Investigating Sedimentation and Rheological properties of Magnetorheological Fluids using various carrier fluids

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Abstract. The present paper focuses on the preparation of Magnetorheological (MR) fluids samples with three types of carrier fluids are silicone, light paraffin and Poly-alpha-olefin (5, 30 and 400 cSt) viscosity oils with 25% volume fraction of carbonyl iron particles and 3% fumed silica as a thixotropic agent to improve sedimentation of the MR fluid. The morphology, magnetic saturation and phase of the carbonyl iron particles were investigated using field emission scanning electron microscopy (FESEM), super quantum interface interference device (SQUID), X-ray diffraction (XRD) respectively. The results found that obtained powder particles spherical in shape, and a high magnetic saturation of 270 (emu/gm) with the applied field of 15000 (Oe). The prepared MR fluids rheological properties were tested using Anton Paar MCR702 Twin drive rheometer fitted with a magneto-rheological module. Sedimentation stability examined using direct observation method.

Keywords: Carbonyl iron, Fumed silica, Silicone, Light paraffin and Poly-alpha-olefin oils

1. Introduction
MR fluids compositions were first discovered by Rainbow the US National Bureau of Standards [1]. A magnetorheological material normally comprised magnetic particles, typically greater than 0.1 micrometres in diameter, dispersed or suspended in a carrier fluid such as synthetic hydrocarbons, mineral and silicone based oils, or aqueous fluids [2]. In the absence of a magnetic field, the magnetizable particles are arbitrarily suspended in carrier fluids and the suspensions shows a Newtonian-like fluid behaviour. In the presence of magnetic field, the magnetic-responsive particles formed chain-like structures within in the carrier fluid [3]. Meanwhile the yield stress of MR fluids is usually between 10 and 100 kPa, and is more than electrorheological (ER) fluids [4]. MR fluids have been proposed for controlling damping in various devices application, such as dampers, shock absorbers, elastomeric mounts, brakes, clutches, and valves [5]. Many of the magnetic-responsive particles available, carbonyl iron is superior candidate in the MR fluids due to its high saturation magnetization, magnetic permeability and soft magnetic property and ease of availability [6, 7]. Sedimentation
represents a significant problem due to the high-density difference between carbonyl iron particles (7.86 g/cm³) and oil medium [8]. Therefore, to improve the stability against sedimentation, Surfactants such as oleic acid and stearic acid to prevent aggregation of Carbonyl iron particles in MR fluids [9]. Furthermore, a few researchers reported coating the magnetic particles with polymers such as poly(methyl methacrylate), polystyrene, polypyrrole, and polyaniline to reduce the density of particles [10]. The various additives such as fumed silica, organ clay, graphite fiber, carbon nanotubes are comparatively lower density and high specific surface area that are beneficial has been adopted towards to improve the dispersion stability[11].

In this study, Preparation of MR fluid samples with three different carrier liquids like Silicone (5cSt), light paraffin (30cSt), Poly-alpha-olefin (400cSt) oil of different viscosity with 25% volume fraction of carbonyl iron particles and previous study suggests that 3% fumed silica as a thixotropic agent which forms a network through hydrogen bonding for improving stabilization of the MR fluid. These specific relative volume fraction of the MR fluid fumed silica particles, and carbonyl iron particles were preferred due to their high sedimentation stability, as demonstrated in literature [12, 13].

2. Experimental

2.1. Preparation of MR fluid

The constituent required for purpose of stabilization in MR fluid fumed silica (0.2-0.3) μm surface area 200m²/g ± 25 m²/g (aggregate) (Sigma Aldrich) are mixed using homogenizer stirring about 15 min in silicone oil (Sigma Aldrich), light paraffin oil (Spectrum chem. Pvt. Ltd) and Poly-alpha-olefin oil (Chemtura Corporation) with a specific Gravity of (0.96, 0.83 and 0.84 g/cm³) and viscosity range of (5, 30 and 400 cSt.) respectively at until a homogeneous obtained. Afterward, carbonyl iron powder particles density: 7.86 ×10³ kg/ m³, CN grade, Avg. particle size (1-9) microns from (Vimal intertrade Pvt. Ltd, India) mixed in the gel using mechanical stirrer at 900 rpm for about 12 hours. In the present work, the 3 types of MRF samples are designated by MRF-1, MRF-2, and MRF-3

| ID      | Type of Carrier fluid (cSt) | Carbonyl iron (Volume %) | Carrier liquid (Volume %) | Fumed silica (Volume %) |
|---------|------------------------------|--------------------------|----------------------------|-------------------------|
| MRF-1   | Silicone oil (5)             | 25                       | 72                         | 3                       |
| MRF-2   | Light paraffin oil (30)      | 25                       | 72                         | 3                       |
| MRF-3   | Poly-alpha-olefin oil (400)  | 25                       | 72                         | 3                       |

