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Magnetorheology for suspensions of solid particles dispersed in ferrofluids

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Abstract

In this work, the magnetorheological properties of suspensions of micron-sized iron particles dispersed in magnetite ferrofluids were studied. With this aim, the flow properties of the suspensions in the steady-state regime were investigated using a commercial magnetorheometer with a parallel-plate measuring cell. The effect of both magnetite and iron concentration on the magnitude of the yield stress was studied for a broad range of magnetic fields. In addition, the experimental values of the yield stress were compared with the predictions from the chain model. With this purpose the values of the yield stress were obtained by means of finite element calculations. Interestingly, it was found that the experimental yield stress increases with the concentration of magnetite nanoparticles in the ferrofluid. Unfortunately, this behaviour is not obtained from calculations based on the chain model, which predict just the opposite trend.

1. Introduction

Magnetic suspensions are complex fluids which exhibit the remarkable property of changing their rheological properties under application of an external magnetic field (Phulé and Ginder 1998). They can be divided into (i) ferrofluids (FF), which are stable colloidal dispersions of ferromagnetic or ferrimagnetic nanoparticles in a liquid carrier (Charles 2002), and (ii) magnetorheological (MR) fluids which are dispersions of micron-sized magnetic particles. Because of their size (∼10 nm) the particles of a FF are single domain and possess a permanent magnetic dipole moment. This fact implies that even under application of high magnetic fields, FF only exhibit small changes in their viscosity and do not develop a yield stress (Rosensweig 1987, 1988, Odenbach 2003). On the other hand, the particles of a MR fluid are magnetically multidomain and the application of a magnetic field induces magnetic dipoles in each particle and strong interparticle interactions, which leads to the formation of a network of particles or aggregates throughout the suspension. As a consequence, MR fluids exhibit a reversible transition from a liquid behaviour to a solid one upon application of an external magnetic field. This transition is characterized by the appearance of high yield stresses and changes in the viscosity of several orders of magnitude (Ginder 1998, Bossis *et al* 2002b).

Due to their relatively low magnetoviscous response, the technological applications of FF are limited to those where only small changes in viscosity are required. That is, for example, the case for ink for magnetic printers. In contrast, the high magnetoviscous response of MR fluids enables them to be used in a wide range of technological applications (Ginder 1996, 1998). Nevertheless, there are still some problems that have to be overcome in order to take full advantage of these applications. These problems arise principally from the high density of the particles that compose a typical MR fluid, meaning MR fluids suffer from irreversible particle aggregation and settling (Ginder 1996). To solve these problems, different procedures have been attempted. Briefly, we can mention the addition of: (i) thixotropic agents (e.g. carbon fibres and silica nanoparticles) (Bossis *et al* 2002b, De Vicente *et al* 2003, Volkova *et al* 2000, López-López *et al* 2006); (ii) surfactants (e.g. oleic acid and stearic salts) (Bossis *et al* 2002b, Dang *et al* 2000, van Ewijk *et al* 1999, López-López *et al* 2005a, 2006); and (iii) the use of viscoplastic media or water-in-oil emulsions as continuous phases (Rankin *et al* 1999, Park *et al* 2001). Although some of these procedures have been proved as effective ways of reducing settling and aggregation, it also has been proved that the magnetic chaining responsible for the magnetoviscous effect can be progressively hindered, as the concentration of stabilizing agent increases (De Vicente *et al* 2003).

In a recent paper (López-López *et al* 2005b), it was demonstrated that the use of magnetite ferrofluids as carrier media is an effective way of reducing the sedimentation of the micron-sized particles of a MR fluid. The aim of this work is to investigate the effect of the magnetite nanoparticles on the rheological properties of extremely bimodal iron–magnetite suspensions. For this purpose the steady-shear flow was investigated and the corresponding yield stresses were obtained. The effect of magnetite concentration as well as the magnetic field strength was investigated. The validity of the chain model, that is, the formation of particle chains induced by magnetic interaction between iron particles (Martin and Anderson 1996, Bossis *et al* 2002b) was checked for these bimodal suspensions. In particular, the experimental results were contrasted with those obtained by means of the finite element method of calculation.

