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Temperature-Dependent Viscoelastic Response of Magnetorheological Grease with Varying Particle Shapes

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Abstract

This study examines the effect of temperatures on the magnetic and rheological properties of magnetorheological greases (MRGs) comprised of different particle shapes, under oscillatory shear mode test. Two samples were prepared with 70 wt.% flake-shaped electrolytic iron particles (EIP) and spherical carbonyl iron particles (CIP) which dispersed in 30 wt.% lithium-based grease, separately. Respective to magnetic properties test, the vibrating sample magnetometry (VSM) revealed that MRG-EIP exhibited 11.4% higher magnetic saturation (M_s), 94.4% higher remanent magnetization (M_r), and 89.1% greater coercivity (H_c) compared to MRG-CIP. These enhancements are attributed to the larger surface area and anisotropic shape of the EIP particles. Meanwhile, under strain sweep tests at 25°C, 45°C, and 65°C, at without (0 T) and with magnetic field of 0.59 T, the linear viscoelastic (LVE) region for both samples display different behaviour. At 25°C, the MRG-CIP possess a wider LVE region, with a limit at 0.0482% strain compared to the MRG-EIP which is limited to 0.0261% strain. Nevertheless, under 0.59 T, both samples exhibited the similar LVE limit, around 0.068% strain due to the magnetization effect experienced by both particles. Albeit being similar in LVE limit, the capability of both MRGs to store modulus (G') at given strains are different, respective to the role of different particle shapes in the grease medium. Despite this, MRG-EIP exhibited a consistent increased in storage modulus at all temperatures, in contrast to the decrement in storage modulus observed in MRG-CIP sample. These results demonstrate that the flake-shaped of EIP improve the thermal and mechanical stability of MRG, offering the enhanced performance for magnetorheological devices that would be operating under variable temperature conditions.

Keywords: Carbonyl iron particles, electrolytic iron particles, linear viscoelastic, magnetic properties, magnetorheological grease, storage modulus, temperatures

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INTRODUCTION

Magneto-sensitive smart materials like magnetorheological (MR) material and ferrofluid are created by dispersing ferromagnetic fillers of nanoscale or micrometre size into various carrier matrices. Their rheological properties can be rapidly and reversibly altered by an external magnetic field [1]. This distinctive characteristic make them valuable in applications that require regulated mechanical behaviour, generating considerable research attention and practical utilisation [2]–[4]. MR materials are typically categorised based on the type of matrix, including MR fluids (MRF), MR elastomers (MRE), and MR greases (MRG), each designed to suit specific operational needs. MRG is a semi-solid magneto-responsive composite, comprising a grease matrix embedded with iron particles [5], [6]. The matrix is formed by a lubricating oil entrapped within a three-dimensional network of a gelling agent, functioning analogously to a polymeric thickener [7] and comparable to fiber- or cellulose-reinforced composited reported in recent studies [8]–[12]. This network imparts thixotropic behavior which can be manifested as time-dependent structural breakdown and recovery resulting in pronounced non-Newtonian rheology in both the absence (off-state) and presence (on-state) of a magnetic field. The dispersion of magnetic fillers within this semi-solid polymer network enables tunable viscosity, yield stress, and modulus in response to an applied magnetic field [13], positioning MRG within the broader category of functional grease-based composites and semi-solid magneto-responsive materials. Previous studies had proven that there is no sedimentation problem occurred in MRG due to the high-off state viscosity attributed to the grease medium which resulted in an excellent dispersion stability between the matrix phase and the magnetic particles by holding the particles from depositing to the bottom part of the material [3], [14], [15].