2.2. Characterization

The morphology of the carbonyl iron particles was identified by FESEM (ZEISS ULTRA55). The magnetic properties of carbonyl iron studied though SQUID (MPMSXL5) magnetometer at room temperature with applied magnetic field of 15kOe. The crystal structure of raw carbonyl iron particles was observed by XRD (D max, Rigaku) with Cu/K-α radiation source (1.5418 Å). The magnetorheological fluid properties was investigated by twin drive rotational rheometer (MCR702, Twin drive Anton Paar, India) connected with a magneto–cell (PS-MR 180/1.2T, Anton paar India) produces a homogeneous magnetic field. A parallel plate measuring device dia 20 mm was used with a gap 0.3 mm at 40°C. The suspension stability of CI/fumed silica based MR fluids was studied by direct observation method.
3. Results and Discussions

3.1. X-ray Diffraction

The crystal structure of the carbonyl iron particles was observed by XRD as shown in Figure 1 only the distinct and intense (110), (200) and (211) diffraction peaks at 44.4°, 64.7° and 82.09° related to the α-Fe phase (ferrite, JCPDS card no. 00-006-0696) were noticed in the XRD patterns of the samples[15].

![XRD pattern of Carbonyl iron powders](image)

**Figure 1.** XRD pattern of Carbonyl iron powders

3.2. FESEM and Particle size distribution

![FESEM image of carbonyl iron and Histogram of Particle size distribution](image)

**Figure 2.** FESEM image of (a) carbonyl iron and (b) Histogram of Particle size distribution
Figure 2(a) presents the FESEM image of pure carbonyl particles showing their spherical surface morphology, and a very smooth surface. Figure 2(b) represents the histogram of particle size distribution through image J processing, it was shown that particles size varies from 2 to 9 microns. As can be seen from figure 2(b) more than 60 % of the CI particles have dia 2-9 microns [16]. Finally, pure CI particles were dispersed in carrier liquids to prepare the MR fluids.

3.3. Magnetic measurements using SQUID
The magnetic saturation properties of dry Iron carbonyl (CI) particles were characterized using SQUID at room temperature. The magnetization curve exhibited the magnetic moment of carbonyl iron particles is 270 (emu/gm) at the magnetic field range, −15000 to15000 (Oe). The obtained results are shown in figure 3 where hysteresis loop is very narrow with a high saturation moment, low remanent magnetization and low magnetic coercivity, which symbolises the soft-magnetic property of the CI particles used in the experimentation [17]. The table result shows saturation magnetization carbonyl iron particles.

| Particulars                  | Pure CI   |
|------------------------------|-----------|
| Magnetic moment (emu/g)      | -270 to 270 |
| Density (g/cm3)              | 7.86      |
| Applied Magnetic field (Oe)  | -15000 to 15000 |

Table 2. Magnetic moment and Density for Pure Carbonyl iron particles [18].

![Magnetization curve of carbonyl particles](image)