2. Experimental details

2.1. Materials

Ferrofluids were prepared as described in a previous work (López-López *et al* 2005c). Briefly, a chemical coprecipitation was performed to obtain magnetite nanoparticles. These nanoparticles were covered by chemisorbed oleate ions. When the carrier is a non-polar liquid with dielectric constant $\varepsilon_r < 5$, the oleate ion is found to be an excellent steric stabilizing agent (López-López *et al* 2005c), which avoids the aggregation of the magnetite particles by van der Waals forces. In the present work, the dispersing medium was kerosene (Sigma-Aldrich, Germany) with viscosity 16.6 ± 1.2 mPa s. The average diameter of the magnetite particles was 7.8 ± 0.3 nm.

Iron powder, obtained from carbonyl iron precursors, was supplied by BASF (Germany) and used without further treatment. The manufacturer indicates the following specifications: (i) chemical composition (wt%): minimum 97.5% Fe, 0.7–1.0% C; 0.7–1.0% N; 0.3–0.5% O; and (ii) density: minimum 7.5 g cm^{-3}. Our scanning electron microscopy (SEM) pictures show that iron particles are spherical and polydisperse, with an average diameter 930 ± 330 nm. Besides, the initial magnetic permeability of these particles is 132 ± 9 (López-López *et al* 2005b). This iron powder was dispersed in either magnetite/kerosene ferrofluids or kerosene.

2.2. Preparation of the suspensions

The suspensions were prepared by mechanical and ultrasonic mixing of iron particles in either ferrofluids or kerosene as described previously (López-López *et al* 2005b). The iron volume fraction Φ, in the so-obtained suspensions, ranged from 10 to 42.4% while the magnetite volume fraction ϕ ranged from 0 to 21.6%. After the suspensions reached the desired homogeneity, 110 μl of them were immediately placed in the measuring system of the magnetorheometer.

2.3. Magnetic properties of the ferrofluids

The magnetization, M, of the ferrofluids used in this work as dispersing media was measured at 20 °C as a function of the magnetic field strength, H, in a Manics DSM-8 magnetosusceptometer (France).

2.4. Magnetorheological properties

Magnetorheological properties of the suspensions were measured at a temperature of 25.0 ± 0.1 °C in a controlled rate magnetorheometer (MCR 300 Physica-Anton Paar, Austria). The measuring system geometry was a 0.02 m diameter parallel-plate set for a gap width of 0.35 mm. The rheological quantities reported are those at the outer radial edge of the plate.

The steady-state regime—viscometry—was investigated as follows: samples were subjected to a shear rate ramp and the corresponding shear stress was measured in suspensions with different iron and magnetite volume fractions in order to obtain the yield stress. To ensure reproducible results, and taking into account the time-dependent behaviour of any suspension, a precise time control is needed in the rheological experiments. For this purpose, samples were placed between the parallel plates and initially presheared for 30 s at a large enough shear rate in the postyield regime and at zero magnetic field strength to ensure a uniform distribution of particles and reproducible initial conditions. Then, an external magnetic field (up to $H_0 = 343$ kA m^{-1}) was applied during a 30 s waiting time with no rate applied. Finally, rheological measurements were started in the presence of the same external magnetic field strength as was used during the waiting time. The elapsed time between successive steps in shear rate ramps was always 2 s.

3. Results and discussion

3.1. Magnetic properties of the suspensions

As an example, figure 1 shows the magnetization curve of ferrofluids containing 3, 12 and 24% magnetite volume fraction. In this figure, the lines correspond to the best fits to the Fröhlich–Kennely law (Jiles 1991):

$$\chi(H) = \frac{M_s}{M_s/\chi_{\text{init}} + H}, \tag{1}$$

where χ is the magnetic susceptibility, χ_{init} is the value of the magnetic susceptibility at $H \to 0$ and M_s is the saturation magnetization of the ferrofluid. The values of χ_{init} and M_s corresponding to the best fits are shown in table 1.