Beyond yield stress and viscosity, another critical parameter for evaluating the mechanical performance of MRG is its viscoelastic behaviour [16], which reflects the ability of the material to store and dissipate energy under deformation. MRG can be classified as a viscoelastic material, as it exhibits both elastic and viscous characteristics depending on the applied strain [3], [17]. It gradually relaxed when a force is applied, and the stress within the material decrease over time until the force is removed. This time-dependent behaviour between stress and strain, when measured in the linear region, is known as the linear viscoelastic (LVE) region [18]. The LVE region is crucial for rheological characterization as it ensures that subsequent measurements are conducted within a strain limit, preserves the microstructure of the materials without causing irreversible damage. Previous studies have explored the use of additives to enhance LVE region, as wider LVE region indicates stronger internal structure and better energy absorption capability [19], which is especially important for MR applications such as MR dampers and isolators. Tarmizi et al [20] modified the composition of MRG by incorporating cobalt ferrite ($CoFe_2O_4$) resulting in a 29% improvement in the LVE region under the off-state condition and a 43% improvement under the on-state conditions. In addition, Nasir et al. [17] introduced graphite particles as additives into MRG forming a composite referred as GMRG. Under a magnetic field, the LVE range of GMRG expanded from <0.03% strain in MRG to ~0.1% strain in GMRG, which is more than a threefold improvement. This enhancement was attributed to the irregular shape and high surface area of graphite which improved the dispersion within the grease medium and strengthen the fibrous structure network.

Other than the utilization of additives, researches have also focused on particles shape to enhance the performance of MR materials. For example, flake-shaped particles consistently demonstrate improved storage modulus and sedimentation stability in MRG when compared to spherical particles, due to their higher surface area per volume and friction-enhanced chain networks. For example, a study by Upadhyay et al. [21] observed that there is an increment of storage modulus in MRF, when they use flake-shaped as magnetic particles, indicating strong solid chain structures due to the friction between flake-shaped particles. In addition, Lee et al [22] reported that the flake-shaped carbonyl iron (CI) microparticles exhibit a higher storage modulus compared to spherical-shaped CI particles attributed to the construction of sturdier clusters and increased solid friction due to the larger surface area per volume of flake-shaped particles. These findings suggest that the shape of magnetic particles directly contributes to the extension of LVE regions, as more energy is required to disrupt the magnetic network under small deformations. However, most of these studies were conducted at ambient temperature, leaving a knowledge gap regarding how temperature variation may affects the viscoelastic properties of MRG with non-spherical shape of magnetic particles. Hence, the present study utilized flake-shaped electrolytic iron particles (EIP) and spherical-shaped CIP as the magnetic particles in MRG to compare their performance in terms of dynamic viscoelastic at different working temperature conditions which at 25°C, 45°C, and 65°C. This work aims to evaluate how particle shape and thermal conditions jointly influenced the linear viscoelastic range and dynamic modulus response of MRG. The insights obtained from this study intended to guide the development of MRG formulations with enhanced thermal stability and mechanical performance for advanced engineering applications.

