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To cite this article: Jianjian Yang *et al* 2018 *Smart Mater. Struct.* **27** 075017

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Ternary solid-ferrofluid-liquid magnetorheological fluids

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Received 11 March 2018, revised 9 May 2018

Accepted for publication 15 May 2018

Published 1 June 2018



CrossMark

Abstract

A new kind of magnetorheological fluid is proposed that exhibits both enhanced magnetorheological effect and kinetic stability against sedimentation. It includes the presence of small amounts of an emulsified aqueous ferrofluid as a third phase in a conventional oil-based magnetorheological fluid prepared by the dispersion of carbonyl iron microparticles.

Supplementary material for this article is available [online](#)

Keywords: ferrofluid, magnetic colloid, emulsion, ternary fluid, magnetorheology, viscoelasticity

(Some figures may appear in colour only in the online journal)

Introduction

Magnetorheological (MR) fluids typically used in applications are mostly formulated by the dispersion of carbonyl iron (CI) microparticles in an oil. In the absence of magnetic fields the particles are not magnetized and interact via conventional colloidal forces. However, in the presence of magnetic fields the particles acquire an induced net magnetic moment and experience anisotropic magnetostatic interactions that result in the formation of elongated structures in the field direction. Because of this structuration, a remarkable change in the mechanical properties of the fluid is observed.

A current trend in magnetorheology research is to enhance the MR effect (i.e. the relative increase of viscosity/yield stress between the on-state and off-state). From a formulation perspective, previous attempts described in the literature concern the use of iron alloys [1], non-spherical particles [2, 3] and particle mixtures [4–6] among others.

Also, a major concern in magnetorheology stems from the fact that the density of the particles (iron) is significantly larger than that of the carrier fluid (oil). As a result of this, the particles sediment under gravity in the course of time. Most frequently, this problem has been addressed by thickening the

carrier fluid up to the point where it exhibits an apparent and sufficiently large yield stress. This is easily achieved by adding hydrophilic fumed silica particles or organoclays to oil-based suspensions [7, 8]. More recently, thermoresponsive carrier fluids have been proposed with a yield stress that can be externally tuned using the temperature as a control parameter [9, 10]. Finally, another tactic involves surface functionalization techniques to fabricate core-shell magnetic particles, hence reducing the density mismatch and also preventing the interparticle aggregation and/or protecting from external degradation such as oxidation [11, 12].

In this manuscript, we report a novel approach to enhance the MR effect and improve fluid stability. The basic idea is to add a small amount of an emulsified aqueous ferrofluid (FF) to the formulation of a conventional oil-based MR fluid, so that when the magnetic field is applied the FF droplets attach to the micrometric iron particles and serve as a bridge between them. As a result, the magnetic field lines are expected to flow more easily along the chain-like structures, facilitating the magnetization of the iron particles and thus enhancing the MR response. Furthermore, in the absence of magnetic fields the FF/oil emulsion remains stable and the CI particles, if diluted enough, tend to sediment more slowly

