



## Rheological Properties of the In-house Prepared Magneto-rheological Fluid in the Pre-yield Region

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### ABSTRACT

The essence of the present work is to study the rheological properties of the in-house prepared magneto-rheological (MR) fluids in the pre-yield region since the rheological properties play a vital role in better understanding of vibration damping capabilities of MR fluids. In the present work, two different compositions of MR fluid samples were prepared with 24 and 30 volume percentages of carbonyl iron (CI) particles. Prepared MR fluid samples contain CI particles as a dispersive medium, silicone oil as a carrier fluid and white lithium grease as an anti-settling agent. The oscillating driving frequency and amplitude strain sweep tests are performed to investigate the rheological properties within the pre-yield region. The influences of driving frequency, strain amplitude, magnetic field and CI particles volume percentage on the rheological properties of the prepared MR fluids were assessed. The linear viscoelastic region of the prepared MR fluid sample was identified and the yield strain obtained was around 0.371%. It is observed that the volume percentage of CI particles in the MR fluid strongly influenced the rheological properties.

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## 1. INTRODUCTION

Magneto-rheological materials change their properties in a significant way when they are exposed to externally applied magnetic fields. Rabinow [1] reported the rheological effect of MR fluids for the first time in 1948. MR fluid typically contains CI particles, carrier fluid and an anti-settling agent (additive). CI particle size used in MR fluids typically vary in the range of 1-10  $\mu\text{m}$  which are spherical for a better MR effect. Additives avoid settling high-density iron particles in the carrier fluid due to gravity in the MR fluid [2-4]. When an external magnetic field is applied, magnetic dipole makes the particles form strong interactions. This causes the iron particles to form a chain-like structure in the magnetic field direction. MR fluids response is in the range of a few milliseconds in the presence of external stimuli. MR fluids can produce higher yield stress values based on the iron particles volume percentage concentration and external field applied [5-10]. Response time of MR fluids

has great importance in most applications. Because of the quick, controllable rheological properties and reversible quality of the MR fluids, they found many potential applications in the field of vibration control devices, dampers, brakes and clutch applications [11-15]. Dynamic characterization studies are conducted on the MR/ER fluids to meet the appropriate application design specifications [16-22]. The rheological properties like storage modulus, loss modulus and loss factor value of MR/ER fluids are found using a sinusoidally oscillating shear strain of frequency. The storage and loss modulus increased with the externally applied magnetic field [23-25]. The extensive usage of MR fluid for the last two decades in medical applications is reviewed and discussed the future perspective [26]. The strain-controlled rheometer was used to analyze the viscoelastic parameters of the MR fluids. The influence of particle size distribution, volume fraction percentage and applied field on the rheological parameters are reported [27-36]. Though few studies exist on commercially available MR

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fluids, the rheological tests on the in-house prepared MR fluids are important because they are cost-effective. The present study aims to prepare the MR fluid samples with 24 and 30% volume fractions of iron particles to study the rheological properties in the pre-yield region. One of the main challenges of the present study is to identify the linear viscoelastic region of the prepared MR fluids and to study the rheological properties in the pre-yield region. The variation in rheological properties of MR fluids in the pre-yield region found potential structural-related damping applications. It is necessary to evaluate the influence of the different parameters on the properties of MR fluid in the pre-yield region. The parameters considered for the present study are driving frequency, shear strain, magnetic field and volume percentage of CI particles. Further, MR fluid samples are tested using an oscillatory strain-based rheometer with a flat plate type configuration. Oscillatory driving frequency and strain amplitude-based tests are performed to analyze the frequency and strain-dependent storage modulus and loss factor for the prepared samples. All the results reported in this work are within the region of yield and also reported the effect of iron particles volume percentage and an externally applied magnetic field on the rheological properties of the in-house prepared MR fluid samples.

