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ORIGINAL CONTRIBUTION

Shear and squeeze rheometry of suspensions of magnetic polymerized chains

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Abstract We present the first experimental results on the magnetorheology of suspensions of non-Brownian magnetic ellipsoidal particles. These particles are made of spherical iron particles linked by polymers and are called polymerized chains. Steady shear, oscillatory shear, and oscillatory squeeze rheological tests have been performed. The rheological properties of the suspension of polymerized chains have been compared with those of the suspension of spherical iron particles. In shear flow, both suspensions develop nearly the same yield stress, while in squeeze flow, the yield stress is several times higher for the suspension of polymerized chains. We show that the squeezing force of a suspension of spherical particles is an increasing function of the magnetic field intensity at low magnetic fields but decreases dramatically at higher fields. Surprisingly, this phenomenon, attributed to cavitation or air entrainment, does not occur in the suspension of polymerized chains.

Keywords Magnetorheological fluid · Fiber suspension · Shear flow · Squeezing

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Abbreviations
MR magnetorheological

Introduction

Magnetorheological damping devices have been broadly developed during the last decade, and nowadays, they are appearing on the market. Some of them work in a squeezed mode, where a magnetorheological (MR) fluid is squeezed by two moving surfaces and offers some viscoelastic resistance to the motion of these surfaces (Wang et al. 2006; Carmignani et al. 2006). The viscoelastic response of the MR fluid can be controlled by magnetic fields. A way to improve MR damping devices is to use novel MR fluids with enhanced MR properties. With this aim, we propose to use suspensions of magnetic fibers, obtained by chemical linking of micron-sized iron particles. From now, we will call these fibers polymerized chains. The synthesis and characterization of these polymerized chains are described in López-López et al. (2007). Because of the presence of thick polymer layers, the magnetization of the polymerized chains is two to three times smaller than the magnetization of the spherical iron particles. In the present work, we are interested in analyzing the rheological behavior of these new MR fluids. Note that besides polymerized chains, two other kinds of micron-sized magnetic fibers have recently been synthesized namely, magnetite microrods (Vereda et al. 2007) and cobalt microfibers (López-López et al. 2007).

Many studies have been carried out on shear and squeeze rheology of nonmagnetic fiber suspensions, helpful reviews being given by Petrie (1999) and Engmann et al. (2005). Shear rheology of classical MR fluids, composed of spherical magnetic particles, has also been studied intensively since the last 40 years of the last century (see for

instance Bossis et al. 2002). There are also some works on the squeeze flow of classical MR fluids under magnetic fields (Tang et al. 1998; Bashtovoi et al. 2002; See 2003; Viera et al. 2003). Detailed investigations of the oscillatory squeeze flow of electrorheological fluids (a physical analogue of MR fluids) were reported by See et al. (1999), Sproston et al. (1994), and El Wahed et al. (1999, 2000, 2003). These authors studied the effect of applied electric fields (DC and AC) on the waveforms of the output force signal. Unfortunately, the works cited above were performed for field-responsive suspensions composed of spherical particles. To our knowledge, there is no rheological study of MR suspensions based on nonspherical magnetic particles. In our paper, we report one of the first results on the magnetorheology of the suspension of polymerized chains, both in shear and in squeeze modes. Finally, we compare the rheological properties of these novel MR fluids with the properties of the classical MR fluids.

Experiments

In our experiments, we used two kinds of MR fluids. They were suspensions of magnetic microparticles in ethylene glycol (Prolabo) stabilized by a silica gel Aerosil 300. The shape, composition, and magnetic properties of the particles were the differences between these two MR fluids. The first MR fluid had spherical carbonyl iron particles (supplied by BASF) with diameters ranging between 0.5 and 3 μm. The second one had ellipsoid-like particles composed of the same spherical iron particles linked by a polymer (ethylene glycol methacrylate phosphate; López-López et al. 2007). The specific saturation magnetization of the polymerized chains was approximately three times smaller than that of the spherical iron particles (López-López et al. 2007). The average particle length and diameter were about 100 and 15 μm, respectively. Both types of suspension were prepared at 5 and 15% particle volume fractions. The sedimentation tests showed that all MR fluids remained well dispersed during at least 30 min, a time sufficient to carry out the rheological measurements—shear and squeeze tests.

