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# A rheometer dedicated for the investigation of viscoelastic effects in commercial magnetic fluids

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#### Abstract

The investigation of viscoelastic properties of commercial ferrofluids based on nanosized magnetite particles in various carrier liquids requires a dedicated rheometer allowing the detection of extremely small changes in viscous and viscoelastic properties. The observation of magnetoviscoelastic behavior in commercial ferrofluids may become particularly important since they exhibit long-term stability allowing reproducibility of the experiments. In addition, they are of relatively simple composition reducing the problems in theoretical description of the observed effects. We will present here the design and main performance data of a rheometer developed for the kind of investigations mentioned above. The quality of the performance will be demonstrated with some new data on rheological properties of a commercial ferrofluid. © 1999 Elsevier Science B.V. All rights reserved.

Keywords: Rheology; Magnetic fluids; Shear thinning

### 1. Introduction

The investigation of viscoelastic behavior in commercial magnetic fluids opens the possibility to obtain data on the dependence of viscoelastic properties as a function of magnetic field strength from fluids with excellent stability. This is advantageous since it enables extended series of investigations without changes in the basic properties of the liquids under consideration. In addition these fluids are nowadays well characterized, and their basic properties are relatively well known compared to those of magnetorheological fluids containing micronsized particles which, e.g. experience sedimentation and therefore may show variations of density within the fluid sample under examination. Thus, the data obtained from rheological investigations of commercial ferrofluids may give rise to new insight into the microscopic reasons for viscoelasticity in ferrofluids by comparing it with theoretical models and numerical results.

The only disadvantage of the investigation of magnetite-based ferrofluids with particles having a mean size about 10 nm is their weak tendency of chain formation and the resulting weak viscoelastic properties. Therefore, it is necessary to design a rheometer allowing the investigation of magnetic fluids under the influence of well-defined variable magnetic fields with maximum precision.

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Beside these basic demands the design of a rheometer for magnetic fluids will have to observe some special problems that may occur if such fluids are investigated under influence of magnetic fields.

#### 2. The principal design of the rheometer

The central part of the rheometer is the fluid cell designed as a modified cone plate arrangement. The outer part of the cell is moved, while the cone is attached to a torque sensor allowing to measure the torque transmitted by the fluid in the cell to the cone. The modification is a Couette region attached to the cone plate part (see Fig. 1), which is built in a way, that the torque exerted by the fluid in the Couette region is negligible compared to that exerted by the cone-plate region. The reason for this modification is the possibility of appearance of spikes due to the normal field instability, if a magnetic field is applied parallel to the symmetry axis of the system. In case of a pure cone-plate arrangement this may force measurement errors due to dewetting of the cone. This problem is excluded by the modification using an additional Couette region (Fig. 1).

The cone is mounted to an axis suspended in an airbearing (see Fig. 2) allowing minimum mechanical friction in the bearing of the cone. This kind of bearing of the cone being provided the use of appropriate torque sensors enables the detection of torque changes as small as  $10^{-7}$  Nm. Preceding experiments with conventional ball bearings showed strong influence of the bearing to the measurement. In particular, magnetic fields applied to the cell magnetized the bearings slightly, giving rise to additional, uncontrollable friction effects.



Fig. 1. Principal sketch of the fluid cell explaining the reason for the modification of the normal cone plate arrangement.



Fig. 2. Sketch of the ferrofluid rheometer, explanations are given in the text.

The whole system consisting of torque sensor, axis running in the airbearing and cone is mounted to an adjustment unit allowing lateral movement as well as tilt in two directions vertical to each other. This unit allows to place the cone relative to the center of the cell with a precision of 1  $\mu$ m and to adjust its main level parallel to the cell with an accuracy of 0.1 mrad. To allow a wide range of fluid viscosities to be investigated, we can attach different torque sensors to the system, providing a total torque range from  $10^{-7}$  to 0.1 Nm.

The outer part of the fluid cell is mounted to an additional axis running in two precision ball bearings. This axis can be driven by electromotors using two different gearings allowing rotating as well as oscillatory motion. The gearing for rotating motion is a combination of a planetary gear with transmission 1:25 with a simple spur gear with exchangeable gears. The driving motor provides a frequency range from 6 to 22 Hz. The exchange of gears allows the change of the frequency range, thus extending the basic range of the motor frequency to

 Table 1

 Characterizing parameters of the rheometer

Diameter of cone	76 mm
Diameter of moved cell	80 mm
Cone-plate angle	3°
Torque range	$10^{-5}$ - $10^{-2}$ Nm
Frequency range rotating	0.037-22 Hz
Frequency range oscillating	0.021–20 Hz

a complete frequency spectrum from 37 mHz to 22 Hz.

The gear for oscillating motion combines a worm gear unit for adjustment of the frequency range with an excenter unit providing the oscillatory motion. It allows the use of frequencies in the range from 21 mHz to 20 Hz with oscillation amplitudes between 1 and 5° corresponding to shear rates 0.13 and 628 s<sup>-1</sup>.

The cell itself is mounted on a second adjustment unit providing lateral motion in two vertical directions to align the cell with the rotation center of the driving unit, and a vertical stage to adjust the distance between the bottom of the cell and the tip of the cone. This adjustment is crucial for the flow profile in the cell and therefore for the accuracy of measurement. With our adjustment unit we can define the distance to  $1 \mu m$ , which is sufficiently good to allow precision measurement. The most important data of the rheometer is compiled in Table 1. A magnetic field can be applied to the fluid by an arrangement of coils placed in the vicinity of the fluid cell (the coils are omitted in Fig. 2 for clearness). All parts within the region of magnetic field are of non-magnetic material to avoid disturbances of the field.

# 3. Some results on viscosity changes in a commercial ferrofluid under influence of a magnetic field

With the rheometer described above we have investigated the change of viscosity in a commercial ferrofluid (APG 513A by FERROFLUIDICS) containing 7.2 vol% magnetite particles in an ester as a function of shear rate and magnetic field strength. As it is well known [1,2] the appearance of rotational viscosity is generally expected for suspensions of magnetic particles under influence of magnetic fields. Shortly after discovery of rotational viscosity numerous experiments verifying this effect have been carried out (see e.g. Refs. [3,4]).



Fig. 3. The increase of viscosity in APG513A as a function of field strength and shear rate.

Experiments [5–7] with commercial ferrofluids showed strongly increased rotational viscosity which could not be explained quantitatively with the common theory [1].

Fig. 3 shows the increase of viscosity for APG513A as a function of the strength of a magnetic field applied parallel to the rotation axis of the rheometer for different shear rates measured in rotating mode. The rotation frequencies have been chosen in a way, that these results extend the findings in Ref. [7] to higher shear rates. It is clearly seen, that a very strong viscosity increase appears, which obviously decreases with increasing shear rates. Compared with our earlier experiments [7] a strongly increased accuracy can be observed, which is directly related to the optimization of the rheometer, and which allows a much more precise investigation of the appearing rheological effects.

The observed effects may be explained by the formation of chains containing primary agglomerates of magnetic particles, and by the rupture of these chains due to shear, like it was done in Ref. [7]. It can be expected, that the rheometer, in its optimized version, will enable the extension of the database on rheological behavior of ferrofluids for stable suspensions dramatically, like it was demonstrated here.

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