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Full length article

Synthesis and characterization of magneto-rheological (MR) fluids for MR brake application

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ABSTRACT

Magneto rheological (MR) fluid technology has been proven for many industrial applications like shock absorbers, actuators, etc. MR fluid is a smart material whose rheological characteristics change rapidly and can be controlled easily in presence of an applied magnetic field. MR brake is a device to transmit torque by the shear stress of MR fluid. However, MR fluids exhibit yield stress of 50–90 kPa. In this research, an effort has been made to synthesize MR fluid sample/s which will typically meet the requirements of MR brake applications. In this study, various electrolytic and carbonyl iron powder based MR fluids have been synthesized by mixing grease as a stabilizer, oleic acid as an antifriction additive and gaur gum powder as a surface coating to reduce agglomeration of the MR fluid. MR fluid samples based on sunflower oil, which is bio-degradable, environmentally friendly and abundantly available have also been synthesized. These MR fluid samples are characterized for determination of magnetic, morphological and rheological properties. This study helps identify most suitable localized MR fluid meant for MR brake application.

1. Introduction

Magneto-rheological fluid (MRF) is a type of smart material whose rheological properties (e.g. Viscosity) can be rapidly varied by applying a magnetic field. It is a free-flowing liquid state in the absence of a magnetic field while its viscosity increases on application of magnetic field [1].

For an MRF, the yield stress can be controlled which can be increased or decreased with the strength of the magnetic field as shown in Equation (1).

$$\tau = \tau_{y(H)} + \mu_p \dot{\gamma} \quad (1)$$

where, τ_y is the yield stress due to the applied magnetic field H, μ_p is the constant plastic viscosity and $\dot{\gamma}$ is the shear-strain rate. The typical properties of MR fluid are as shown in Table 1.

Many MR fluid applications operate under different modes like valve mode, shear mode and squeeze mode. The MR brake (MRB) is a device to transmit torque by the shear stress of MRF. Hence, MRB operates in a direct-shear mode, shearing the MR fluid filling gap between the two surfaces.

In the shear mode, the MR fluid is located between surfaces moving (sliding or rotating) in relation to each other with the magnetic field owing perpendicularly to the direction of motion of these shear surfaces as shown in Fig. 1 The characteristic of shear stress versus shear rate can be controlled by the magnetic field [5].

As shown in the Fig. 2, MRB consists of a rotating disk immersed in MRF, enclosed in an electromagnet. The yield stress of a fluid varies as a function of magnetic field applied by an electromagnet.

In MRB, the gap between stator and rotor is filled with low (off-state) viscosity MRF. On the application of magnetic field, MRF changes its state from liquid to semi-solid. Each magnetic particle forms north and south poles and hence, the opposite poles attract each other which results in strong bonding between them. This aligns them in a strong chain. Due to such chaining action, yield strength of fluid increases, which opposes the friction between stator and rotor and hence fulfills the braking function [2]. The strength of chain formed by magnetic particles due to bonding is a function of relative speed between stator and rotor, applied magnetic field and volume percentage of magnetic particle.

MRBs have been explored recently as an alternative to conventional hydraulic brakes for road vehicle applications. Park et al.

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Table 1
Properties of typical MR fluids [2–4].

Property	Typical value
Initial viscosity	0.2–0.5 [Pa s] (at 25 °C)
Density	3–4 [g/cm^3]
Magnetic field strength	150–250 [kA/m]
Yield point	50–100 [kPa]
Reaction time	15–25 ms
Work temperature	−50 to 150 °C
Typical supply voltage and current intensity	2–25 V, 1–2 A

[7] and Karakoket al. [8] have assessed MRBs for a typical medium sized car; however the braking torque generated by this brake application has been found to be inadequate. Sukhwani and Hirani [9] experimentally evaluated MRB performance parameters for high speed MR brake application and advocated MR gap of 1 mm. Attempts have also been built in the recent past to optimize MRB design torque and weight meant for automotive application. Patil and Sawant [10] have made an attempt to evaluate MRB system intended for vehicular application from a reliability perspective.