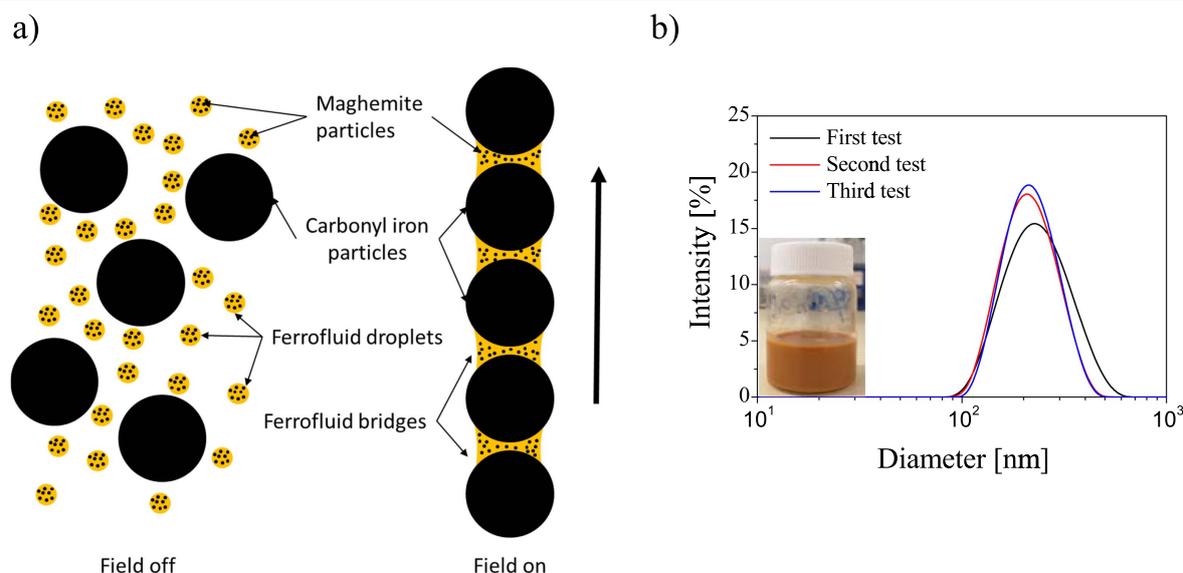


Figure 1. (a) Schematics of the FF bridges under magnetic fields. (b) FF droplet size distribution. Mean droplet diameter is 198 nm. Inset is the 8 vol% FF emulsion. A 1/1000 (volume ratio of FF emulsion to oil solution) dilution of that emulsion was used for the dynamic-light-scattering measurements.

because the effective carrier fluid increases its viscosity. Interestingly, the process is reversible, i.e. when the field is switched off the FF droplets detach themselves from the iron microparticles (see figure 1(a)).

Experimental

Materials

The CI used in this work was kindly provided by BASF SE Germany. In particular, we used the EW grade CI powder. The saturation magnetization of this particular grade was 209 emu g⁻¹.

Mineral oil, Span 80, Tween 60 and absolute ethanol were obtained from Sigma-Aldrich and Scharlau and used without further purification.

Suspension formulation

First, an aqueous citrate-stabilized FF was prepared following the procedure described by Ménager *et al* [13]. The first step in the preparation of the FF consists of the synthesis of magnetite nanoparticles by means of the precipitation method. In this procedure, ferric (FeCl₃·6H₂O, Sigma-Aldrich, >99.0%) and ferrous (FeCl₂·4H₂O, Sigma-Aldrich, >99.0%) chlorides were mixed at room temperature in a molar ratio 2:1 in an acidic aqueous solution and the pH was raised quickly by the addition of a NH₃ solution (Sigma-Aldrich, reagent grade, 28%–30%). The black precipitate that forms upon the addition of the base was then washed with water, treated with an acidic solution (HNO₃, Panreac, analysis grade) and oxidized to maghemite in a boiling Fe(NO₃)₃ (Scharlau, Spain, extra pure) solution. Next, the brown precipitate was dispersed in a citrate (Na₃C₆H₅O₇·2H₂O, Sigma-Aldrich, reagent grade) solution and the temperature was raised to 80 °C. The

functionalization of the particles' surfaces with citrate provides stability at pH = 7. The saturation magnetization of these maghemite nanoparticles was 52 emu g⁻¹. After the citrate treatment, the precipitate was washed with water, acetone and diethyl ether, and redispersed in water. Once the residual ether was removed by heating the dispersion, the pH was adjusted and the suspension was centrifuged at 12 000 g for 60 min. The resulting FF was stable for months and had a maghemite concentration of 2 vol%.

Second, Span 80 and Tween 60 surfactants were blended together in mineral oil to prepare a 2.4 wt% total surfactant concentration oil solution. For all samples the weight ratio of Span 80 and Tween 60 was 9:1. A systematic investigation had been previously carried out to arrive to this optimal concentration (see comments below in the *Results and Discussion* section).

Third, a small amount of FF was dropwise added to the oil solution by vigorous agitation using a homogenizer (SilentCrusher M) in order to obtain a sufficiently stable FF/oil emulsion. Then, an assistant surfactant, ethanol (1:1 with respect to emulsifier volume) was added. The stirring speed was 15 000 rpm and the duration was 5 min. The largest FF concentration in the emulsion that we studied was 8 vol%. Smaller FF concentration emulsions were also prepared by diluting the stock 8 vol% FF emulsion with surfactant solutions in oil.

Finally, a given amount of CI particles was added to the FF emulsion by stirring at 1000 rpm for 5 min in a centrifugal mixer (SD-500).