## 2. MATERIALS AND METHODS

### 2. 1. Materials and Equipment Used for The Preparation of MR Fluid Samples

MR fluid contains iron powder particles suspended in a carrier fluid medium. In this work, Iron particles are carbonyl iron particles (CPIs) and silicone oil is the carrier fluid. The CI particles powder, low in magnesium and manganese compounds with a purity of 99.5% is purchased from Sigma Aldrich with a density of 7.86 g/cm<sup>3</sup>. Merck Life Science Private Limited supplied the silicone oil. The viscosity of the Silicone oil is 340 Cst (at 25<sup>o</sup>C) and the density is of 0.970 g/cm<sup>3</sup> (at 25<sup>o</sup>C). Scanning Electron Microscopy (SEM) analysis is conducted using the ZEISS FESEM instrument to study the surface morphology and size of the CI particles. Additionally, a small amount of white lithium grease is used as the surfactant (surface modifier or additive for the iron particles) to avoid the sedimentation of the iron particles [37, 38]. Permatex is supplied the white lithium

grease, it looks as a paste-like substance. This surfactant will accumulate around the iron particles to make them suspend in the carrier fluid. Two types of MR fluid samples are prepared with the different compositions (different volume percentage). Table 1 shows the details of the sample composition.

### 2. 2. MR Fluid Preparation Procedure

For the present study, MR fluid samples are prepared at a room temperature. Two types of fluids are prepared with different compositions to analyze the rheological properties in the region of pre-yield. Volume fraction calculations have been used to prepare MR fluid samples. The MRF-I sample is prepared with the 30% volume fraction of carbonyl group-based iron particles, 70% volume fraction of silicone oil and 2 grams of white lithium grease. The MRF-II sample is prepared with the 24% volume fraction of iron particles, 76% volume fraction of silicone oil and 2 grams of white lithium grease. The desired volume fraction calculations are converted into weight fractions using the density property of the iron particles and silicone oil. Weight fractions of the materials are easy to measure using the weighing machine. The required weight fraction proportions of the constituents for both the samples are measured using high precision weighing machine tool.

For MRF-I fluid sample, 44.8 grams of silicone oil is taken in the container. The container which contains the carrier fluid is kept under a Mechanical stirrer for the stirring. REMI RQ-5 Plus mechanical type stirrer is used to stir MR fluid. The stirrer shaft or rotator of the mechanical stirrer is arranged in such a way that it should not touch the base of the container. The 2 grams of white lithium grease is initially added to the carrier fluid while stirring. The speed maintained for this stirring process is 600 rpm. This process is continued for 2 hours for the uniform mixing of surface modifier in the carrier fluid. Then, to the stirred fluid, 155.2 grams of CI particles are added in small amounts while the fluid is stirring at 800 rpm. The stirring process is continued for at least 12 hours without interruption for uniform mixing of CI particles in the carrier fluid. For the MRF-II sample, 56.24 grams of silicone oil, 2 grams of white lithium grease, and 143.76 grams of CI particles are taken. The same procedure is followed for the preparation of the MRF-II sample. The quantity of additive added in the two types of fluid compositions is not changed. The small

TABLE 1. MR fluid samples composition

Sl.No.	Volume percentage (Vol%)		Weight percentage (Wt%)		Additive
	CI Particles	Silicone Oil	CI Particles	Silicone Oil	
MRF-I	30	70	77.6	22.4	2 grams
MRF-II	24	76	71.88	28.12	2 grams

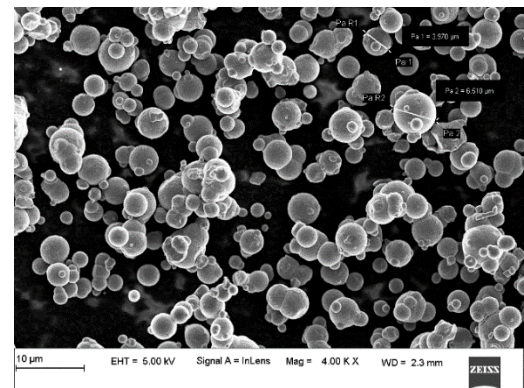
amounts of additive added will not have much influence on the rheological properties [38]. The additive will provide additional suspension action to MR particles. The mixing process of silicone oil and white lithium grease without adding CI particles is shown in Figure 1(a). The whitish color indicates the uniform mixing of white lithium grease in the carrier fluid. The mixing process of iron particles into the already prepared solution is shown in Figure 1(b). After this mixing process, the fluid is turned into black color because of the added CI particles.