To reduce the sedimentation of MR fluid particles Fang et al. [11] introduced single-walled carbon nanotube (SWNT) in the CI based MR fluid. Shetty and Prasad [12] synthesized MR fluid with a non-edible vegetable oil such as Honge oil as a carrier liquid and they reported that the yield stress produced is only 25 kPa. Sarkar and Hirani [1] discussed the synthesis of MR fluid and its application in braking point of view. Choi et al. [13] encapsulated CI particles with poly methyl methacrylate as core–shell structured particles to improve dispersability of the MR fluid. Jiang et al. [14] added wire-like iron nanostructures into the conventional CI based MR fluid and thus, synthesized a type of dimorphic MR fluid. Present work makes an endeavor to explore best suited MRF synthesized typically for MRB. Earlier, some research studies based on MR fluid synthesis and characterization for general purpose have been attained; however, past literature doesn't provide evidence of MRFs synthesized for vehicular MRB [1,2,4,12–14].

In the present work, research findings of synthesis and characterization of MRF samples aimed at MRB, based on carbonyl iron (CI) and electrolytic iron (EI) powder with oleic acid and grease as additives have been reported.

2. Synthesis of MR fluid

Six MRF samples were synthesized based on the following requirements from brake application point of view.

2.1. Requirements

2.1.1. Low off-state viscosity

The field-independent viscosity (η) is the most critical off-state property of MRFs. The MR-fluid viscosity is most influenced by two factors: the intrinsic viscosity of the carrier fluid and the particle volume fraction [2]. Higher the particle volume fraction, higher is the MR-fluid viscosity. In case of high OFF state viscosity, the vehicle will experience more drag as compared to the low OFF state viscosity even though brake is not applied.

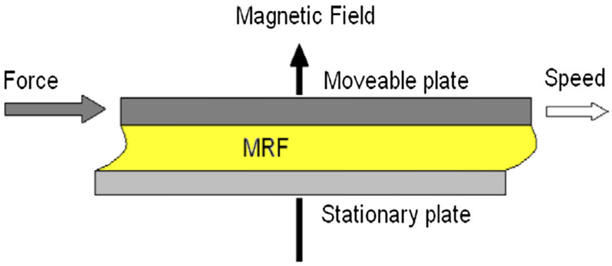


Fig. 1. Shear Mode [6].

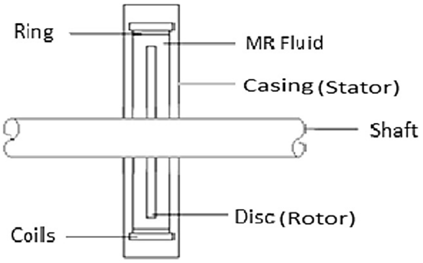


Fig. 2. Schematic of MRB.

2.1.2. High yield stress

The material of the particles has an impact on the maximum yield stress since its value increases with the square of the saturation magnetization of the particles. Another factor influencing the maximum yield stress is the particle volume fraction [2,6]. An increase in the particle volume fraction leads to an increase in the output torque of the MRB. A number of researchers have shown that the maximum yield stress increases non-linearly with growing particle volume fraction and its value is nearly 50–90 kPa [13,14]. For brake application, high yield stress results into high braking torque.

2.1.3. Less In-Use-Thickening (IUT)

If an ordinary MRF is subjected to high stress and high shear rate over a long period of time, the fluid will thicken. This phenomenon is called In-Use-Thickening (IUT) [2,6]. Due to this increased OFF state viscosity owing to IUT, drag increases and results in the power loss when brake is not applied.

2.1.4. Wide temperature range

MRF meant for MRB should withstand to broad temperature range as the brake may be operated at subzero temperature as well as prolonged time which shall result in temperature rise. Commercial MRF is reported to be able to withstand from 80 °C [3].

2.2. Selection of MRF components

MRF typically comprises a liquid carrier, magnetic particles and additives. The subsection below highlights the selection of these components.

2.2.1. Liquid carrier

Carrier liquid is the major constituent of MRFs (50–80 percent by volume) [12]. The commonly used carrier liquids are mineral oil, synthetic oil and silicone oil.

The mineral oils are neither biodegradable nor environmentally friendly, whilst synthetic oils cost more [2]. Synthetic oil possesses important properties like higher flash point, does not thicken at higher temperatures, lower friction, high shear strength and high viscosity index. Silicone oil has good temperature-stability and good heat-transfer characteristics, oxidation resistance [12,13], very low vapor pressure, and high flash points. There is little change in physical properties over a wide temperature span and a relative flat viscosity temperature slope and serviceability from −40 to 204 °C [2,4]. Thus, for the proposed study, synthetic oil and silicone oil were selected.

