Characterization of sedimentation and high magnetic field flow behavior of some magnetorheological fluids

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Abstract: Preparation and characterization of an improved sedimentation rate MR fluid is presented. Three different commercial additives were used for dispersion/stabilization of iron particles with sizes between 4-6 µm in synthetic oil carrier. The structural and magnetorheological properties of the sample (MRF-LM5) were compared with the same characteristics measured for a commercial MR fluid (MRF-132DG, LORD Co., USA) of similar saturation magnetization. The sedimentation rate of particles measured by X-ray transmission in MRF-LM5 is twice slower than in MRF-132DG, while the MR effect is higher for the commercial MR sample.

1. Introduction
One of the important characteristics of MR fluids is their significant gravitational sedimentation rate. Slowing down the particle settling velocity is an actual research task and involves various solutions concerning the morphology and size range of magnetic particles, as well as the use of various additives. A good compatibility of additives with the carrier and the iron particles reduces sedimentation rate and irreversible agglomeration of particles. In order to ensure long-term controllable and reproducible MR effect, the carrier liquid with additives has to keep its stability, while the suspended iron particles should be magnetically soft [1-4].

In this work a laboratory prepared MR fluid, with improved sedimentation behavior is investigated and compared with the properties of a commercial MR fluid (MRF-132DG, Lord Co., USA).

2. Experiments and discussion
2.1. MRF samples
For the preparation of MRF-LM5 samples micropowder of iron (99.5% Fe, 80g) with particle sizes between 4-6 µm [5] was used. Three different carrier liquids (20g) were experimented (Castrol oil, transformer oil and silicon oil), which are stable in the temperature interval - 40 °C, + 130°C. Also, several additives were considered to prevent sedimentation and coagulation, as well as to facilitate dispersion of particles (liquid and/or powder); finally there were selected three additives denoted Thix (1.5g), HT (1g) and Pur 8050 (3g). These additives are commercial products (Q Speciality) designed for structuring the carriers and there were selected taking into account their characteristics and the observed improvements of the sedimentation stability of MRF samples. The density of the selected MRF-LM5 sample was 2.75 g/cm³. The MRF-132DG fluid, product of Lord Co.(USA), is a...
suspension of micron-sized (mean size 8.87 µm), magnetizable particles in a hydrocarbon carrier fluid. This sample has the following characteristics [6]: density 2.98-3.18 [g/cm³], solid content by weight 80.98 [%].

2.2. Dimensional and morphological analysis of particles in MRF-LM5

In order to determine the shape and mean size of iron particles of the powder dispersed, several MRF-LM5 type samples were analyzed. These were deposited on 300 mesh supports for transmission electron microscopy investigations (Tesla BS-613; U = 50 kV, I = 80mA). Investigations performed at 1000X magnification resulted in TEM images (Fig. 1) which show that the iron particles in suspension are of spherical shape and have close sizes. Data analysis referring to more than 400 particles conducted to the mean size \( \bar{d} = 4.32 \mu m \) and to the histogram of relative frequency (Fig. 2).

For the determination of the state of internal tensions the iron powder was supposed to X-ray diffraction investigations using a „Dron 3” type diffractometer with molybdenum radiation (\( \lambda_{\alpha1,\alpha2} = 0.71 \) Å, \( U = 40 \) kV, \( I = 30 \) mA) in the angular interval \( 2\theta \in (14^\circ, 65^\circ) \), the detector having the speed of 1°/min (Fig 3).

In Fig. 3.a the spectral lines of the doublet \( \lambda_{\alpha1,\alpha2} \) are splitted already at small 20 angles (2\( \theta \approx 35^\circ \)) and at large 2\( \theta \) angles are almost completely splitted. That means that the particles of this sample do not have internal stresses and, consequently, they have the characteristics of a soft magnetic material. A similar structural behaviour is specific also to the commercial sample MRF-132DG (Fig. 3.b.)

![Figure 1. Shape, size and distribution of particles in a sample of MRF-LM5](image1)

![Figure 2. Relative frequency histogram of particle sizes in MRF-LM5](image2)

![Figure 3. X-ray diffraction spectra](image3)

a) MRF-LM5; b) MRF-132DG

2.3. Analysis of gravitational settling by X-ray transmission

For the analysis of gravitational sedimentation of particles the set-up of the „Dron-3” X-ray diffractometer was used, with the X-ray radiation generated by the molybdenum tube.

