MAGNETIC SUSCEPTIBILITY AND MAGNETIC HYSTERESIS LOOP OF SOME CARBONYL IRON POWDERS USED IN MAGNETORHEOLOGICAL FLUIDS

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Abstract
Some commercial carbonyl iron powders, from BASF AG, were characterized through their magnetic susceptibility and saturation magnetization. The saturation magnetization increases in the order: CC < HQ < OX < CS < SM. Although all the powders have 96% minimum of iron content, this result suggests that an investigation at higher magnetic fields, under simultaneous shear is very important. The reversibility and the reproducibility of the MR effect were satisfactory, confirming that carbonyl iron powders are good ferromagnetic materials to prepare magnetorheological fluids.

1 Introduction
Magnetorheological fluids (or magnetorheological suspensions, in a physical-chemistry context) are considered a new material, although their applications were first reported by Rabinow at the end of the forties. [1] Other interesting applications about MRFs, pictures showing a microscopic representation and the macroscopic effect of a MRF, can be found at the web site of Lord Rheonetic magnetic fluids. [2]

MRF contain basically four types of substances: a ferromagnetic powder (such as carbonyl iron powder, cobalt, or nickel); a carrying fluid (mineral or silicone oils, in general); a dispersant (to avoid or minimize particle coagulation); and gel-forming additives (as hydrophobic silica powder, for example). [3]

The rheological properties of such fluids are changed by the application of magnetic fields in a reversible way. For example, under a field of 250 kA/m, they can present yield stresses of up to 100 kPa1. Besides, the change of the semi-solid state for fluid state and vice-versa happens in ~ 5 milliseconds. [4]

These characteristics allow the use of these fluids in controllable mechanical devices, such as conventional shock absorbers, mountings, brakes and clutches [5]; dampers to reduce damages in civil engineering constructions caused by earthquakes [6]; polishing in optics [7]; reduction of vibrations in helicopters [8]; and biomedical applications, [9,10] among others.

Since the active material inside MRF is a ferromagnetic powder, it is necessary to evaluate their magnetic properties in order to better understand the MRF formulation development.

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1 To give some idea of the sizes of these units, the Earth’s magnetic field is typically $H = 56$ A/m ($0.7$ Oe) and the most formulations of toothpaste shows yield stresses around 40 Pa.
2 Experiment

2.1 Magnetic measurements

The powders, without any treatment, were weighted and the magnetization hysteresis loop were measured using a SQUID (superconducting quantum interference device) type magnetometer (Quantum Design MPMS XL7). From the hysteresis loop data, magnetic susceptibility was calculated and other magnetic parameter were obtained.

2.2 Rheological measurements

The yield stress, viscosity curves, and the MR effect (considered in this work as the viscosity increase when the magnetic field is applied) were measured using a controlled stress rheometer (Paar Physica MCR 300), with the plate-plate configuration (50 mm) and 1 mm gap. The magnetic field was applied through a Helmholtz coil system and the magnetic flux density ($B$) was measured with a Hall effect probe and a portable gaussmeter. In order to yield reproducible results, the experiments were performed using triplicate sample. For each sample, triplicate measurement were obtained.

3 Results and discussion

3.1 Magnetic measurements

The evaluation of the magnetization is important, because several parameters of the mechanical design of devices using MRFs directly depend on this intrinsic property. Carlson et al. [5] showed that the minimum active fluid volume $V$ in valve mode devices, like a damper, is related with the yield stress $\sigma_y$ and the viscosity $\eta$ of the MRF through the equation 1, where ‘c’ is a factor that depends on the device type, $\Delta P$ is the pressure drop and $Q$ is the flow rate.

$$V = \frac{12}{c^3} \left( \frac{\eta}{\sigma_y^2} \right) \left( \frac{\Delta P_{\sigma_y}}{\Delta P_{\eta}} \right) Q \Delta P_{\sigma_y}$$

Equation 1

On the other hand, Shulman et al [11], and Rosensweig [12] associated the yield stress $\sigma_y$ with the magnetic susceptibility $\chi$ of the fluid, as showed in the equation 2:

$$\sigma_y \propto \phi \mu_0 H_0^2 f(\phi, \chi)$$

Equation 2

where $\phi$ is the volumetric fraction of the magnetic powder, $\mu_0$ is the vacuum permeability, $H_0$ is the applied magnetic field strength, and $f(\phi, \chi)$ is a function involving various terms on $\phi$ and $\chi$. Since the magnetic susceptibility is defined by the ratio between magnetization $M$ and the field strength $H$, the magnetic response of the powders plays an important role in the performance of MRFs, as demonstrated in previous studies [13].

