RHEOLOGICAL AND STABILITY PROPERTIES OF MAGNETORHEOLOGICAL FLUID WITH SUPERPARAMAGNETIC MAGHEMITE NANOPARTICLES

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Specially dedicated to
My beloved mother, father, husband, son and all my family

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ABSTRACT

This research is focused on the development of a new magnetorheological (MR) fluid which contains maghemite (γ-Fe_2O_3) nanoparticles so as to improve its performance. The performance of MR fluid is presented in terms of physical and rheological properties and its application in MR device. In this work, the γ-Fe_2O_3 has been synthesized using co-precipitation method and coated with oleic acid. Two types of MR fluids were prepared, bidisperse MR fluid containing carbonyl iron (CI) microparticles substituted with γ-Fe_2O_3 and MR fluid utilizing γ-Fe_2O_3 additive. MR fluid containing γ-Fe_2O_3 showed great improvement exhibiting reduced sedimentation rate and enhanced re-dispersibility. During the period of 50 hours, the bidisperse MR fluid with 5 wt% of γ-Fe_2O_3 reduced 15% of sedimentation rate and MR fluid with 1 wt% of γ-Fe_2O_3 additive reduced 9.6% of sedimentation rate compared to pure CI MR fluid. The rheological properties of the MR fluid were analyzed with respect to the rheological models of Bingham Plastic, Herschel Bulkley and Casson models. The rheological properties of bidisperse MR fluid revealed that the substitution of 5 wt% γ-Fe_2O_3 increased the yield stress by 8.5% but further substitution of γ-Fe_2O_3 would slightly decrease the yield stress. On the other hand, the MR fluid added with γ-Fe_2O_3 additive showed improvement in yield stress over the entire range of magnetic field applied. The results indicated that the addition of 1 wt% of γ-Fe_2O_3 in MR fluid increased the yield stress by 11.7%. The performance of MR fluid using MR valve equipped with a hydraulic bypass damper resulted in improvement of damping force when γ-Fe_2O_3 is added. The MR fluid with 1 wt% γ-Fe_2O_3 additive improved the maximum damping force up to 11.1% compared to the pure MR fluid. Therefore, the substitution and addition of γ-Fe_2O_3 nanoparticles in the MR fluid improved both its physical and rheological properties, hence it can potentially be used in commercial application as a simple and reliable damping device.

ABSTRAK

Kajian ini diberi tumpuan kepada penghasilan bendalir magnetorheologi (MR) baru yang mengandungi nanopartikel *maghemite* (γ-Fe_2O_3) untuk meningkatkan prestasinya. Prestasi bendalir MR ditunjukkan dari segi sifat fizikal dan reologi dan aplikasinya dalam peranti MR. Dalam kajian ini, γ-Fe_2O_3 telah disintesis dengan menggunakan kaedah pemendakan dan dilapisi dengan asid oleik. Dua jenis bendalir MR disediakan, bendalir campuran MR yang mengandungi micropartikel besi karbonil (CI) digantikan dengan γ-Fe_2O_3 dan bendalir MR yang ditambah dengan bahan tambahan γ-Fe_2O_3. Bendalir MR yang mengandungi γ-Fe_2O_3 menunjukkan peningkatan di mana kadar pemendapan dikurangkan dan penyebaran semula dipertingkatkan. Dalam tempoh 50 jam, bendalir campuran MR dengan 5% γ-Fe_2O_3 mengurangkan 15% kadar pemendapan manakala bendalir MR dengan 1% bahan tambahan γ-Fe_2O_3 mengurangkan 9.6% daripada kadar pemendapan berbanding bendalir MR CI tulen. Sifat-sifat reologi dari bendalir MR dianalisis dengan model rheologi iaitu model Bingham Plastic, Herschel Bulkley dan Casson. Sifat rheologi bendalir campuran MR menunjukkan bahawa penggantian 5% γ-Fe_2O_3 meningkatkan tegasan alah sebanyak 8.5% tetapi penggantian γ-Fe_2O_3 seterusnya akan mengurangkan sedikit tegasan alah. Sebaliknya, bendalir MR yang ditambah dengan bahan tambahan γ-Fe_2O_3 menunjukkan penambahan tegasan alah apabila kekuatan medan magnet yang berbeza dikenakan. Keputusan menunjukkan bahawa penambahan 1% γ-Fe_2O_3 dalam bendalir MR meningkatkan tegasan alah sebanyak 11.7%. Prestasi bendalir MR menggunakan injap MR yang dilengkapi dengan peredam pintasan hidraulik menghasilkan peningkatan daya redaman apabila γ-Fe_2O_3 ditambah. Bendalir MR dengan bahan tambah 1 % γ-Fe_2O_3 meningkatkan daya redaman maksimum hingga 11.1% berbanding bendalir MR tulen. Oleh itu, penggantian dan penambahan nanopartikel γ-Fe_2O_3 dalam bendalir MR menambah baik ciri fizikal dan rheologinya, maka ia berpotensi untuk digunakan dalam aplikasi komersil sebagai peranti redaman yang ringkas dan boleh dipercayai.