The steady shear test was performed with a controlled-stress rotational rheometer Thermo Haake RS150 in a titanium cone-plate geometry with 35 mm in diameter and 2° in cone angle. A sketch of the measuring cell is shown in Fig. 1a. A magnetic field, with intensity ranging between 0 and 31 kA/m and direction perpendicular to the flow, was applied using a solenoid. Because of its diamagnetic nature, the cone did not appreciably perturb the magnetic field lines, which ensured the uniformity of field in the measuring gap. Before each measurement, the MR fluid was presheared in the absence of magnetic field at a constant shear stress of 150 Pa during 1 min. After that, a

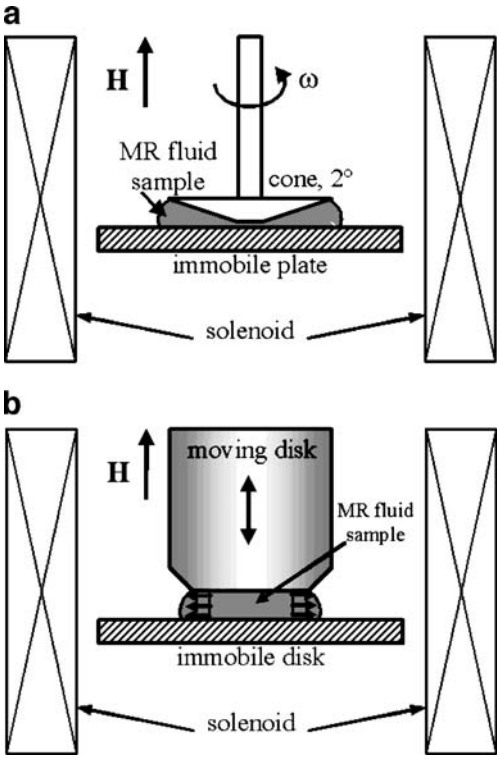

Fig. 1 Measuring cells for shear rheometry (**a**) and squeeze rheometry (**b**)

magnetic field was applied, and the MR fluid was left at rest during 30 s—a sufficient time for MR fluid structuring. Then, a shear stress ramp, with a 30-s interval between each stress value, was applied, and the corresponding shear rate was measured. In this manner, shear stress vs shear rate curves (flow curves) were recorded. First, a series of flow curves was recorded at increasing magnetic fields (from 0 to 31 kA/m), and then the flow curve was measured once again in the absence of magnetic field. We found the same results for zero field rheometry before and after magnetic field application. This confirms that particle sedimentation and particle migration are negligible during the experiments.

To study the linear viscoelastic behaviour of the MR fluids, shear oscillometry was performed using the same experimental setup (Fig. 1a). A sinusoidal shear stress was applied to the MR fluids; this stress provoked an oscillatory torsional deformation of the MR fluid sample, sandwiched between the cone and the plate, and the corresponding shear strain signal was measured by the rheometer. First, the linear domain of viscoelasticity was determined by applying a ramp of shear stress amplitudes, τ =0.01–100 Pa, at fixed frequency of oscillation f=25 Hz. This domain is characterized by a linear response of the shear strain amplitude vs the amplitude of the applied shear stress. Then, a frequency ramp, f=0.05–100 Hz, was applied at a fixed shear stress amplitude in the viscoelastic region, τ = 1 Pa, and the dynamic moduli G' and G'' were measured as a function of frequency. Shear oscillometry was performed

for both types of MR fluids at 5% particulate volume fraction and in the presence of a magnetic field of the intensity H=30.6 kA/m.

The oscillatory squeeze test was performed with a viscoanalyzer RAC815A (Metravib Instruments). A MR fluid was confined between two parallel nonmagnetic disks, as shown in Fig. 1b. The upper disk, driven by the viscoanalyzer, oscillated in the vertical direction with imposed amplitude of displacement and frequency. The MR fluid squeezed film created a viscoelastic response, which was transmitted to a force transducer connected to the fixed lower disk. The experiments consisted of measuring the squeezing force amplitude and the phase angle between the periodic force and the displacement signals in response to the imposed harmonic oscillations of the upper disk. In our experiments, the excitation frequencies f were 5, 15, 25, 35, and 45 Hz, the displacement amplitude Δh of the upper disk was 50 μm, the initial gap between the disks was h_0=0.5 mm, and the upper disk radius was R= 8.5 mm. These measurements were carried out both in the absence and in the presence of a uniform magnetic field (0–32 kA/m). The magnetic field, normal to the squeezing surfaces, was created by a solenoid placed around the disks.

To check whether the polymerized chains wore out under flow, two independent tests were performed: (1) Polymerized chains were observed in the microscope before and after the flow, and no important changes in their integrity were observed; (2) suspensions were subjected to shear ramps in the absence of the field before and after the tests, and identical curves were obtained. Therefore, it can be concluded that the polymerized chains did not wear out under flow.

