Properties of plate-like carbonyl iron particle for magnetorheological fluid

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Abstract. This work experimentally discussed the characterization, magnetic, and rheological properties of plate-like carbonyl iron particle (CIP) in comparison with conventional spherical CIP. Plate-like CIP was produced by using ball milling method. The effect of plate-like shape on the magnetic behavior of CIP was firstly investigated by vibrating sample magnetometer (VSM). The results indicated that the plate-like CIP obtained higher saturation magnetization (about 8%) than that of the spherical particles. In addition, the field-dependent rheological properties such as yield stress were investigated and the results are compared between two particles as a function of the magnetic field intensity.

1. Introduction
Magnetorheological fluid (MRF) is a magnetic field responsive fluid containing polarizable particles and a liquid carrier. MRF is typically used in automobile application such as dampers to provide varied damping forces, depending on a magnetic field strength. In general, the apparent viscosity of MRF is changeable with the application of magnetic field strength [1]. This happens due to the suspended magnetic particles in non-magnetic medium of MRF which is aligned as a chain-like formation and restricted the fluids flow [2]. This phenomenon developed the yield stress in MRF. The magnetic and rheological properties of MRF are highly dependent on the magnetisable particles features. It has been stated that they play the important roles in achieving high yield stress and low sedimentation of MRF [3]. In recent researches, numerous types of magnetisable particles are widely used as constituents of MRF, such as cobalt alloy, nickel alloy, soft ferrite, manganite and CIP [3, 4]. CIP is the most common magnetic particles used in MRF fabrication due to its promising permeability, relatively low electrical conductivity, high Curie temperature, high saturation magnetization and low cost in comparison with other metallic particles [2, 3, 4]. CIP based MRF is also capable to operate with the yield stress of 100 kPa and has a high saturation magnetization about 2.1 T [5].

The development of MRF with the aim of achieving high yield stress has extensively been studied in order to enhance the maximum output force of the application devices or systems [6]. It is well known that the yield stress can be increased by increasing the particles amount and particle size. However, this may cause high yield stress at the off-state and severe sedimentation problem [7, 8]. The high yield at the off-state restricts controllable range of the field-dependent force or torque in application systems. Therefore, many researchers have focused on improving the rheological properties of MRF through adjusting magnetisable particles behaviours such as coating of magnetic particles, addition of additives and bi-dispersing of nano size particles [9].

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Recently, the particle shape has been considered to be the most important factor to overcome the sedimentation problem and enhance the output force [3, 9, 10, 11]. As reported by Upadhyay et al. [9], the flake-based MRF exhibited a higher storage modulus compared to sphere-based MRF. This is due to the field dependent solid friction between the flake shaped particles. In addition, the sedimentation rate decreased about 50% once the flake-like shape particles were used. With this regard, Gomez-Ramirez et al. [10] prepared CoNi-based MRF with different shape of magnetisable particles; CoNi nanofibers (56 nm length, 6.6 nm width) and nano spheres (24 nm diameter). It was found that the MR effect remarkably strengthened once nanofibers were utilized as dispersed phase instead of nano spheres due to the formation of the strong effect of fibre orientation on the demagnetizing field. Comparing the rod-based and sphere-based MRF revealed that the rod based MRF had an enhanced MR performance under oscillatory shear [11].

The main technical contribution of this work is to introduce plate-like CIP based MRF and compare the field-dependent rheological properties with the spherical particles based MRF. The plate-like particles are prepared through the solid state powder processing technique which involves repeated welding, fracturing and re-welding of powder particles in a high-energy ball mill. After testing X-ray diffraction of the particles before and after milling process, the particle shapes are observed via SEM (Scanning Electron Microscopy). Then, the yield stress are evaluated and compared between two different particle shapes based MRF. It is noted here that to the best of our knowledge, no study has been conducted to prepare the plate-like CIP and investigated the effect of the plate-like CIP on the rheological behaviours of MRF.

2. Experimental

In this study, the OM series of CIP manufactured by Basf Company was used with average size of diameter and density are 4 µm and 7.874 g/cm$^3$ respectively. In order to synthesize plate-like shape of CIP, rotary ball mill method was used. The milling process was operated at 200 rpm for 20 hours in room temperature by using wet milling method. Zirconia ball was used as grinding media with ball to powder weight ratio is 20:1. Pure ethanol was added as process controlling agent (PCA) to overcome adhesion between particles and to improve the efficiency in synthesizing plate-like shape particles.

Polyalphaolefin (PAO) oil produced by Ferrotec Company was used as carrier fluid in MRF preparation, and oleic acid from R&M Chemicals with density of 0.9 g/ml is adopted as additive. Two types of MRF are individually prepared using spherical and plate-like CIP. The phase structure analysis of both particles are identified (within 2° range of 35–90°) using powder X-ray diffractometer (PANalytical XRD) employing Cu-Ka X-radiation of wavelength 1.5418Å. A scanning electron microscopy (JEOL JSM-7001F SEM) was used to analyze the surface morphology and the average size of the particle. Magnetic behavior of the particles were characterized using a vibrating sample magnetometer (Lakeshore 7404 series VSM) in magnetic field range of 10000 Oe. In addition the rotational flow tests are carried out at 25 °C using a parallel-plate configuration in the MCR 302 Anton Paar rheometer. The 20-mm diameter plate and Magneto-Rheological Device (MRD 70 Anton Paar) are attached for on-state measurement. The on-state measurement for the shear stress and viscosity is carried out at conditions; magnetic field from 100 mT until 500 mT and rate sweep until 1000/s.