In summary, the FF used in this work consisted of an aqueous maghemite suspension at 2 vol% concentration. This FF was emulsified in mineral oil containing Span 80 and Tween 60 in a ratio 9:1 at a concentration of 2.4 wt%. To this FF emulsion, CI particles were added in different concentrations. In this work, both FF and CI particle concentrations

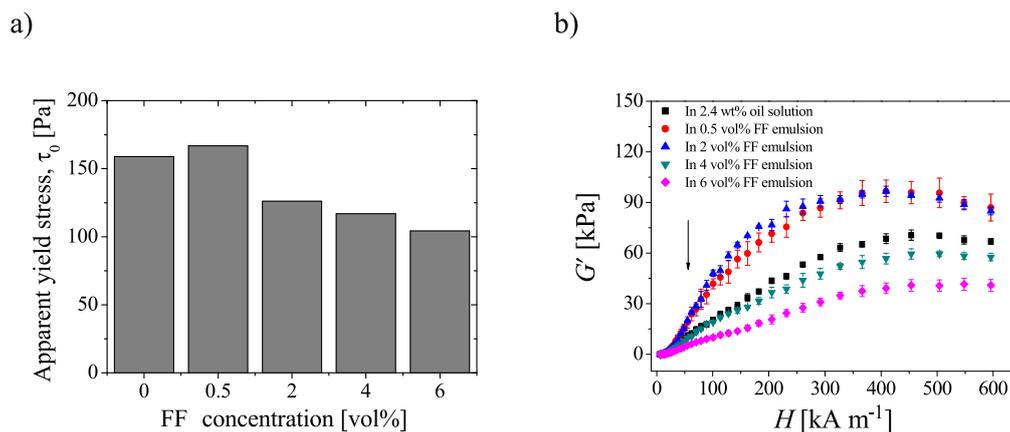


Figure 2. (a) Apparent yield stress (τ_0) as a function of FF concentration for 5 vol% CI suspensions at $H = 100 \text{ kA m}^{-1}$. (b) Storage modulus (G') as a function of external magnetic field strength (H) for 5 vol% CI suspensions with different FF concentration levels. Vertical arrow corresponds to the magnetic field strength for microscopy observations (56 kA m^{-1} ; see figure 5).

were changed in ranges from 0 vol%–8 vol% and 0 vol%–30 vol%, respectively.

Kinetic stability

The droplet size distribution of the FF droplets in mineral-oil-based surfactant solutions (i.e. FF emulsions) was estimated using a Zetasizer (Malvern Instruments) at 25°C . To this end, the FF emulsions were diluted to 1/1000 by using the oil surfactant solution and avoiding an additional input of mechanical energy, i.e. avoiding vigorous mixing, stirring or ultrasound treatment.

The gravitational sedimentation over time was investigated by visual observation of the sedimentation front at different times.

Rheometry

A commercial magnetorheometer (MCR501, Anton Paar, SE Germany) was used in a plate-plate configuration (20 mm diameter, $300 \mu\text{m}$ gap) and in isothermal conditions (25°C). The plates were sandblasted to prevent wall slip.

A typical protocol consists of three stages. First, the samples are preconditioned at a constant shear rate (100 s^{-1}) for 30 s. Later, the rotating plate is stopped for another 60 s in order to equilibrate the samples. Finally, the test starts. Two different tests were carried out to elucidate both the steady and dynamic oscillatory shear properties of the MR fluids: (i) in the steady shear flow tests the stress was logarithmically increased at a rate of 10 s/point from 20 Pa to 2 kPa. (ii) in the magnetosweep tests the excitation frequency was 1 Hz and the strain amplitude was 0.001%, well within the linear viscoelastic region of the suspensions [14].

At least three independent measurements with fresh new samples were taken. Error bars in the figures correspond to the standard deviation of the different repetitions.

Fluorescence microscopy

Fluorescence microscopy was used to visualize the structure of the ternary MR fluids both in the absence and presence of a

magnetic field (ca. 56 kA m^{-1}). We first added a small amount of fluorescein (6.8 g l^{-1}) to the polar phase, i.e. the aqueous FF, and then prepared the emulsion and a diluted ternary MR fluid (iron microparticles/FF/mineral oil) following the procedures described above. The sample was observed in a confocal laser scanning microscope (DMI600D Leica).

