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RESEARCH ARTICLE

Rheological Analysis on Hardening of Magnetorheological Grease with Kerosene

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ABSTRACT - Magnetorheological grease (MRG) with dilution oils may suffer from reduced storage stability caused by oil separation. This phenomenon potentially causes a performance reduction of the material due to possible accelerated degradation of the grease medium. The long-term rheological behavior of MRG with kerosene (MRGK) was investigated in this study. MRGK was prepared with 10 wt% kerosene as the dilution oil, together with a sample of MRG without any kerosene as the control. A modular compact rheometer (MCR) was used to obtain rheological data from the MRG samples in an oscillatory strain sweep mode under the influence of magnetic fields, which ranged from 0A to 3A. After one year, the measurement was repeated to observe any changes to the rheology of MRG samples. Results showed a significant hardening of the sample diluted with kerosene, which mainly showed a drastic increase in off-state storage modulus at low strain. This was shown by the off-state storage modulus of MRGK, which after one year showed an increase of 15% in the initial storage modulus, and an increase of 2438% in the storage modulus at 10% strain. The MRG sample showed an increase of 50% and 47%, respectively. The on-state storage modulus did not appear to experience such a drastic change after one year. The study concluded that while dilution oil may be a promising candidate to reduce the initial viscosity of MRG, the resulting performance difference may compromise the long-term performance, and may even cause accelerated degradation when in use.

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1.0 INTRODUCTION

Magnetorheological grease (MRG) is a novel material used as an alternative to magnetorheological fluid (MRF) in various applications. These materials consist of microparticles of a soft magnetic material, commonly carbonyl iron particles (CIP), which are suspended in a fluid or semi-solid matrix and are able to organize into column-like structures under the influence of a magnetic field. These columnar structures provide added resistance to flow or shear within the carrier fluid, which greatly increases the apparent viscosity of the material. The ability of the material to change its viscosity under a magnetic field allows for its applications in torque transfer [1]–[3] or variable dampening [4]–[7] purposes. As such, the optimal material for such purposes requires a low initial viscosity and a high apparent viscosity after the introduction of a magnetic field. Since the CIP chains that align to the magnetic field provide most of the structural integrity, a low initial viscosity ensures as little resistance as possible when the device is not activated, which in turn not only provides a higher change in apparent viscosity and improves the MR effect, but also a faster response time of the material when the magnetic field is switched on. The response time also depends on the strength of the magnetic field applied. Meanwhile, by optimizing the MR effect of MRG, studies mainly focused on additives [2], [8] or parameters [9]–[11] which either lowered the initial viscosity or transient response time of the material.

As a measure to lower the initial viscosity of commercial grease, dilution oils are proposed as a solution [12], [13]. The dilution oils can effectively lower the concentration of grease thickener in the MRG mixture, which in turn lowers the overall apparent viscosity. It was reported that low viscosity oils such as kerosene are initially very effective at lowering the apparent viscosity of MRG [12]. As such, an increase in performance was also noted in terms of the magnetorheological (MR) effect. However, it was observed that excess dilution oils caused oil separation as the grease thickener could not structurally hold on to the amount of liquid in the MRG. Different dilution oils seemed to have different effects on the initial viscosity and oil separation as studies reported that the use of hydraulic fluid, which has a higher viscosity than kerosene, would hold a higher weight percentage of dilution oil without separation [13], [14]. The sample with kerosene started to separate at around 15 wt% kerosene in the MRG, while the hydraulic fluid sample stayed intact throughout the study. The effect of this behavior was only discussed in passing as the studies had only focused on initial rheological performance.

The breaking down of MRG, as stated in the previous studies [12]–[14], was a strong indication of grease degradation. Several studies have established the degradation of grease in various conditions, including high temperature [9], repeated shear [15], and even decomposition due to electrical discharge [16]. While temperature [9], [17] and electrical discharge [16] altered the grease chemically, the effect of repeated shear would only result in physical degradation because of the

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grease thickener structure destruction due to excessive shear [17], [18]. However, in the case of MRG, studies on the shear degradation of grease medium were limited. Direct studies on prolonged use or storage of MRG were also lacking. It is well documented that grease with high static oil separation is undesirable for lubrication purposes, as the grease fails to structurally hold the oil in the area designated for lubrication. Similarly, for MRG, it is not desirable for the grease to flow to places that are not intended, or even leak out of the device altogether. Current research on MRG focuses more on improving the grease by using additives [8], [19]–[21], or immediate effects of external factors such as temperature [9], [17]. As such, it is necessary to further investigate the effects of additives on the shear degradation of MRG, and subsequently towards the long-term stability and performance of MRG.