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LIST OF ABBREVIATIONS

γ-Al_2O_3	-	Alumina
γ-Fe_2O_3	-	Maghemite
α-Fe_2O_3	-	Hematite
Co	-	Cobalt
$CoFeO_4$	-	Cobalt Ferrites
CI	-	Carbonyl Iron
CNT	-	Carbon nanotube
Cu	-	Copper
CuO	-	Copper Oxide
ER	-	Electrorheological
Fe	-	Iron
Fe^{2+}	-	Iron with oxidation number +2
Fe^{3+}	-	Iron with oxidation number +3
Fe_3O_4	-	Magnetite
$FeCl_2$	-	Iron (II) chloride
$FeCl_3$	-	Iron (III) chloride
$Fe(NO_3)_3$	-	Iron (III) Nitrate
FESEM	-	Field Emission Scanning Electron Microscope
FT-IR	-	Fourier Transform Infrared Spectroscopic
HCl	-	Hydrochloric acid
HNO_3	-	Nitric acid
Mn-Zn Ferrites	-	Manganese-Zinc ferrites
MR	-	Magnetorheological
MWCNT	-	Multiwalled carbon nanotube
NH_3	-	Ammonia solution
Ni	-	Nickel
PANI	-	Polyaniline

PEG 4000	-	Polyethylene glycol 4000
PMMA	-	Poly methyl methacrylate
PVA	-	Polyvinyl alcohol
R^2	-	Coefficient of determination
TEM	-	Transmission Electron Microscopy
TiO_2	-	Titanium Oxide
VSM	-	Vibrating Sample Magnetometer
XRD	-	X-ray Diffraction

LIST OF SYMBOLS

°C - Degree celcius
τ - Shear stress
τ_y - Dynamic yield stress
$\dot{\gamma}$ - Shear rate
η_p - Plastic viscosity
η_∞ - Fluid viscosity at infinite shear rate
µm - Micrometer
$\rho_{particle}$ - Density of the nanoparticle
ρ_f - Density of the ferrofluid
ρ_c - Density of the carrier liquid
$\emptyset_m$ - Particle mass fraction
χ - Magnetic susceptibility
μ - Permeability
μ_r - Relative permeability
π - Pi
β - Full width of half maximum values
θ - Diffraction angle
λ - Wavelength
A - Ampere
A/m - Ampere per meter
B - Magnetic flux density
cSt - Centistokes
cm^{-1} - per centimeter
emu/g - Magnetic moment over weight
emu/cm^3 - Magnetic moment over volume
g/cm^3 - Density

H	-	Magnetic Field Strength
Henry/m	-	Henry per meter
Hz	-	Frequency (Hertz)
g/mL	-	Density
kN	-	Kilo Newton
kA/m	-	Kilo Ampere per meter
kPa	-	Kilo Pascal
K	-	Consistency index
K	-	Kelvin
mm	-	Milimeter
mm^2	-	Area in milimeter
M	-	Magnetization
M_r	-	Remanent magnetization
M_s	-	Saturation magnetization
n	-	Model constant
nm	-	Nano meter
Pa.s	-	Pascal second
s^{-1}	-	per second
T	-	Tesla
v %	-	Volume percentage
wt %	-	Weigth percentage
Wb	-	Weber

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Magnetorheological (MR) fluids fall in the class of smart materials, due to its controllable rheological properties. MR fluid rheological properties can be continuously, rapidly, and reversibly changed with the present of a magnetic field, which makes this material of high interest, due to its real-time MR response [1]. MR fluid typically consist of micron-sized particles suspended in a non-magnetic fluid. MR fluid has an apparent yield stress up to 100 kPa depending on the composition, concentration of the particle and magnetic field strength [2]. The rheology of MR fluids has attracted much attention since its properties can be monitored by the application of magnetic field. Due to the improvement in MR technology, research on the MR characteristic and its applications are increasing, ranging from the automotive and civil engineering to the biomedical applications [3].

The important characteristics of magnetic particles described and used in the MR fluid are includes the saturation magnetization, distribution of particles size and shape and coercive field [4]. Besides the magnetic particles itself, carrier fluid, surfactants and additives are other important factors that can influence the rheological properties, stability and re-dispersibility of MR fluid [1]. In the absence of magnetic field (off-state), the magnetic particles in MR fluid are randomly dispersed in the carrier fluid. Under the influence of magnetic field (on-state), the dispersed particles formed a chain-like structure in the direction of the field with the pole of one particle being attracted to the opposite pole of another particle [5,6]. The inert-particle forces originating from the alignment of this magnetic particles lead to a material with higher yield stress and apparent viscosity [7]. The chain-like structure formed by the particles during application of magnetic field resist to a certain level of shear stress without breaking and the fluid behave as a solid-like liquid [8]. When the shear stress exceeds a critical level, the chain structure breaks and the fluid starts to flow. The value of shear stress at this critical level is known as apparent yield stress of the fluids [9].

