20], nanosheets [21], nanocubes, nanoplatelets, octahedral particles [22–24] and olivary particles [25] have been reported. Although the magnetic

saturation of the Fe_3O_4 is smaller than the CI particle, it's compatible surface, small size, and small density match well with the carrying fluid thus presents better stability [26]. To this end, the study of the Fe_3O_4 based magnetic fluid is very important not only for their application but also for fundamental interest [27]. Magnetic particles played the key role in determine the magnetorheological effect for the magnetic fluid [28–30]. Under applying the magnetic field, the magnetic particles assembled to form chain-like structures thus enhanced the viscosity. It was found that the shape of the magnetic particles highly influenced the MR performance. For example, the rod-like particles led to higher MR effect while the wire-like particles improved the stability [31,32]. In comparison to the spherical particles, the anisotropic magnetic particles could result in stronger chains structure due to the larger friction force [33]. The inner structure of the magnetic particles was another critical parameter for the magnetic fluid [34]. Due to its low intensity, the hollow structure would be favorable in magnetic fluid because it could improve the stability [35–38]. Moreover, the chains structure of the hollow particles based magnetic fluid must be very different from the nanoparticles [39]. Therefore, the study of the superparamagnetic fluids based on the Fe_3O_4 hollow nanospheres is valuable.

In this work, a novel magnetic fluid based on the superparamagnetic Fe_3O_4 hollow nanospheres was developed. The hollow structured

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Fe_3O_4 nanospheres were synthesized by a simple hydrothermal method and they were further dispersed in the water to form uniform magnetic dispersion. The rheological testing indicated the as-prepared magnetic fluid presented well MR effect with high stability. In comparison to the Fe_3O_4 nanoparticles, the hollow nanospheres based magnetic fluid gave out a better MR performance. A possible enhancing mechanism was proposed to analyze the nanostructure dependent rheological properties in the magnetic fluid. At last, a molecule dynamic simulation was conducted to analyze the magnetic responds of the MF based on hollow particles. These results supplied much valuable information for designing high performance magnetic fluid. Fe_3O_4 was attractive in magnetic fluids because it's easy.

2. Experiment

2.1. Materials

All the chemical reagents were analytical graded and used without further purification. Iron(III) chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$; Sinopharm Chemical Reagent Co, Ltd), urea ($\text{CO}(\text{NH}_2)_2$; Sinopharm Chemical Reagent Co, Ltd), Citric acid trisodium salt dehydrate ($\text{C}_6\text{H}_5\text{Na}_3\text{O}_7 \cdot 2\text{H}_2\text{O}$; Sinopharm Chemical Reagent Co, Ltd), PolyVinylPyrrolidone ($(\text{C}_6\text{H}_9\text{NO})_n$; Model K-90; Sinopharm Chemical Reagent Co, Ltd), PolyAcrylaMide-3000000 ($(\text{C}_3\text{H}_5\text{NO})_n$; Sinopharm Chemical Reagent Co, Ltd) and distilled water were used for the synthesis of Fe_3O_4 hollow nanospheres.

2.2. Synthesis of Fe_3O_4 hollow nanospheres

Fe_3O_4 hollow nanospheres were synthesized by a hydrothermal method. Typically, 4 mmol $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, 12 mmol $\text{CO}(\text{NH}_2)_2$ and 8 mmol $\text{C}_6\text{H}_5\text{Na}_3\text{O}_7 \cdot 2\text{H}_2\text{O}$ were dissolved in 40 ml distilled water. After being vigorously stirred to a uniform solution, 0.3 g Polyacrylamide-3000000 and 0.1 g PolyVinylPyrrolidone were added into the solution. Keep stirring until all the reagents were dissolved completely. Then, the mixture was transferred into a 50 ml Teflon-lined autoclave. The autoclave was sealed and then maintained at 200 °C for 12 h. After the autoclave was naturally cooled down to room temperature, the black precipitation was washed with distilled water and absolute ethanol for three times. Finally, the black precipitation was dried in vacuum desiccation oven at 60 °C overnight to get the Fe_3O_4 hollow nanospheres.

For the synthesis of magnetic fluids, the above Fe_3O_4 hollow nanospheres were dispersed into water via sonication. After a uniform suspension was obtained, the final product was sealed in a vial for the further measurement. The magnetic fluids based on Fe_3O_4 hollow nanospheres with weight fraction of 10%, 15%, 20% were prepared. Moreover, in order to compare the difference of the rheological properties in magnetic fluids based on the hollow nanospheres and nanoparticles, the hollow Fe_3O_4 nanospheres was treated under a continuous ball milling for 24 h to achieve the Fe_3O_4 nanoparticles. The Fe_3O_4 nanoparticles based magnetic fluid was prepared after being dispersing in the water. The two magnetic fluids were prepared via a similar process.