The limitations of commonly used liquids have motivated exploration of sunflower oil as an alternative carrier liquid for said application. After comparing properties of various alternative carriers like soybean oil, sunflower oil, honge oil; edible vegetable oil like Sunflower oil, which is bio-degradable, environmentally friendly, and abundantly available in many parts of India was selected as a carrier liquid in the present work. Past literature has not reported use of the same as MRF carrier liquid; hence, samples were synthesized with sunflower oil as a carrier fluid.

2.2.2. Magnetic particles

The size of magnetic particles is of the order of 1 μm–10 μm [2]. The sedimentation increases with an increase in the size of magnetic particles and hence, to get optimum effects EI and CI powders of 10 micron, were used for this study.

With the help of the hysteresis loop, the magnetic properties of the material can be determined. A hysteresis loop shows the relationship between the amount of induced magnetization (M) and the magnetic field (H) applied to a magnetic material as shown in Fig. 3.

Where, the retentivity is a material's ability to retain a certain amount of residual magnetic field when the magnetizing force is removed after achieving saturation. The coercive Force is the amount of reverse magnetic field which must be applied to a magnetic material to make the magnetic flux return to zero. Permeability is a property of a material that describes the ease with which a magnetic flux is established in the component and the reluctance is the opposition that a ferromagnetic material shows to the establishment of a magnetic field. Reluctance is analogous to the resistance in an electrical circuit [2,14,15].

Low retentivity and coercivity, high saturation magnetization, high permeability, small remnance and high electrical resistivity are the preferred magnetic properties in the formulation of MR fluids as it bounds smaller hysteresis loop [2,15].

The percentage of these particles varies from 30 to 50 percent by volume [2,3]. OFF state viscosity increases with increase in volume of magnetic particles, however, increased magnetic particle volume also increases the attainable yield strength by MR fluid which is desired for braking application. Hence, for first four samples as shown in Table 3, magnetic particles of 45% by volume are selected as the OFF state viscosity obtained is within 0.5 Pa s. However, the viscosity of sunflower oil is far less as compared to silicone oil and synthetic oil and hence, the OFF state viscosity of MR fluid prepared with Sunflower oil as a carrier fluid with 45% by volume of magnetic particles is found to be far less as compared to the first four samples (Refer Table 3). Thus, volume percentage of magnetic particles is increased to 60% to obtain higher yield strength for last two MR fluid samples (Refer Table 3) and OFF state viscosity is also maintained within 0.5 Pa s.

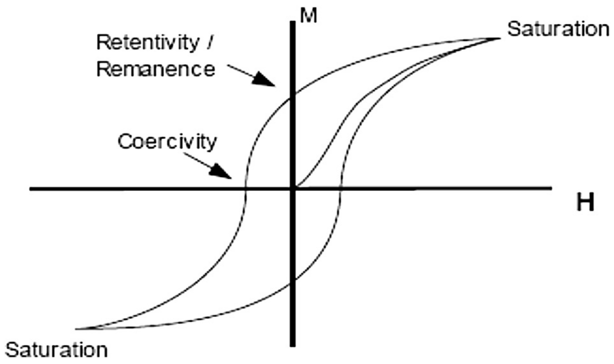


Fig. 3. M–H Curve [2].

2.2.3. Additives

The stabilizers serve to keep the particles suspended in the fluid, whilst the surfactants are adsorbed on the surface of the magnetic particles to enhance the polarization induced in the suspended particles upon the application of a magnetic field [3]. In this study, highly viscous material grease was used to improve settling stability while oleic acid was used as an antifriction additive. Coating of magnetic particles improves the sediment stability and reduces the agglomeration of the MR fluids [16]. For this study, gaur gum was selected to coat the EI and CI powders.

2.3. Nomenclature system

The nomenclatures used in this work for samples, is produced in Table 2.

2.4. MRF synthesis plan

Table 3 presents MRF samples synthesized for this study.

3. Characterization

MRF samples as well as Lord's 132DG MRF were characterized using rheometer, Scanning Electron Microscope (SEM) and Vibrating Sample Magnetometer (VSM). Rheological measurements include the determination of yield stress, viscosity, shear rates and were carried out using Parallel Plate Rheometer. Determination of the morphological state of the sample was done with the help of SEM. Magnetic properties like magnetic saturation (Ms), coercivity (Hci), retentivity (Mr), permeability were measured using VSM.