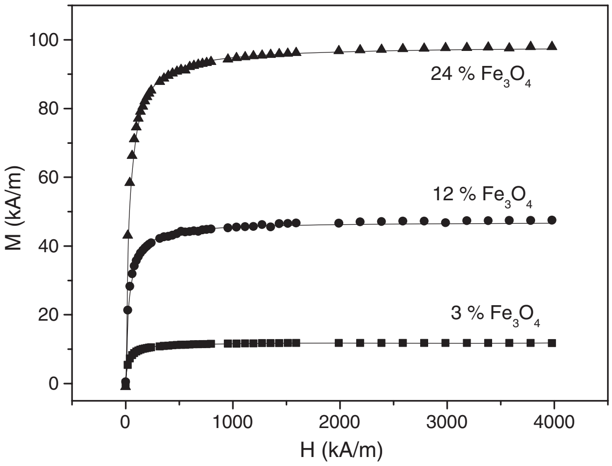

Figure 1. Magnetization data for ferrofluids containing the indicated magnetite volume fractions. The lines correspond to the best fits to the Fröhlich–Kennely law (equation (1)).

Table 1. Values of χ_{init} and M_s obtained by fitting the experimental data of figure 1 to the Fröhlich–Kennely law (equation (1)).

Fe_3O_4 volume fraction (%)	χ_{init}	M_s (kA m^{-1})
3	0.413 ± 0.008	11.925 ± 0.022
12	1.42 ± 0.05	47.01 ± 0.16
24	3.73 ± 0.08	98.24 ± 0.24

Using the values in table 1, and taking into account that a ferrofluid can be considered as a hydrodynamically and magnetically continuous medium (De Gans 2000), the susceptibility of the iron/ferrofluid suspensions can be calculated using the Maxwell-Garnett theory (Garnett 1904):

$$\mu_r = \mu_{r,m}\frac{1+2\Phi\beta}{1-\Phi\beta}; \qquad \beta \equiv \frac{\mu_{r,p}-\mu_{r,m}}{\mu_{r,p}+2\mu_{r,m}}; \tag{2}$$

where μ_r is the relative permeability of the iron/ferrofluid suspension, $\mu_{r,m}$ the relative permeability of the continuous medium (ferrofluid in this case), $\mu_{r,p}$ the relative permeability of the iron particles, and β is the so-called magnetic contrast factor. We need this permeability to calculate the mean field, H, inside the cell which is well represented by $H = H_0/\mu_r(H)$. Actually the permeability of the suspension will depend on the field, not only because of the dependence expressed by equation (1) but also due to the formation of alignment of particles in the presence of the field. This situation is not described by equation (2) which is valid only for an isotropic medium, so we have used a value obtained from a finite element simulation of a unit cell, composed of two half-particles in quasi-contact immersed in a ferrofluid whose permeability is given by equation (1). The use of periodic boundary conditions along the field axis amounts to considering infinite chains of particles. The average permeability obtained from this FEM calculation is reported in figure 2. Note that the values are significantly higher than the ones predicted by equation (2); for instance the FEM initial permeability of the suspension with 20.8% Fe and 21.6% Fe_3O_4 is about 10 whereas equation (2) would predict (taking $\beta = 1$) $\mu_r = 8.45$. In figures 4–9 it is the internal field $H = H_0/\mu(H)$ that is used. The values of H_0 are those indicated in the caption of figure 3.