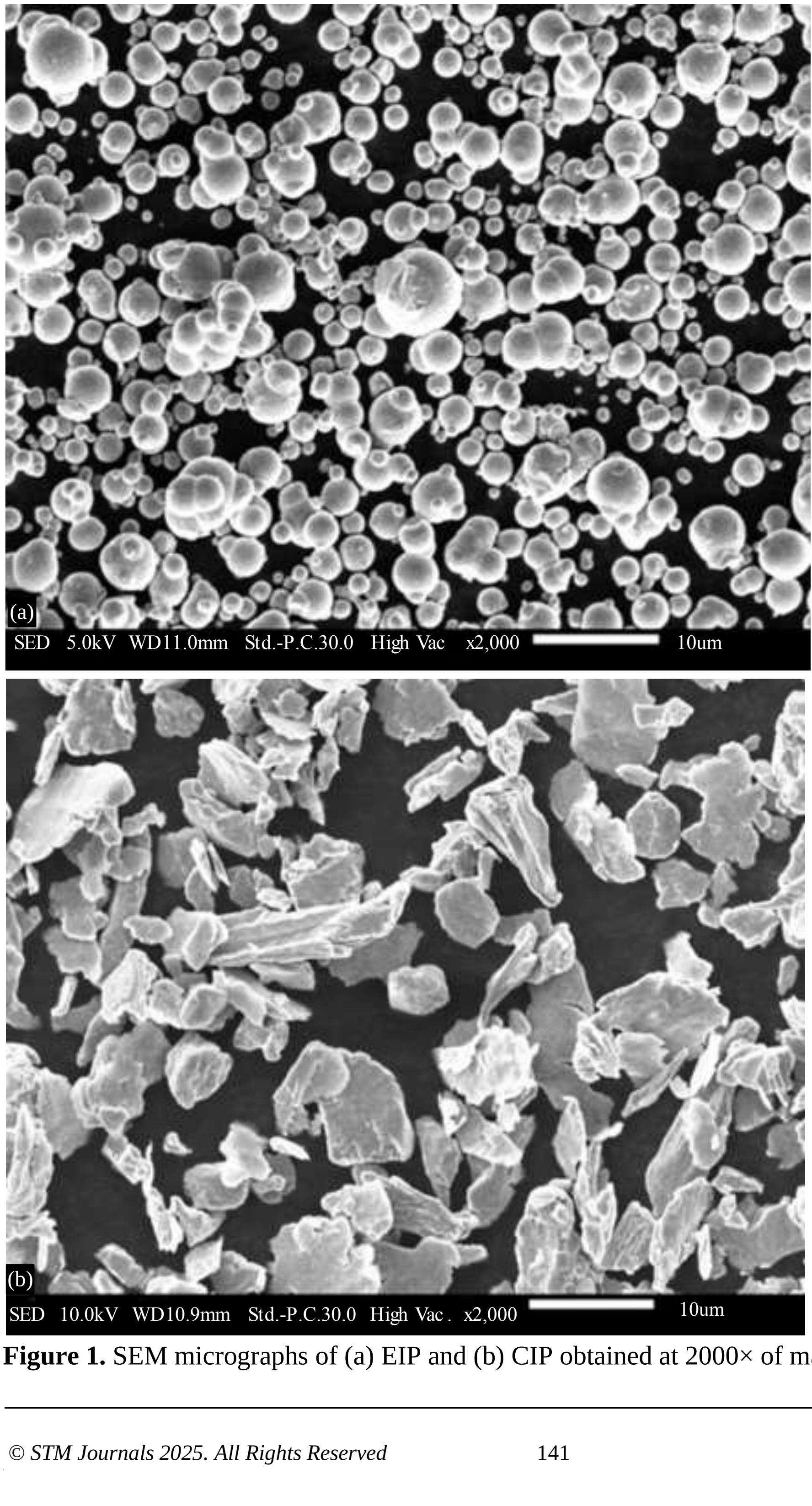

Figure 1. SEM micrographs of (a) EIP and (b) CIP obtained at 2000× of magnification.

MATERIALS AND METHODOLOGY

Preparation of MRG Samples

Irregular shape of EIP were purchased from Industrial Metal Powder (India), while spherical-shaped CIP (OM type) were purchased from BASF Germany as magnetic particles. The morphology, in terms of particle shape and size, is depicted in Figure 1(a) and 1(b). The EIP exhibit an irregular flake-like shape with faceted edges and an average size of 3–12 µm, whereas the CIP display a smooth spherical shape with the range of size from 2–7 µm. NPC Highrex HD-3 grease (Nippon Koyu Ltd., Japan) was utilized as the main base medium for MRG fabrication by using a high-speed mechanical stirrer (HS-30 D, DAIHAN Scientific Co., Ltd.). In order to fabricate the MRG-EIP, 30 wt.% of grease was stirred for 5 minutes to loosen the internal structures of the base grease. Then, 70 wt.% of EIP was added into the stirred grease and continuously stirred with mechanical mixing stirrer at 300 rpm for 2 hours to ensure homogenous distribution of MRG-EIP. The similar procedure was repeated for MRG-CIP. It should be remarked that all sample preparations conducted in this study were carried out at room temperature.

Magnetic Testing

The magnetic properties of MRG-EIP, MRG-CIP, and base grease were examined using a Vibrating Sample Magnetometer (VSM, LAKESHORE model 7404). Approximately 4.8 mg of each MRG samples and grease were carefully loaded into a non-magnetic sample holder. This sample mass was chosen in accordance with standard VSM operating guidelines, which recommend 2–10 mg to ensure a strong signal-to-noise ratio while avoiding overfilling or compromising the magnetic uniformity of the sample. The samples were subsequently vibrated continuously at room temperature under applied magnetic field ranging from -14 to 14 kOe. The details of the magnetic properties including coercivity, retentivity, and saturation magnetization were obtained and recorded in section 3.1.

