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Synthesis and magnetorheology of suspensions of submicron-sized 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 measurements 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. The MR properties of these MR fluids were investigated. 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; a theory based on the change of the aggregates' shape with the size of the particles could explain these observations.

1. Introduction

Suspensions of colloidal magnetic particles are complex fluids that exhibit magnetic field-dependent rheological (flow) properties. This feature is known as magnetorheological (MR) effect. According to the size of the dispersed particles, these suspensions can be divided in: (i) ferrofluids (FF), which are dispersions of ferro- or ferrimagnetic nanoparticles (approx. 10 nm in diameter) in a carrier liquid (Odenbach 2006); and (ii) MR fluids, which are dispersions of micron-sized particles of magnetizable materials in a carrier liquid (Bossis *et al* 2002). Upon magnetic field application, particles of FF and MR fluids experience attractive magnetostatic forces, which lead to the formation of particle structures aligned in the field direction. The formation of these structures, which strengthen the suspension and oppose to the flow, is the basic phenomenon underlying the MR effect. 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:

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

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:

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$$\beta = \frac{\mu_p - \mu_f}{\mu_p + 2\mu_f} \quad (2)$$

μ_p being the relative permeability of the particles. Generally, particles of FF and MR fluids have large permeability and, therefore, $\beta \approx 1$. Thus, taken $T = 300$ K and $\mu_f = 1$, 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 $H = 130$ kA/m. Therefore, the Brownian forces normally dominate the magnetic forces in FF and, consequently, they only exhibit weak magnetorheological effect (Shahnazian and Odenbach 2007, Zubarev and Iskakova 2006a). 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. 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* (1995) reported some experimental data on the influence of particle size on the MR properties of suspensions of magnetisable particles. These authors measured the shear stress as a function of the shear rate for three different suspensions' sizes, 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, respectively. They found that, for a given shear rate, the apparent shear stress slightly increased with the diameter of the particles. In the micron range, Bombard *et al* (2002) investigated the effect of polydispersity on the yield stress of MR suspensions based on different carbonyl iron powders but their results are not conclusive due to the different magnetic susceptibilities of the suspensions. For iron particles having mean sizes of 1.8 and 6.7 microns, Trendler and Bose (2005) found a higher yield stress for the biggest particles. Rosenfeld *et al* (2002) and Wereley *et al* (2006) investigated the MR properties of suspensions of iron particles based on two different diameters :30 microns and 26 nm respectively. These authors 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. Finally, some investigation of the effect of particle sizes were conducted on suspensions of non magnetic particles in a ferrofluid by de Gans *et al* (2000) and Saldivar-Guerrero *et al* (2006) They found an important effect of particle size on these properties for particle diameters of the order of 100 nm. but due to the low permeability of the ferrofluid the magnetic forces are much lower than in classical MR suspensions and Brownian motion could still be efficient to break the chains of particles (Lemaire *et al* 1995).

The difficultness of performing a rigorous experimental study about the influence of particle size on the MR properties lies in obtaining monodisperse spherical particles with different average particle diameter. The aim of the present work is to rigorously analyze this dependence for particle diameters in the range 50 nm to 1 μm . 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

2.1. Materials

The cobalt precursors used in the synthesis of the particles were cobalt(II) hydroxide ($\text{Co}(\text{OH})_2$; Sigma-Aldrich, technical grade) and cobalt(II) acetate tetrahydrate ($\text{Co}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$; Sigma-Aldrich, reagent grade); potassium tetrachloroplatinate(II) (K_2PtCl_4 ; Sigma-Aldrich, purity 99.99%) and silver nitrate (AgNO_3 ; VWR Prolabo, Rectapur[®]) were used as nucleating agents; the liquid polyols used for the reduction of the cobalt ions were diethylene glycol (DEG) ($(\text{HOCH}_2\text{CH}_2)_2\text{O}$; Sigma-Aldrich, ReagentPlus[®]) and 1,2-propanediol (12PD) ($\text{CH}_3\text{CH}(\text{OH})\text{CH}_2\text{OH}$; Sigma-Aldrich, ReagentPlus[®]); sodium hydroxide (NaOH ; VWR Prolabo, Rectapur[®], pellets) was also used in the synthesis of the cobalt particles. For the preparation of the suspensions of cobalt particles, silicone oil (viscosity at 25 °C: 0.479

Pa-s; VWR Prolabo, Rhodorsil[®]) was used as carrier liquid and aluminum stearate ($\text{Al}(\text{C}_{18}\text{H}_{35}\text{O}_2)_3$; Sigma-Aldrich, technical grade) as dispersant.