### 2.3. The Microstructure of Carbonyl Iron Particles

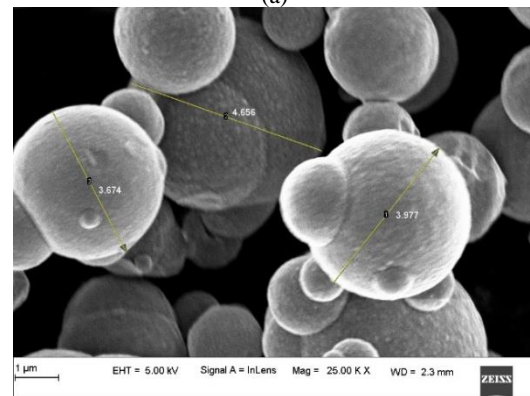
Scanning Electron Microscopy (SEM) test is performed on CI particles to confirm the surface morphology and particle size. It is observed that iron particles are spherical in shape. Figures 2(a) and 2(b) illustrate the shape of iron particles viewed at 4000x and at 25000x, respectively. The size of the few particles is marked in the SEM image to ensure the iron particles size. It is observed that the average iron particles size is around 4 to 9  $\mu\text{m}$ .

### 2.4. Experimental Setup

Rheological characterization of prepared MR fluid samples is done using Modular Compact Rheometer (MCR 702 Anton Paar make). Figure 3 shows the Rheometer setup with the magnetorheological device (MRD) cell used for the rheological study. The measuring system includes mainly MRD cell, a Chiller for the cooling, data processing sensors and a computer with the rheocompass software to take data from the sensors of the rheometer. The Rheometer is connected to RheoCompass software through which the user can specify the test conditions and store the acquired data. A MRD cell is a DC power supply device that provides the required magnetic field to the MRF sample poured on the stationary plate of the



(a)



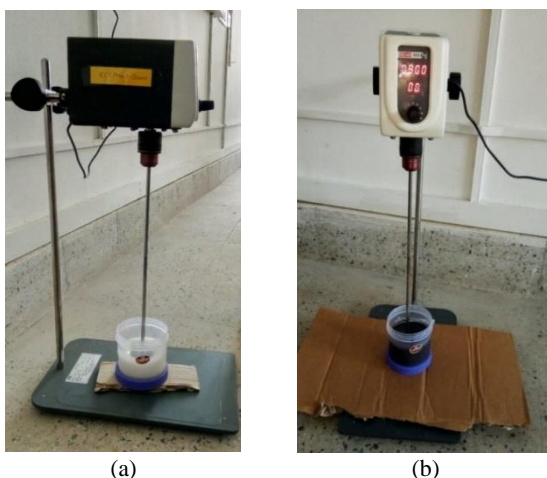
(b)

**Figure 2.** CI particles microstructure magnified view (a) at 4000x and (b) at 25000x

rheometer. During the tests, a small amount of MR fluid samples is poured in the bottom stationary parallel plate. A measuring gap of 0.5 mm is maintained for all the tests. For every test, approximately 0.4 ml of MR fluid sample is filled in the gap of two parallel plates configuration for rheological properties measurement.

