Journal of Magnetism and Magnetic Materials 465 (2018) 421–429

Contents lists available at ScienceDirect

Journal of Magnetism and Magnetic Materials

journal homepage: www.elsevier.com/locate/jmmm

Research articles

Rheological response of magnetorheological suspensions sediments and its implications for redispersibility

Fang-Hao Niu[a], Zhi-De Hu[a,*], Hua Yan[b], Jian-Jian Yang[c], Han-Song Zhang[c]

[a] Department of Military Infrastructure, Army Logistics University, Chongqing 401311, PR China
[b] Department of Basic Science, Army Logistics University, Chongqing 401311, PR China
[c] Department of Military Petroleum, Army Logistics University, Chongqing 401311, PR China

ARTICLE INFO

Keywords:
Magnetorheological suspensions
Sediment
Redispersibility
Particle size distribution
Viscoelasticity
Thixotropy
Recovery

ABSTRACT

This work reports an investigation into the redispersibility of magnetorheological suspensions (MR suspensions) by testing the rheological properties of suspension sediment. By using a new freeze-sampling method, slices of sediment were taken from the bottom of the container with little changes on shear history which makes it possible to employ rheological measurement. Size distribution of particles contained in sediment was studied by a laser scattering method. Oscillatory and steady shear was employed to studying the rheological viscoelastic and flow behavior. Also, thixotropy and recovery behavior was also studied. The results obtained proved that redispersibility is a multifactor-dependent property which can be reflected from rheological responses of MR suspensions' sediment. Hydrodynamic force and particle interaction which have significant influence on those properties were analyzed in this study. In addition, indexes correspond to different rheological properties were put forward to make a comparison quantitatively between traditional MR suspensions.

1. Introduction

Magnetorheological suspensions (MR suspensions) are typically suspensions of magnetizable particles dispersed in carrier fluids which shows a reversible transition from liquids to a semi-solid state upon the application of magnetic field [1]. These changes on rheological properties, which known as "magnetorheological effect", are caused by the formation of anisotropic aggregates through magnetic forces between magnetizable particles. These outstanding properties makes them good candidates for applications in shock absorbers, seal, dampers and some other torque transferring devices [2–4].

However, the large mismatch of density between magnetizable particles and carrier fluids makes it unavoidable to settle under gravity. During the sedimentation process, magnetizable particles are prone to aggregate and form a compact solid "cake" result from small levels of residual magnetism and van der Waals force, which is to say, it is quite difficult to redisperse the suspension [5]. Magnetorheological suspensions are no longer functional once the suspension has not been redispersed in time. Hence, poor redispersibility has become a serious problem facing the technical applications.

Many recent researches have pay significant attention to the promotion of redispersibility through different approaches as well as characterization methods. Early in 1999, Phule et al. [5–6] made a calculation of magnetic dipole–dipole and van der Waals force interaction energy, they got the conclusion that enlarging the distance between particles is the key to improve redispersibility. As for experiment, it has reach an agreement that rheological test for the sediment is appropriate for estimating redispersibility. López-López et al. [7] employed a vane-in-cup measure system and a rotation test was applied after sample settling in the cup for different range of time. They found that both oleic acid and aluminum stearate could significantly enhance redispersibility due to repulsion effect although these surfactants could hardly prevent sedimentation. In contrast, gel-forming agent such as silica nanoparticles enhance the sedimentation stability to a large extent. But once the MR suspensions are sheared, it quickly breaks down and particles could settle easily. As a result, iron-silica sediment is formed and redispersibility is hindered. It should be noticed different concentration zones exist in the MR suspensions sediment column during the settling process. Rheological properties measured by this kind of vane-in-cup system are the mean value of a considerable large section of sediment which could not represent the "stiff cake forming zone (with extremely high concentration of dispersed phase and hard to redisperse)" [8–10]. There is another method based on recording the stiffness of the sediment by applying a penetration standard needle (ASTM D5 05a) connected to a digital balance provided by Iglesias et al. [11]. According to their research, nano magnetite surrounding the large

* Corresponding author.
E-mail address: huzd6503@163.com (Z.-D. Hu).

https://doi.org/10.1016/j.jmmm.2018.05.101
Received 12 March 2018; Received in revised form 27 April 2018; Accepted 30 May 2018
Available online 05 June 2018
0304-8853/

particle protect them from short-range attraction, thus hindering the aggregation. Later, described by Bombard et al. [12–13], the measure of the redispersibility can be estimated by recording the normal force while a four blade vane tool penetrating into the simple at a constant speed of 1 mm/s. Another index of redispersibility in their experiment is the yield stress obtained from rotating the vane tool at a constant angular speed (0.001 rpm). Applying this method, 1-octanol was found to be an effective additive to improve redispersibility although high 1-octanol concentration may cause depletion flocculation [14]. MR suspensions based on ionic liquids with coated carbonyl iron (CI) particles has also been proved to be easy to redisperse [15,16].