2.2. Synthesis of cobalt particles

Reduction of metallic ions in a liquid polyol has been extensively used for the preparation of spherical particles of magnetic and non-magnetic materials (Fiévet 2000, Fiévet *et al* 1989, Sun *et al* 2005, Viau *et al* 1996). We used this method to synthesize spherical cobalt particles with different average particle diameter. The synthesis was carried out in the following way: (i) when K_2PtCl_4 was used as nucleating agent, appropriate amounts of the cobalt precursor, the nucleating agent and NaOH were mixed and dissolved in a liquid polyol. When AgNO_3 was used as nucleating agent, two different solutions in liquid polyol were prepared, one that contained the cobalt precursor and another that contained the nucleating agent; (ii) 250 mL of the solution that contained the cobalt precursor were poured into a 500 mL flask and bubbled with N_2 for 30 minutes; (iii) afterwards, this solution was brought to boiling point under continuous mechanical stirring; (iv) when AgNO_3 was used as nucleating agent, 10 minutes after boiling was reached 10 mL of the solution of AgNO_3 were injected into the flask's content; (v) after some time (reaction time), the reaction was stopped by cooling down the mixture to room temperature. During the reaction, the volatile products resulting from the oxidation of the polyol were distilled off, while the evaporated polyol was continuously refluxed. Finally, the black metal powder was taken by magnetic decantation, washed four times with ethanol, two more times with acetone, and then dried at $\sim 50^\circ\text{C}$. Reagent concentrations and other synthesis details are specified in table 1 for the different samples of cobalt powder. Note that other average diameters were obtained but we have chosen those with the narrow polydispersity in the range 60-600 nm, in order to minimize the overlap between different sizes.

Table 1. Synthesis details for the different samples of cobalt powder.

Sample name	Polyol	Concentration of NaOH	Cobalt precursor / concentration	Nucleating agent / concentration	Reaction time	Yield of synthesis
S1	DEG	0	$\text{Co}(\text{OH})_2 / 0.1 \text{ M}$	$\text{AgNO}_3 / 0.0007 \text{ M}$	4 h	86%
S2	DEG	0	$\text{Co}(\text{OH})_2 / 0.1 \text{ M}$	$\text{AgNO}_3 / 0.001 \text{ M}$	4 h	95%
S3	12PD	0.25 M	$\text{Co}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O} / 0.1 \text{ M}$	$\text{K}_2\text{PtCl}_4 / 0.001 \text{ M}$	4 h	90%
S4	12PD	0.25 M	$\text{Co}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O} / 0.1 \text{ M}$	$\text{K}_2\text{PtCl}_4 / 0.0026 \text{ M}$	1 h	95%

2.3. Preparation of the suspensions.

The synthesized cobalt powders were used for the preparation of MR suspensions (solid concentration 5 vol.%). These suspensions were prepared by dispersing appropriate amounts of cobalt powders in silicone oil. Based on discussions reported in a previous paper (López-López *et al* 2008), aluminum stearate was used as dispersant. A detailed description of the procedure followed to coat metallic particles with aluminum stearate and to prepare the final suspension can be found in López-López *et al* (2008).

2.4. Magnetorheology

The magnetorheological properties of the suspensions were measured using a Haake RS150 controlled stress rheometer. The measuring system geometry was a titanium plate–plate configuration of diameter 20 mm and gap 0.300 mm. Due to their diamagnetic nature, neither the plates nor the axe appreciably perturbed the magnetic field lines. The magnetic field was applied in the vertical direction using a non-commercial electromagnet, specially designed for its use in the above-mentioned rheometer. This electromagnet allows reaching fields up to 580 kA/m in the measuring gap –the radial distribution of the magnetic field in the measuring gap for a given electric current in the coil is shown in López-López *et al* (2009). Rheological experiments were carried out as follows: (i) in order to ensure reproducible results,

suspensions were shaken using a vortex mixer, then immersed in an ultrasonic bath, and finally shaken again. Immediately afterwards, suspensions were placed in the measuring system and presheared for 30 s at a large shear rate in the absence of applied magnetic field; (ii) a magnetic field was applied during 30 s waiting time with no rate applied; (iii) suspensions were finally subjected to shear stress ramps, in the presence of the same magnetic field applied during the waiting time, and the corresponding shear rates and dynamic viscosities were measured.