Rheological Testing

Rheological test of MRG samples was performed by using a Physica MCR 302 rheometer (Anton Paar, Graz, Austria) that was equipped with a magneto-controllable accessory (MRD 70/1T) to generate magnetic fields to the sample, and parallel plate measuring system (PP20/MRD/T1/P2) for the sample place during the testing. A Viscotherm VT2, a temperature control unit would enable precise temperature adjustment during the setting of temperatures at 25°C, 45°C, and 65°C. Samples were loaded between parallel plates of 1 mm gap, where the top plate was movable or rotatable, while the bottom plate remained stationary. The magnetic field strength was generated by applying controllable currents to the electromagnetic coil and the generated magnetic field was measured using a Teslameter. The rheological tests were performed in oscillatory mode with shear rates ranging from 0.01 to 100 s^{-1} at a fixed frequency of 1 Hz and frequency ranging from 1 Hz to 100 Hz under a constant strain of 0.01%. The magnetic field strength applied for each test was varied from 0 to 0.59 T by adjusting the coil applied current from 0 to 3 A. The applied currents that corresponded to magnetic field strengths were as follows: 0 A = 0 T, 1 A = 0.22 T, 2 A = 0.41 T, and 3 A = 0.59 T.

RESULTS AND DISCUSSION

Magnetic Properties Analysis

The magnetic properties of MRG-EIP and MRG-CIP was investigated by using VSM analysis. The corresponding magnetization curves are shown in Figure 2, and their magnetic parameters are provided in Table 1. It is evident that MRG-CIP exhibits a lower saturation magnetization (M_s) as compared to MRG-EIP. This is because the irregular flake-shaped EIP and higher surface area tend to form denser grease matrix and interlock between particles which facilitates a better magnetic particle chaining under the influence of external magnetic field [2]. In addition, MRG-EIP also shows higher remnant magnetization (M_r) and coercivity (H_c) measured at 7.7987 emu/g for and 62.499 Oe, respectively. The enhanced M_r and H_c can be attributed to the increased magnetic anisotropy and domain wall pinning effects which is related to the irregular flake-shaped EIP [23]. Unlike spherical-shaped CIP that magnetize uniformly in all directions, the flake-shaped EIP preferred magnetization directions along their major axes known as shape anisotropy. This anisotropy contributes to the greater resistance against demagnetization which resulting in higher M_r and H_c [24].