In this study, the effect of dilution oils on the long-term rheological performance of MRG and MRGK was investigated. It was anticipated that the study results would enlighten the long-term behavior of diluted MRG under storage and establish the knowledge further for future studies on the effects of additives.

2.0 LITERATURE REVIEW AND THEORETICAL FRAMEWORK

2.1 Overview of Magnetorheology

Magnetorheology is the property of a material that allows it to change its viscosity under the influence of a magnetic field. Over the years since its advent, it has received attention as a controllable, self-contained mechanism to provide resistance without the use of mechanical moving parts. This mechanism exploits the tendency of magnetizable particles to align themselves along the direction of a magnetic field. Conveniently, the aligned particles form columns that increase in structural strength under the presence of a stronger magnetic field [22]. As a result, the material exhibits a higher apparent viscosity or tensile strength when is magnetized. The difference in rheology in materials is called the MR effect. It is useful to quantify the performance of the MR material. To further expand the novelty of the resulting material, different matrices are used to house the magnetic particles.

Currently, materials with drastic changes in viscoelasticity are MR fluids [23], greases [2], [10], [24] and plastomers [25], [26]. The fluid nature of the materials allows CIP to move around freely enough within the matrix, allowing more defined formations of CIP chains. Low viscosity materials, such as MR fluids and greases are used for torque transfer purposes. In recent studies, most devices that utilized MR materials were called for the use of MR fluids and had the highest performance due to their low initial viscosity. However, the use of semi-solids, such as MRG and MR plastomers were being investigated to reduce or even eliminate the sedimentation of magnetic particles within the MR fluids.

2.2 Rheology of MR Materials

Rheology is defined as the study of flow behavior. Typically, a fluid is described as a substance that can easily yield to external forces and does not have a fixed shape. For example, liquids, such as water, oil and similar materials are usually called fluids. However, there are fluids that flow a lot slower than those liquids, such as honey, tar, or even pitch. Ideally, fluids flow in a Newtonian behavior such that their flow velocity increases proportionally to the shear force. This proportionality constant is called viscosity. However, most fluids in the real world are non-Newtonian and do not have constant viscosities due to small irregularities, such as intermolecular forces between big molecules and sometimes even physical interactions within the microstructural level [27]. Consequently, those fluids also exhibit properties of elastic solids. To further consolidate the study on rheology, the term viscoelasticity is coined.

The viscoelasticity of a material can be measured in terms of a complex modulus (G*). This modulus, similar to the viscosity measurements, is a direct measurement of the change in stresses (τ) within the material over a change in strain (γ) (Equation 1).

$$|G^*(\omega)| = \frac{\tau_\alpha}{\gamma_\alpha} \tag{1}$$

The complex modulus is a vector quantity, which can be decomposed into several other parameters that are characteristic of vectors. These parameters are further interpreted as various moduli that can represent how close a material is to a solid or liquid. The instantaneous slope of the measured complex modulus, commonly noted as tan δ, is the loss tangent of the material and is a direct indication of how much energy is lost due to dampening. Storage modulus (G') represents the tendency of a material to store energy and is analogous to the elasticity of a material. On the other hand, loss modulus (G") reflects the tendency of a material to dissipate energy, which in turn is similar to the viscosity of the material. The derivation of these parameters is shown in Equation 2 – Equation 4.