Data analysis of the squeeze test

All the results of squeeze test are presented in the form of squeezing force amplitude vs frequency dependencies, like the one shown in Fig. 4. For both MR fluids, all these dependencies are perfectly approximated by a linear function F_0=F_Y+Af giving some nonzero force amplitude F_Y when interpolated to zero frequency. Furthermore, the phase lag angle between force and displacement signals lies between 85 and 90° for any measurement, which is characteristic for a weak elastic response. Such linear behavior of the squeezing force amplitude possessing a yield value F_Y depending on the magnetic field strength allows us to assume a Bingham model of MR fluids in squeeze flow: $\tau = \tau_Y\, sgn(\dot{\gamma}) + \eta \cdot \dot{\gamma}$ with τ_Y and η being the MR fluid yield stress and plastic viscosity, respectively. To extract the yield stress and the plastic viscosity from these data, we must consider a fluid mechanics problem of a Bingham fluid flow between two parallel disks. A good first-order approximation to this problem is given in the classical work of Covey and Stanmore (1981). Using lubrication approximation, they obtained the following expression for the squeezing force F as a function of the upper disk speed $\dot{h}$:

$$F = -\frac{\pi\tau_Y R^3}{h}\, sgn(\dot{h}) - \frac{3\pi\eta\dot{h}R^4}{2h^3} \tag{1}$$

This formula derived for weak Bingham numbers $Bn = \frac{\tau_Y h}{\eta\cdot\dot{h}} << 1$ can be applied safely for a very wide range of Bingham numbers upon introduction of appropriate correction factors in both terms of Eq. 1 (Matsoukas and Mitsoulis 2003; Engmann et al. 2005). We estimated that the formula 1 can be applied to our experiments with a 14% error, acceptable for the semiquantitative analysis.

Let the upper disk oscillate with a frequency f such that the MR fluid film thickness varies as $h = h_0 + \Delta h\cos(2\pi ft)$, and therefore, the upper disk velocity is $\dot{h} = -2\pi\,\Delta h f \sin(2\pi ft)$. Substituting these expressions into Eq. 1 and taking into account that $\Delta h << h_0$, we arrive to the final expression for the squeezing force amplitude as a function of the excitation frequency:

$$F_0 = \frac{\pi\tau_Y R^3}{h_0} + \frac{3\pi^2\eta\,\Delta h R^4}{h_0^3} f = F_Y + Af \tag{2}$$

The first term in Eq. 2 represents the yield force F_Y corresponding to the dynamic yield stress, and the second one corresponds to the Newtonian term of the Bingham law. Therefore, once the values of F_Y and A were obtained by a linear fit of the experimental data, we calculated the yield stress τ_Y and the plastic viscosity η from the formula 2 by putting the first term equal to F_Y and the second one to (Af).

To validate our experimental technique, we performed measurements on two calibrated Newtonian liquids and found that their viscosity measured by the oscillatory squeeze test corresponded to the one obtained using standard rheological measurements.

Results and discussion

The shear oscillometry results are presented in Fig. 2 in the form of the frequency dependence of the MR fluids' storage and loss moduli at 5% particulate volume fraction and at a magnetic field intensity H=30.6 kA/m. As is seen in this figure, the suspension of spherical particles has stronger viscoelastic properties than the suspension of polymerized chains. This should be connected with the lower magnetization and, therefore, MR response of the polymerized chains. For both suspensions, the storage modulus G' is found to be larger than the loss modulus G'', indicating that the behavior of both MR fluids is closer to solid than to liquid. This is not surprising if we recall that the applied shear stress τ=1 Pa is much lower than the yield stress of

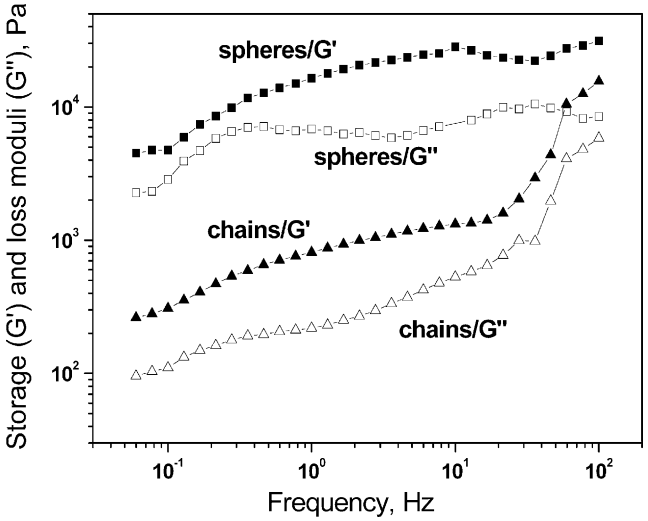

Fig. 2 Frequency dependence of the dynamic moduli of MR fluids for 5% particulate volume fraction and at a magnetic field intensity H=30.6 kA/m

the MR suspensions. The complicated shape of the curves $G'(f)$ and $G''(f)$ (Fig. 2) suggests that the MR fluids do not follow a Maxwellian solid behavior and possess, at least, a few relaxation times. To estimate the relaxation time, creep experiments were performed in the linear viscoelastic domain by applying an instantaneous jump of the steady shear stress from 0 to 1 Pa and recording the shear strain vs time at the same conditions (H=30.6 kA/m, ϕ=5%). The relaxation times were estimated from these measurements by fitting the experimental data with a biexponential function, in the way described by Jolly et al. (1999). The shortest time was found to be around 30 and 200 s for the MR fluids composed of spherical particles and polymerized chains, respectively. The possible reason for such a difference in relaxation time is the solid friction between the polymerized chains that slows down their response to variations in the applied mechanical stress.