### 3. RESULTS AND DISCUSSION

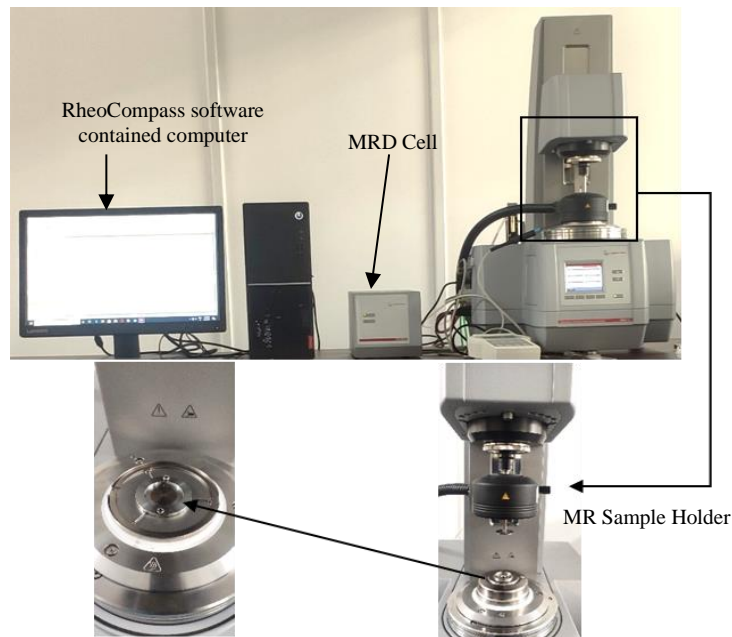
Initial tests are conducted at a constant shear rate (1/s) to check the relation between the applied current (A) and the magnetic field. For this, the current is increased from 0 to 5A at a constant shear rate of 1/s. There will be a particular relationship between the applied current and the magnetic field produced for every MR fluid. MRF-132LD has a linear relation between the coil current and magnetic field developed [27]. Based on the results obtained, Figure 4(a) shows the linear relationship between applied coil current and the magnetic flux (T) developed for the prepared MR fluid. All the experimental results presented in this article are against the current. The current dependency of viscosity in the measuring gap is shown in Figure 4(b). From 0 to 2A, there is a rapid increment in the viscosity of the prepared



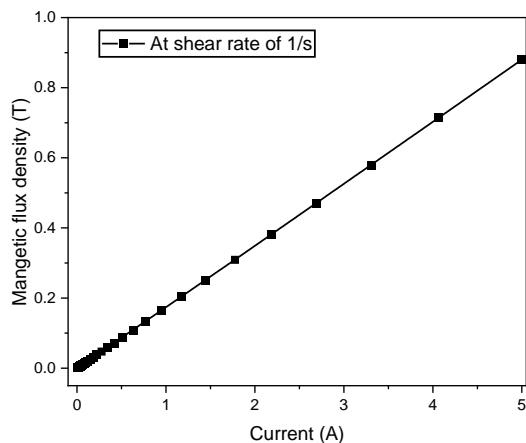
(a)

(b)

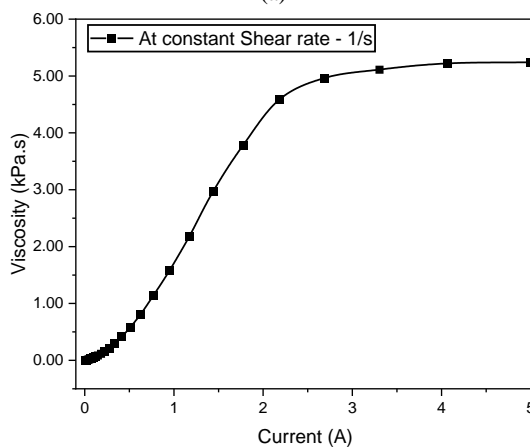
**Figure 1.** (a) Silicone oil and white lithium grease solution (b) Mixing of CI particles in the prepared solution



**Figure 3.** Rheometer experimental setup with MR cell



(a)



(b)

**Figure 4.** Variation in (a) Magnetic flux density and (b) Viscosity of the MRF-II sample with the applied current

sample. After 2A of current, change is not much in the viscosity curve. This means that the fluid's viscosity will not improve further even if the current value is increased. This implies that the change in the rheological properties beyond 2A current is not much appreciable for the prepared MR fluid sample.