$$G' = G^* \cos\delta \tag{2}$$

$$G" = G^* \sin\delta \tag{3}$$

$$\tan\delta = \frac{G"}{G'} \tag{4}$$

2.3 Oscillatory Strain Sweep Experiment

In an oscillatory strain sweep, the independent variable is strain amplitude. By varying the amplitude while under a constant oscillating frequency, the experiment can determine the amount of strain it takes before the material starts to behave differently. From this experiment, the phase angle of the material could be measured directly by measuring the time displacement of material response with regard to the oscillatory input given through the rheometer. A schematic of the results is shown in Figure 1. From the data, the different moduli mentioned earlier could be derived in a straightforward manner. In the storage modulus on the strain graph, the region in which the material behaves elastically is called the linear viscoelastic region (LVE), which comes from the graph of the storage modulus being linear. For future testing purposes, this region represents the safe region, whereby the strain can be applied without damaging the material significantly. This metric is used to determine the strain values for future rheological tests. Right after the LVE, the point at which the material starts to behave non-linearly is called the yield point. This is the point where the material starts to break down. This point is the upper limit of strain, which is applicable to a material before it undergoes plastic deformation. In a semi-solid, it signifies the point, whereby the material starts to flow and behave more like a liquid. Furthermore, if there exists a point at which the magnitude of storage modulus meets the magnitude of loss modulus, the intersection is called the flow point, whereby the material exhibits more fluid-like properties and less solid properties.

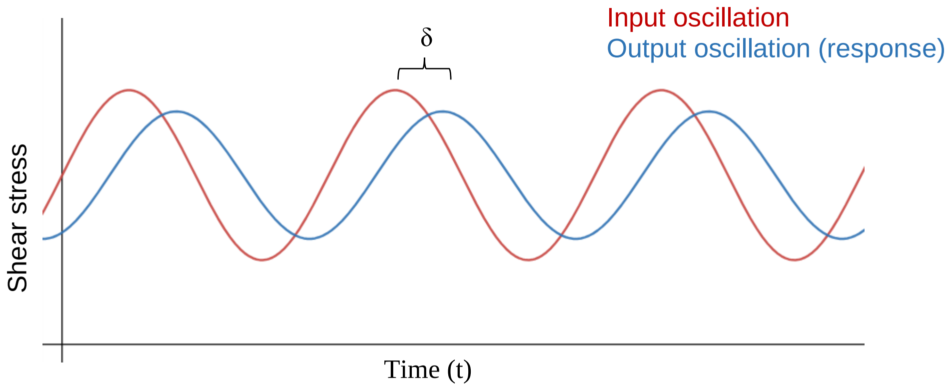


Figure 1. Schematic of phase angle measurement in oscillatory rheological experiments

3.0 MATERIALS AND METHODS

3.1 MRG Preparation

The MRG samples in this study were prepared by using OM-type CIP obtained from BASF (Germany) and suspended in NPC Highrex HD-3 lithium grease (Nippon Koyu Ltd, Japan). The grease has a lithium soap thickener with refined mineral oil as the base oil. Off-the-shelf kerosene was used as the dilution oil. The compositions of the samples are shown in Table 1. The components were combined and mixed with a mechanical impeller for 1 hour at 200 rpm to obtain a homogeneous mixture. A sample of lithium grease and MRG is shown in Figure 2. The prepared MRG and MRGK samples were divided into two portions, one portion for immediate analysis, and another portion was stored in an inert container for 1 year in ambient conditions. A schematic of the material preparation is shown as a flow chart in Figure 3.

Table 1. Compositions of test samples

Sample	*Grease (wt%)*	*CIP (wt%)*	*Kerosene (wt%)*
MRG	30	70	-
MRGK	20	70	10

Figure 2. Comparison between lithium grease (left) and MRG (right)