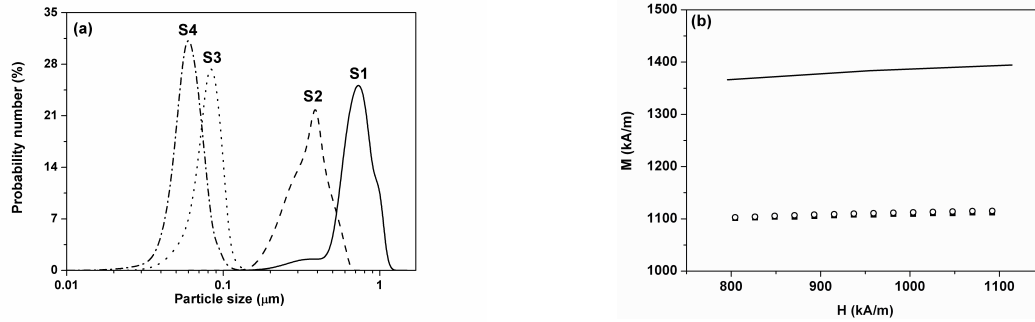


Figure 1. (a) Particle size distribution obtained by electron microscopy. (b) Magnetization at saturation for sample S1 (open circles), sample S4 (full squares) and for bulk cobalt (continuous line).

3. Results and discussion

3.1. Particle characterization

The synthesized powders consisted of relatively monodisperse spheres of submicrometer size, as inferred from electron microscopy observations (not shown here for brevity). Figure 1a shows the particle size distribution obtained by electron microscopy. Table 2 shows the average particle size obtained from electron microscopy and light scattering. As observed, there is a quite good agreement between the data obtained by electron microscopy and the data obtained by light scattering. Magnetometry measurements showed that the saturation magnetization of the powders was approximately the same for all particles sizes. As an example, the magnetization curve at saturation is shown in Figure 1b for the two extreme cases (samples S1 and S4). The magnetization of bulk cobalt is also included for comparison.

Table 2. Mean diameter (nm), measured by different techniques, of cobalt particles synthesized by reduction in polyols.

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

3.3. Magnetorheology

The steady-shear flow of suspensions containing 5 vol.% of cobalt powders of samples S1-S4 dispersed in silicone oil, was investigated as a function of the applied magnetic field. As an example, figure 2 shows the shear stress as a function of the shear rate for an applied magnetic field strength of 431 kA/m. As observed, for a given shear rate, the shear stress decreases as the average particle diameter is diminished. This decrement is negligible for the highest particle sizes, and becomes quite significant for average diameter smaller than 100 nm. Similar trends were observed for the other magnetic fields. Curves like those shown in figure 2 are characteristic of plastic behaviour and can be characterized by the Bingham equation:

$$\sigma = \sigma_y + \eta_{pl}\dot{\gamma} \quad (3)$$

Here σ is the shear stress, σ_y the dynamic (Bingham) yield stress, η_{pl} the plastic viscosity, and $\dot{\gamma}$ the shear rate. Alternatively, the MR effect of MR fluids can be characterized by the values of the static (frictional) yield stress. In this article, the static yield stress is identified with the value of the shear stress that corresponds to a shear rate of 0.1 s^{-1} .

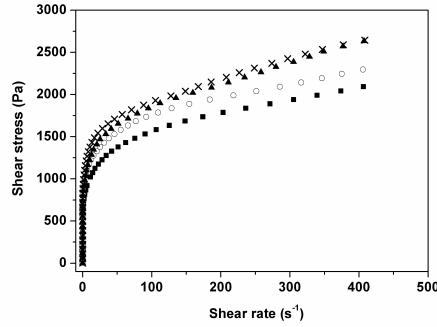


Figure 2. Shear stress plotted as a function of shear rate for suspensions containing 5 vol.% of cobalt particles under the presence of a magnetic field strength of 431 kA/m. Crosses: sample S1; full triangles: sample S2; open circles: sample S3; full squares: sample S4.

The values of the static and dynamic yield stress of suspensions containing 5 vol.% of cobalt powders (samples S1-S4) are plotted as a function of the magnetic field strength in figure 3.