**Figure 3.** Magnetization curve of carbonyl particles

3.4. Study on Magnetorheological Flow Curves
The Rheometer MCR 702 Twin Drive consists of the stationary bottom plate and rotating top parallel plate dia 20 mm with MRD cell (70/1T). The fluid sample is placed in the gap between the plates. Finding shear stress, for three different compositions like MRF-1, MRF-2 and MRF-3 as follows:
Figures 4 (a) and (b) represent the shear stress versus shear rate graphs and shear stress versus the applied magnetic field in the range 0 to 0.9 Tesla at 40°C. The experiment conducted with the magnetic field and without magnetic field (i.e. off-state and on-state). Figure 4 (a) shows a linear increase in shear stress with shear rate varied from 0 to 200 (γ = 1/sec) at no magnetic field the characteristics of the MR fluid behaves like Newtonian fluid. We found that the shear stress obtained from MRF-1 and MRF-2 were relatively low, which proposes the low viscosity of the base fluid. In the case of MRF-3, a shear stress limit is very high due to high viscosity of base fluid. When magnetic field applied, a different consequence appeared in figure 4(b). Clearly, the as the shear stress increases with increasing applied magnetic field (i.e 0 to 0.9T) for all three samples. The prepared MRF-1 shows higher shear stress 15000 (Pa) compared to MRF-2 12000 (Pa) and MRF-3 9000 (Pa) at a maximum applied magnetic field of 0.9 Tesla. MR performances may effect due to particles size, surface morphology of particles [19]. The MR fluids exhibited the Bingham plastic a minimum yield stress is needed for fluid flow behavior shear stress under applied magnetic field strength, representing the stable chain structures of magnetized particles [20].The equation for the constitutive behaviour is given below.

$$\tau = \tau_\gamma + \mu \dot{\gamma} \quad \dot{\gamma} \geq 0$$

The two constraints of the Bingham-plastic model are where $\tau_\gamma$ is the yield stress as a function of magnetic field, $\dot{\gamma}$ denotes the shear rate and $\mu$ is the shear viscosity [21] The below tables shows results found by MR effect at on-state rheology and off state rheology of tested MR fluids.

| Table 3. Properties of investigated off-state rheology MR fluids [22]. |
|-----------------|---------|--------|-------|
| ID              | Shear stress (Pa) | Shear rate (1/sec) | Temperature °C |
| MRF-1           | 60      | 0-200  | 40    |
| MRF-2           | 125     | 0-200  | 40    |
| MRF-3           | 1400    | 0-200  | 40    |
Table 4. Properties of investigated on-state rheology MR fluids [22].

| ID    | Shear stress (Pa) | Shear rate (1/sec) | Magnetic flux density (Tesla) | Temperature °C |
|-------|-------------------|--------------------|--------------------------------|----------------|
| MRF-1 | 15000             | 100                | 0-0.9                          | 40             |
| MRF-2 | 12000             | 100                | 0-0.9                          | 40             |
| MRF-3 | 9000              | 100                | 0-0.9                          | 40             |

3.5. Sedimentation stability of MR fluids

In order to investigate the effect of sedimentation of MR with three different carrier liquids, sedimentary ratio (R) can be investigated by pouring the magnetorheological fluid in 10 ml of measuring cylinder without disturbance at room temperature [23]. To identify the sedimentation dispersion stability, experiment is performed placing the MR fluid samples ideal for 750 h and sedimentation rate of particles in MR suspension was taken at a regular interval. Sedimentation ratio can be defined as:

\[
\text{Sedimentation ratio (\%) = \left(\frac{\text{Volume of supernatant fluid}}{\text{Total volume of MR fluid}}\right) \times 100}
\]

![Figure 5](image)

Figure 5. Sedimentation ratio curves three different carrier based MR fluids

Figure 5 shows the sedimentation curves of three different carrier based MR fluids samples were used for the examination of dispersion stability varying in treatment time (i.e. MRF-1, MRF-2 and MRF-3). It is clear from Figure 5 that the MRF-3 based fluid lowest sedimentation ratio of 97% compared to MRF-1 and MRF-2 due to high viscosity of the base oil. The inset figure shows final results of the sedimentation after 750 hour for MRF based suspensions.
4. Conclusions
We prepared MR fluids containing carbonyl iron/fumed silica with three different carrier liquid, viscosity (5, 30 and 400 cSt) and FESEM, XRD confirmed the spherical morphology and excellent crystalline structure of the carbonyl particles. The magnetic property with a saturation magnetization (270 emu/g) was confirmed by SQUID.
The MR performance for prepared MR fluids was studied by using a twin drive rheometer. The maximum shear stresses of reaching 15000, 12000 and 9000 Pa respectively in the magnetic field range 0–0.9 T at 40°C.
The sedimentation stability during direct observation we found that the MR fluid with fumed silica as thixotropic additive slower sedimentation stability and the ratio was observed 90, 93 and 96% of MRF-1, MRF-2 and MRF-3 respectively during 750 hrs.

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