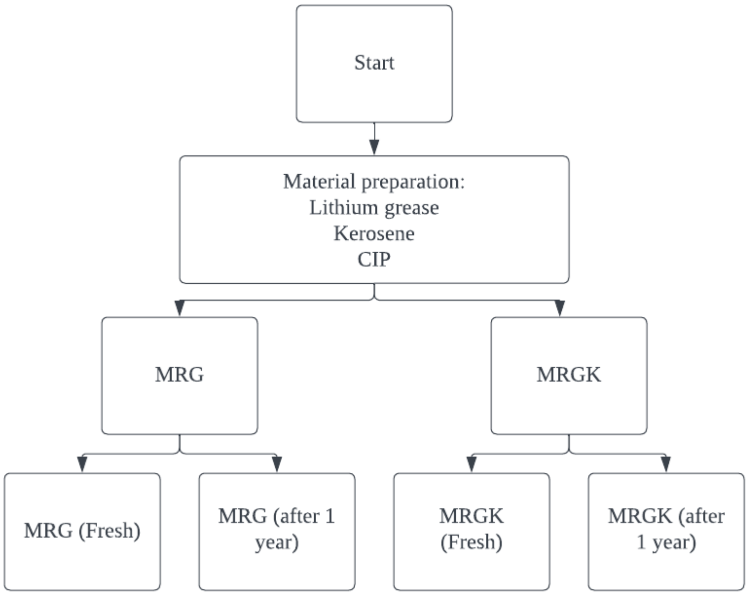


Figure 3. Flow chart of sample preparation

3.2 Rheological Analysis

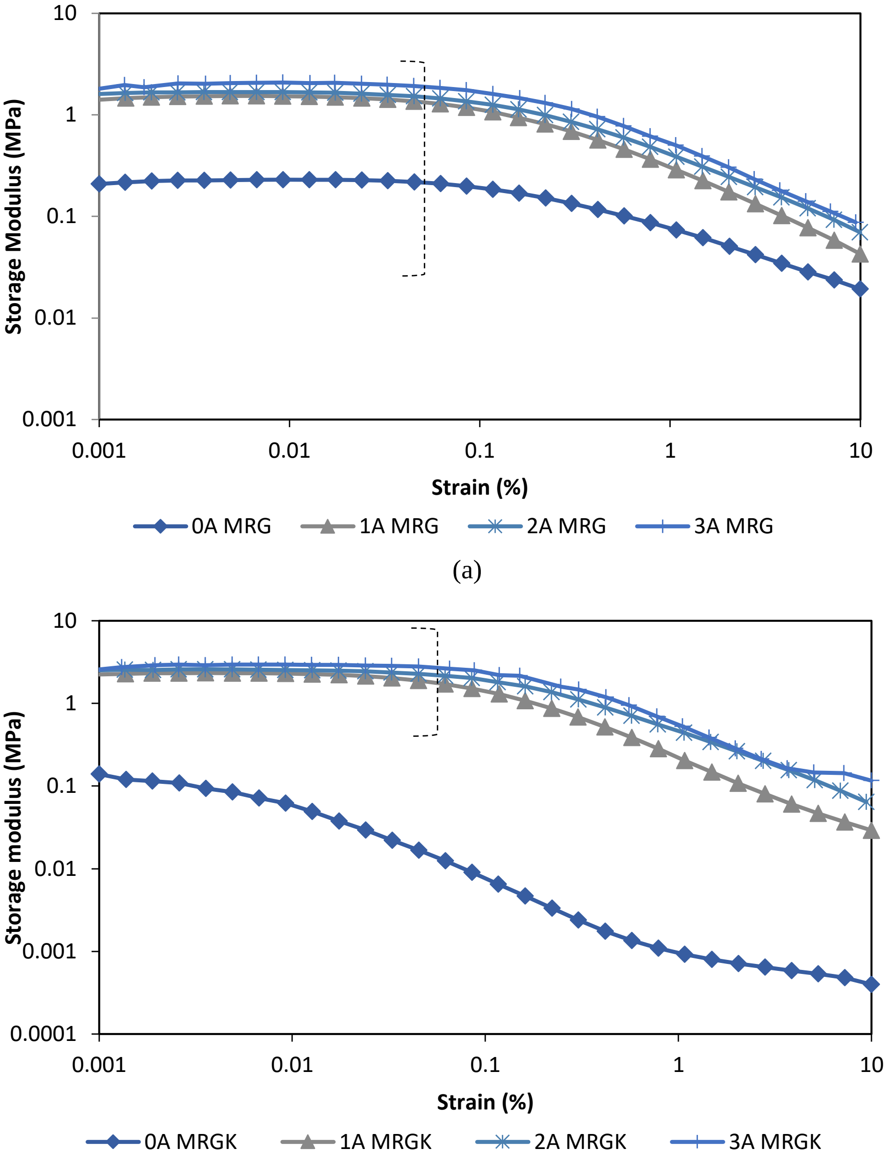
Once the samples were prepared, rheological experiments were carried out in a modular compact rheometer (MCR) (Physica MCR 302, Anton Paar, Graz, Austria) with a parallel plate attachment (PP20). The temperature of the samples was maintained at a constant temperature of 25°C by using a water-cooling system (Viscotherm VT2, Anton Paar). Finally, the magnetic field strength was controlled by using a magnetorheological device MRD 70/1T by varying the current supplied to the device. A photograph of the rheometer set-up is shown in Figure 4. An oscillatory strain sweep was carried out on the samples with a constant frequency of 1Hz and a strain that ranged from 0.001% to 10%. The magnitude of currents used in this study ranged from 0A to 3A, which corresponded to a magnetic flux density from roughly 0T to 0.6T, respectively. A graph of storage modulus overstrains was plotted and roughly analyzed to visualize the LVE region of the samples as well as a rough tabulation of the storage modulus at 0.001% and 10%. This was because the instrument was prone to some variation during the data collection at low strain values. The loss moduli were also collected, graphed, and analyzed with the same parameters. The same rheological analysis was repeated after 1 year.