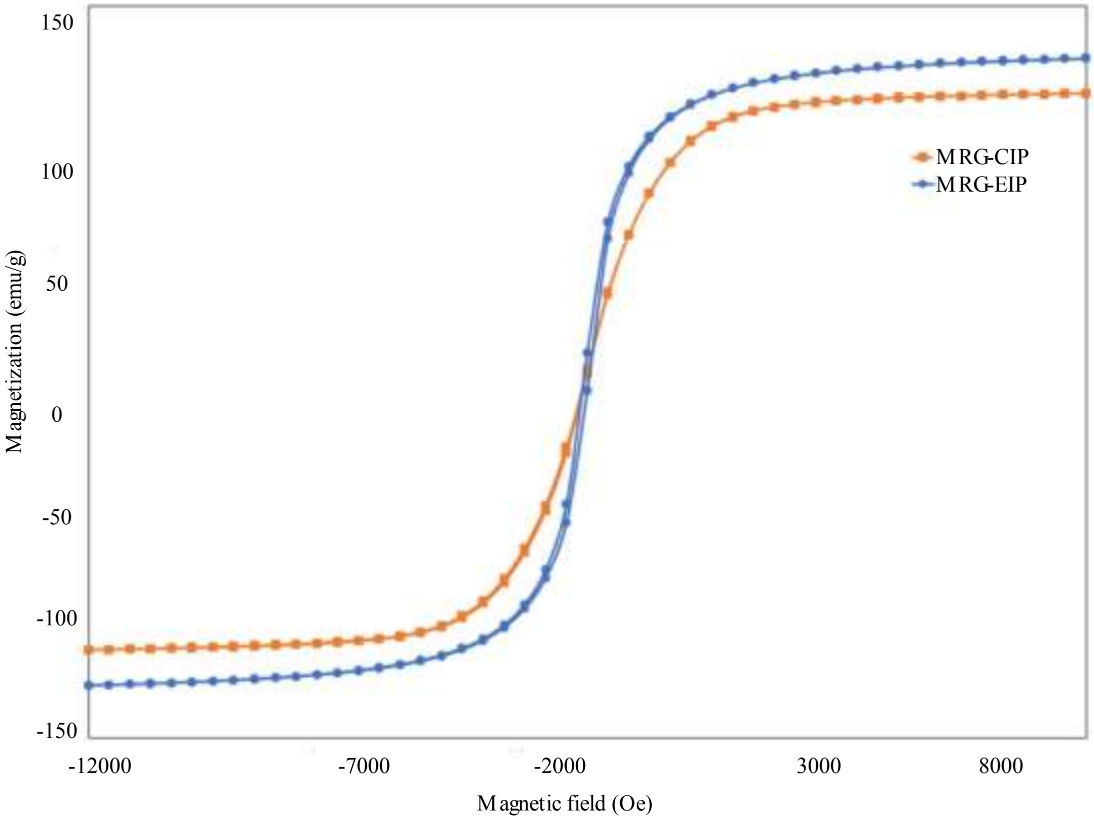

Figure 2. Hysteresis loop for MRG-EIP and MRG-CIP samples.

Table 1. Magnetic properties values of MRG-EIP and MRG-CIP obtained from VSM analysis.

Samples/parameter	MRG-CIP	MRG-EIP
M_s (emu/g)	114.33	129.04
M_r (emu/g)	0.43528	7.7987
H_c (Oe)	6.8032	62.499

Strain Sweep Analysis

The variation of storage modulus (G′) with increasing shear strain was analysed to investigate the influence of particle shape on the linear viscoelastic (LVE) region of MRG at 25 °C, 45°C and 65 °C. Figure 3(a) illustrates the storage modulus for MRG samples incorporating EIP and CIP, under both 0A and 3A current conditions at 25°C. It is observed that there is increment from 0.0261% to 0.0675% strain for LVE region from 0A to 3A which is equivalent to 61.3%% increment. As for MRG-CIP, the increment is from 0.0482% to 0.0675% which is equivalent to 28.5%. During the off-state condition at ambient temperature, MRG-CIP has a wider LVE region compared to MRG-EIP. This is attributed to the uniform spherical shape of CIP, which tend to have an even dispersion and lower internal friction within the grease medium leading to more uniform particle distribution [2]. However, during on-state condition, both MRG-EIP and MRG-CIP have same LVE region due to the magnetic dipole-dipole interactions that dominate the rheological response. This indicates that once an external magnetic field is applied, particle chaining and alignment become the primary governing factors of the viscoelastic response, minimizing the differences between spherical and flake-shaped geometries in terms of LVE limit. In terms of storage modulus, MRG-EIP exhibits higher values of storage modulus as compared to MRG-CIP in both off-state and on-state conditions, due to the larger surface area of flake-shaped EIP which promotes stronger particle-particle interactions and stronger chain alignment under the influence of magnetic field [25]. The rough facets and anisotropic geometry of EIP also allow stronger interlocking and frictional contacts, enhancing chain stability under magnetic fields, whereas the smoother CIP particles form weaker chains that are more vulnerable to disruption.

