THE EFFECT OF GRAPHITE CONTENT ON THE RHEOLOGICAL PERFORMANCE OF MAGNETORHEOLOGICAL GREASE

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DEDICATION

This thesis is dedicated to my family that always gives a great support for me to complete my Master Journey. A special dedication to my Husband, Hairi who continually grant a motivation for me to follows my dream and taking a good care of our child, Hasif.

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ABSTRACT

Magnetorheological grease (MRG) is classified as a smart material because its properties can be changed by applying a magnetic field. MRG is made up of magnetic carbonyl iron particles (CIPs) dispersed in a grease medium. The utilization of grease with high viscosity as a medium in MRG has benefit in preventing the settlement of CIPs from occurring. However, it limits the increment of yield stress in the on-state condition, thus reducing application performance during operation. Therefore, the introduction of graphite as an additive was investigated in this study to improve the rheological properties of MRG including apparent viscosity, shear stress and viscoelastic properties. Apart from that, the performance of MRG in term of yield stress was determined as well as the increment of yield stress from off-state and on-state condition were also evaluated to see the effect of different graphite contents on the properties of MRG. MRGs with graphite weight percentages ranging from 5, 10, and 15 wt.% were developed through conventional mixing method, namely MRG5, MRG10, and MRG15. The properties of all fabricated samples were then compared to those of a reference MRG sample. The microstructure of MRG and MRG15 was characterized using an environmental scanning electron microscope (ESEM). The rheological properties of all samples, including apparent viscosity and shear stress were examined using a shear rheometer in the rotational mode with shear rate ranging from 0.01 to $100s^{-1}$. While the viscoelastic properties in term of storage and loss modulus of all samples were carried out through shear rheometer under oscillatory mode with varied strains range from 0.001 to 10% at fixed frequency of 1Hz for strain sweep and frequency range from 0.1 to 80 Hz at fixed 0.01% strain for frequency sweep. The results demonstrated a uniform distribution of CIPs in MRG and CIPs with graphite in MRG15 under ESEM analysis at the off-state condition. Based on rheological testing, addition of graphite displayed a slight increment in the apparent viscosity of MRG5, MRG10, and MRG15, and a significant improvement in the yield stress. The highest yield stress achieved in this study is 61.778 kPa with increment of yield stress of 52.645 kPa from 0A to 3A. An expansion of the linear viscoelastic region from 0.01% to 0.1% was also observed for the MRG10 and MRG15 samples, credited to the domination of the elastic properties on the sample. Furthermore, all samples displayed a strong solid-like (elastic) behavior due to high value of storage modulus, G' acquired compared to loss modulus, G'' at all frequency ranges. These obtained results were confirmed based on ESEM under on-state condition, which described the contribution of graphite to constructing a more stable chain structure in the MRG. In conclusion, the findings highlight the influence of the addition of graphite on improving the rheological properties of MRG. Hence, the addition of graphite in MRG is a great potential to be applied in many applications such as in brake, damper and clutch.