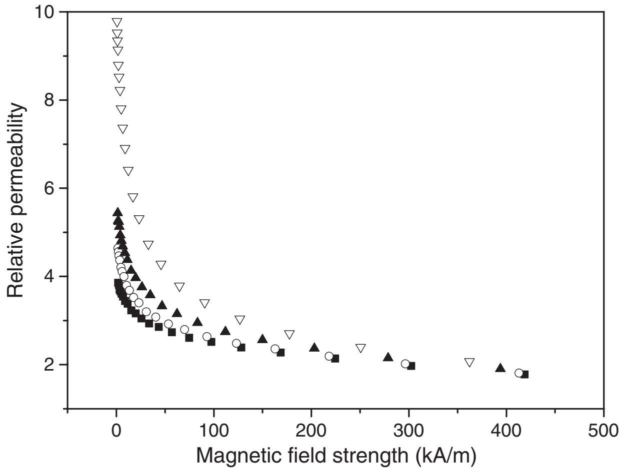

Figure 2. Relative permeability of suspensions as a function of magnetic field strength. ■: 20.8% Fe + 0% Fe_3O_4; ○: 20.8% Fe + 2.7% Fe_3O_4; ▲: 20.8% Fe + 10.8% Fe_3O_4; ▽: 20.8% Fe + 21.6% Fe_3O_4.

3.2. Magnetorheological effect

The steady-shear flow of samples containing different amounts of micron-size iron particles and magnetite nanoparticles (iron/ferrofluid suspensions) was investigated, both in the presence and in the absence of applied magnetic field. As an example, figure 3 shows the shear stress as a function of the shear rate for a suspension containing 10% iron volume fraction in kerosene (figure 3(a)) and for another suspension containing the same amount of iron and 21.6% magnetite volume fraction (figure 3(b)). As can be seen, both iron and iron–magnetite suspensions show a magnetorheological effect (that is, the application of a magnetic field provokes changes in the viscosity of several orders of magnitude and the appearance of yield stress). The transitions from solid-like (the state where the sample does not flow; values of shear stress smaller than the yield stress) to liquid-like (the state where the sample flows like a Newtonian liquid; values of shear stress larger than the yield stress) are different in the two systems. While in iron suspensions (see figure 3(a)) this transition is very sharp, it takes place in a step of shear rates of only 10 s^{-1}, in iron–magnetite suspensions (see figure 3(b)) the transition is completed after a step of 100 s^{-1}. It is important to note that the same difference was also observed for the other iron and iron/magnetite suspensions. This could be a consequence of the existence of complex iron–magnetite structures in these bimodal suspensions. The existence of complex particle structures in iron–magnetite suspensions was proved earlier (López-López *et al* 2005b).

It was demonstrated (López-López *et al* 2005b) that the use of magnetite ferrofluids as carrier media is an effective way of reducing the sedimentation of the micron-sized particles of a MR fluid. Now, the question that arises is that of how magnetite nanoparticles affect the magnetorheological effect of these extremely bimodal systems. *A priori*, it is difficult to predict the effect of the ferrofluid on the yield stress. On one hand, the force of interaction between two magnetic dipoles mediated by a fluid is proportional to the relative permeability of this fluid; this should increase the yield stress, but in dense suspensions we cannot represent the iron particles as point dipoles immersed in a ferrofluid. On the other hand the increase of permeability of the suspension decreases the average field in the suspension; that should