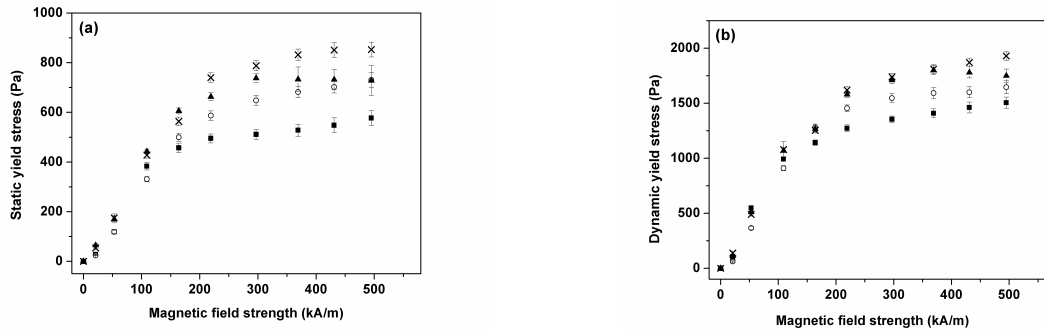


Figure 3. Yield stress plotted as a function of the magnetic field strength for suspensions containing 5 vol.% of cobalt particles. (a) Static yield stress; (b) dynamic yield stress. Crosses: sample S1; full triangles: sample S2; open circles: sample S3; full squares: sample S4.

As expected for MR fluids, the values of the yield stress showed by samples S1-S4 increase with magnetic field strength. In all cases, the values of the static yield stress are considerably smaller than the values of the dynamic yield stress, which indicates the existence of wall slip of the field-induced particle chains. Let us now focus on the comparison of the values of the yield stress for the different samples. As observed, both the static and dynamic yield stress decreases as the average particle diameter is diminished (see particle diameter in table 2). This decrement is negligible for the highest particles, and becomes quite significant for average diameter smaller than 100 nm. For example, taken as the reference the value of sample S1, at $H = 297 \text{ kA/m}$ the static and dynamic yield stress are respectively: 94 % and 98 % for sample S2; 82 % and 89 % for sample S3; 65 % and 78 % 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 (Lemaire *et al* 1995). 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 was developed by Zubarev and Iskakova (2006b) for ferrofluids and is transposed to the case of MR suspensions in the next section.

3.4. Theory

If Brownian motion does not explain the decrease of yield stress for $\lambda > 1000$ we have to consider an other explanation taking into account an external parameter which is the size of the structure induced by the field which depends both on the size of the cell and on the size of the particles through the surface energy of particles located on the surface of the aggregates. We shall use a theory previously developed to predict the size of the aggregates in the presence of a magnetic field (Grasselli *et al* 1994). Supposing a network of ellipsoidal aggregates aligned on the field direction, the magnetic energy per unit volume is given by:

$$U_t = -\frac{\phi}{\phi_a} \frac{H_0^2}{8\pi} \left[\frac{\mu_a - 1}{1 + n_z(\mu_a - 1)} \right] \left[\frac{1 - n_\sigma}{1 + n_r(1 - n_\sigma)} \right] \quad (4)$$

where n_z is the usual demagnetization factor:

$$n_z = \frac{(1 - e^2)}{2e^3} \left[\ln \left(\frac{1 + e}{1 - e} \right) - 2e \right] \quad (5)$$

with e the eccentricity of the ellipsoid defined by: $e = \sqrt{1 - c^2}$ and c is the aspect ratio of the ellipsoid. The quantity n_r is a demagnetization factor expressing the decrease of the field induced by the presence of the other aggregates around the reference one and given by (Grasselli *et al* 1994):

$$n_r = \left[\frac{(\mu_a - 1)}{1 + n_z(\mu_a - 1)} \right] \frac{\phi}{\phi_a} \left[\left(1 + \frac{\phi_a c^2}{\phi} \right)^{1/2} - \frac{c}{2} \left(\frac{2\phi_a}{3\phi} \right)^{1/2} \right] \quad (6)$$

Finally, the quantity n_σ expresses a decrease of the Lorentz magnetic field on the particles which are on the surface of the aggregates and is given by:

$$n_\sigma = \frac{\pi}{3\sqrt{3}} a \frac{S_e}{V_e} \frac{\mu_p - 1}{\mu_p + 2} \quad (7)$$