ABSTRAK

Gris Reologi Magnet (MRG) dikelaskan di bawah bahan pintar kerana sifatnya boleh diubah melalui penggunaan medan magnet. MRG terdiri daripada partikel besi karbonil magnetik (CIPs) yang tersebar di dalam media gris. Penggunaan gris yang mempunyai kelikatakan yang tinggi sebagai media di dalam MRG memberi kelebihan dalam menghalang pemendapan CIPs daripada berlaku. Walaubagaimanapun, ia telah mengehadkan kenaikkan hasil tekanan pada keadaan pengaruh magnet, seterusnya mengurangkan prestasi operasi aplikasi. Oleh sebab itu, pengenalan grafit sebagai adiktif telah disiasat di dalam kajian ini untuk menaikkan sifat reologi MRG termasuk kelikatan ketara, tekanan rincih dan sifat kelikatan elastik. Selain itu, prestasi MRG dalam bentuk hasil tekanan ditentukan termasuk peningkatan hasil tekanan dikira melalui perbezaan dalam kedaan tanpa pengaruh medan magnet dengan pengaruh medan magnet untuk melihat kesan kepelbagaian kandungan grafit terhadap sifat MRG. MRG dengan peratus berat grafit dipelbagaikan dari 5, 10, dan 15 wt.% telah dihasilkan dan diberi nama sebagai MRG5,MRG10 dan MRG15. Kemudian, kesemua sampel dibandingkan sifatnya dengan sampel rujukan, MRG. Mikrostruktur sampel MRG dan MRG15 telah dicirikan diuji dengan pengunaan Mikroskrop Pengimbasan Elektron (ESEM). Sifat reologi untuk kesemua sampel termasuk kelikatan dan tekanan rincih diperiksa menggunakan rheometer dalam mod putaran dengan kadar rincih dipelbagai dari 0.01 hingga $100s^{-1}$. Disamping itu, sifat kelikatan elastik dijalankan dengan penggunaan rheometer rincih dalam mod ayunana dengan keterikan pelbagai dari 0.001 hingga 10% dengan frekuensi tetap, 1Hz dan frekuensi pelbagai dari 0.1 hingga 80 Hz dengan strain tetap 0.01%. Keputusan menunjukkan taburan seragam CIPs dalam MRG dan CIPs dengan grafit dalam MRG15 melalui analisis ESEM tanpa pengaruh magnet. Berdasarkan ujikaji reologi, penambahan grafit menunjukkan penambahan sedikit kepada kelikatan dalam sampel MRG5, MRG10, dan MRG15 dan peningkatan yang ketara dalam tekanan yang dihasilkan. Tekanan hasil yang tertinggi diperoleh dari kajian ini ialah sebanyak 61.778 kPa dengan perkembangan stress hasil sebanyak 52.645 kPa dari 0A hingga 3A. Rantau pengembangan linear likat kenyal (LVE) dari 0.01% kepada 0.1% turut diperhati disebabkan oleh dominasi sifat elastik sampel tersebut. Selain itu, kesemua sampel menunjukkan kekuatann sifat seperti pepejal (elastik) disebabkan oleh nilai modulus penyimpanan yang tinggi berbanding nilai modulus kehilangan dalam semua lingkungan frekuensi. Keputusan yang diperoleh ini telah dipastikan dengan ESEM dalam keadaan pengaruh medan magnet yang menunjukkan grafit memberisumbangan dalam penghasilan struktur rantai yang lebih stabil dalam MRG. Secara konklusinya, kajian ini menunjukkan pengaruh penambahan grafit dalam memperbaikisifat reologi MRG. Oleh itu, penambahan grafit dalam MRG menunjukkan kebolehan untuk diaplikasikan dalam pelbagai aplikasi seperti brek, peredam dan klac.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

MR	-	Magnetorheological
MRF	-	Magnetorheological fluid
MRE	-	Magnetorheological elastomers
MRP	-	Magnetorheological plastomers
MRG	-	Magnetorheological grease
CIPs	-	Carbonyl iron particles
UTM	-	Universiti Teknologi Malaysia
NGLI	-	National Lubricating Grease Institute
ESEM	-	Environmental Scanninng Electron Microscope
EDX	-	Energy dispersive X-ray
ANOVA	-	Analysis of variance

LIST OF SYMBOLS

Pa - Pascal

H - Magnetic field intensity

M_s - Magnetic saturation

M_r - Magnetic retentivity

H_c - Coercivity

G' - Storage modulus

G'' - Loss modulus

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Research background

Up to this date, the study regarding smart materials has been extensively carried out by numerous researchers due to their wide application in engineering and daily life. Smart materials can be defined as a material that can changes its properties correspond to external stimuli such as heat, light, temperature, stress, electrical field, and magnetic fields. Examples of smart materials are shape memory alloy, piezoelectric, photochromic, thermoactive, electrorheological, and magnetorheological (MR) materials. In these few decades, the study regarding MR materials has attracted interest by researchers due to their unique properties in which its rheological properties can be controlled through the application of magnetic fields [1]. Generally, MR material was made up of ferromagnetic particles, usually carbonyl iron particles, CIPs dispersed in various types of carrier medium or matrix. Based on the utilization of different carrier mediums or matrix, MR materials can be categorized under several groups which are Magnetorheological fluid (MRF), Magnetorheological elastomers (MRE), Magnetorheological grease (MRG), and Magnetorheological plastomers (MRP).