Figure 4. Experimental setup for MCR

4.0 RESULTS AND DISCUSSION

The results of the freshly prepared MRG storage modulus are shown in Figure 5. The samples showed a typical Bingham plastic fluid behavior, whereby the samples showed the presence of an LVE. This region ended at a yield point, with the storage modulus decreasing rapidly after that point. This shows that MRG on its own is a shear-thinning fluid.

The MRG samples alone experienced a sudden increase in storage modulus once a magnetic field was introduced. As the current increased, the storage modulus increased correspondingly, although the change was less drastic than the increase from 0A to 1A. This could be credited to the formation of structural chains of CIP during the on-state that resisted the shear forces supplied to the MRG samples. The MRGK sample showed a very low initial storage modulus, nearly up to an order of magnitude. The structural integrity of the material was also weak, as shown by the near absence of an LVE. However, once under the influence of a magnetic field, the increase in storage modulus was larger, reaching a higher on-state storage modulus as compared to MRG alone. The increase could be attributed to the lower structural integrity of MRGK, which allowed the CIP particles to move more easily in the grease medium. The facilitated movement was theorized to result in stronger CIP chains, which increased resistance. The results obtained in this part of the study were validated by comparing it with previous studies [13], [14].

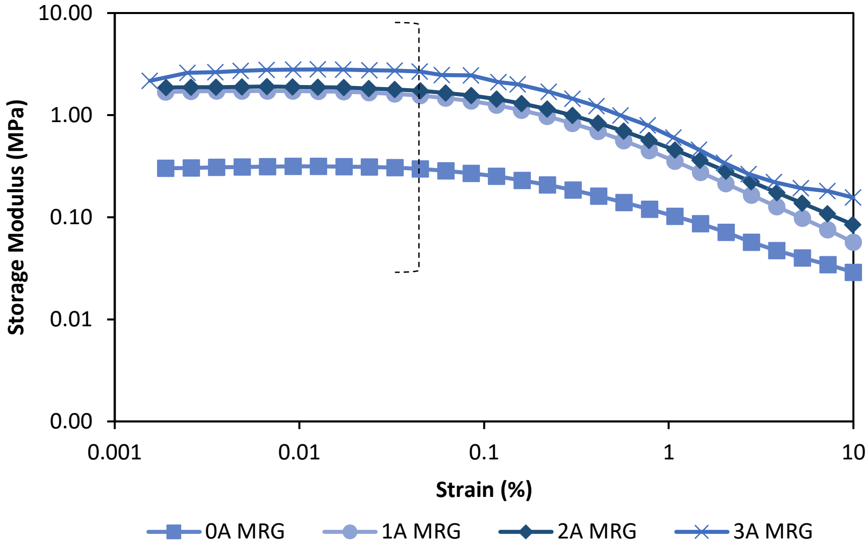


(a)

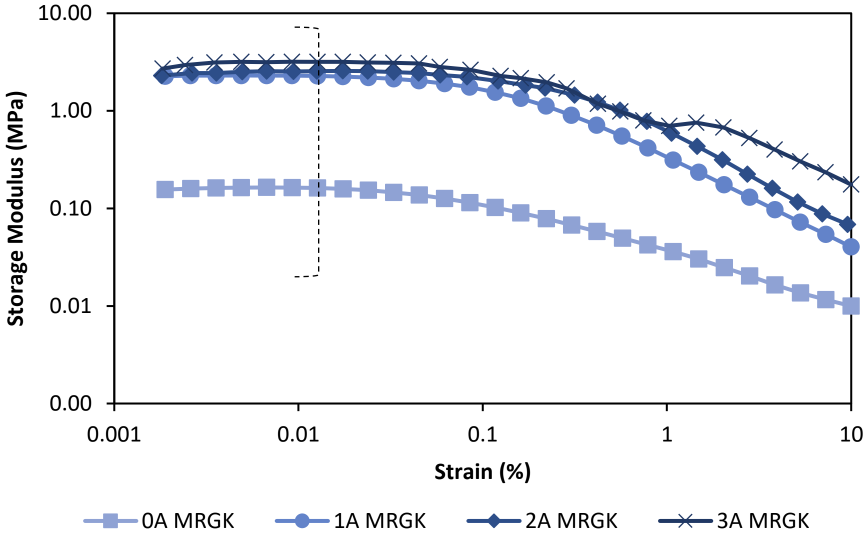
(b)