The MR fluid flow curves of the shear rheometry are presented in Fig. 3a for the suspension of spherical iron

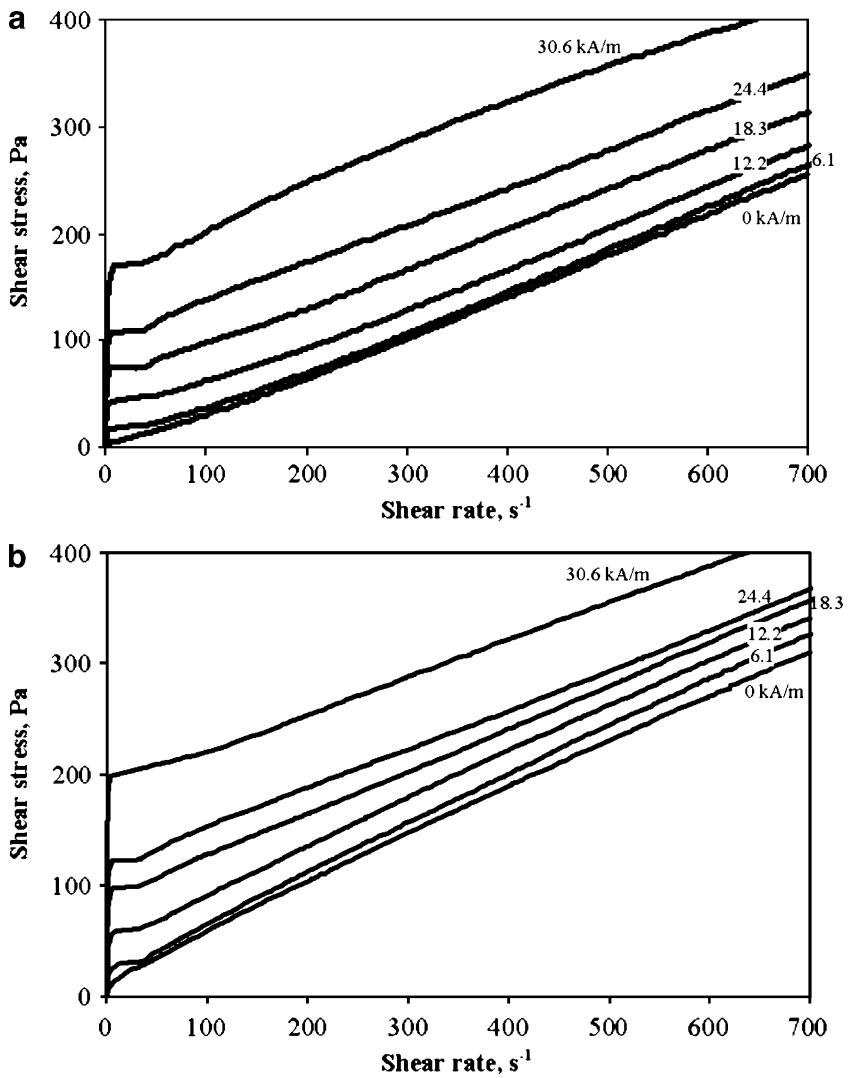

Fig. 3 Flow curves of the MR suspension of spherical iron particles (**a**) and of polymerized chains (**b**) at 15% volume fraction

particles and Fig. 3b for the suspension of polymerized chains, both at the same volume fraction of 15%. As expected, both suspensions experience solid-to-liquid transition in the presence of a magnetic field and possess a yield stress. We performed a linear fit of the flow curves (Bingham model) in the domain of the shear rates $\dot{\gamma}>$ 200 s^{-1}, and we calculated the dynamic yield stress as the shear stress interpolation to zero shear rate. The magnetic field effect on the shear yield stress of both MR fluids is illustrated in Fig. 5 and will be discussed below in comparison with the yield stress obtained in squeeze rheometry.