Results and discussion

FF emulsion stability

FF emulsions prepared in this work had to be stable over long periods of time. For this reason, the droplet size had to be sufficiently small and surfactants carefully chosen. On the other hand, the droplets had to be sufficiently large to ensure a magnetophoretic response upon the application of a magnetic field of the order of a few kA m^{-1} . Hence, a balance had to be reached with regard to the droplet size. In this context, in the present work, we used a high-energy emulsification method together with a mixture of emulsifiers [15, 16] in order to prepare a stable microemulsion with the droplet size in the nanoscale regime. A cosurfactant (ethanol, see *Experimental* section) was also added to reduce the interfacial tension and consequently help to decrease the size of droplets resulting from flow-induced rupturing [17, 18]. The droplet diameter of the FF emulsion was approximately 200 nm, as illustrated in figure 1(b). The inset shows that the FF emulsion is brownish due to the multiple scattering phenomenon and the absorption properties arising from the iron oxide nanoparticles enclosed within the droplets. The FF emulsion prepared presented a very good stability without obvious phase separation, sedimentation or creaming over the period of at least one month.

FF concentration

In this section, we discuss the rheological behavior, in steady and dynamic oscillatory shear, of ternary solid-FF-liquid MR

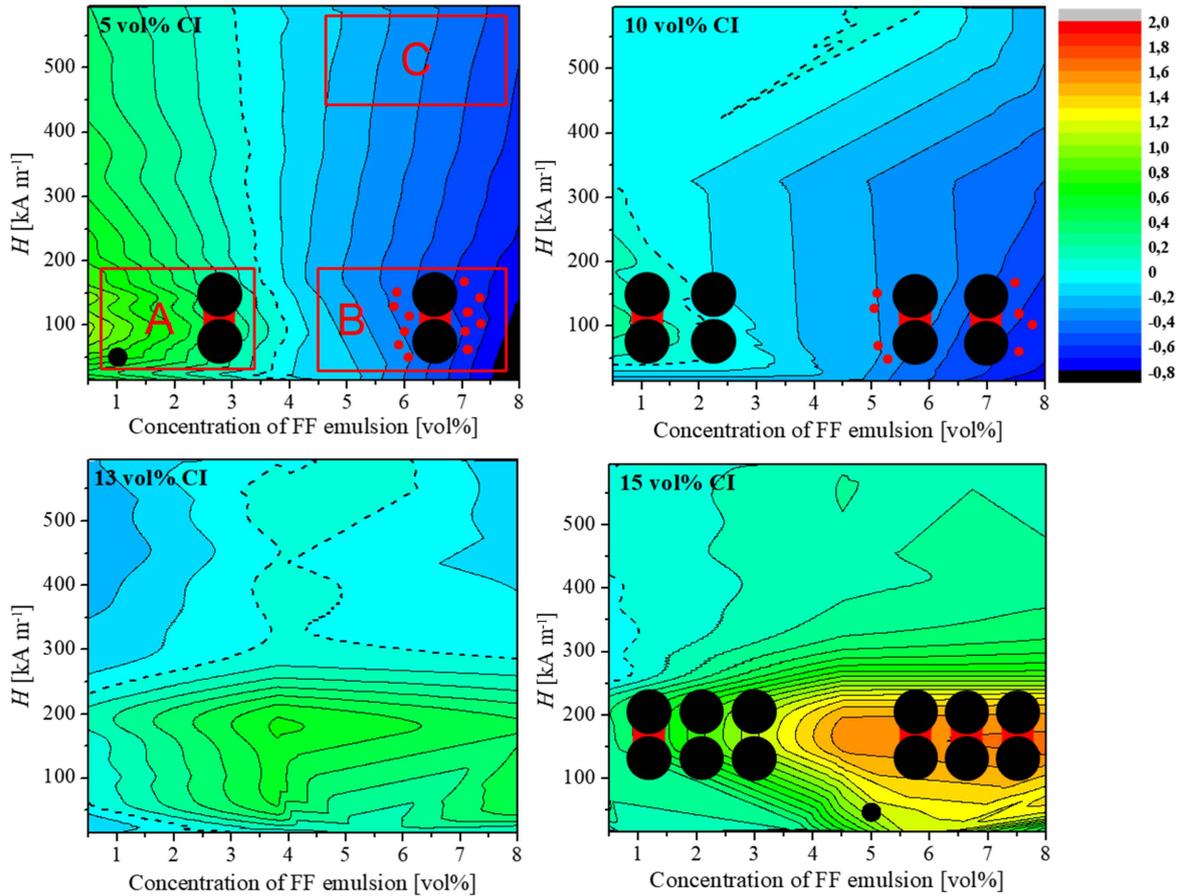


Figure 3. Contour plots corresponding to the relative storage modulus ($\Delta G'$) as a function of external magnetic field strength (H) and FF concentration for four different CI concentrations (5, 10, 13 and 15 vol%). Small black circles correspond to microscopy observation tests in figure 5. Dashed line corresponds to $\Delta G' = 0$.

fluids prepared by the addition of small amounts of FF to a mineral-oil-based suspension of CI particles.

