Magnetic Properties of Carbonyl Iron Particles in Magnetorheological Fluids

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Abstract. Knowledge of the magnetic properties of dispersed magnetic particles is a prerequisite to the design an MR fluid with desired performance. A term specific susceptibility is introduced for characterization of particle susceptibility. The study was performed with the Bartington MS2B magnetic susceptibility system on small samples volume. Specific magnetic susceptibility of iron particles was found to be a linear function of median particle size. Structural change in the fluid, including particle organization, led to susceptibility drift and may affect fluid performance. It was shown that susceptibility data can be used for evaluation of the concentration of carbonyl iron particles in MR fluids.

1. Introduction
Dispersed magnetic particles are the main components of MR fluid and knowledge of their magnetic properties is required to design MR fluids with the desired performance [1 - 3]. Here we report initial susceptibility data for dispersed particles of commercial powders. In MR fluid applications, it is desirable to trace the drift or aging of particle properties (e.g., due to particle oxidation/abrasion/agglomeration while the fluid is in use) that might be responsible for MR fluid performance change. We show that this can be detected by simple magnetic susceptibility measurements.

Reported methodology is based on a small scale (sample volume) and low cost technical means. Two basic cases are considered: first, dilute fluid samples, where individual particles mainly contribute to the magnetic properties of the system, and second, neat concentrated fluids, for which particle interactions and ordering may introduce complex effects.

2. Problem configuration
The Maxwell-Garnett equation for magnetic susceptibility of a suspension of non-interacting spherical particles has the following form [4]: \( \chi = \frac{3\phi\chi_p}{3 + \chi_p(1 - \phi)} \), where \( \chi_p \) is the susceptibility of the particles and \( \phi \) is the volume fraction of the particles. For dilute suspensions, when \( \phi \) is a low number, the equation can be reduced to \( \chi = \phi\frac{3\chi_p}{3 + \chi_p} \). This equation can be further transformed to the linear form

\[ \chi = \chi_s \phi \quad \text{or} \quad \chi_s = \frac{\chi}{\phi} \quad (1) \]
where the slope $\chi_s = 3\chi_p/(3 + \chi_p)$ is named as specific magnetic susceptibility for convenience.

Hence, one can evaluate the specific susceptibility $\chi_s$ from the measured sample susceptibility $\chi$ and the particle volume fraction $\phi$ of the fluid samples and then use this value of $\chi_s$ as a measure for the particle susceptibility $\chi_p$.

3. Experimental: magnetic materials and techniques
Carbonyl iron powders (CIP) of different grades and median particle size, $D_{50}$, were received from BASF (Germany) and ISP (USA). The powders are polydisperse and particles are spherical in shape as observed with optical microscope and scanning electron microscopy images. According to manufacturer’s specification CIP chemical composition includes $> 97\%$ w/w of Fe and the density is $7500$ kg/m$^3$. In one case, the BASF EW iron powder of $D_{50} \sim 3.5$ µm was fractionated to produce samples of large particle sizes with $D_{50}$ up to $\sim 4.5$ µm.

The measurement of the initial magnetic susceptibility was performed with a Bartington MS2B system [5]. Particle sizing for slurries of CIP, involved in the study, was carried out using an acoustic absorption spectrum method (Colloidal Dynamics, Acoustosizer™). Otherwise, CIP manufacturers’ particle size data were used. The concentration of CIP in samples was determined by weight using mass balance, or moisture measurements for water based fluid samples.

4. Results and discussion
4.1. Experiments with diluted samples
The standard 10 ml cell was used. The discussed below results for specific susceptibility were obtained using diluted ($< 2\%$ v/v of CIP in glycerol) fluid samples. The inert and viscous glycerol carrier retarded the sedimentation of dense CIP enabling consistent, multiple measurements. The average values were calculated from the 10 measurements on each sample.

Typical experimental characteristics for three different types of CIP are shown in the figure 1 where magnetic susceptibility values are plotted as a function of concentration for each particle type. The solid line is calculated using equation (1) at $\chi_s = 4$ and well coincides with experimental data for BASF HQ CIP. At the same time, each type of CIP is characterized by a different slope $\chi_s$, which suggests that particles have different susceptibility.

All results of testing are listed in table 1. Even though various commercial CI powders may differ due to some difference in component compositions and internal structure (mechanically hard and soft materials), a significant trend in the specific susceptibility - a dependence on particle size is immediately apparent. This trend is shown in the figure 2 and data for all samples are fitted by the following empirical linear equation: $\chi_s = 0.668D_{50} + 0.961$. Thus, knowing the median CIP size (reported in µm) one can estimate the specific susceptibility of the particles.