Figure 5. Storage modulus results for freshly synthesized (a) MRG (b) MRGK. Line marker shows the extent of LVE region

After 1 year, the storage modulus obtained is shown in Figure 6. The MRG behavior was similar to when it was freshly prepared. A rough calculation of changes in the storage modulus at 0.001% and 10% strain was made and the data was tabulated in Table 2. There was a 50% increase in the storage modulus at 0.001%, which showed that the material had slightly hardened and became more resilient to shear. At around 10% strain, there was a 47% increase in storage modulus, which similarly showed a slightly increased resistance to shear. However, the behavior of the MRGK sample showed a significant difference, while the storage modulus near 0.001% strain experienced an increase of up to 15% in the off-state, the storage modulus around 10% strain experienced up to 2438% as compared to the storage modulus upon preparation. The appearance of the LVE signified that the grease had shown significant hardening as the material had returned to a semi-elastic state under low strains. Similarly, the on-state storage modulus for MRGK also experienced a slight decrease. This followed the previous justification of stronger CIP chains in lower viscosity media.



(a)



(b)

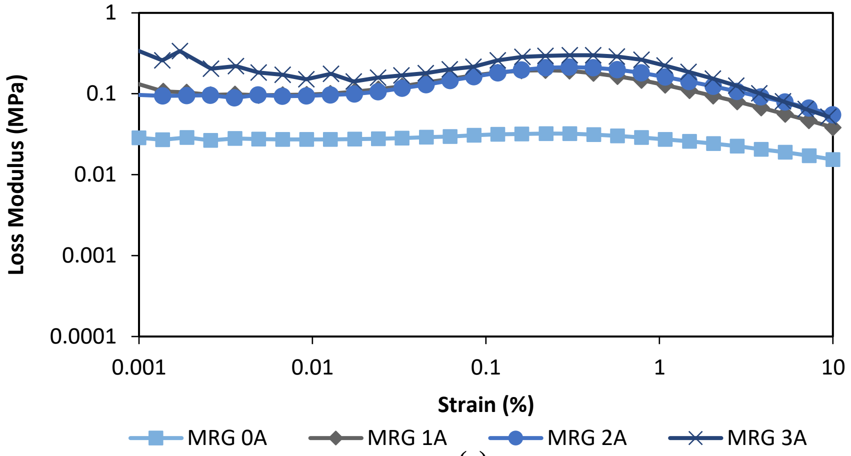
Figure 6. Storage modulus results for (a) MRG (b) MRGK after one year. Line marker shows the extent of LVE region

Table 2. Comparison of off-state storage modulus between MRG and MRGK

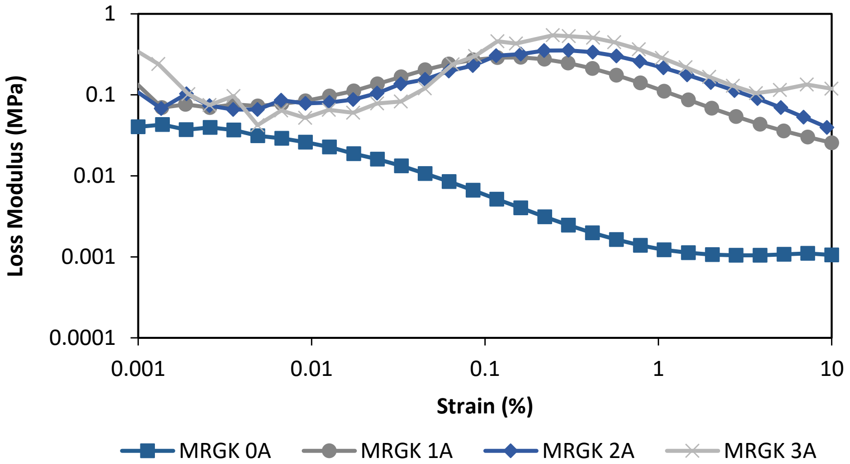
Condition \ Sample	MRG		MRGK	
	Storage modulus at 0.003% strain (MPa)	Storage modulus at 10% strain (MPa)	Storage modulus at 0.003% strain (MPa)	Storage modulus at 10% strain (MPa)
Fresh	0.20	0.019	0.13	0.00039
After 1 year	0.30	0.028	0.15	0.0099