3. Results and Discussion

3.1. Structural characterization

Figure 1 shows the X-ray diffraction (XRD) pattern for spherical and plate-like carbonyl iron particle (CIP) at 25 °C. The diffraction peaks for both samples can be indexed to the body centre cubic structure (BCC α Fe) with Im-3m space group. The intensity peak was observed to increase as the CIP undergoes the milling process. A sharper and broaden of diffraction peak was obtained by plate-like CIP due to the decreasing of the crystallite size and the increasing of the internal lattice strain [12]. Furthermore, the peaks widths broaden are also attributed to the accumulation of lattice defects such as grain boundaries and dislocations during the milling process [12]. Herewith, the changes observed in
the XRD patterns suggest that the particle shape is changed from the spherical to the plate-like. Although, there is no impurity peak was observed and the structure of CIP was preserved where the diffraction peak for both shapes of CIP was aligned at the same angle.

![Figure 1 X-ray Diffraction graph for spherical CIP and plate-like CIP at T = 293 K.](image)

The SEM micrographs for both spherical and plate-like CIP are shown in Figure 2. It is evident that the particles are flattened and varied to the plate-like shape with more homogeneous distribution after the ball milling process. It is also obvious that the particles become more dispersed after ball milling (Figure 2b). This is because the collision between balls, vial and powders leads to break the cluster form of particles. From the SEM micrograph, the average particle size is measured and the diameter size for spherical and plate-like CIP is 2 µm and 3.6 µm respectively. The particle length was increased about 80% after the ball milling process with an average thickness about 0.86 µm.

![Figure 2 SEM micrograph for a) spherical CIP and b) plate-like CIP at x3000.](image)
3.2. Magnetic characterization
M-H curve for both spherical and plate-like CIP was shown in Figure 3. Magnetic saturation was changed as the shape of CIP changed, which is induced by the milling process. The plate-like CIP exhibits higher saturation magnetization compared to the spherical CIP. Indeed, the saturation magnetization for the plate-like CIP is 211.3 emu/g while the spherical shape CIP is only 195.7 emu/g. This happens due to the shape anisotropy in magnetic anisotropy where it can only be obtained by non-spherical particles. Not perfectly spherical particles lead to the unequal direction of demagnetization field. Hence, one or more easy axis can be created where the magnetic domains tend to align. In addition, the magnetised particles will align with their long axes in the field direction, which results in the polarization charge concentration at the upper and lower edges.

3.4. Rheological properties
Flow curves of shear stress versus shear rate for both plate-like and spherical CIP based MRF are shown in Figure 4. Different magnetic field strengths are applied to investigate their MR effect. It is found that the shear stress increases much faster at a lower shear rate and then changes slowly at a high shear rate. This is due to the fact that disordered arrangement of particles leads to the enhancement of flow resistance at lower shear rate, while particles begin to arrange their orientation along the direction of shear at higher shear rate, causing to the decrease of viscosity [13]. Further, the flow curves obtained by both samples are best fitted with Herschel-Bulkley (H-B) model to describe the shear yield stress value [6]. In this model, the shear stress is given by

$$\tau = \tau_0 + k\dot{\gamma}^n$$  \hspace{1cm} (1)

where $\tau_0$ is the shear yield stress in the state of presence and absence applied magnetic field, $\tau$ is the shear stress, $k$ is the consistency index, $n$ is the shear thinning exponent and $\dot{\gamma}$ is the shear rate. The term $k\dot{\gamma}^n$ shows the shear thinning behaviour of the suspensions fluid under the magnetic field. The $n$ describes the degree to which a material is shear thinning ($n < 1$) or shear thickening ($n > 1$). The values of $n$ analysed from this both samples are lower than 1, indicating that the rheological behaviour for both fluids are shear thinning. The shear yield stress values, which are obtained by fitting the H-B model to the experimental curves, are plotted as a function of magnetic field strength as shown in Figure 5.
As presented in Figure 5, the shear yield stress for both MRF samples is observed to increase with increase of the applied magnetic field strength. This is more probably due to the stronger pull force among magnetic particles in the presence of applied magnetic field. As the magnetic field intensity is greater, a larger shear stress is needed to break the column-like structures and fluid to start to flow. It is interesting to notice that the plate-like CIP based MRF exhibits a sharp increase of shear yield stress value compared to the spherical CIP based MRF. Hence, a greater yield stress about 81494 Pa obtained by plate-like CIP based MRF compared to the spherical CIP based MRF (about 21979 Pa) at 700mT. Indeed, the yield stress achieved by plate-like based MRF is increased up to 270% compared to its original spherical shape. This is because the plate-like particles have larger contact surface areas which lead to a strong attraction between particles and consequently the clusters become hard to break and flow.
4. Conclusion
In this work, two different shape of CIP for MRF composition were prepared and experimentally investigated the field-dependent rheological properties. It has been observed from XRD and SEM that no new phase is occurred by milling the spherical shape to the plate-like shape and the plate-like CIP is flattened resulting in 86% of the length enhancement. This resulted in higher saturation magnetization compared to the original un-milled sample. This happen because the plate-like CIP has larger contact surface which formed stronger clusters due to magnetic field. Finally, the field-dependent rheological test has been demonstrated that the yield shear stress of the plate-like CIP based MRF is increased by 270% compared to the spherical particles based MRF.

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6. References
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