Among all, MRF is the most popular type of MR material due to its excellent performance which gives fast responses time as well as higher MR effect. MRF was first introduced by Rabinow in 1948 that consists of CIPs dispersed in non-magnetic carrier fluid [2]. Carrier fluid such as silicone oil, mineral oil, or other synthetic oil with low viscosity was commonly utilized in MRF [3]. The utilization of low viscosity carrier fluid in MRF has offered benefits as it allowed a free movement of CIPs to ease the formation of a columnar chain structured under influence of magnetic fields. These led to fast changes of its rheological properties as magnetic fields being applied and the changes are reversible when the magnetic fields are removed. The outstanding properties owned by MRF have made it widely applied in engineering applications such as brake, clutches, shock absorber, and engine mounts [4]. Nevertheless, MRF will undergo sedimentation after some time due to the density mismatch between CIPs with a low viscosity carrier fluid and thus lowered the stability and performance in MRF [5,6]. Aside from that, the usage of MRF in an application demanded additional seals to prevent leakage of the devices which increases the cost of the production in MRF-based devices [6,7]. The listed above drawbacks have led to the invention of other types of MR material named MRE that diffused CIPs in the solid rubber matrix. This invention was able to solve the sedimentation issues in MRF as CIPs are being locked within the matrix [8]. Nonetheless, as CIPs are fixed within the matrix, it is not free to move, instead it just vibrates, thus, it can only experience small changes of its properties with influence of magnetic fields. As consequences, low MR effect was obtained in MRE [8,9].

Alternatively, another type of MR material known as MRG has been introduced. MRG was early discovered by Rankin et al. in 1999 which utilizing high viscosity liquid, grease to suspend the CIPs [10]. The sedimentation problem that occurred in MRF can be encountered in MRG as the employment of non-Newtonian liquid grease in MRG can suspend the CIPs against gravity thus prevent it from being settled down [11]. This indirectly, result in excellent dispersion stability in MRG. On top of that, compared with MRE, CIPs in MRG have a certain freedom of movement, which can overcome the lower MR effect in MRE [10,12]. As consequence, a higher MR effect of 952.38% was recorded on MRG compared to 71.7% of solid-like state MRE [13,14]. Apart from that, by comparing with MRF, the MRG does not require additional sealing to prevent leakage of the devices as MRG owns self-sealing property due to their thick viscosity. This property can maintain the stability of the equipment for long-term usage, thus reduce the manufacturing cost [6].

The merits mentioned above have directed MRG to become a potential candidate to be applied in engineering applications such as seismic dampers, brakes, and clutches [4]. However, the utilization of grease as a medium for MR material has made the MRG experience high off-state viscosity and apparently, limits the expansion of yield stress in the on-state condition. This phenomenon in return has led the MRG to exhibit poor performance such as torque output [15].

1.2 Motivation of the Study

Despite of owing several advantages, MRG also own some limitation. Due to the utilization of grease with high viscosity, MRG experienced high off-state yield stress compared to MRF [15]. Conversely, in on-state conditions, it is seen that MRF gives a high value of yield stress compared to MRG. The reason of this because the movement of CIPs was hindered by high viscosity medium in MRG which in return led difficulty for the CIPs to align and form columnar chain structure with the influence of applied magnetic fields. Consequently, this resulted in reducing the increment of yield stress in on-state condition of MRG.

A development of MRG with high achievable yield stress and broad range from off to on-state are required to provide wide control in absorbing vibration [16,17]. Study done by Mohamad et al. [13] has proved that the yield stress of MRG can be improved by increasing the CIPs' weight percentage by up to 70wt% with range of increment of about 27.6kPa from off to on-state (0 to 3A). On other hand, an improvement of the rheological properties of the MRG can be undertaken by the addition of additives. Kim et al. [18] investigated the influence of kerosene oil as an additive on the rheological properties of MRG. They discovered that the apparent viscosity of MRG was reduced by the addition of 5 wt% of kerosene, which indicated a better dispersion of CIPs in the grease medium. However, at the same time, the dynamic yield stress and viscoelastic properties of MRG were also decreased.