These results are then discussed in context of the braking application. Emphasis has been given on the yield strength produced by MR fluid and the stability of MR fluid as they affect the braking action to a greater extent. More is the yield strength produced by MR fluid, more is the torque generated by MR brake and hence, discussion is particularly made to correlate the suitability of the different MR fluid components and hence, MR fluid samples for braking action.

3.1. Magnetic properties measurements

The magnetic properties of EI and CI powders have been determined by using VSM as shown in Fig. 4.

A cylindrical sample of the magnetic particles have been prepared and placed between strong magnetic coils of VSM. Under a constant magnetic field, when the mechanical vibrations are applied to a sample of magnetic material the voltage is induced in the pickup coils which is proportional to the magnetic moment of the material. Thus hysteresis (M–H) curve has been drawn to know different magnetic properties such as saturation magnetization, coercivity, retentivity, etc.

Table 2
Nomenclature system.

Sr. No.	Nomenclature	Interpretation
1	ESi 45%	45% EI and silicone oil
2	CSi 45%	45% CI and silicone oil
3	ESy 45%	45% EI and synthetic oil
4	CSy 45%	45% CI and synthetic oil
5	ESu 60%	60% EI and sunflower oil
6	CSu 60%	60% CI and sunflower oil

Table 3
Synthesis plan.

Sample name	Carrier fluid	Metal particles	Additive package (grease + oleic acid)
MRF ESi 45% (250 ml)	Silicone oil [49% by vol.]	EI powder [45% by vol.] ~10 μm	1% by vol.
MRF CSi 45% (250 ml)	Silicone oil [49% by vol.]	CI powder [45% by vol.] ~10 μm	1% by vol.
MRF ESy 45% (250 ml)	Synthetic oil [49% by vol.]	EI powder [45% by vol.] ~10 μm	1% by vol.
MRF CSy 45% (250 ml)	Synthetic oil [49% by vol.]	CI powder [45% by vol.] ~10 μm	1% by vol.
MRF ESu 60% (250 ml)	Sunflower oil [35.20% by vol.]	EI powder [60% by vol.] ~10 μm	3% by vol.
MRF CSu 60% (250 ml)	Sunflower oil [35.20% by vol.]	CI powder [60% by vol.] ~10 μm	3% by vol.

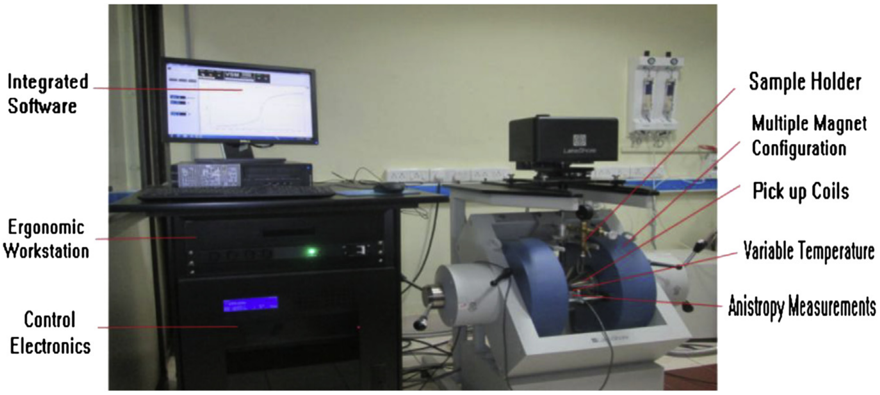


Fig. 4. Vibrating sample magnetometer (VSM).