From the graphs of the MRGK sample, it was noted that at higher magnetic fields, there was a sudden increase in storage modulus of around 1% strain and onwards. This behavior could be attributed to slipping between the sample and rheometer stage. This slippage appeared to be erratic, as the MRGK sample exhibited different points of inflection before and after 1 year. In addition, this behavior was indicated in the MRG sample after 1 year. Since the structural integrity of MRG under the influence of magnetic fields depended highly on the formation of CIP chains within the grease matrix, it was possible that the magnetic flux density was dense enough to cause the erratic breaking and forming of existing chains in the matrix once introduced to an oscillating shear of significant magnitude.

The loss modulus graphs for freshly synthesized samples are shown in Figure 7. The graphs indicated that kerosene had a significant effect on the integrity of the grease medium. This could be seen through the on-state behavior of the MRGK sample, in which it showed a lower initial energy loss at lower strain values. This could be contributed to the fact that under a strong magnetic field, CIP had already formed the structural chains needed to support the material. This behavior was not observed in the MRG sample as the erratic initial loss modulus only showed up at 3A. This showed that the grease matrix was structurally strong enough to resist the influence of magnetic fields. However, at higher strains, the energy loss was proportional to the magnetic field strength, which might accounted for the increase in heat loss through friction at higher magnetic fields. The off-state loss modulus for MRGK was also significantly different from that of MRG, which showed a lower loss of energy once the grease medium was completely broken down by the higher oscillatory shear of the experiment. In contrast, the MRG sample showed the minimum change in loss modulus within the test parameters, showing that the grease medium was still functioning as a thickener even at higher shear strains.



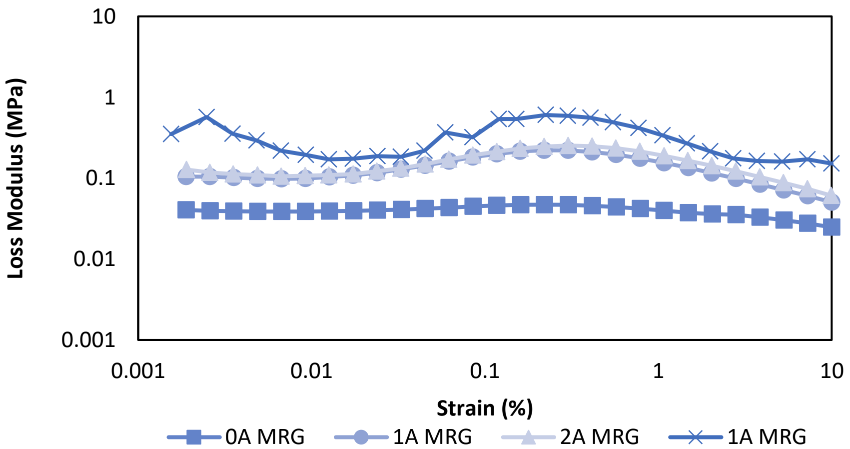
(a)



(b)