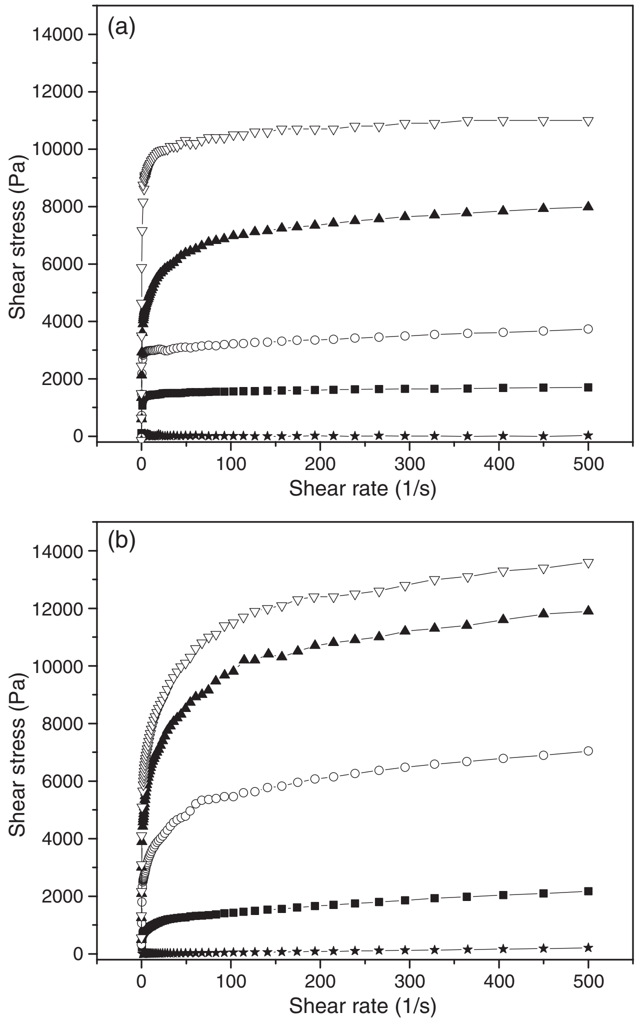

Figure 3. Shear stress as a function of shear rate for suspensions containing: (a) 10% iron volume fraction; (b) 10% iron volume fraction and 21.6% magnetite volume fraction. Magnetic field strength in air: ★: 0 kA m^{-1}; ■: 85.7 kA m^{-1}; ○: 171 kA m^{-1}; ▲: 257 kA m^{-1}; ▽: 343 kA m^{-1}.

decrease the yield stress. In practice some previous experimental results report an increase of yield stress (Chen *et al* 1998, Chin *et al* 2001, Ginder *et al* 1996). Nevertheless, in our system there are iron–magnetite structures (López-López *et al* 2005b). Therefore, a detailed analysis of the effect of magnetite addition in the values of the yield stress is worthy of attention.

Since dynamic yield stress gives a quantitative idea of the point where all internal structures in suspensions are broken (suspensions flow like Newtonian liquids), while static yield stress corresponds to the point where suspensions start to flow, this section will be devoted to the dynamic yield stresses. The values of the yield stress, obtained by fitting the Bingham model equation (Barnes *et al* 1998) to experimental curves like those shown in figure 3, are plotted as a function of the magnetic field strength inside the sample in figures 4–6. First, let us analyse the effect of magnetite addition on suspensions containing 10% of iron (figure 4). As can be

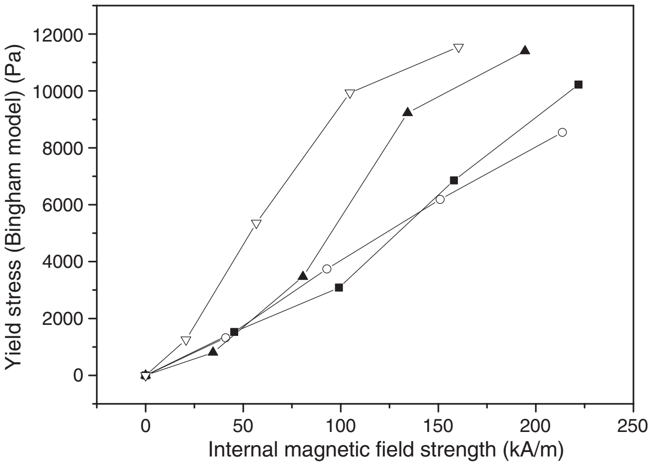

Figure 4. Yield stress (Bingham model) as a function of the internal magnetic field strength for suspensions containing 10% iron volume fraction and the following Fe_3O_4 volume fractions: ■: 0%; ○: 2.7%; ▲: 10.8%; ▽: 21.6%.