The force vs excitation frequency curves of the squeeze rheometry, being somewhat analogous to the flow curves of the shear rheometry, are presented in Fig. 4a and b for the suspensions of spherical particles and polymerized chains, still at the same volume fraction of 15%. As stated above, all these curves are linear and suggest a Bingham behavior of the MR fluids in the experimental range of parameters. As observed in Fig. 4, the squeeze flow curves are shifted upward with increasing magnetic field, signifying a viscosity increase of MR fluid. For the suspension of spherical particles, this tendency reverses at a critical value of the magnetic field ($H\approx$14 kA/m), and a strong decrease in force is observed for higher magnetic fields (Fig. 4a). On the other hand, such force decrease is not observed for the suspension of polymerized chains even at higher fields (Fig. 4b). This phenomenon is connected with cavitation and will be discussed in detail below. Note that both, input (displacement) and output (transmitted force) signals, were sinusoidal and did not possess significant higher harmonics. In contrast to our observations, See at al. (1999) and Sproston et al. (1994) observed flat, close-to-rectangular waveforms of the output force of a squeezed electro-

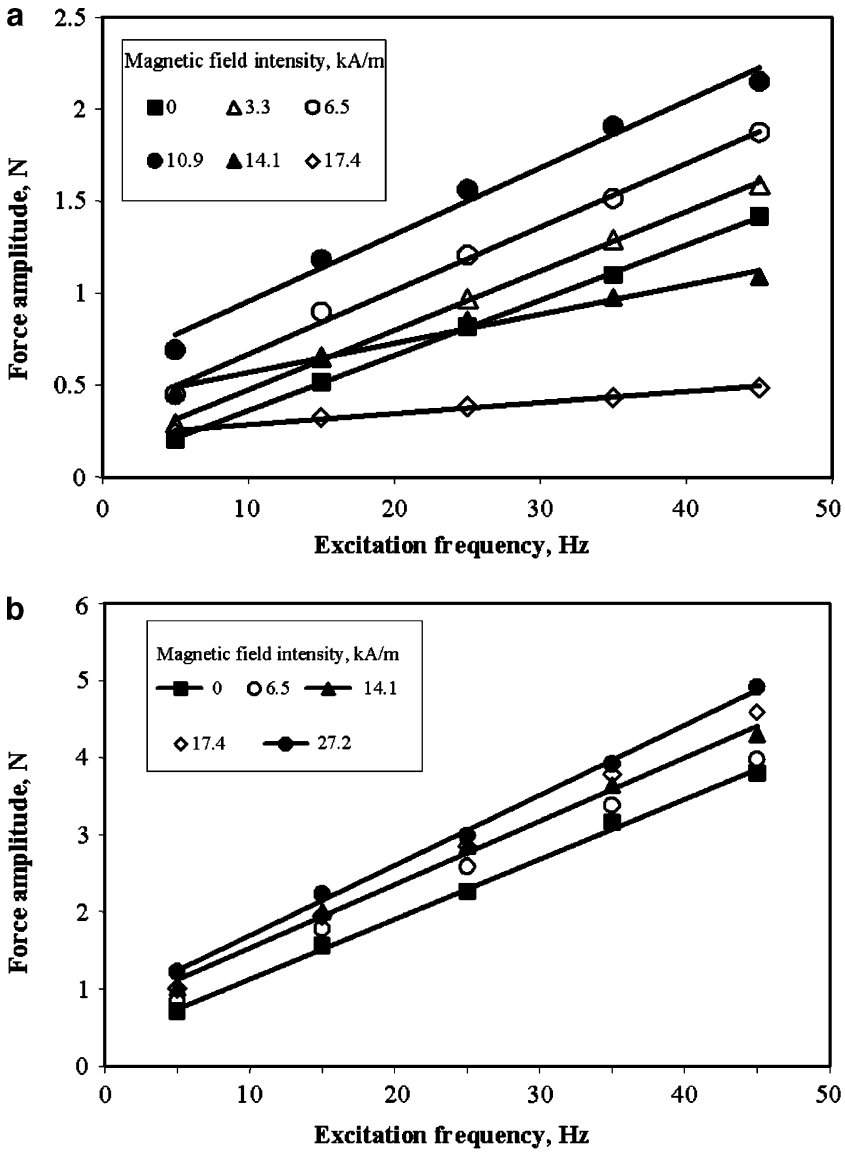

Fig. 4 Squeezing force amplitude vs excitation frequency for the suspension of spherical iron particles (**a**) and for the suspension of polymerized chains (**b**), at 15% volume fraction. The displacement amplitude is Δh=50 μm, the initial gap is h_0=0.5 mm

rheological fluid and explained it in terms of a biviscous model of a Bingham fluid. We suppose that higher harmonics of the output force are connected to the nonlinear terms of the force vs disk velocity dependence, Eq. 1, which appear at high yield stress or high Bingham numbers. In our case, the magnetic field is not strong enough to generate such nonsinusoidal response of the transmitted force.