4.2. Experiments with concentrated MR fluid
The small 0.27 ml special cell was used in this set of experiments to overcome the problem with the limit of instrument output signal. A large volume (~ 1 liter) of fresh MR fluid with CIP concentration of more than $35\%$ v/v was sheared continuously, by pumping through a closed loop, to simulate real application conditions while small portions were taken periodically, and susceptibility measurements were performed.
Figure 1. Magnetic susceptibility vs. concentration: (▲) ISP S-1000; (●) BASF EW; (■) BASF HQ; (line) by Maxwell-Garnett equation (1) at $\chi_p = 4$.

Figure 2. Specific magnetic susceptibility of different grades CI vs. particle size with linear fit (line).

Table 1. Specific magnetic susceptibility $\chi_s$ of various types of CIP and their particle size

| CI powder ID | Size (D50, µm) | $\chi_s$ (SI unit) | CI powder ID | Size (D50, µm) | $\chi_s$ (SI unit) |
|--------------|----------------|--------------------|--------------|----------------|--------------------|
| BASF HQ      | 1.1            | 1.77               | BASF CC      | 5              | 3.83               |
| BASF HF      | 1.7            | 2.68               | ISP S-1281   | 5              | 3.55               |
| BASF HS      | 2              | 2.53               | ISP R-1521   | 6              | 4.38               |
| BASF EW      | 3.5            | 3.58               | BASF OR      | 6              | 4.68               |
| BASF EW *    | 4.5            | 3.63               | BASF CL      | 6.5            | 4.8                |
| ISP S-1701   | 4.7            | 3.77               | ISP S-1000   | 9              | 8.03               |
| BASF OS      | 4              | 3.82               |              |                |                    |

*Mechanically hard; ^mechanically soft; *fractionated.

It was noticed that the susceptibility increased by 4 to 5% from the initial values within first 30-50 hrs and leveled afterwards. This drift seemed to be related to particle and fluid “structural changes” that occurred in the fluid during use.

The ability of agglomerated/structured CI particles to affect the susceptibility was confirmed in separate tests. Small samples were briefly placed in the field of a permanent magnet. In order to assess the effect of particle organization, two types of samples based on BASF EW CIP were tested: (i) using viscous liquid carrier, glycerol, and (ii) using a rigid composite carrier, polyester resin (Castin' Craft Casting Resin with Catalyst [6]).

As it shown in the figure 3, particles mobility and structuring results in increased susceptibility (by ~12%). The effect was persistent and was not completely diminished by vibratory agitation out of field (left). At the same time the magnetic field did not affect the susceptibility for particles locked in the rigid composite sample (right). A similar effect in permeability measurements has been reported elsewhere [1].

An attempt was made to use equation (1) for analyzing the concentration of neat (concentrated) MR fluid with CIP of defined specific susceptibility by measuring fluid sample susceptibility. This approach could be considered as a simple method for probing fluid concentration for various purposes. Four fluid samples were prepared using CIP with $\chi_s$ of 3.9 and “true” concentration of 0.38, 0.42, 0.44 and 0.46 v/v. Samples were subjected to shear to account for the initial drift in the susceptibility (see previous section). The sample susceptibility $\chi$ was measured for each composition (figure 4),
and CIP concentration was computed using equation (1) and susceptibility data. Finally, the error $\Delta = \frac{(\text{true concentration} - \text{computed concentration})}{\text{true concentration}}$ was calculated. Both computed concentration and error $\Delta$ are plotted in the figure 4. Up to CIP concentration of 0.44 v/v the error $\Delta$ doesn’t exceed 1%. Thus, the simple and quick susceptibility measurements can be used to determine the volume concentration of MR fluid samples, at least for a practical range of volume concentration of $0.38 < \phi < 0.44$ v/v.

![Figure 3. Normalized by initial specific magnetic susceptibility for MR fluid samples of different states based on liquid carrier (left) and rigid carrier (right).](image)

![Figure 4. Sample magnetic susceptibility (open triangles) and computed CIP concentration (solid diamonds) with the error $\Delta$ as a function of the true CIP volume concentration.](image)

5. Summary
- Bartington MS2B magnetic susceptibility system is a convenient tool to evaluate initial susceptibility of carbonyl iron; it requires only a small sample volumes providing quick reproducible measurements;
- Specific magnetic susceptibility, $\chi_s$, of iron particles is found to be a linear function of median particle size. The following empirical equation can be used to define specific magnetic susceptibility if the particle diameter, $D_{50}$[µm] is known: $\chi_s = 0.668D_{50} + 0.961$;
- Structural change, including particle organization such as translation, aggregation and chaining, occurring in the MR fluid during use led to susceptibility drift and may affect fluid performance;
- The linear relationship between sample susceptibility and specific magnetic susceptibility can be used for screening of the particles concentration for a fluid with well defined specific susceptibility.

Acknowledgements
We thank Prof. S. Jacobs at University of Rochester for scanning electron microscopy and for access the Colloidal Dynamics Acoustosizer™ equipment for measurement of particle size.

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