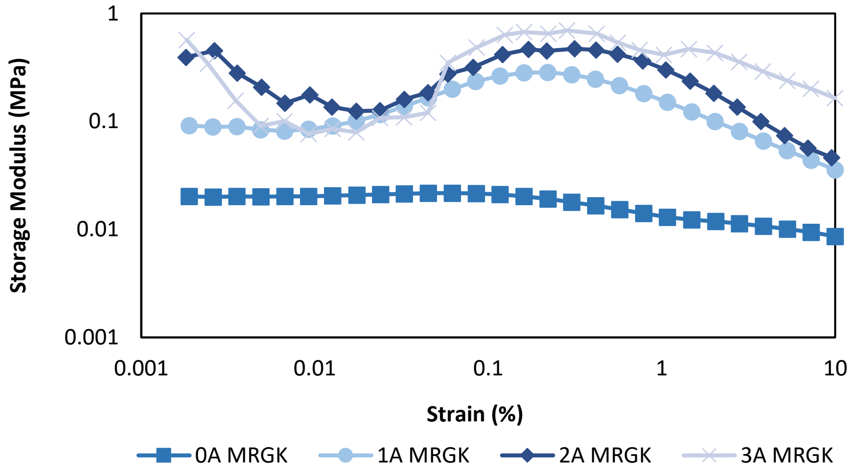
Figure 7. Loss modulus results for freshly synthesized (a) MRG (b) MRGK

The loss modulus of samples after 1 year is shown in Figure 8. The MRG sample showed a similar behavior as compared to when it was freshly prepared. Meanwhile, the overall behavior of the MRGK sample remained similar, the off-state loss modulus showed a huge difference. The MRGK loss modulus returned to a similar behavior to MRG, albeit with a slight decline after 0.1% strain, which was most likely due to some remaining kerosene still present in the mixture.



(a)

Figure 8. Loss modulus results for (a) MRG



(b)

Figure 8. (cont.) (b) MRGK after one year

Previous studies regarding MRG with added kerosene showed a general positive, since the additive could help to achieve a lower viscosity, which increased the overall performance [12]–[14]. While the same conclusion regarding the performance of MRGK could be made in this study, it was noted that the MRGK sample exhibited a significant hardening over time while under storage. The significant hardening of MRGK under storage showed that the use of kerosene as the dilution oil was unsuitable for long-term applications. While the MRGK sample still showed a slightly lower storage modulus as compared to MRG after 1 year, the difference in behavior between freshly prepared and year-old MRGK was very drastic. This might interfere with any modeling or calculations to implement a controllable device by using the material. Besides, the hardening might suggest the loss of oil. This further suggested that evaporation of the base oil might occur in MRGK. As a result, instability of MRGK with high amounts of dilution oils not only will show a decrease in performance after a period in storage, but also will probably degrade while operating in a device.

Furthermore, the relatively low storage modulus of fresh MRGK sample at higher strain suggested a high degree of grease thickener degradation, as the structure of the grease greatly contributed to the rheological properties of the material in the off-state. While the initial storage modulus did not experience a drastic increase as compared to MRG, the storage modulus at 10% reflected a poor resistance to shear. In lithium grease, the intertwined fibrous structuration of lithium hydroxystearate thickener provided a matrix to hold onto the base oil when not in use. Without the sponge-like structure of the lithium grease thickener, the ability of the grease to hold in the liquid oil components would decrease. It was theorized that such behavior would further reduce the stability of grease while in operation.

5.0 CONCLUSIONS

MRG samples were prepared with the addition of kerosene as the dilution oil. From the study, it was evident that the hardening of MRG was an issue after dilution with kerosene. The increase in storage modulus of over 2400 % after 1 year of storage was not only a huge increase, but also a huge change in storage modulus as compared to the 50% increase of MRG. This finding outlined the drastic change in the rheological behavior of MRGK over time, which has caused concern for any potential applications using MRG. Even so, the overall storage modulus of MRGK was still significantly lower than MRG. Therefore, it is essential to further explore this behavior as well as investigate the effect of other dilution oils on the long-term stability of grease. This allows for a better understanding of diluting MRG, and thus the selection of an optimal dilution oil for use in different applications. As such, it will be a valuable development in knowledge not only to further the advancement of MRG as a viable MR material but also to hopefully allow for the opportunity to explore new additives and their effects on performance.

Future work on this study was planned on microstructural analysis of grease, which will paint a better picture of the effect of kerosene or other dilution oils on the microstructural integrity of grease. Furthermore, chemical studies, such as the Fourier transform infrared (FTIR) spectroscopy may be conducted to further analyze the nature of the structural degradation of MRGK.

6.0 DECLARATION OF COMPETING INTERESTS

The authors declare that there are no known competing interests and personal relationships that could have potentially influenced the work done and reported in this paper.

7.0 ACKNOWLEDGEMENT

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