In figure 2, we show typical curves for 5 vol% CI concentration and FF concentrations in the range 0 vol%–6 vol%. By way of example, in figure 2(a), we show the FF concentration dependence of the apparent yield stress (τ_0) under a magnetic field of $H = 100 \text{ kA m}^{-1}$. The apparent yield stress was obtained from the steady shear rheograms by extrapolation of the stress to zero shear rate. Interestingly, the yield stress exhibits a maximum with the FF concentration. Similarly, in figure 2(b), we show the magnetic field dependence of the storage modulus (G') for the same MR fluids. As expected, the storage modulus increases with the external magnetic field as a result of the formation of field-induced structures. Again, the FF concentration has a remarkable effect in the response. Slightly increasing the FF concentration increases the modulus. However, a more noticeable increase of the FF concentration results in a reduction in the viscoelasticity.

This effect is more clearly observed with the help of contour plots and through the use of the relative storage modulus ($\Delta G'$), which can be defined as the ratio between the modulus increment under the addition of FF and the modulus for the conventional MR fluid (i.e. without FF in the

formulation):

$$\Delta G' = \frac{G'(\text{with FF}) - G'(\text{without FF})}{G'(\text{without FF})}. \quad (1)$$

Here, $G'(\text{without FF})$ stands for the storage modulus of CI suspensions in a surfactant solution in mineral oil (i.e. binary systems), while $G'(\text{with FF})$ stands for the storage modulus of CI suspensions in FF/oil emulsions (i.e. ternary systems). The solution of surfactant in mineral oil was chosen as the continuous phase of the reference systems (binary systems) because this same solution was the continuous phase of the FF emulsion in which the CI particles were dispersed to prepare the ternary systems. The relative storage modulus $\Delta G'$ is plotted in figure 3 as a function of FF concentration and external magnetic field strength. Each contour plot corresponds to a different concentration of iron. As observed, there is a well-defined region in each plot where the MR effect is increased.

In the absence of magnetic fields, the storage modulus is negligible (below the torque resolution of the rheometer) suggesting that there is not a space-filling network of particles in suspension. However, upon increasing the magnetic field strength, the CI particles form chain-like structures that eventually span the gap between the plates, successfully transmitting the stress. For very large magnetic fields the CI

microparticles become fully magnetized (i.e. magnetically saturated) and therefore the storage modulus levels off, reaching a constant value (see figure 2).

For sufficient small magnetic field strengths (H) and FF droplet concentration (below ~ 4 vol%)—see region A in figure 3, 5 vol% CI—the storage modulus is enhanced with respect to the case of binary systems (without FF). The reason for this is the formation of bridges of FF between CI particles, which serve as a pathway for the magnetic field lines to pass along the aggregates. A similar explanation was reported by Chin and Park [19] for ER fluids. The presence of the FF bridges was confirmed with confocal microscopy observations of the ternary suspensions at 56 kA m^{-1} (see figure 5 below).

When the FF concentration is too large, with respect to the iron content—see region B in figure 3, 5 vol% CI—, there is a surplus of FF that is not well accommodated between the iron particles that form the chain-like structures. This surplus of FF remains essentially in the form of isolated droplets within the mineral oil solution, which increases the permeability of the carrier fluid (mineral oil + FF droplets) and brings down the so-called contrast factor $\beta = (\mu_p - \mu_c)/(\mu_p + 2\mu_c)$, i.e. the permeability mismatch between the iron particles and the carrier. Under these circumstances, G' decreases, since it is known that in the linear magnetization regime the magnetostatic interaction F_m has a quadratic dependence on β : $F_m \propto \beta^2$.

When the magnetic field strength is too large—see region C in figure 3, 5 vol% CI—, the field-induced aggregates are very compact and interparticle separation becomes negligible (of the order of the particle roughness). Hence, the surface area that is available for the FF droplets to adsorb decreases. As a result of this, there is again a surplus of FF that is not accommodated between the iron particles and remains in suspension as isolated droplets, hence decreasing the storage modulus of the suspension because of the decrease in the magnetic permeability mismatch.

