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Synthesis and magnetorheology of suspensions of cobalt particles with tunable particle size

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Abstract. Different samples of cobalt powder were synthesized. Particle size and shape were characterized by electron microscopy and light scattering. These measures showed that the synthesized powders consisted of monodisperse spheres with average diameters ranging between 63 and 760 nm. These powders were used for the preparation of magnetorheological (MR) fluids by dispersing them in silicone oil. It was found that particle size did not have much influence on the MR response of MR fluids for average particle diameters larger than 100 nm. On the other hand, the MR response decreased appreciably when average particle diameter was diminished below 100 nm.

1. Introduction

Suspensions of colloidal magnetic particles are complex fluids that exhibit magnetic field-dependent rheological (flow) properties. Upon magnetic field application, these particles experience attractive magnetostatic forces, which lead to the formation of particle structures aligned with the field direction. The formation of these structures, which strengthen the suspension and oppose to the flow, is the basic phenomenon underlying the magnetorheological (MR) effect [1-4]. The formation of field-induced particle aggregates, i.e. the intensity of the MR effect, will depend on the ratio of the magnetic interaction energy to the thermal energy. For two magnetic dipoles this ratio is given by [3]:

$$\lambda = \frac{\pi\mu_0\mu_f\beta^2 a^3 H_0^2}{2kT}. \quad (1)$$

Here, a is the particle radius, μ_0 the permeability of vacuum, μ_f the relative permeability of the carrier liquid, H_0 the external magnetic field strength, k the Boltzmann constant, T the absolute temperature, and β the magnetic contrast factor. Generally, the particles have a large permeability and, therefore, $\beta \sim 1$. Thus, taken $T = 300$ K and $\mu_f \sim 1$, for MR fluids ($a \sim 1 \mu\text{m}$) $\lambda = 1$ is obtained for $H = 0.046$ kA/m, whereas for FF ($a \sim 5$ nm) the same value is obtained for $H = 368$ kA/m. Therefore, the Brownian forces normally dominate the magnetic forces in FF and, consequently, they only exhibit weak MR effect [5-6]. On the other hand, even at low magnetic field, the magnetic forces are much higher than the Brownian forces in MR fluids and, consequently, they exhibit strong MR effect [7-8].

From the experimental viewpoint, little is known about the effect of particle size on the MR properties of suspensions intermediate between FF and MR fluids. Lemaire et al. [9] measured the shear stress as a function of the shear rate for three different suspensions, made of polystyrene particles containing inclusions of magnetite, under the presence of an applied magnetic field of

approximately 10 kA/m. The only difference between the suspensions was the average particle diameter: 0.5, 0.8 and 1.0 microns. They found that, for a given shear rate, the apparent shear stress slightly increased with the diameter of the particles. Rosenfeld et al. [10] investigated the MR properties of two suspensions of iron particles, the average particle size being the only difference between both of them: 30 microns and 26 nm. They found that, even at high magnetic field strength, the suspension of microparticles exhibited yield stresses up to 2 times higher than the suspension of nanoparticles. According to Equation 1, even for a particle diameter of 25 nm the parameter λ is still larger than unity for magnetic fields of interest ($\lambda \sim 10$ for $H = 100$ kA/m). Thus, from the theoretical viewpoint, the MR effect should not show any size dependence for diameters larger than 25 nm.

The difficultness of performing a rigorous experimental study about the influence of particle size on the MR properties, lies in obtaining monodisperse and well dispersed spherical particles with different average particle diameters. For instance, Kormann et al. [11] obtained a quite large yield stress (3 kPa at 0.3 Tesla) with ferrite nanoparticles of 30 nm diameter likely because of the presence of aggregates of nanoparticles. The aim of the present work is to rigorously analyze this dependence for particle diameters in the range 50 nm to 1 μ m. With this objective, we synthesized different samples of cobalt powder with controlled diameter, by reduction of cobalt ions in polyols. The so-synthesized powders were used for the preparation of MR suspensions. Finally, the MR properties of these suspensions were investigated as a function of the applied magnetic field.

2. Experimental methods

Reduction of metallic ions in a liquid polyol has been extensively used for the preparation of spherical particles of magnetic and non-magnetic materials [8,12-16]. This method was used in this work to synthesize spherical cobalt particles with different average diameter. Particles size and shape were characterized by means of scanning electron microscopy (SEM) and transmission electron microscopy (TEM). In addition, the hydrodynamic diameter of the particles was obtained by light scattering measurements using a Mastersizer 2000 instrument (Malvern Instruments, UK). The magnetization curves measured on the powders with a vibrating sample magnetometer show no noticeable difference between the different sizes and we find M_s between 13900 and 14000 Gauss whatever the size. Nevertheless it is worth noting that this value is significantly lower than the one of bulk cobalt ($M_s = 17900$ G) which means that some part of the catalyst has been included into the cobalt nanoparticles.

MR suspensions ($\phi = 0.05$) were prepared by dispersing the synthesized cobalt powders in silicone oil. Based on discussions reported in previous papers [17-18], aluminum stearate was used as dispersant. The MR properties of the suspensions were measured using a Haake RS150 controlled stress rheometer. The measuring system geometry was a plate-plate configuration of diameter 20 mm and gap 0.300 mm with a controlled temperature $T = 20^\circ\text{C}$. The magnetic field was applied in the vertical direction using a home-made electromagnet similar to the one sold by Anton Paar for the rheometer MCR301, which allows reaching fields up to 580 kA/m in the measuring gap.