To perform the sedimentation rate measurement, each sample was first homogenised and after it was introduced in a rectangular Plexiglas sample holder having the dimensions 9x4x400mm. The filled sample holder was placed in vertical position in front of the X-ray detector, the radiation passing through the 4 mm thick layer of MRF. For having the maximum radiation intensity \( I_t \) transmitted through the MRF layer at the bottom of the sample holder, the detector was rotated in the angular interval \( 2\theta \in (-0.5^\circ, +0.5^\circ) \) with the speed of \( (1/2)^\circ/\)min.

The X-ray transmission experiment was repeated four times, at one week time intervals, keeping the same experimental conditions for each sample. The variation of particle concentration is represented in Fig. 4 by the dependence of the maximum intensity of the transmitted radiation as a...
function of time. Also, in Fig 4 the mass density ($\rho$) of the samples is represented after each time interval.

**Figure 4.** Gravitational sedimentation at 1, 2, 3 and 4 weeks time intervals ($\rho$ - the corresponding density values) a) MRF-LM5; b) MRF-132DG

By comparing the transmitted X-radiation intensities and the corresponding density values for the two types of MR fluids analyzed, it follows that the laboratory prepared sample MRF-LM5 has better gravitational sedimentation behaviour than the commercial sample MRF-132DG.

2.4. Magnetic and magnetorheological characterization

The full magnetization curves of MRF samples were determined by vibrating sample magnetometry (VSM 880, DMS(USA)). The samples have close values of saturation magnetization, 620 kA/m for MRF-LM5 and 660 kA/m for MRF-132DG.

Magnetorheological measurements were performed using a PHYSICA MCR300 rheometer with plate-plate MR cell (MRD180), having 20 mm diameter and 0.5 mm gap size. Oscillatory (Amplitude Sweep) and rotational (Flow Curve) tests were performed at the temperature $T = 20 ^\circ C$, in the absence of the field ($B=0$) and in applied field ($B = 0, 64.8, 125, 187, 253, 379$ and $502$ mT).

To describe the behavior of the two MR fluid samples, two analytical models were used (Fig 5). In the absence of the field the model of Carreau-Yasuda [7], while for the flow behaviour in magnetic field the model developed by Susan-Resiga proved to be adequate [8]:

**Figure 5.** Viscosity curves at $T=20 ^\circ C$: a) MRF-LM5; b) MRF-132 DG
\[ \eta = \eta_0 \left[ 1 - \tanh \left( \frac{\gamma}{\gamma^*} \right) \right] + \left[ \left( \frac{\tau_0}{\gamma} \right) + \left( \frac{\tau_1}{\gamma} \right) \left( \frac{\gamma}{\gamma^*} \right)^{n-1} \right] \tanh \left( \frac{\gamma}{\gamma^*} \right) \]

where: \( \eta_0 \) - zero field viscosity; \( \gamma \) - shear rate; \( \gamma^* \) - transition shear rate between two different flow behaviours; \( \tau_0 \) - threshold shear tension; \( \tau_1 \) - fit parameter; \( n \) - pseudo-plastic exponent.

The flow diagrams (Fig.5) evidence that the two MR fluid samples have quasi-Newtonian behaviour at low shear rate; such behaviours can be observed for various systems, including nanocomposites [9]. At high shear rate, both samples have pseudo-plastic (shear thinning) properties, the change in flow behaviour being observed at \( \gamma = 10^{-3} \text{s}^{-1} \). Increasing the shear rate the magnetic field induced particle structures are progressively destroyed. Both samples show large MR effect under the action of applied magnetic field in the interval \( B = 0-502 \text{mT} \). The relative increase of viscosity \( \Delta \eta/\eta_0 \) (Fig. 6) evidence that the observed MR effect is larger in the case of the commercial sample MRF-132DG.

3. Conclusion

Sedimentation rate of particles in the case of the sample MRF-LM5 is twice slower than for the commercial sample MRF-132DG, of similar magnetic properties. The flow properties of samples in zero magnetic field are fitted by the Carreau-Yasuda model, while their flow behaviour in magnetic field is well described by a newly developed model [8].

Acknowledgement: The present research has been supported by the Romanian National Authority for Scientific Research through the CEEX- C2-M1-1185, C64/2006-2008 „i-SMART-flow” project.

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