In summary, for a 5 vol% CI system, low FF concentrations and low magnetic field strengths are required for the ternary suspensions to exhibit an enhanced MR effect. Under these conditions, basically all of the FF droplets in dispersion serve as bridges between the iron particles in the field-induced structures.

CI concentration

Similar tests were also carried out for larger CI concentrations that are closer to those of interest in commercial applications (10, 13 and 15 vol%). The results are also shown in figure 3.

When the particle concentration increases from 5 vol% to 10 vol%, region A becomes smaller and region B enlarges. The reason for this is a balance between the increase in the number of contact points between iron particles in the structures and the formation of more (compact) aggregates. On the one hand, for low FF concentrations there is insufficient FF for all contact points. On the other hand, for high FF concentrations, a larger number of FF droplets still exist in

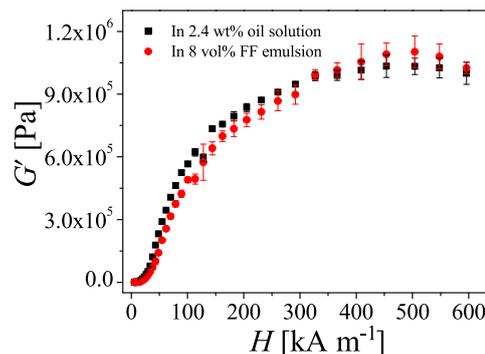


Figure 4. Storage modulus (G') as a function of external magnetic field strength (H) for 30 vol% CI suspensions based on oil solution and 8 vol% FF emulsion.

dispersion if compared to those bridging the gap between the iron particles in chain-like structures.

When the iron concentration further increases above 10 vol%, we observe that the FF concentration needed for the enhancement in the relative storage modulus grows with iron concentration. This is especially noticeable for the 15 vol% iron MR fluid, which exhibited the enhancement at an FF concentration of 4 vol%–8 vol%. For this iron concentration, nearly all of the FF droplets are bridging the gaps between the iron particles and there is no FF left in suspension. As a result, the relative storage modulus is maximized. It should be noted that in this case (figure 3, 15 vol% CI) an enhancement of the MR response was observed for almost all the fields and FF emulsion concentrations we studied.

Finally, for very large iron concentrations, of the order of 30 vol%, the rheological behavior of the suspension is not significantly affected for the concentrations of FF investigated in this work. An example is shown in figure 4. In this case, FF concentrations studied in this work are so small if compared to the CI loading, so that interparticle gaps are mostly depleted of FF and the rheological behavior is very similar in both binary and ternary systems.

Fluorescence microscope observations

The enhancement of the rheological response when dispersing the CI particles in the FF emulsion is presumably due to the FF droplets bridging the gap between the CI particles forming chain-like structures. In an attempt to confirm this model, we tried to visualize these bridges by observing ternary MR fluids in a fluorescence microscope.

Some representative images are shown in figure 5. In the absence of magnetic fields, FF droplets are perfectly spherical in shape and randomly distributed within the carrier fluid. We do not observe a preferable aggregation of the droplets to the CI particles. However, in the presence of magnetic fields, the FF droplets are attracted by the primary chain-like structures formed by the CI particles. In particular, the FF droplets move to fill in the gaps between the CI particles. This is clearly appreciated in figure 5 for a 5 vol% CI suspension in a 1 vol% FF emulsion (see figure 5(a)) and also for 15 vol% CI suspensions in a 5 vol% FF emulsion (see figure 5(b)). The