Although there are many literatures about dynamics of sedimentation by tracking the boundary between supernatant and sediment as well as comparing the difference of light (λ = 880 nm) transmitted and backscattered through the MR suspensions [17–21], little attention has been paid to the redispersibility which is equally important. Also, previous studies took the whole fluid column as the experiment subject rather than specific suspension layer. So it is quite necessary to give a comprehensive research about the rheological properties of the sample at specific position in a fluid column. The aim of this study is to introduce a rheological method to estimate redispersibility systematically and quantitatively. In order to take a sample from the bottom of the container without changes in shear history, liquid nitrogen is used to freeze the MR suspensions column (see experiment section). Storage modulus and loss modulus were tested to analyze viscoelasticity of sediment that correspond to microstructure formed in sediment. Then flow behavior was studied through a rotation test. It is known that dynamic viscosity and yield stress is a microscopic reflection of microstructure such as magnetizable particle aggregations which hinder redispersity, which could be feasible index. Also, thixotropy and recovery of the sediment were also studied which indicate the damage extent of microstructure after rotation as well as recover extent. As for MR suspensions, we prepare samples with different additives which have been reported used commonly in traditional MR suspensions. In addition, MR suspensions we prepared are actually polydisperse suspensions, in which larger particles settle quicker. As a result, median particle diameter and distributions of MR suspensions after sedimentation are totally different from original one. So it is quite necessary to test the median particle diameter and distribution of the sample taken from sediment that do affect rheological properties to a large extent.

2. Experiments

2.1. Materials

Considering the magnetizable particles used most commonly, soft magnetic CI particles (HT-1quility, supplied by Xinghua chemical co., LTD, China. The information below is provided by the manufacturer: chemical composition: 99.77% Fe, 0.042% C, 0.01% N, 0.16% O; density: 7.8 g/cm^3.) was used in the preparation of the MR suspensions. Scanning electron microscopy (SEM) images (Fig. 1) shows the iron particles are spherical and polydisperse. Mineral oil (dynamic viscosity at 20 °C: 0.307 Pa·s, density: 0.8895 g/cm^3, supplied by Chongqing Yiping Company, China.) was employed as carried fluids for the preparation of MR suspensions. Stabilizing additives we used are as follows: (i) oleic acid (OA, $CH_3(CH_2)_7CH = CH(CH_2)_7COOH$), analytical quality. (ii) silica nano particles, 50 nm. OA and silica particles are both supplied by Aladdin Company, China.

2.2. Preparation of MR suspensions samples

The modification of iron particles was as follows: (i) iron particles were dried for 12 h in a vacuum to remove the physical absorbed water on the surface (vacuum degree: 0.08 MPa; temperature: 60 °C); (ii) the dried particles were immersed in the mixture of isopropyl alcohol and oleic acid (2 vol% of MR suspensions) and mechanical ball-milling (300 rpm, 6 h) was employed to allow the adsorption of oleic acid; (iii) mixture from (ii) was dried to get the modified particle.

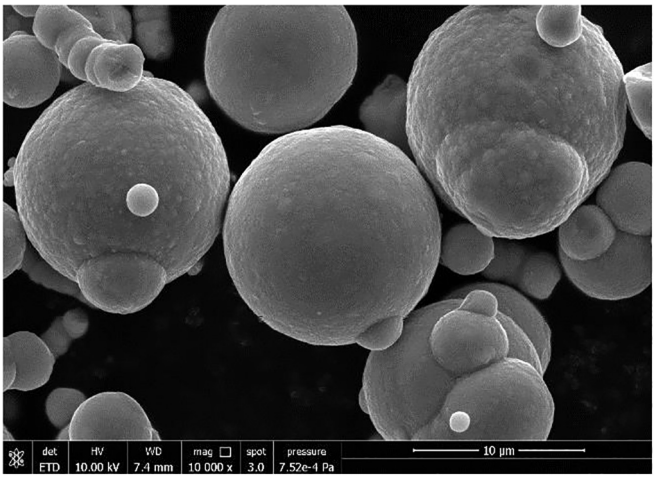

Fig. 1. SEM picture of iron particles.

The preparation of MR suspensions was as follows [7]: (i) a)iron particles (modified, 20 vol%) and mineral oil, b)iron particles (bare, 20 vol%), nano-silica (0.705 vol%) and mineral oil were mixed in a beaker; (ii) the mixture was shaken by hand and immersed in an ultrasonic bath. This step was repeated for three times; (iii) samples were immersed in a sonifier (Branson 450, USA) until suspensions reach homogeneity.

2.3. Freeze sectioning

In order to take a piece of sample from sediment area without changes in shear history, liquid nitrogen was employed to freeze the sample in a plastic cuvette (made from polypropylene). As shown in Fig. 2, 10 ml homogenous MR suspensions in the cuvette was centrifuged at 500 rpm (approximately 42*g*) in the centrifuge (Changsha xiangzhi TDZ5-WS, China) for 40 min to make sure particles settle completely. The equivalent time of gravity sedimentation is more than 6 months. Thereafter, the cuvette was immersed into the liquid nitrogen for 45 s to make sure the sample was solidified and not too brittle. Then the sample together with the cuvette was sectioned into a slice for 1.5 mm thick at a height of 5 mm above the bottom by a cutting tool. Finally, the slice was moved to the measuring cell and the residual part of the cuvette surrounding the sample was removed slightly once the slice begin to melt. It is essential to keep a dry environment in case of the condensation of water vapor, so a dehumidifier (MORII MDH-790B, China) was employed.

2.4. Particle size distribution and volume fraction

Particles in the slice were washed by petroleum ether for several times and dried to ensure that mineral oil was removed. 4 g particles were then dispersed in ethanol by ultrasonic bath and sonifier. Here it is worth noting that all samples should be subjected to the same dispersion protocol. After that the suspension was added into the measuring chamber of a laser scattering particle size distribution analyzer and tested (HORIBA LA-300, Japan). In addition, the slice was weighed before and after washing. The volume fraction could be calculated as:

$$\phi(h) = \frac{w_2(h)/\rho}{w_2(h)/\rho + (w_1(h) - w_2(h))/\rho_0} \quad (1)$$

where ϕ is volume fraction, ρ is the density of CI particles, ρ_0 is the density of carrier fluid, w_1 and w_2 are weight of the slice before and after washing, respectively.