Magnetorheological fluids exhibit linear rheological properties within the region of pre-yield [27]. MR fluid exhibits linear viscoelasticity when the developed shear strain is within the region of yield strain amplitude. So, MR fluid's yield strain amplitude is determined before conducting the other rheological measurement tests. The oscillatory shear strain amplitude method is used to determine yield strain [39-42]. The current of 2A is applied to determine the yield strain for the prepared MRF sample. Before applying the amplitude sweep, the magnetic field is set up by applying the suitable current for the tested fluid placed in the MR cell's parallel plate configuration. Amplitude sweep is taken logarithmically up to 1% shear strain amplitude. Strain amplitude is applied up to 1% because the experimentally obtained yield strain for most of the MR fluids is well below this range [8, 27]. From the stress-strain curve, the experimental yield strain of 0.371% is obtained as shown in Figure 5. The obtained yield strain for the prepared MR sample is in good agreement with the reported data in literature [27]. According to literature [27], the yield strain obtained is in the range of 0.2-0.6%. Once, the yield strain is determined from the strain amplitude test, remaining tests needed to be conducted with in the region of yield strain. For all the further experiments, maximum shear strain amplitude ( $\gamma_0 = 0.04\%$ ) is applied to ensure the obtained results are within the pre-yield region.

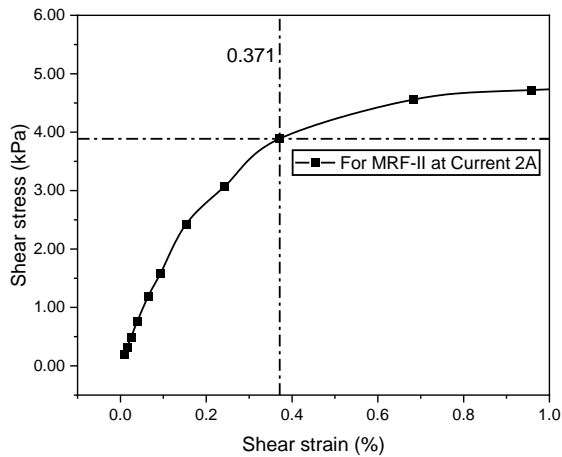


Figure 5. Yield strain from the shear stress-strain curve

**3. 1. Frequency-dependent Rheological Properties**

A frequency sweep test is conducted for the prepared MR fluid samples at a constant shear strain amplitude ( $\gamma_0 = 0.04\%$ ). Frequency sweep is applied logarithmically from 1 to 100 Hz to evaluate the influence of driving frequency on rheological properties. Figures 6(a) and 6(b) illustrate the storage modulus and loss factor value plots with respect to the driving frequency at four different applied magnetic field values. From the results obtained, the storage modulus increases with the increment in the externally applied field. This is due to the alignment of CI particles in the MR fluid as the magnetic field is applied. This alignment of CI particles transforms liquid state of MR fluid into a semi-solid. This chain-like formation increases the storage modulus of the MR fluid. At any particular applied current, the value of the storage modulus is increasing with the oscillatory driving frequency, as we can observe from the Figure 6(a). The loss factor value at any particular magnetic field initially decreases and then increases as the driving frequency increasing [37]. At certain frequencies, the fluid becomes more viscous due to internal structure of the iron particles in the MR fluid. This attributed to the higher loss factor values [27]. In this case, the increase in loss factor values is observed after the frequency of 60 Hz. A similar trend is followed for the storage modulus and loss factor for all the applied magnetic fields.

At the current of 2A, the storage modulus attained a maximum value of 2.01 MPa as the frequency reaches 100 Hz. Storage modulus at current 2A is considerably very high compared to the value of storage modulus at current 0.5A for an oscillating driving frequency of 10Hz. The loss factor at current 0.5A is two times more than the value of the loss factor at 2A for an oscillating driving frequency of 10Hz.