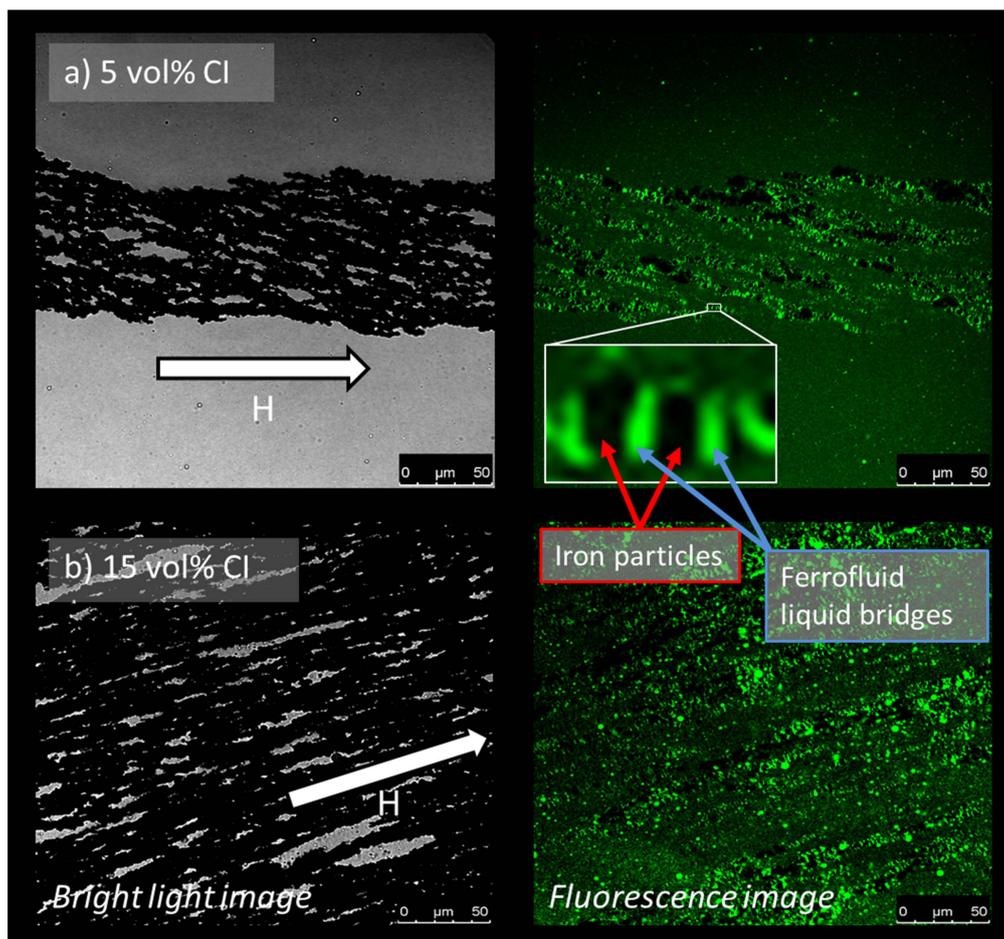


Figure 5. Fluorescence microscopy images of 5 vol% (15 vol%) CI MR fluids containing fluorescently dyed FF droplets with concentration of 1 vol% (5 vol%). Sample was prepared according to the method mentioned in the text. Picture corresponds to a typical aggregate under the presence of a magnetic field of 56 kA m^{-1} . Left column: bright light image. Right column: fluorescence image. Green regions correspond to the FF droplets.

conditions under which the microscopy observations were carried out are represented by small black circles in figure 3.

Sedimentation stability

We also studied the sedimentation stability of the suspensions by visual observation. In figure 6 we show the sedimentation profiles of three suspensions at two particle loadings (5 and 15 vol%): (i) CI particles in mineral oil, (ii) CI particles in a 2.4% oil solution, (iii) 5 vol% CI particles in a 1 vol% FF emulsion and (iv) 15 vol% CI particles in a 5 vol% FF emulsion. A visual inspection of the curves suggests that the dominant factor is the surfactant concentration. From the height of the sediment at long times, it can be inferred that suspensions containing surfactants pack very well, while suspensions in mineral oil form a loose cake after sedimentation.

Differences are also observed at short and medium times. In order to interpret this behavior, we determined the viscosity of the different carrier fluids employed in the preparation of the CI suspensions. The results obtained in a cone-plate geometry (50 mm diameter) are summarized in table 1. As observed, the differences in shear viscosities between the

samples are small, but compared to the pure mineral oil, viscosity tends to increase with the addition of surfactant and the presence of the emulsified FF. However, when plotting L/L_0 data as a function of the time/viscosity ratio, the sedimentation curves did not collapse. This is a strong proof that the viscosity of the carrier is not the only factor driving the sedimentation behavior of the suspensions. Instead, the surfactant concentration must play an important role.

For the lowest iron concentration investigated (5 vol%, figure 6(a)), the short time behavior is clearly improved when the iron particles are dispersed in the FF emulsion. This is expected because of the slight increase in carrier fluid viscosity. However, for the most concentrated suspension (15 vol%, figure 6(b)) the sedimentation is faster for the suspensions prepared in the FF emulsions when compared to the MR fluid prepared in the pure (i.e. surfactant free) mineral oil. The reason for this could be that the suspensions in mineral oil truly form a colloidal gel that spans the whole volume because of the interparticle interactions. Still, when compared with the CI in 2.4 wt% surfactant oil solution, the sedimentation of the ternary system is also slower at 15 vol% CI.