2.3. Characterization

The crystal structure of the prepared nanospheres was examined via X-ray diffraction (X-RD) on a Philips X'pert Pro SUPER rotation anode with Cu-K α radiation ($\lambda = 1.541874 \text{ \AA}$). A scan rate of $0.02^\circ \text{ s}^{-1}$ was applied to record the pattern with the 2θ range of $10\text{--}70^\circ$. Transmission electron microscopy (TEM) images were obtained on JEOL-2010 with an accelerating voltage of 200 kV. The magnetic property was measured on a superconducting quantum interference device (SQUID) magnetometer at 300 K with the applied field sweeping from -20 to 20 kOe .

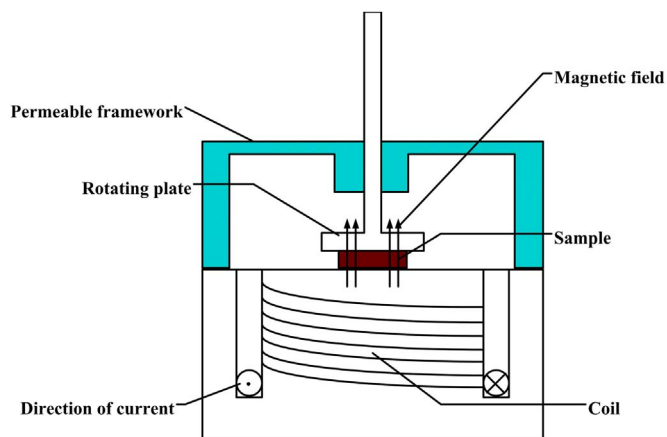


Fig. 1. Principle of testing part for Physica MCR 301.

The rheological properties of the magnetic fluids were tested by a commercial rheometer (Physica MCR 301, Anton Paar Co., Austria). Fig. 1 shows the schematic of the testing part. The samples were placed between two parallel plates and a shear loading was applied on the sample through the rotating plate, which is made of non-permeable material and the diameter of the rotating plate was 20 mm. The rotating plate can also transmit other signals (such as stress, displacement, and strain, etc.) by the sensors connected to it. The magnetic field is generated by an inbuilt coil and the intensity of the magnetic field is controlled by the current in the coil. Magnetic induction lines pass through a permeable framework and sample to form a closed magnetic circuit.

A certain amount of magnetic fluid was taken and put between the two parallel plates of the commercial rheometer. The test was taken out under different shear rate, current and parallel plate gap. All the tests were carried out at room temperature.

3. Result and discussion

3.1. Characterization of the hollow Fe_3O_4 nanospheres

As shown in Fig. 2(a)–(c) are the TEM images of the Fe_3O_4 hollow particles. Clearly, the particles in the images are spherical and the nanospheres had a pale center region and a dark edge, which indicated that the nanospheres had a hollow nanostructure. The average diameter of the magnetite hollow nanospheres was about 300 nm, while the shell thickness was about 40 nm. A higher magnification TEM with one single hollow sphere was used to further investigate the inner nanostructure of the hollow particles. It was clearly observed that the hollow sphere was composed of irregular shaped primary particles with the diameter of approximately 15 nm, which agreed well with the XRD analysis which would be shown later. The shell of the Fe_3O_4 hollow sphere was aggregated by large amount of irregular nanocrystals, thus it was porous. In order to compare the difference of the rheological properties in magnetic fluids based on the hollow nanospheres and nanoparticles, the hollow Fe_3O_4 nanospheres was treated under a continuous ball milling for 24 h to achieve the Fe_3O_4 nanoparticles. Fig. 2(d) showed the TEM image of the final Fe_3O_4 product after the ball milling. Only Fe_3O_4 nanocrystals with size about 15 nm were found. No hollow nanosphere was found in the image, indicated all the particles were broken. This result also supposed that the Fe_3O_4 hollow spheres were aggregated by the secondary Fe_3O_4 nanocrystals.

There are two reasons for the ball milling treatment. Firstly, the ball milling could break the hollow nanospheres into nanoparticles because the nanospheres were composed of large amount of secondary Fe_3O_4 nanocrystals. Secondly, the as-obtained Fe_3O_4 nanocrystals exhibited similar crystal nanostructure and magnetic properties to the Fe_3O_4 hollow spheres, thus they could be used to prepare Fe_3O_4 nanocrystals

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