Their findings were consistent with the study conducted by Mohamad et al. [19] that utilized and compared three different types of dilution oils namely kerosene oil, castor oil, and hydraulic oil in MRG. Even though they discovered the usage of these types of dilution oils was able to reduce the off-state viscosity of the MRG, however, the dynamic yield stress of the MRG was also reduced. In other words, the addition of dilution oils in MRG could lower their apparent viscosity, however, it would make the CIPs less attached to the grease medium and expected to experience a slipping effect under the influence of shear stress that caused a drop on the resultant yield stress. Consequently, it would reduce its performance especially under the influence of low magnetic fields strength. Recently, Wang et al. [20], have optimized the method to fabricate MRG through an ANOVA analysis by many parameters such as CIPs fraction and size, and silicone oil viscosity. They found that the optimum yield stress could be obtained by manipulating the CIPs fraction and silicone oil viscosity, but the influence of CIPs size was negligible. However, it was noted that as the utilized silicone oil viscosity was higher, the yield stress of MRG was seen to drop at a higher magnetic field strength. The reasons were possibly due to the utilization of high viscosity of silicone oil, up to 1000 m^2s^{-1} in MRG has contributed to the rise of MRG's apparent viscosity, which finally restricted the alignment of CIPs in the medium under influence of magnetic fields.

Apart from utilizing different types of dilution oil as an additive in MRG, several studies incorporated solid-type additives in order to enhance the rheological properties of MRG. For example, Park et al. [21] revealed that by adding nanoparticle's additive, CrO_2 in MRG has helped to improve the stability of MRG that resulted from the steric repulsion effect between CIPs and CrO_2. Though, the dynamic yield stress of MRG showed insignificant improvement. Nevertheless, Mohamad et al. [22] introduced another type of nanoparticle's additive in MRG namely, super-paramagnetic, γ-Fe_2O_3. The addition of 1 wt. % of additive capable to lower the off-state viscosity and at the same time, increased the viscosity at on-state viscosity of MRG. The result reflected the effect of nano-sized particles that filled in the voids between the CIPs under the influence of magnetic fields and thus, contributed towards the formation of stronger chain-like structures inside the medium. Later, Tarmizi et al. [23] utilized a micron-sized additive of cobalt ferrite, $CoFe_2O_4$ that has further lowered the off-state viscosity of the MRG by up to 86% with 1 wt%. Their result reported the highest yield stress obtained, about 12 kPa with the incorporation of 5 wt.% $CoFe_2O_4$ at 0.64 T of the applied magnetic field. However, it was noted that the range of expansion of yield stress in MRG with the incorporation of 5 wt% $CoFe_2O_4$ was considered low, which was from 0.8 to 12 kPa by increasing magnetic field from 0 to 0.64 T. Apparently, this yield stress range limit the material to be applied in a wide range of applications.

Aside from adding magnetic particles to improve the rheological properties of MR material, alternatively, incorporation of non-magnetic, carbon-based additives such as graphite could also enhance the rheological performance of MR materials [24–27]. Graphite possesses excellent properties such as good thermal and electrical conductivities, mechanical properties, chemically inert, and low density, which are capable to maintain the existing mechanical properties of the material. Moreover, graphite can be classified as an economical additive attributable to the high availability and low-cost production [28]. An experimental study conducted by Tian et al. [24] presented that the initial mechanical properties of MRE have been improved by the addition of 20 wt% graphite. Then, a noticeable improvement of MR effect up to 60% towards field-dependent modulus of MRE has been confirmed by Shabdin et al. [25] with corresponding to the 33 wt% graphite. In their study, the MR effect was improved by 176% as compared to the previous study in [24].

Other MR materials such as MRP and MRF have also benefiting from the utilization of graphite as an additive. With addition of 15 wt% graphite in MRP has increased the saturated storage modulus by 0.8 MPa compared to pure MRP, and the viscosity was remarkably improved due to strengthen effect exhibited by graphite [26]. In another study performed on MRF by Thakur [27], a high on-state viscosity and shear stress values could be obtained with increasing the weight percentage of graphite flakes by up to 3%. The authors stated that this was caused by the contribution of graphite that involved in improving the formation of columnar chain structure by filled the empty gap between CIPs to form more strong structures, and as a result, the yield stress was elevated.