**3. 2. Amplitude Strain Dependent Rheological Properties**

An amplitude strain sweep is conducted for the MR fluid samples at a constant angular

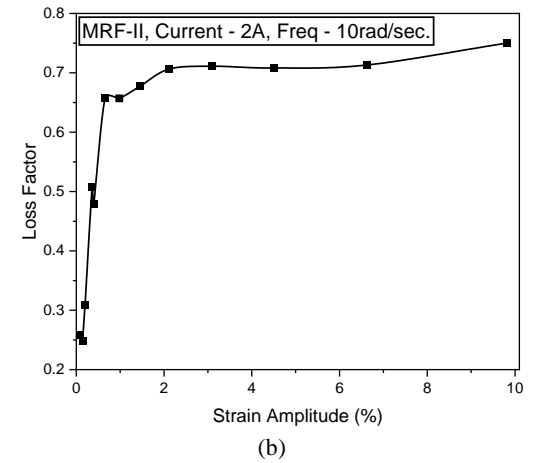
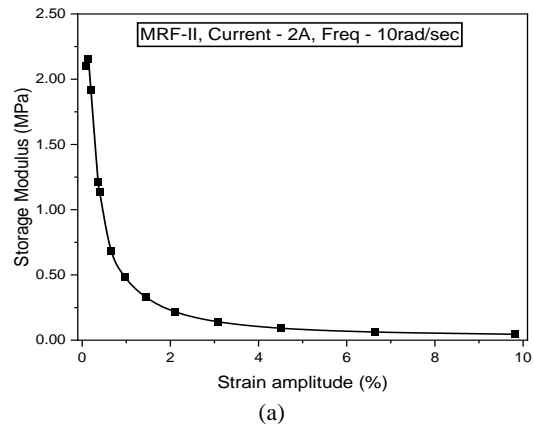


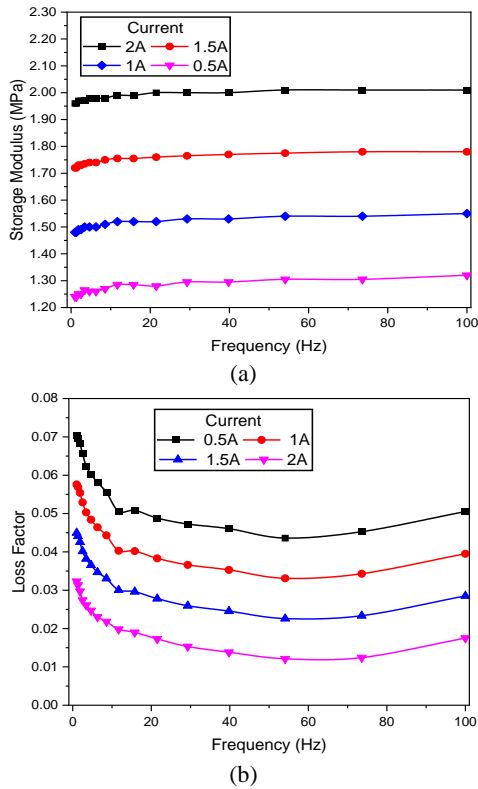
Figure 6. Frequency dependency of (a) Storage Modulus and (b) loss factor at different currents for MRF-II sample

frequency of 10 rad/s. Amplitude sweep is applied logarithmically from 1 to 10% of strain amplitude. Figures 7(a) and 7(b) illustrate the strain amplitude dependent storage modulus and loss factor values, respectively.