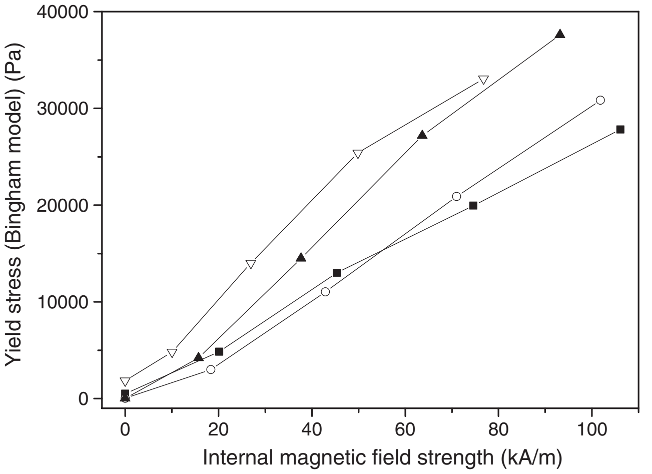

Figure 5. Same as figure 4 but for suspensions containing 31.6% iron volume fraction and the following Fe_3O_4 volume fractions: ■: 0%; ○: 2.7%; ▲: 10.8%; ▽: 21.6%.

observed, the addition of low magnetite content (2.7%) does not provoke important changes in the yield stress, as compared with the values obtained in the absence of magnetite. However, the addition of higher magnetite contents (10.8 and 21.6%) provokes a significant increment in the yield stress, especially at intermediate values of the magnetic field strength. Figure 5 shows the results for suspensions containing 31.6% iron volume fraction and different amounts of magnetite. As expected, the values of the yield stress are approximately three times higher than those obtained for suspensions containing 10% of iron—the chain model predicts a linear dependence of the yield stress with the concentration of multidomain magnetic particles (Bossis *et al* 2002b). As regards the effect of magnetite addition, trends similar to those obtained for suspensions containing 10% of iron are observed; that is, the addition of low magnetite content (2.7%) does not provoke significant changes in the yield stress, whereas it increases

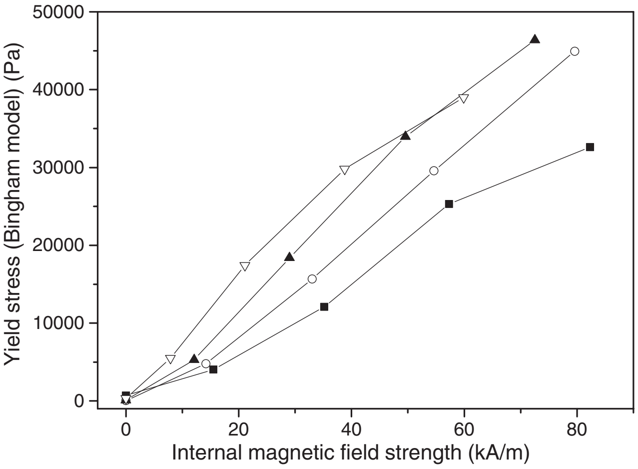

Figure 6. Similar to figure 4 but for suspensions containing 42.4% iron volume fraction and the following Fe_3O_4 volume fractions: ■: 0%; ○: 2.7%; ▲: 10.8%; ▽: 21.6%.

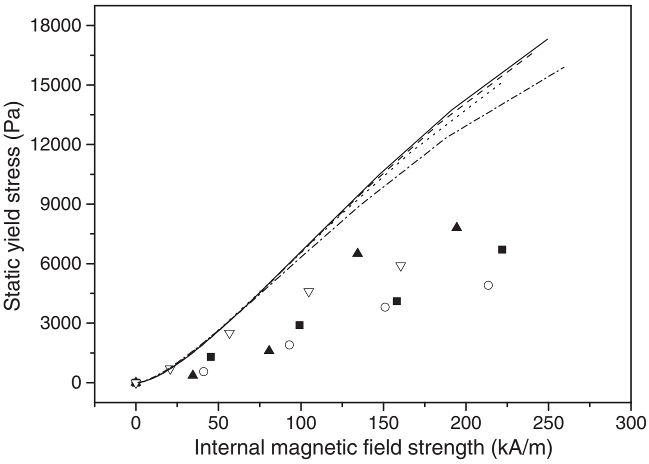

Figure 7. Static yield stress as a function of the internal magnetic field strength for suspensions containing 10% iron volume fraction and the following volume fractions of Fe_3O_4 (experimental/simulation): ■/——: 0%; ○ /- - - -: 2.7%; ▲/· · · · · ·: 10.8%; ▽ /— · —: 21.6%.

considerably at high magnetite content (10.8 and 21.6%). Finally, the results for the highest iron content (42.4%) are shown in figure 6. In this case, an important difference with respect to the previous set of suspensions (figures 4, 5) is observed: even the addition of low magnetite content provokes a significant increase of the yield stress, while for higher magnetite contents, the trends are similar to those obtained for 10 and 20.8% of iron.