Therefore, it can be concluded that the incorporation of graphite is proven to improve the rheological properties of MR materials, and it is expected that rheological properties of MRG will be improved too. Although the off-state viscosity of MRG is presumed to slightly increases with the addition of graphite powder, the field-dependent yield stress of MRG can be enhanced due to the strengthen effect exhibited by the graphite.

1.3 Problem Statement

Due to the tunable properties of MRG, it is potentially to be applied as a vibration damper. However, a development of MRG with high yield stress is required to provide a large variation in damping force in order to protect the body of the system. Aside from that, the range of yield stress’s increment from off to on-state also an important matter to be considered for technical utilization. Based on the previous study, the achievable yield stress of MRG and the range of yield stress from off to on state is still considered low. Meanwhile, additives were proven to enhance the properties of MRG, yet research related to additives that can improve the yield stress of MRG has not been carried out yet. Graphite on other hand are potential additives to be utilized in MRG as it is proven in improve the interaction between magnetic particles with influence of magnetic fields. Therefore, in this study graphite are introduced as an additive in MRG and their rheological properties was investigated to indicates the influence of graphite on the arrangement of magnetic particles in MRG that subsequently led to enhancement of the yield stress of MRG.

1.4 Research Objectives

The main objective of this research is to enhance and increase the range of yield stress from off to on state condition by introduce graphite additives in MRG. The specific objectives of this study are listed as below:

a) To confirm the uniform distributions of particles in MRG via morphological analysis.

b) To analyse the rheological properties of MRG under rotational and oscillatory mode at various magnetic field.

c) To evaluate the effect of graphite composition on the yield stress of MRG.

1.5 Research Scope

The scope of this study is focussed on experiment evaluation and characterization of MRG with graphite additives to indicate their potential ability in real application. The scopes are listed below:

a) Preparation of four (4) types of MRG with different weight percentages (wt.%) of graphite (0, 5, 10 and 15 wt%) via conventional mixing method by using mechanical stirrer for 2 hours.

b) The morphologies of MRG samples were examined via Environmental Scanning Electron Microscope (ESEM) under absence and presence of magnetic fields by pre-treated the sample with 0.1T magnetic fields for 5 minutes at room temperature.

c) The rheological properties of MRG samples are analysed in off-state and on-state through oscillatory and rotational mode of testing. The measure parameter are strain sweep and frequency sweep in terms of storage and loss modulus and viscosity in term of shear rate and shear stress. The performance of MRG in term of yield stress are determine from the extrapolation of graph at zero shear rate.

1.6 Significance of Research

Present study has introduced graphite additives in MRG and how different graphite content can enhance the rheological properties of MRG. Different from previous additives that has been utilized in MRG, graphite able to improve the yield stress as well as increase the yield stress range from off to on-state condition. This is due to the features owned by graphite led in developing a great interaction between the grease medium and CIPs thus results in creation of strong polarized chain structure within the medium with influence of magnetic fields. Therefore, the utilization of graphite in MRG with high yield stress and larger yield stress range can be potentially used in application of vibration damper.

1.7 Thesis outline

This thesis is structured into five chapters. The first chapter provides a brief introduction of the material studied, motivation of the study related to the previous related research, problem statement, objectives that wants to be achieved in this research scope and significance of the research. In this chapter, a summary of each chapter also being explained.

Chapter Two present the literature review related to the MR, additives that has being utilized in MRG, utilization of graphite in MR material as well as explanation on rheological properties of MR material and several examples of application that are potentially applied using MRG.

Chapter Three describes the planning of the experiment including material selection, fabrication of MRG samples through conventional mixing method, brief on the morphological testing on the samples and particles via Environmental Scanning Electron Microscope (ESEM) as well as rheological testing on all the samples under rotational and oscillatory mode in various magnetic fields strength.

Chapter Four displayed the result obtained from morphological and rheological testing in absence and presence of magnetic fields. The obtained result was interpreted in a graph, table, figure and was carefully discussed to provide an understanding of behaviour of graphite on the rheological properties of MRG.

Chapter Five conclude the result acquired from the research as well as stated the contribution and providing recommendation for potential future works.

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