The flow curves of shear and squeeze rheometry for 5% volume fraction MR suspensions are similar to the flow curves at 15% and are not presented here for brevity. At this point, it is important to remark that we verified our squeeze rheometry results for a range of deformations, ε=0.05–0.2, corresponding to a displacement amplitude Δh=25–100 μm. We found that for both types of MR fluids, the yield force F_Y and consequently the yield stress do not depend, within the experimental error, on the deformation applied.

The main result of our study—the magnetic field effect on the dynamic yield stress of both types of suspension—is presented in Fig. 5. In this figure, we compare at the same time the MR effect for both types of MR fluids and the yield stress obtained by means of two different methods: shear rheometry and squeeze rheometry.

In shear rheometry, we observe a parabolic-like growth of the dynamic yield stress with the magnetic field intensity for both types of MR fluids. We note that the dynamic yield stress of the dilute suspension (ϕ=5%) of spherical particles is higher than that of the equivalent suspension of polymerized chains. For the most concentrated suspension (ϕ=15%), it is the suspension of chain particles that offers the highest yield stress. However, for both concentrations, the yield stress of the suspension of spherical particles experiences a more rapid growth with the magnetic field. The higher yield stress of the concentrated suspension of polymerized chains (ϕ=15%) is associated with a signifi-

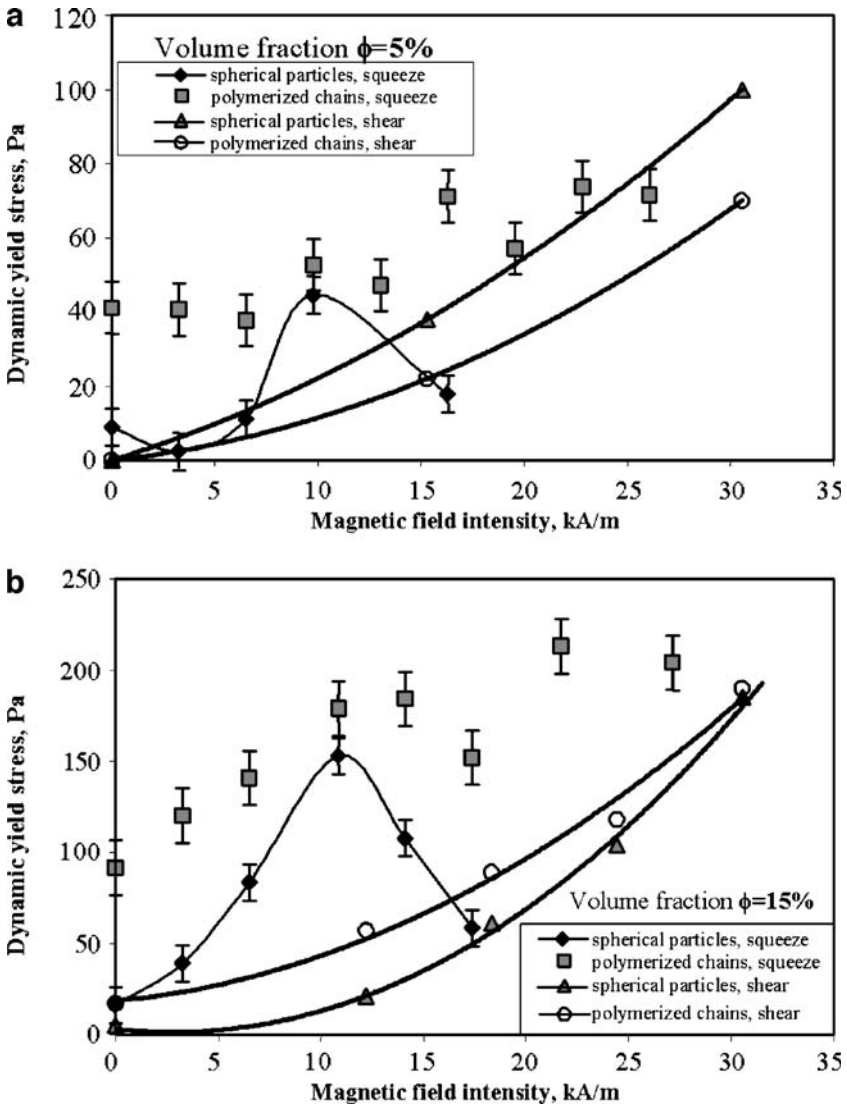

Fig. 5 Field dependence of the dynamic yield stress of suspensions of spherical particles and polymerized chains at 5 (**a**) and 15% volume fraction (**b**): comparison between shear and squeeze rheometry. In squeeze tests, the displacement amplitude is Δh=50 μm, the initial gap is h_0=0.5 mm

cant initial yield stress $\tau_Y \approx 20$ Pa in the absence of magnetic field. This yield stress can be explained by a solid friction between the chains in shear deformation that can appear even at apparently low chain volume fractions (Servais et al. 1999). On the contrary, there is no significant yield stress for the suspension of spherical particles at zero magnetic field.