3.2. ON state measurements

A parallel-plate rheometer as shown in Fig. 5 was used to measure the characteristics of the MR fluids. The setup enclosed 5 major parts: a fixture for the MR fluid, a mechanical input, an electromagnetic circuit, a compression load cell and a data acquisition system. The fixture for the MR fluid consisted of 2 parallel plates. The lower plate was connected to a linear motor that adjusted the dimensions of the gap between the 2 plates. An optical laser sensor used to monitor the movement of the lower plate so as to ensure a constant gap throughout tests. Shear actions were produced by a D.C. servo motor. An optical encoder was used to measure the speed of the motor. An electromagnetic circuit with 3500 turns of coil providing about 10 mT to 1 T was designed by using ANSYS. Magnetic pole radius was 20 mm. A magnetometer was used to measure the magnetic field (B-field). However, magnetometer probe inserted directly into the MR fluid couldn't be used to measure the real magnetic field. Within the MR fluid, it was inserted into the gap between the upper pole and also the upper plate. A calibration was done before the experiments were conducted. A compression load cell was fixed beneath the shaft of the lower plate to measure the normal force of the excited MR fluid. Signals from the load cell and the incremental encoder of the motor were captured by data acquisition units and recorded in a laptop for analyses.

3.2.1. Measuring procedure

Experiments were performed to study the responses of the MR fluid samples along the field direction. The specimen MR fluid contained in its original bottle was shaken and de-gassed in a vacuum before its use. A specific MR fluid quantity was taken onto the lower plate using a clean syringe. Elevation to the lower plate is then made to form a gap of 1 mm between the plates, which was monitored by the laser sensor. Pre-shearing was produced by revolving the upper plate with 2.2 s^{-1} was applied to the MR fluid for10 min to ensure good dispersion. To start with, the readings for the load cell under no field and zero shear strain conditions were recorded for the first 3 min. The desired field was then applied and the normal force data were captured from the load cell with a sampling rate of 10 Hz for 30 min. The experiment was carried at a constant shear rate of 100 s^{-1}which is imposed on the MR fluid by a

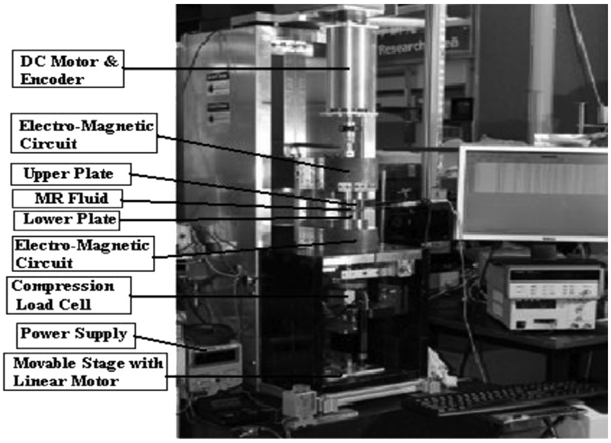


Fig. 5. Parallel plate rheometer.

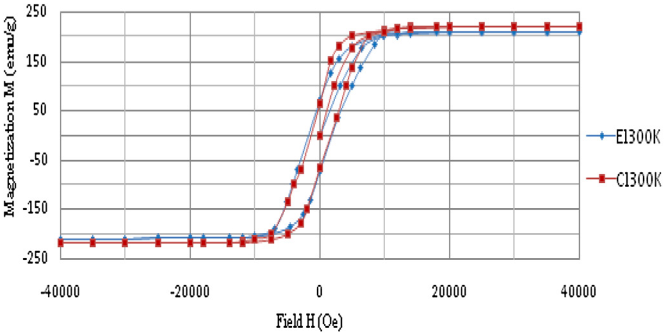


Fig. 6. Hysteresis curves for EI and CI powders at 300 K (27 °C).

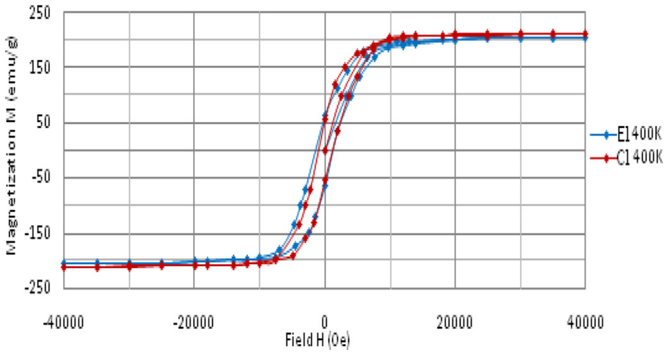


Fig. 7. Hysteresis curves for EI and CI powders at 400 K (127 °C).

motor. The normal force was first recorded for 5 min. After the experiments, the MRF was demagnetized by applying an inverse impulse magnetic field. The experiment was repeated for other magnetic fields.