Table I. Average diameter (nm), measured by different techniques, of the synthesized cobalt particles.

Sample name	S1	S2	S3	S4
Electron Microscopy	760	330	83	63.6
Light Scattering	629	498	126	69

3. Results and discussion

The synthesized powders consisted of relatively monodisperse spheres of submicrometer size. Table I shows the average particle size obtained by electron microscopy and light scattering. As observed, there is a quite good agreement between the data obtained by electron microscopy and by light scattering which confirms the absence of aggregation in liquid phase.

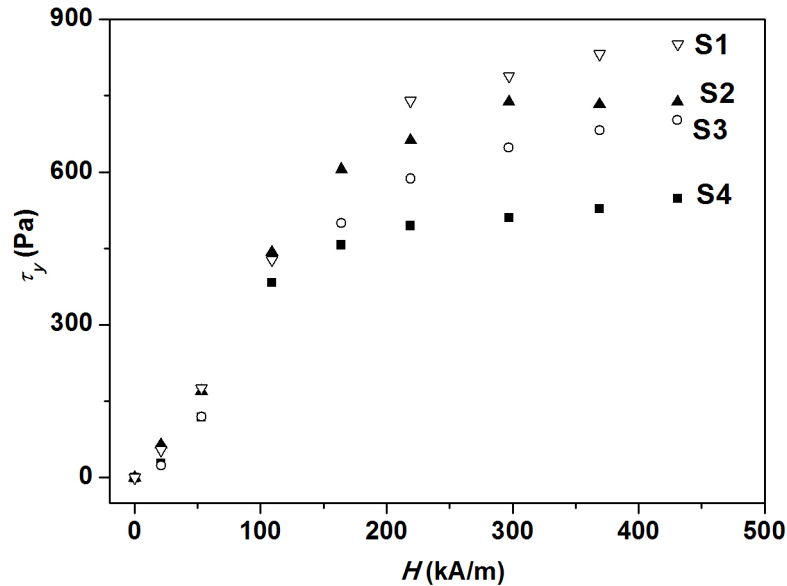


Figure 1. Yield stress, τ_y , plotted as a function of the magnetic field strength, H , for suspensions containing 5 vol.% of cobalt particles (samples S1-S4; see Table I).

The steady-shear flow of suspensions containing 5 vol.% of cobalt powders of samples S1-S4 dispersed in a silicone oil of viscosity: 0.479Pa.s and density = 0.97g/cm³, was investigated as a function of the applied magnetic field. The values of the shear stress were obtained as a function of the shear rate, for the different applied magnetic field strengths (up to 431 kA/m). From extrapolation at zero shear rate, of the shear stress vs. shear rate curves in the range 0.01 s⁻¹ - 0.1 s⁻¹ on a logarithmic scale, the values of the static yield stress were obtained. These values are plotted as a function of the magnetic field strength in Figure 1. As expected for MR fluids, the values of the yield stress shown by samples S1-S4 increase with magnetic field strength. Let us focus on the comparison of the values of the yield stress for the different samples. As observed, the yield stress decreases as the average particle diameter is diminished (see particle diameter in Table I). This decrement is negligible between S1(760nm) and S2 (330nm)except at the highest fields, but becomes quite significant for average diameter smaller than 100 nm. For example, taken as the reference, the value of sample S1 at $H = 297$ kA/m, the yield stress is respectively: 94 % for sample S2; 82 % for sample S3; and 65 % for sample S4. The reason for this decrement could be the Brownian motion, which is able to generate fluctuations on a large scale and promote the presence of gaps between the particles, which will decrease the strength of the columnar structures under strain and, consequently, the strength of the MR effect [9].Nevertheless, considering that, even for the smallest diameter, we have $\lambda > 1000$ for $H = 297$ kA/m it is not obvious that such a low Brownian motion can explain this size dependence; furthermore, this decrease should be less pronounced at the largest fields, which corresponds to the highest values of λ , and actually we observe the contrary. Another explanation stands on the surface energy of the aggregates, which determines their size and the condition of their rupture under strain. The energy difference produced by the breakdown of an aggregate in two smaller ones will be a decreasing function of the size of the particles as it is the case for the surface energy. The consequence is that aggregates constituted of smaller particles will break at lower strain and consequently at lower stress. This approach developed by Zubarev et al. [19] for ferrofluids could likely explain the observed behavior and will be developed in a future paper.

4. Conclusions

Different samples of spherical cobalt particles with average diameter in the range 60 nm – 800 nm, have been synthesized by reduction of ions in liquid polyols. Being dispersed in silicone oil, these particles allowed us to carry out the first systematic study on the effect of particle size on the yield stress of MR suspensions. This effect has been shown to be almost negligible for average particle diameter higher than 100 nm. For smaller diameters, a significant decrement of the MR effect is observed. Brownian motion is probably not responsible for the observed behavior but rather the change of surface energy when an aggregate is broken and form two half aggregates of the same length. Besides the academic interest of this study, it has also been evidenced that nanometric particles have large potential applications in smart MR devices, especially those having narrow flow channels, such as active hydrostatic bearings for precision machinery [20].

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