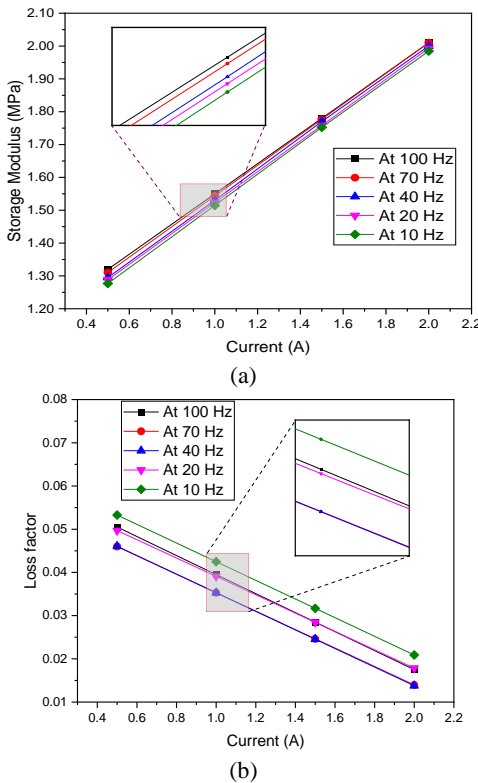
Experimental results concluded that the storage modulus decreases as the strain amplitude is increasing. For higher amplitude of shear strain, the chain formed between the iron particles will shear off due to higher displacements. This leads to the lower values of storage modulus at higher strain amplitudes. However, as the strain amplitude increases, there is an increment in the loss factor. Amplitude strain-dependent properties like storage modulus and loss factor values trend obtained in this research work are good agreement with the reported data in literature [27].

**3. 3. Magnetic Field Dependent Rheological Properties**

In this section, magnetic field dependent storage modulus and loss factor at different driving frequencies 10, 20, 40,70, and 100 Hz at a constant strain amplitude of 0.04% are determined. Figure 8(a) shows that the storage modulus at any driving frequency increases with the applied magnetic field



**Figure 7.** Strain amplitude dependency (a) storage modulus (b) loss factor at Current 2A for MRF-II sample



**Figure 8.** Magnetic field dependency of (a) Storage modulus (b) loss factor for the MRF-II sample at different driving frequencies

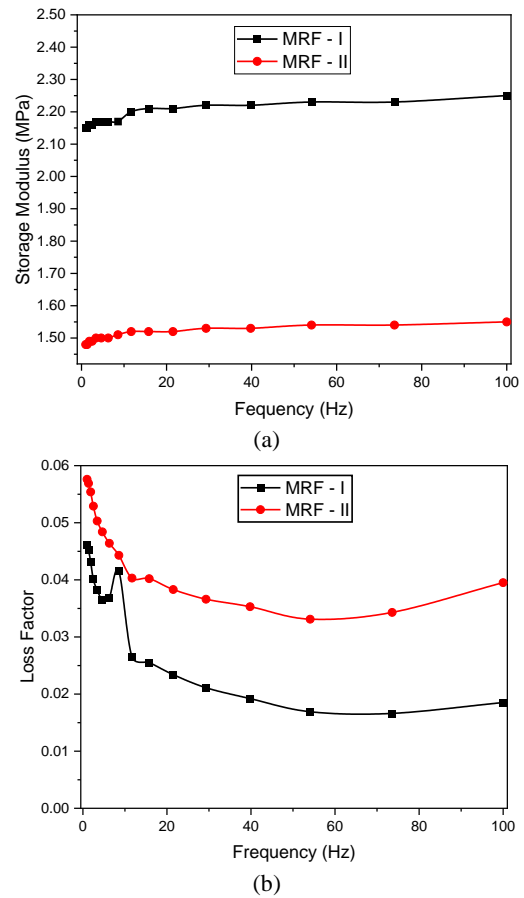
value. Iron particles in the MR fluid start to arrange in a chain-like structure with the applied magnetic field value. Because of this, fluid started to behave semi-solid. The semi-solid nature of the MR fluid increases the value of the storage modulus.

Storage modulus value increases with the driving frequency at any particular magnetic field value. However, the Loss factor value decreases with the applied field value at any driving frequency [27, 37].

### 3. 4. Volume Fraction Dependent Rheological Properties

The rheological properties depend on the percentage of CI particles in the MR fluid [27, 32, 37]. The storage modulus and loss factor for two MR fluid samples with respect to frequency are shown in Figures 9(a) and 9(b).