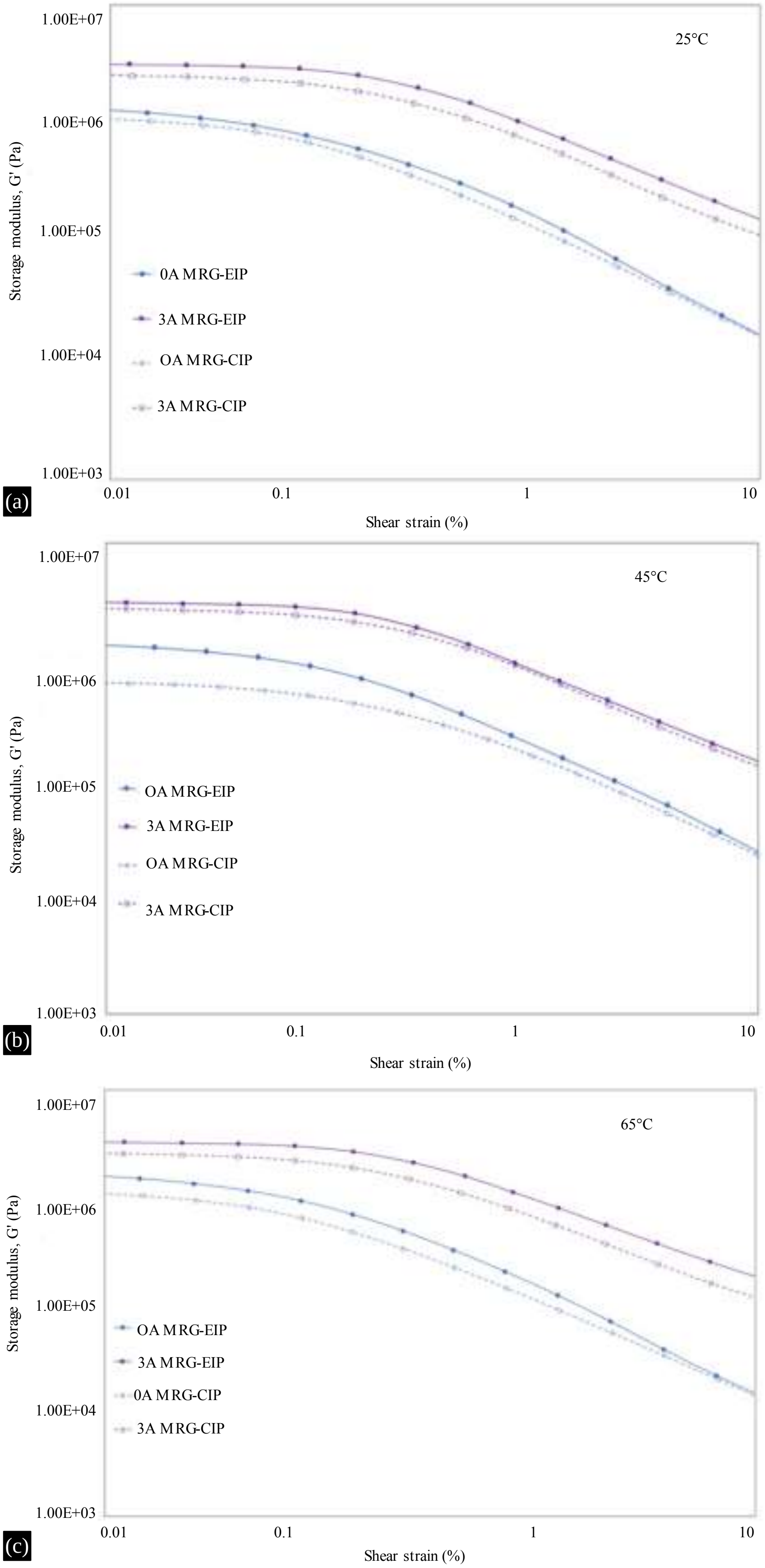

Figure 3. Storage Modulus of MRG-EIP and MRG-CIP at (a) 25 °C, (b) 45 °C and (c) 65 °C.