Figure 1 shows the magnetization $M$ (emu/g) as a function of the applied magnetic field strength $H$ (Oersted). The results show that the magnetization of the powders increases in the following order: CC < HQ < OX < CS < SM, but it is difficult to see the difference between
the curves at lower magnetic fields. So, as pointed by Carlson et al. [14], a graphic of \( M^2 \) vs. \( H \) (square of the magnetization vs. magnetic field) show the best point to energize the MRF, if a secant line and tangent to the curve is traced. The associated abscissa value is the best point to work and to design the magnetic circuit of the MR device. Figure 2 shows this type of plot, applied to the five BASF carbonyl iron powders studied in the present work. Figure 3 shows the magnetic susceptibility \( \chi \) of the powders as a function of the field strength \( H \) in an expanded view (between 10 and 6000 Oersted) that is the usual field region for practical devices using MRF’s [14].

Figure 1. Magnetization (M) of the BASF carbonyl iron powders as a function of the applied magnetic field strength (H).

Figure 2. The square of the magnetization as a function of applied magnetic field strength of five BASF carbonyl iron powders: CC, HQ, OX, CS and SM.

Figure 3. Magnetic susceptibility \( \chi \) as a function of the applied magnetic field strength \( H \) of five different carbonyl iron powders.
Figure 2 shows that for the powders CS and SM, the best point is around 2000 Oe, but for the powders OX, HQ and CC, this point is ~ 3800 Oersted. Based on this result, we can propose that MRFs based on CS and SM powders can show better performance than MRFs prepared with HQ, CC or OX powders at the same mass fraction. The MR effect must be larger at the same field or the MR effect can be set to the same mechanical response, but with lower power consumption. At this point we are trying to confirm experimentally this prediction.

Figure 3 shows that the magnetic susceptibility increases in the following order: CC < HQ < OX < SM. It was expected that the larger the magnetic susceptibility, the larger the yield stress, according to the equation 2. So, one should expect that a MRF prepared with SM powder had showed a larger yield stress, compared with CC powder, for example. However, our preliminary results, working at low fields, did not confirm this prediction [15]. Up to now, we do not have a good explanation for this contradictory result.

3.2 Rheological measurements

In order to evaluate the reversibility of the MR effect, a MRF was prepared according to the composition listed on the tables 1 and 2. The viscosity was measured under a constant shear stress, above the yield stress, during 15 minutes, turning the applied magnetic field ‘on’ (constant magnetic induction = 300 Gauss) and ‘off’. The results are shown in figure 4.

<table>
<thead>
<tr>
<th>Table 1: Composition of the Gel base.</th>
<th>Table 2: MRF formulation.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oil (Texaco ‘C1’) 90 ml</td>
<td>BASF carbonyl iron powder ‘SM’ 20,007 g 66,1% w/w</td>
</tr>
<tr>
<td>Oleic acid 16 ml</td>
<td>Gel base 10,2576 g 33,9% w/w</td>
</tr>
<tr>
<td>Hydrophobic silica (Cab-O-Sil TS 610) 4,7354 g</td>
<td></td>
</tr>
</tbody>
</table>

Figure 4. Viscosity measured as a function of the time, under constant shear stress = 100 Pa, and alternating applied magnetic field ‘on’ (B = 300 Gauss) and ‘off’. MRF formulated with BASF carbonyl iron powder, grade ‘SM’ at 66% w/w.
Figure 4 shows that the MR effect (here, the viscosity increase when the magnetic field is turned ‘on’, or the viscosity decrease when the field is turned ‘off’) is almost perfectly reversible, and the effect has good reproducibility. The viscosity increases more than 5000 times, under 300 Oersted.

Table 3: Composition of the three similar MRF based on BASF iron powder ‘CL’.

<table>
<thead>
<tr>
<th>Formulation</th>
<th>BASF iron powder ‘CL’</th>
<th>Gel base</th>
<th>% w/w iron</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>20,0131 g</td>
<td>10,2237 g</td>
<td>66.19</td>
</tr>
<tr>
<td>B</td>
<td>20,0713 g</td>
<td>10,0507 g</td>
<td>66.63</td>
</tr>
<tr>
<td>C</td>
<td>20,0676 g</td>
<td>10,0991 g</td>
<td>66.52</td>
</tr>
</tbody>
</table>

To check if our results are reliable, three similar MRF were also prepared, with the same amount of carbonyl iron powder and all the other constituents of the formulation. The details of the formulations are described in table 3. The viscosity curves as functions of the shear rate in the range of $10^{-4}$ to $10^3$ s$^{-1}$ were measured under two magnetic field strengths. Each data set represents the results measured using a different sample. The result is shown in figure 5.

As expected, figure 5 shows that the viscosity increases with the magnetic field and decreases when the shear rate increase (MRF are shear thinning). The MR effect is more pronounced at lower shear rates. The reproducibility is quite well at 300 gauss and a bit worst at 100 gauss.
4 Conclusion

The results about the magnetic properties suggest that the MRF’s based on BASF carbonyl iron powders ‘CS’ and ‘SM’ must have a better performance than the other grades, as ‘CC’, ‘HQ’ and ‘OX’. To confirm this prediction, is necessary more investigation, specially at higher magnetic field strengths and with simultaneous shear. The MR effect of MRF formulation ‘SM’ showed a reversible behaviour. MRF formulation ‘CL’ showed good reproducibility.

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