3.3. *Stability of MR fluid*

Stability of MR fluid samples depends on the content of iron particles and the type of the oil used. Sedimentation is one of the undesired properties of MRF which makes it unstable [9]. The sedimentation was measured by visual observation of the position changes of boundary between clear and turbid part of carrier oil. Prepared samples were placed into cylindrical glass test tubes (length 0.5 m, diameter 40 mm) for 3 days. As a result sedimentation ratio (R) was calculated. Thus, sedimentation ratio can also be defined as a proportion between length of clear and turbid part of MR fluid as shown in Equation (2):

$$R[\%] \ = \frac{A}{B} \times 100 \tag{2}$$

where: R[%]—sedimentation ratio, A—length of the clear part, B—length of the turbid part.

4. Results and discussions

This section deals with results of characterization studies of MRF samples.

4.1. *Measurements of magnetic properties*

The measurements of EI and CI powders were made at 300 K (27 °C) and 400 K (127 °C). These temperatures correspond to room temperature and rise in temperature after braking. As the temperature involved into the braking is more so the purpose is to only check weather at higher temperature, MR fluid employing CI or EI powder sustain their magnetic properties or not and how the behavior of these powder changes from room temperature to higher temperature.

The hysteresis loops for the EI and CI powders at a temperature of 300 K (27 °C) have been shown in the Fig. 6.

Hysteresis loop found in case of CI and EI powders at 300 K (27 °C) temperature is almost similar only the value of magnetic saturation is considerably more in case of CI powder which is desired in the formulation of MR fluid for brake application. Magnetic saturation of particles is directly proportional to the strength of the chains formed among these magnetic particles which ultimately increases the attainable ON state shear strength and hence, braking torque generated.

The hysteresis loops for the EI and CI powders at a temperature of 400 K (127 °C) have been shown in Fig. 7.

At a higher temperature (400 K (127 °C)), this small difference in magnetic properties of CI and EI powders changes proportionally. At this temperature also magnetic saturation of CI powders remains slightly higher than that of CI powder.

Magnetic properties for EI and CI powders at 300 K (27 °C) have been given in Table 4.

The best-in-row values of stated magnetic properties which favor the braking action have been highlighted with gray shading as shown in Table 4. Thus, with above properties MR fluid sample employing CI powder produces considerably good magnetic properties as compared to the samples which employ EI powders.

4.2. *Morphological measurements*

Morphological properties of the magnetic particles used in the formulation of MR fluid contributes a lot as they directly relates to the amount of yield strength produced by the fluid. Fig. 8 shows characteristic morphologies of pure EI and CI particles. Flakelike shape is observed in case of EI powder as shown in Fig. 8(a) while the spherical shape is observed in case of CI powder as shown in Fig. 8(b). The spherical shape helps to minimize magnetic shape anisotropy and hence depicts better properties [2]. This helps to increase the strength of the bonds which are formed among magnetic particles which further leads to increase the yield strength of the fluid.

Fig. 9(a) shows the difference between the morphology of coated EI and coated CI powders. As shown in the Fig. 9(a) and (b), the coating layer of gaur gum has been formed on the surface of the EI and CI powders. This guar gum coating not only greatly improves the sedimentation stability, but also strengthens the yield stress of the MR fluid.

4.3. *Rheological measurements*

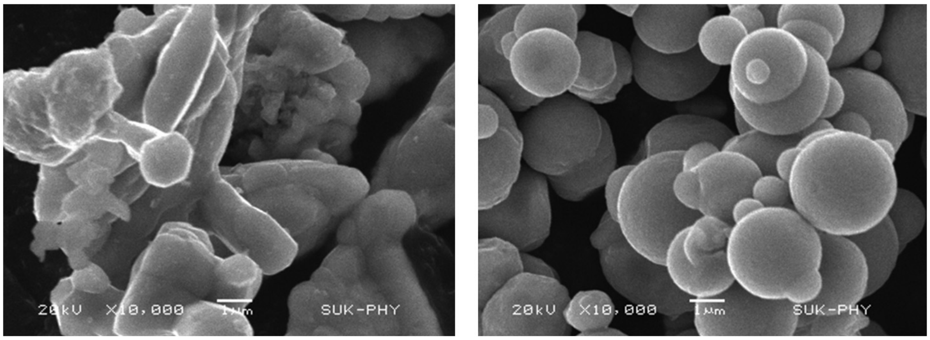
The OFF state and ON state measurements of the properties like yield stress, shear strain rate, viscosity of all the samples were carried out.