The permeability μ_p of the cobalt particles depends on the magnetic field. We have measured the saturation magnetization of the particles which was equal to $M_s = 1110 \pm 5$ kA/m for all sizes. The initial susceptibility χ_i of bulk cobalt is taken equal to 70 (Brown 1958) and the overall magnetization curve is represented by a Fröhlich–Kennelly law:

$$\mu_p(H_0) = 1 + \frac{M_s}{M_s \chi_i^{-1} + H_0} \quad (8)$$

It is worth noting that the value of n_σ is not sensitive to the initial permeability as long as it is much larger than unity. The permeability μ_a , appearing in U_t and n_r , is the permeability of an aggregate of internal volume fraction ϕ_a . We have taken also the Fröhlich–Kennelly law but with the initial permeability and saturation magnetization of a compacted powder with $\phi = 0.7$, assuming that the volume fraction, ϕ_a , inside the aggregates is close to this value. The aspect ratio, c , of the ellipsoid can be obtained for a given applied field, H_0 , and a given radius of the cobalt particles, by a minimization of the energy U_t . We find in particular that the aspect ratio decreases when the size of the nanoparticles increases. It comes from the

fact that large particles increase the surface energy (which is proportional to a) and so the number of aggregates (for a given length of the aggregates which is equal to the distance between the plates of the rheometer) has to decrease. The final result is then an increase of the thickness of the aggregates.

Now we have to relate the aspect ratio of the aggregates to the yield stress. Firstly we write that the shear stress is the derivative of the magnetic energy U_t versus the strain γ with $U_t(\gamma)$ given by (Bossis *et al* (1997):

$$U_t(\gamma) = -0.5M_z(\gamma)H_0 \frac{\phi}{\phi_a} \quad (9)$$

with

$$M_z(\gamma) = \mu_0 H_0 \chi_a \left[\left(\frac{1}{1+\gamma^2} \right) \frac{1}{(1+\chi_a n_z)} + \left(\frac{\gamma^2}{1+\gamma^2} \right) \frac{1}{1+\chi_a n_x} \right] \quad (10)$$

In this expression, n_z and $n_x = (1-n_z)/2$ are the depolarization factors, which depend on the aspect ratio, c , of the ellipsoid, which was previously calculated for ellipsoids aligned with the field. To take into account the constancy of the volume of the aggregate during strain we write $c(\gamma) = c(1+\gamma^2)^{3/2}$; but we did not try to find $c(\gamma)$ by a minimization of the energy for every γ because, as we are going to see, the values of γ remain quite small.

The last step is to consider the fact that the aggregates can break before the strain for which the stress reached its maximum. This happens if the structure made of aligned aggregates with their thickness divided by two (or equivalently $c'=2c$) has a lower energy that the strained initial structure. We call this rupture strain γ_r . In practice we find that it is always the case and that γ_r is quite small (between 0.07 and 0.12 depending on the size of the particles). In figure 4 we have compared the predictions of this theory for the static yield stress to the experimental results for the two extreme sizes of 64 nm and 760 nm. The theory predicts values which are intermediate between the experimental curves obtained for static and dynamic yield stress. Taking into account that there is no free parameter in this theory, the agreement is rather good and, above all, it actually predicts an increasing difference in stresses between the two different sizes when the field increases, which was the more intriguing result. Actually this behaviour could not be explained by the introduction of Brownian motion since the larger is the field the smaller would be the influence of Brownian motion.

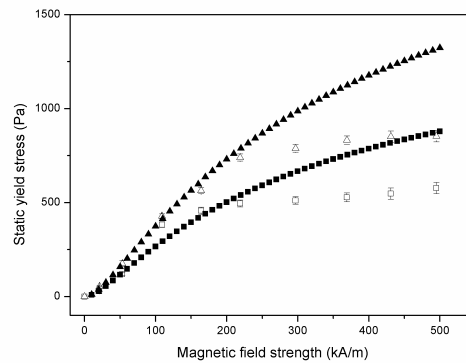


Figure 4. Comparison between theory and experiment: static yield stress plotted as a function of the magnetic field strength for suspensions containing 5 vol.% of cobalt particles (triangles: sample S1; squares: sample S4). Open symbols correspond to the experimental values and full symbols to the theoretical values.

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. Average particle size can be controlled by the type and concentration of the nucleating agent. The synthesized particles have been used for the preparation of MR fluids. Rheological measurements have shown that the effect of particle size on the MR effect is small for average particle diameter higher than 100 nm. For smaller diameters, a significant decrement of the MR effect is observed which increases with the magnetic field amplitude and this behavior can't be explained by Brownian motion. We have proposed an explanation of this behaviour by considering the effect of surface energy on the aspect ratio of the aggregates formed in a magnetic field. Besides the academic interest of this study, it appears that a MR fluid with a diameter of particle between 50 and 100nm would have large potential applications, especially when abrasion is an important problem such as active hydrostatic bearings for precision machinery (Ochonski 2005).

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