The results show that the storage modulus value is higher for the MRF-II sample compared to the MRF-I sample. The magnetic field effect will be greater for the higher vol% of the CI particles MR fluid sample for any applied magnetic field value. However, the loss factor value for higher vol% of CI particles is less compared to the low vol% of CI particles MR fluid sample. This may



**Figure 9.** Volume fraction dependency of (a) Storage modulus (b) loss factor at Current 1A for MRF-I and MRF-II with respect to frequency

be due to more carrier fluid in the low vol% of CI particles sample. The results analyzed in this work confirmed that iron particle concentration in the MR fluid strongly influences the rheological properties.

The present study identified the linear viscoelastic shear strain region and the obtained yield strain is around 0.371% for the MRF samples under the amplitude strain sweep test. The driving frequency and strain amplitude have a considerable influence on the rheological properties. This study suggests that the volume percentage of CI particles in the MR fluid strongly influences the rheological properties. Though the present study explored the influence of different parameters on the rheological properties, the current study is limited to only two different compositions of MR fluid samples. Availability of the rheometer with MRD cell and testing cost of the prepared samples are one of the main limitations when it comes to MR fluids study.

#### 4. CONCLUSIONS

The current research work presents rheological properties within pre-yield for the in-house prepared MRF samples. The variation in rheological properties like storage modulus and loss factor are determined for the applied driving frequency, amplitude strain, current, and volume percentage of CI particles to check the dependency of these factors on the prepared MR fluid samples. The results obtained in this research work can be summarised as follows:

- The linear viscoelastic shear strain region (pre-yield) is identified and the obtained yield strain is around 0.371% for the in-house prepared MRF samples under the amplitude strain sweep test.
- For all the magnetic fields applied, there is an increment in the storage modulus value with the driving frequency. This confirms that the driving frequency has a considerable influence on the rheological properties.
- For both the samples, the storage modulus value is increasing and the loss factor is decreasing with the strain amplitude.
- The storage modulus is increasing and the loss factor value is decreasing with the magnetic field and for any applied magnetic field, the loss factor is greater at low frequencies than at higher frequencies.
- The Storage modulus obtained for the MRF-II sample is relatively high compared to the MRF-I sample. This concludes that the volume percentage of CI particles in the MR fluid strongly influences the rheological properties.

The present study helps in identifying the linear viscoelastic region for the in-house prepared MR fluid samples and understanding the influence of different parameters on the rheological properties in the pre-yield region. The results obtained further can be utilized for the numerical/experimental studies of the sandwich beam or

plate problems, where there is necessity of isolate the natural frequency from the resonance or reducing the amplitude of vibration.

#### 5. ACKNOWLEDGMENTS

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### Persian Abstract

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#### چکیده

ماهیت کار حاضر بررسی خواص رئولوژیکی سیالات مغناطیسی رئولوژیکی (MR) آماده شده داخلی در منطقه پیش تسلیم است زیرا خواص رئولوژیکی نقش حیاتی در درک بهتر قابلیت‌های میرایی ارتعاش سیالات MR ایفا می‌کند. در کار حاضر، دو ترکیب مختلف از نمونه‌های سیال MR با ۲۴ و ۳۰ درصد حجمی ذرات کربونیل آهن (CI) تهیه شد. نمونه های مایع MR آماده شده حاوی ذرات CI به عنوان یک محیط پخش کننده، روغن سیلیکون به عنوان مایع حامل و گریس لیتوم سفید به عنوان یک عامل ضد ته نشینی هستند. فرکانس حرکت نوسانی و آزمون‌های جایجایی کرنش دامنه برای بررسی ویژگی‌های رئولوژیکی در ناحیه پیش تسلیم انجام می‌شوند. تأثیر فرکانس مورد ارزیابی قرار گرفت، دامنه کرنش، میدان مغناطیسی و درصد حجم ذرات CI بر روی خواص رئولوژیکی سیالات MR تهیه شده مورد ارزیابی قرار گرفت. ناحیه ویسکوالاستیک خطی نمونه سیال MR تهیه شده شناسایی شد و کرنش بازده بدست آمده حدود ۰٫۳۷۱ درصد بود. مشاهده شده است که درصد حجمی ذرات CI در سیال MR به شدت بر خواص رئولوژیکی تأثیر می‌گذارد.

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