4.3.1. *OFF state measurements*

Lord Corporation 132 DG MRF and locally synthesized MRF samples were characterized on ANTON PAAR Modular Compact Rheometer. OFF state viscosity measured for various MR fluid samples are shown in Table 5.

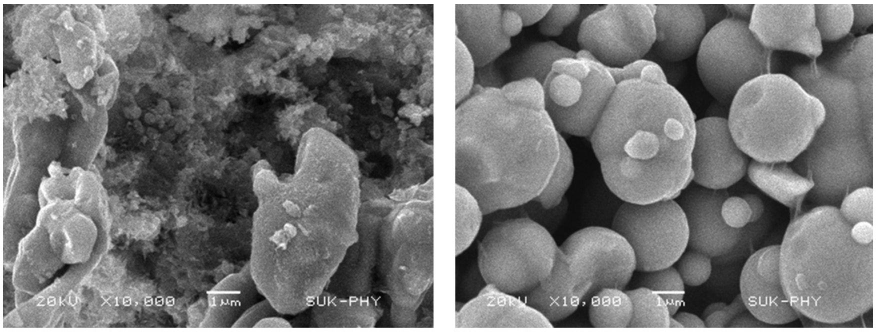
Table 4
Magnetic properties for EI and CI powders at 300 K (27 °C) and 400 K (127 °C).

Magnetic properties	300 K (27 °C)		400 K (127 °C)		Remark
	EI	CI	EI	CI	
Ms (emu/g)	210.3	220.0	204.1	211.2	CI is favorable for braking application
Hci (Oe)	1775.2	1477.1	1207.1	907.3	
Mr (emu/g)	72.15	65.17	62.15	54.99	
Field at Ms (Oe)	20.00E+3	25.00E+3	22.00E+3	28.00E+3	EI is favorable for braking application



(a) (b)

Fig. 8. SEM images of (a) EI powder (b) CI powder.



(a) (b)

Fig. 9. SEM images of (a) Coated EI powder (b) Coated CI powder.

Table 5
OFF state viscosity measurements.

Property	MR fluid samples						
	MRF ESi 45%	MRF CSi 45%	MRF ESy 45%	MRF CSy 45%	MRF ESu 60%	MRF CSu 60%	Lord MRF 132DG
OFF state viscosity (Pa s)	0.26	0.27	0.28	0.3	0.32	0.35	0.2

The shear stress for various samples was then plotted against varying shear rate which is shown in Fig. 10.

As shown in Fig. 10, as the shear rate increases, shear stress goes on increasing. This makes the fluid to offer more resistance to the rotation of the disc with an increase in shear rates. The highest value of shear stress is found to be 118.02 Pa as in case of MRF CSi 45%, while the value of Lord 132 DG MRF is 104.2 Pa. The MRF ESu 60% and MRF CSu 60% indicate the comparatively lowest shear stress values which lie in between 70 and 90 Pa. The shear stress value of MRF samples with EI powder is less as compared to CI powder.

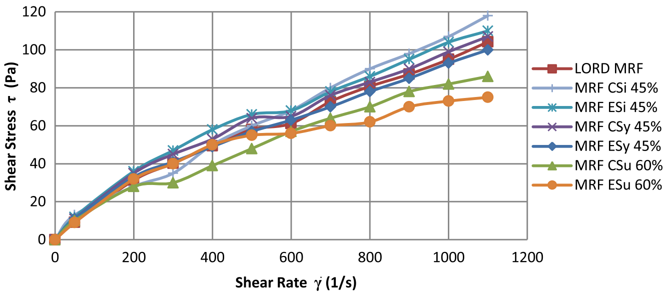


Fig. 10. OFF state shear stress change with varying shear rates.

4.3.2. ON state measurements

The rheological measurements were carried out after the application of magnetic field and that is termed as ON state.

As shown in the Fig. 11, the MRF samples with synthetic oil and silicone oil could develop the yield stresses above 70 kPa, while the samples which use sunflower oil as a carrier fluid couldn't exceed 45 kPa. As shown in the Fig. 11, MRF CSi 45% shows the maximum yield stress of about 92.34 kPa whereas MRF ESu 60% shows the lowest yield stress which is 40.64 kPa.