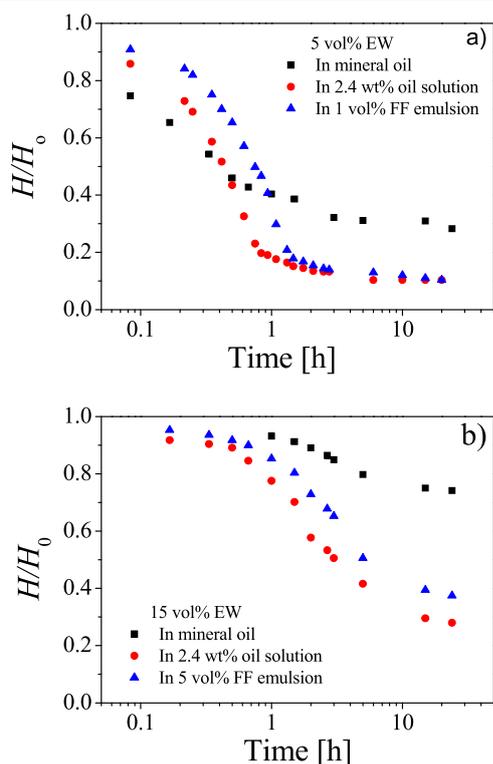


Figure 6. Sedimentation stability of CI suspensions in surfactant oil solution, FF emulsion and pure mineral oil, respectively. (a) 5 vol% CI and (b) 15 vol% CI.

Table 1. Shear viscosity of the CI suspensions. Viscosity of the carrier fluids was measured with a cone-plate geometry (CP-50) and the viscosity was averaged in the shear rate range from 3–100 s⁻¹.

Sample	Viscosity (mPa · s)
Mineral oil	24.4 ± 0.2
2.4 wt% surfactant oil solution	25.4 ± 0.1
1 vol% FF emulsion	26.2 ± 0.1
5 vol% FF emulsion	28.8 ± 0.4

To sum up, for the two CI concentrations that were studied and are shown in figure 6, the sedimentation was slower for the ternary systems, i.e. for the iron microparticles dispersed in the FF emulsion, compared to the CI particles dispersed in the solution of surfactant in mineral oil. The latter system is the most appropriate reference, since the ternary system under study contained the same surfactants in solution. The CI particles in oil (without surfactant) settled down more slowly, especially at long times, because the lack of a surfactant facilitates the formation of loose aggregates that form a sediment that is also loose.

Conclusions

We prepared a novel MR fluid by adding micrometric CI particles to a w/o emulsion of an aqueous FF in mineral oil. Thus, compared to the conventional basic formulation of an

MR fluid formed by iron particles dispersed in an oil, our system contained aqueous FF droplets as a third phase.

An enhancement of the MR response was observed for particular ranges of FF concentration and magnetic field strengths. This enhancement can be ascribed to the fact that upon the application of a field, the FF droplets tend to fill the gaps between the iron particles, which facilitates the flow of the field along the chain-like structures. Observations of the field-induced structures in a fluorescent microscope support this model.

Since the role of the FF droplets is to serve as a pathway to the flux lines and thus facilitate the magnetization of the iron particles, the enhancement in the MR response occurs at low and medium fields and is smaller or does not happen at large fields, when the iron particle's magnetization has saturated. A plausible explanation for this is that at large fields, when the space between the iron particles is constrained, some FF is expected to be released from the gaps between the particles into the carrier, which hinders the MR response due to the reduction of the contrast factor (β). The same reasoning, i.e. the decrease in β , explains the lower MR response observed at larger concentrations of emulsified FF.

It should be emphasized, however, that for the ternary fluid with an iron concentration of 15 vol%, an enhancement of the MR response was observed for almost all field intensities and FF droplet concentrations under consideration.

The ternary system reported in this work may be of interest to better understand the pendular to capillary state transition in capillary suspensions because of the external magnetic driving force that competes with the wetting characteristics of the suspensions.

Acknowledgments

This work was supported by MINECO MAT 2016-78778-R and PCIN-2015-051 projects (Spain), the European Regional Development Fund (ERDF) and by the Junta de Andalucía P11-FQM-7074 project (Spain). J J Yang acknowledges the Chinese State Scholarship Fund. J R Morillas acknowledges the FPU14/01576 fellowship.

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