In squeeze rheometry, we observe a rather high dynamic yield stress for suspensions of polymerized chains in the absence of magnetic field (45 Pa for ϕ=5% and 80 Pa for ϕ=15%). This dynamic yield stress is much higher than the one observed in shear rheometry. This suggests that the resistance of chain particle suspensions to squeeze deformation is much higher than to shear deformation. Similar to shear rheometry, we also observe an increase of the dynamic yield stress of both MR suspensions with the magnetic field, meaning field-induced structuring. For both volume fractions (5 and 15%), the suspensions of polymerized chains show higher values of τ_Y than the suspensions of spherical iron particles. However, at magnetic fields $H<$ 10 kA/m, the yield stress τ_Y of the suspensions of spherical particles increases much faster with the field than that of the suspension of polymerized chains. We explain it by the higher magnetization of the spherical particles as compared to the polymerized chains, which contain a lot of polymer (López-López et al. 2007). At higher fields, we observe a dramatic decrease in the dynamic yield stress of the suspensions of spherical particles, which is not the case for the suspensions of chain particles. To explain these observations, we shall consider the squeeze flow of both suspensions.

Suspension of spherical particles In the presence of the magnetic field, spherical iron particles become magnetized, and they attract each other and form chain-like aggregates extended in the direction of the magnetic field. The MR fluid becomes a Bingham one, and, according to Scott (1931) and Engmann et al. (2005), the squeeze flow contains two distinct regions—a shear zone and a central plug zone, as depicted in Fig. 6a. In the plug zone, the radial component of the fluid velocity varies only in radial direction suggesting purely extensional flow (Engmann et al. 2005). The microstructure of the MR suspension is different in these zones and is determined by a competition between hydrodynamic and magnetic interparticle forces. Such competition is described by the Mason number, which scales as: $Mn_{\text{shear}} \propto \frac{\eta_0 \dot{\gamma}}{\mu_0 H^2}$ for shear flow and, by analogy, $Mn_{\text{ext}} \propto \frac{\eta_0 \dot{\varepsilon}}{\mu_0 H^2}$ for extensional flow. Here, η_0=0.32 Pa s is the viscosity of the dispersing liquid (ethylene glycol with silica particles of Aerosil 300), and $\mu_0 = 4\pi\ 10^{-7}$ Henry/m is the magnetic permeability of vacuum. In complex squeeze flow, shear rate $\dot{\gamma}$ and extension rate $\dot{\varepsilon}$ fields are not homogenous, and their characteristic values scale as: $\dot{\gamma} \propto \frac{\dot{h}R}{h_0^2} = \frac{2\pi \Delta h f R}{h_0^2}$ and $\dot{\varepsilon} \propto \frac{\dot{h}}{h_0} = \frac{2\pi \Delta h f}{h_0}$ (Macosko 1994). For the given range of experimental parameters, we estimated the order of magnitude of the Mason numbers in the squeeze flow of MR suspension: $10^{-2} < Mn_{\text{shear}} < 10$ and $10^{-3} < Mn_{\text{ext}} < 1$. These relatively low values of the Mason numbers indicate that in our experiments, magnetic interaction between particles either dominates over hydrodynamic interaction or is of the same order of magnitude, except for $Mn_{\text{shear}} > 1$. This suggests that in squeeze flow, the MR fluid aggregates resist against hydrodynamic forces and do not split into individual particles. To learn more about the MR structure in squeeze flow, a more detailed investigation, using either optical visualization or dynamic molecular simulation, is needed. Such simulations were performed by Kim et al. (1999) for an electrorheological fluid subjected to a continuous squeeze at constant squeeze velocity. The simulations confirm the existence of aggregates in the ER fluid under squeeze and predict chain-like structures aligned with the magnetic field in the central domain of the ER fluid sample, and rib-like structures extended in the radial direction near the periphery.