As we shall see in the next section, the increase of magnetic permeability due to the addition of ferrofluid cannot explain the increase of dynamic yield stress with the volume fraction of ferrofluid. It seems quite surprising to find a noticeable increment of the yield stress of the suspension containing 42.4% of iron and 2.7% of magnetite with respect to the suspension containing only 42.4% of iron, whereas this difference does not exist for lower volume fractions of iron particles. An explanation could be that the ferrofluid plays the

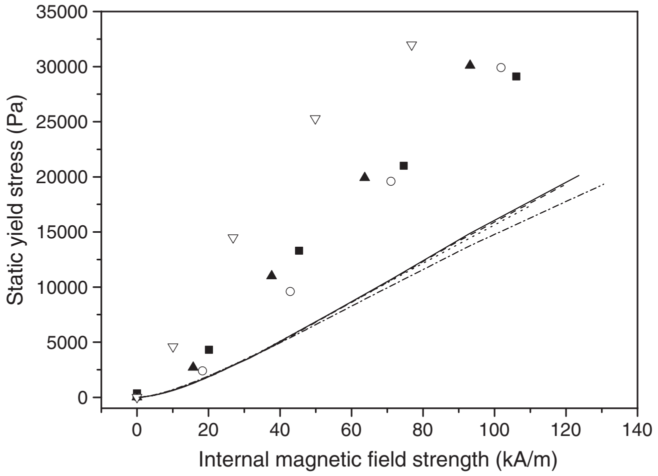

Figure 8. Same as figure 7 but for suspensions containing 31.6% iron volume fraction and the following volume fractions of Fe_3O_4 (experimental/simulation): ■ /——: 0%; ○ /- - - -: 2.7%; ▲ /· · · · · ·: 10.8%; ▽ /— · —: 21.6%.

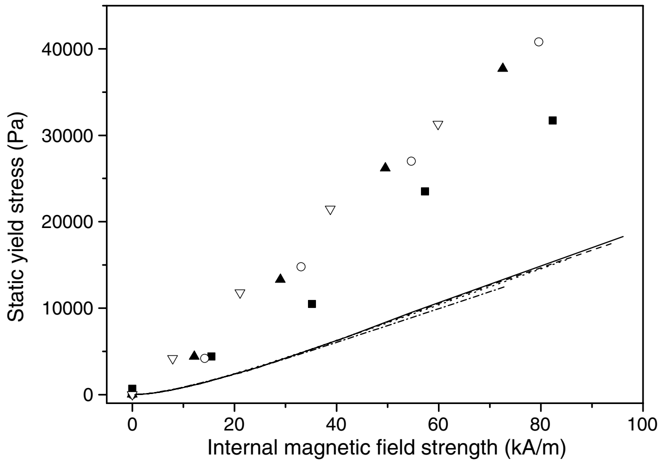

Figure 9. Same as figure 7 but for suspensions containing 42.4% iron volume fraction and the following volume fractions of Fe_3O_4 (experimental/simulation): ■ /——: 0%; ○ /- - - -: 2.7%; ▲ /· · · · · ·: 10.8%; ▽ /— · —: 21.6%.

role of a surfactant: in the absence of magnetite, iron particle aggregation is favoured by magnetic attraction, which results from the remnant magnetization of the iron particles, and van der Waals interaction (Phulé *et al* 1999). As a consequence of this aggregation, particle chaining in the presence of a field is rather hindered and, therefore, the magnetorheological effect is lowered. In contrast, irreversible iron aggregation is prevented in such systems when magnetite nanoparticles are present (López-López *et al* 2005b) and, therefore, the formation of chains induced by the application of a field is favoured. This mechanism must operate in all iron/magnetite suspensions. However, it is just for the highest iron content (42.4% iron; figure 6) that it is observable because it is especially at very high particle loading when irreversible particle aggregation becomes more dramatic.