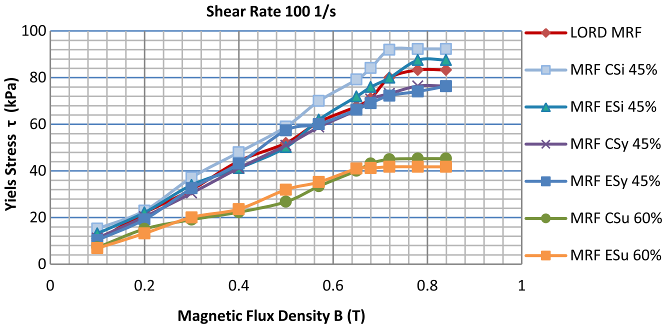


Fig. 11. Yield stress at constant shear rate.

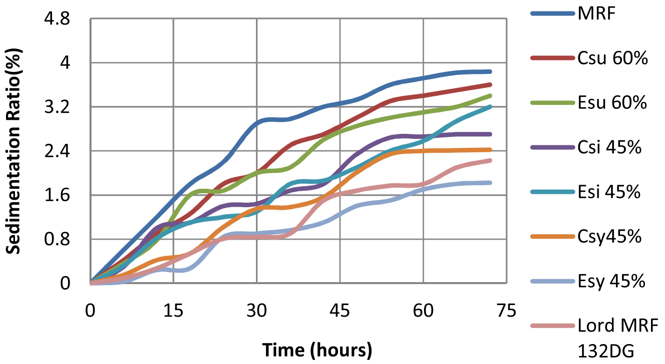


Fig. 12. Sedimentation ratios versus time.

Table 6
Concluding remarks.

MRF samples properties	Csi 45%	Esy 45%	Csu 60%	Esu 60%	MRF 132DG
Yield stress	Best	Better	Worse	Worse	Better
Magnetic properties	Best	Better	Better	Better	Better
Temperature sustainability	Best	Better	Worse	Worse	Better
Sedimentation	Better	Best	Worse	Worse	Best
OFF state viscosity	Best	Best	Better	Better	Best

The yield stress produced by the MRF CSu 60% is 41.7 kPa which is slightly more than the yield stress produced by MRF ESu 60%. The samples with EI powder have found to possess less yield stress as compared to CI powder. The MRF samples CSu 60% and ESu 60% have shown the peak on the graph at about 0.68 T. This peak indicates the magnetic saturation for that material. The magnetic saturation is also higher as in case of MRF CSi 60% which is at 0.72 T while LORD MRF 132 DG shows the point of magnetic saturation at 0.78 T. The magnetic saturation should be as more as possible since, it directly affects the yield stress produced by the fluid and thus, the braking torque.

4.4. *Stability of MR fluid*

Fig. 12 represents the sedimentation ratios of the particles for various samples for 3 days (72 h) with and without additives.

MRF without additive is named as 'MRF'. This MRF has the highest sedimentation ratio (3.84%), while the lowest sedimentation ratio (1.82%) is observed in case of ESy 45%. This means, the additive and coating of guar gum improved the sediment stability of the MRFs. Almost all the MR fluid samples saturate after 65–65 h.

Sedimentation of the MRF should be as low as possible. Increased sedimentation of MRF particles directly affects the yield strength produced by the MRF and hence, the torque produced. Thus, from the sedimentation point of view, Lord MRF and MRF ESy 45% seem suitable for the braking application.

5. Conclusions

MR fluid samples with different compositions preferably to suit braking application have been synthesized. Based on this synthesis and characterization results, qualitative analysis (worse, better & best) for various MR fluid samples from braking context have been presented in Table 6.

MRF samples Csu 60% and Esu 60% cannot meet the requirements of braking application; however, they may be used for low yield stress applications. The difference between the OFF state viscosities of examined samples is very small, however the ON state shear strengths produced by them are significant. MR fluid sample Esu 60% with OFF state viscosity 0.32 Pa s could produce shear strength of only 40.64 kPa whereas, with 0.7 Pa s OFF state viscosity MR fluid sample Csi 45% has developed highest shear strength of 92.34 kPa. CI powder is better for MR fluid samples used for braking application as compared to the EI powder as it produces considerably higher saturation magnetization.

Thus, based on the overall study CSi 45% is recommended for the brake application as the most suitable MR fluid.

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