Suspension of polymerized chains In the presence of a magnetic field, polymerized chains become aligned with the field and, as for spherical particles, can form aggregates containing a few chain particles. These aggregates are expected to be much longer than the aggregates of spherical particles. Such big aggregates cannot sustain large shear forces. Therefore, if the aggregates of spherical particles are completely destroyed at the Mason numbers of the order of 1 (Volkova et al. 1999), the aggregates of polymerized chains should be destroyed at $Mn_{\text{shear}} \ll 1$. Therefore, we

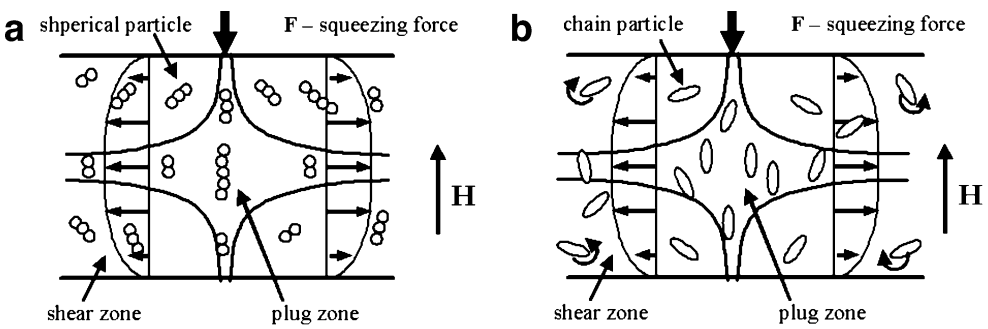

Fig. 6 Squeeze flow of the suspension of spherical iron particles (**a**) and of the suspension of polymerized chains (**b**) in the presence of a magnetic field. Two flow zones coexist—a shear zone near the walls and a central plug zone of purely extensional flow

expect no aggregates of polymerized chains in the shear zone of the squeeze flow (Fig. 6b)—notice that, as mentioned in the experimental section, polymerized chains are not destroyed under shear. However, there should be some remarkable increase in the shear viscosity because the magnetic field alters the orientation distribution of the chain particles and makes them spending more time perpendicular to the flow. In the plug zone, in the absence of magnetic field, chain particles align with the flow maximizing the extensional viscosity of the suspension. At low extensional Mason numbers, the magnetic field can maintain the chain particles perpendicular to the flow, and in contrast to shear flow, the extensional viscosity in the plug zone is decreased. However, in our experiments, in most cases, the yield force F_Y is several times smaller than the squeezing force amplitude F_0 and the shear zone must dominate over the plug zone. Thus, the resulting effect is a field induced increase in the viscous dissipation in squeeze flow.

Cavitation An oscillatory squeeze flow consists of periodic compression and traction of the fluid film. Under traction, when the plates move apart, an inward radial flow occurs in the fluid film, provoking a pressure decrease inside the film. A pressure decrease can induce a vapor or gaseous cavitation and even an air entrainment into the fluid film and the formation of a bubbly fluid mixture. It is exactly what happens in squeeze-film dampers, affecting dramatically their load capacity (Diaz and San Andrés 2001). The same phenomena are observed in traction of viscous fluids and adhesive materials (Gay and Leibler 1999). In our case, cavitation/air entrainment must occur at some critical magnetic field corresponding to a critical traction force, at which the pressure decreases below a cavitation pressure. The dramatic decrease in the squeezing force amplitude of the suspension of spherical particles can be explained by fluid film inflation with air bubbles sucked from the ambient air or formed by evaporation of the liquid at overcritical magnetic fields. We do not have a clear explanation why it does not happen to the suspension of polymerized chains. We can suppose that under a magnetic field, chain particles form much more deformable structures than spherical particles. This filament structure, spanning the gap, could better transmit the force between the upper and the lower disk and prevent cavitation from provoking a rupture between the fluid and the disks.

Conclusions

The present work is devoted to the effect of the magnetic field on the yield stress of MR suspensions of polymerized chains, in both shear and squeeze flow. In steady shear flow, the dynamic yield stress of the MR suspension of polymerized chains increases monotonically with the magnetic field and is of the same order of magnitude than the yield stress of the equivalent suspension of spherical iron particles. The oscillatory squeeze flow of non-Newtonian MR suspensions appears to be rather complex, combining regions of both shear and extensional deformation. The dynamic yield stress of MR suspensions is extracted from the extrapolation of force vs excitation frequency curves, using lubrication approximation. Both in the presence and in the absence of a magnetic field, chain particle suspensions offer much higher yield stress than spherical particle suspensions under squeeze. This is explained in terms of a solid friction between chain particles, which form more intricate aggregates. Because of the stronger magnetization of spherical iron particles, the yield stress of the suspension of spherical particles experiences a more rapid growth with the magnetic field than the suspension of polymerized chains. An abrupt decrease in squeezing force amplitude with increasing magnetic field is observed for the suspension of spherical iron particles at magnetic fields higher than 14 kA/m. Surprisingly, this phenomenon, attributed to cavitation or air entrainment, does not occur in the suspension of polymerized chains, at least at magnetic fields $H<30$ kA/m. This could make suspensions of elongated magnetic particles more preferable for use in squeeze film dampers. Finally, creep experiments in the linear viscoelastic domain allowed us to estimate the relaxation time of both MR fluids. It was found to be seven times longer for the suspension of polymerized chains as compared to the suspension of iron spheres.

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