3.3. Comparison with theoretical calculations

In order to contrast the experimental results with theory, we calculated the values of the yield stress predicted using the standard chain model (Ginder 1998, Bossis *et al* 2002b), applying the finite element method (FEM) calculation procedure to a cell formed of two half-spheres with periodic boundary conditions in order to represent infinite chains of spherical ferromagnetic particles (Bossis *et al* 2002a, 2003). Under shear, the chains are supposed to deform affinely with the strain, that is, the motion of the particles take place only along the velocity lines. Then, the maximum of the projection of the interparticle force on the direction of the shear corresponds to the rupture of the chain and the corresponding shear stress is the yield stress.

Notice that theory predicts the values of the static yield stress and, therefore, the results of our simulation must be compared with the values of the stress plateau obtained from *log–log* flow curves in the limit of zero shear rate. Figures 7–9 show the results. Firstly, it is worth mentioning that the trends obtained for the values of the experimental static yield stress are different to those obtained for Bingham (dynamic) yield stresses. For example, the values of the Bingham yield stress for the suspension containing 10% of iron and 21.6% of Fe_3O_4 are clearly higher than those for the suspension containing the same amount of iron and 10.8% of Fe_3O_4 (see figure 4), whereas this does not happen with the values of the static yield stress (see figure 7). The fact that the static yield stress is not very sensitive to the amount of ferrofluid is also well reproduced by the chain model of yield stress for the three volume fractions of iron (cf figures 7–9).

Secondly, it can be observed that at low iron content (10%; figure 7) the theory overestimates the values of the yield stress. In contrast, at high iron content (31.6 and 42.4%; figures 8 and 9) the simulation underestimates the yield stress. This is true also at zero ferrofluid content. Between 10% iron and 31.8% iron content, the chain model would predict an increase of yield stress by a factor of three, but experimentally there is a factor of ten. The existence of this disagreement between theory and experiment for iron suspensions at weak magnetic field was previously reported (Volkova *et al* 2000) and it is a first indication of the failure of the standard approach to predicting the magnitude of the yield stress. This is probably due to a reinforcement of contacts in multichain structures. More important is the fact that, in the presence of ferrofluids, the trends obtained by simulation are opposite to the experimental ones: whereas the experiments show a global tendency to an increase of yield stress as magnetite concentration increases, the simulation predicts the reverse behaviour. This is further evidence of the failure of the chain model for describing the magnetorheological effect in suspensions of micron-sized magnetic particles dispersed in ferrofluids. Actually it is difficult to tell whether the increase of yield stress is due to some bridge structure of magnetite particles between surfaces of iron particles or simply to the fact that the ferrofluid, by acting like a surfactant, prevents the formation of defects in column-like structures formed during the application of a field.

4. Conclusions

In this work, the magnetorheological response of suspensions of micron-sized magnetic particles dispersed in ferrofluids has been studied. It has been found that the values of Bingham yield stress increase with the concentration of solid nanoparticles in the ferrofluid carrier. This is not due to the increase of permeability of the suspension, as can be demonstrated using a chain model, but probably to the formation of heterogeneous iron–magnetite structures that avoid the irreversible aggregation between iron particles and facilitate field-induced chaining.

The experimental values of the static yield stress have been compared with those theoretically predicted by means of the standard chain model using finite element calculations. Here too, the increase of yield stress with the amount of ferrofluid is not predicted by the theory, showing that either the ferrofluid strengthens the structure by forming strong magnetite bridges or decreases the number of defects in chain structures. Microrheology using a small number of iron particles in a ferrofluid should help to answer this question and is planned for future work.

Acknowledgments

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