At 45°C, the LVE region for MRG-EIP and MRG-CIP becomes shorter compared to at 25°C which is around 0.0133% and 0.0240%, respectively in Figure 3 b. This reduction occurs because heating increase particle mobility within the grease medium and weakening the van der Waals interaction leading to disruption of the original microstructure [26].The thermo-softening experienced by the grease matrix further reduce its elastic resistance to deformation and causing the material to yield at smaller strains thereby shortening the LVE region. The proposed mechanism is that the influence of particle shape on viscoelastic behavior is governed by the balance between shape of magnetic particle and interparticle interactions with thermal softening of the grease matrix. Flake-shaped particles, with their larger surface area and anisotropic geometry, promote stronger interlocking and frictional resistance, which stabilize particle chains against thermal agitation. In contrast, spherical particles provide smaller contact areas and smoother surfaces, forming weaker chains that deteriorate more rapidly as the matrix softens with increasing temperature. This difference explains why MRG-EIP retains higher viscoelastic strength than MRG-CIP at elevated temperatures. In contrast, under the on-state condition, for both MRG-EIP and MRG-CIP, the LVE region maintains the same as at 25°C. When a magnetic field is applied, the magnetic effect dominates the elastic response of MRG and override the thermal softening effect [18]. The magnetic dipole-dipole interactions are much stronger than thermal energy resulting in stable and strong chain of alignment that resist open disruption, thus maintaining the LVE region even when temperature increases. Consistent with the trend observed at 25 °C, MRG-EIP retains a higher storage modulus than MRG-CIP, even at elevated temperature, with values of approximately 3.15×10^6 Pa and 2.54×10^6 Pa, respectively. This suggests that the anisotropic shape of EIP provides greater thermal resilience to particle chaining and energy storage capacity, even as the grease matrix softens.

At 65°C, the LVE region for MRG-EIP and MRG-CIP decrease slightly as compared to 45°C, with values of 0.0131% and 0.0238% in figure 3c indicating that once thermal softening and increased particle mobility occurs at 45°C, further temperature increase has minimal effect on LVE region. During the on-state condition, for both MRG-EIP and MRG-CIP, the LVE region remain unchanged as at 25°C and 45°C. As for storage modulus, a similar trend is observed at 25°C and 45°C where higher storage modulus value for MRG-EIP with value of 3.21×10^6 Pa while MRG-CIP is reduced to 2.52×10^6 Pa. Meanwhile, during the off-state condition, particle shape was observed to influence the LVE region, whereas temperature has minimal effect [27]. However, during the on-state condition, neither particle shape nor temperature significantly affected the LVE region. As for storage modulus, MRG-CIP shows a reduction with increasing temperature, while MRG-EIP displays a slight increase from 25 °C to 65 °C. In MRG-CIP, the spherical particle shape provides a smaller contact area and smooth surface, making the magnetic chains more vulnerable to thermal agitation, hence the reduction in modulus with increasing temperature [2]. Conversely, the flaky shape of MRG-EIP which has larger surface area and irregular facet edges promote stronger particle-particle contacts and stronger chain stability [21], allowing the storage modulus to increase slightly from 25 °C to 65 °C despite the thermal softening of the matrix.

CONCLUSION

The rheological performance of MRG is strongly influenced by particle shape and temperature in the absence of a magnetic field. However, at on-state condition, the role of particle shape and temperature does not significantly affect the LVE region but affects storage modulus. Vibrating sample magnetometry (VSM) revealed that MRG-EIP exhibited a higher magnetic saturation (Ms) of 129.04 emu/g compared to 114.33 emu/g for MRG-CIP, a significantly higher remanent magnetization (Mr) of 7.7989 emu/g compared to 0.43528 emu/g for MRG-CIP, and a greater coercivity (Hc) of 62.499 Oe compared to 6.8032 Oe for MRG-CIP owing to the irregular flaky shape morphology that promotes stronger particle–particle interactions within the grease medium and chain stability under magnetic fields. Although MRG-CIP exhibited a wider off-state LVE region at 25 °C, this advantage diminished in the on-state where magnetic interactions dominated. Increasing temperature reduced the LVE range for both formulations in the off-state, with MRG-CIP decreasing from 0.04815% to 0.02376% and MRG-EIP from 0.0261% to 0.01305%. However, the storage modulus followed contrasting trends, with a decrease observed for MRG-CIP and a slight increase for MRG-EIP. During the on-state condition, MRG-CIP exhibited a ~9.57% decrease in storage modulus from 25°C to 65°C, whereas MRG-EIP showed a ~2.92% increase. Overall, the findings confirm that flake-shaped particles enhance the mechanical strength and thermal resilience of MRG, making them preferable for advanced MR applications requiring reliable performance over a broad temperature range.

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