Most of the success of MR fluid used in the devices is largely due to the advancement in fluid technology. The biggest challenges of MR fluid are to have high turn up ratio, high maximum yield stress and producing a stable and re-dispersible MR fluids [1,10]. To achieve all these criteria, researchers have to find a way to produce the best MR fluid, suitable for commercial applications where manufacturing cost and maximum yield stress are critical issues. Considering high density of microparticles dispersed in the MR fluid, their stability and re-dispersibility are the main issues. Therefore there are severe need to find improved methods for facilitating their stabilization [11]. To overcome these drawbacks, various methods have been reported to improve stability of MR fluid includes adding surfactant such as oleic acid and stearic salt to prevent aggregation [12], and thixotropic agent or thickening agent such as silica nano and arabic gum [12,13] to prevent particle settling. Moreover, the use of viscoplastics media such as grease [14], water in oil emulsions as continuous phase [15] and ionic liquid as carriers [16,17] have also been investigated. Besides that, some researchers also used different shapes of magnetic particles (flake shape) to improve its stability [18,19]. Furthermore, a few researchers improved the magnetic particles by coating them with polymers such as poly methyl methacrylate (PMMA) [20] and polyaniline (PANI) with multiwalled carbon nanotube (MWCNT) [21]. However, because the coating process using polymers are rather complicated, the additive method using various materials has been adopted. The use of these additives such as carbon nanotube, CNT [22], organoclay [23] and nanowires [24,25] have been found to effectively prevent sedimentation problem [26]. However, it is reported that by adding non-magnetic additive into the MR fluid will hinder the formation of chains, thus, decreases the MR effect [27].

In order to find a new way to enhance the performance, stability and re-dispersibility of the MR fluid, the focus has been shifted on suspension composed magnetic nanoparticles rather than non-magnetic nanoparticles. Thus, researches have been conducted on the advantages of using the mixture of magnetic nanoparticles and microparticles, called bidisperse MR fluids. Bidisperse MR fluid is a fluid that contain both micro- and nanoparticles, where part of microparticles is replaced with nanoparticles [17]. Chin et al. [28] reported that it is possible to maintain high level of MR effect while reducing the sedimentation rate by replacing only part of the microparticles in MR fluid with nanoparticles. The optimum concentration of nanoparticles at which the highest yield stress is reached is depends on total magnetic particles concentration. Based on previous researches, Wereley et al. [29] reached highest dynamic yield stress with 7.5 wt% of nanoparticles concentration when total particle loading is 60 wt%, whereas, Chauduri et al. [30] measured highest yield stress at 5 wt% of nanoparticles concentration with 45 wt% total particle loading, while Ngatu et al. [31] achieved highest yield stress at 15 wt% of nanoparticles concentration with 80 wt% total particle loading.

Although bidisperse MR fluid was reported to improve the sedimentation rate and re-dispersibility of the suspension, this substitution also offers different results in the enhancement of yield stress. Over the years, most researchers concluded that the substitution of magnetic nanoparticles has both improved the stability and increased the value of yield stress. Trihan et al. [32] and Wereley et al. [29] reported that substitution of 20% magnetic nanoparticles to the MR fluid increase the yield stress. Meanwhile, Chaudhuri et al. [30] reported that substitution of 5% magnetite nanoparticle (Fe_3O_4) increased the yield stress but decreased when the magnetite is 7.5%. Furthermore, Lopez et al. [33] also reported an increased in yield stress for magnetite varied from 0 to 21.6%. Recent research by Jonkkari et al. [17] also reported that 5% substitution of magnetite increased the yield stress up to 13%. On the other hand, several other researchers found that the substitution of magnetic nanoparticle into MR fluid would decrease the value of yield stress eventhough the sedimentation stability is increased. Rosenfelt et al. [34] reported that the yield stress has reduced to 11% for bidisperse suspension compared to the monodisperse suspension. Ngatu et al. [31] also found that magnetic nanoparticles reduced the yield stress of MR fluid up to 64% and Iglesias et al. [35] also reported that the yield stress is decreased when 7% of magnetic nanoparticle is substituted into MR fluid. Upon comparing the results from all the researches, the enhancement of MR fluid yield stress is strongly dependent on the magnetic saturation value of the magnetic nanoparticles itself. Higher value of magnetic saturation in magnetic nanoparticle tends to increase the value of yield stress of MR fluid. Most of the researchers that reported the improvement of MR fluid yield stress used magnetite nanoparticles that have high magnetic saturation, while in contrast, the researchers that reported in reduction of MR fluid yield stress mostly used the iron nanoparticles synthesized from carbonyl iron that have lower value of magnetic saturation. For example, Park et al. [36] reported that the iron nanoparticles synthesized from carbonyl iron precursor have magnetic saturation of 4.58 emu/g or 9 kA/m, far lower than magnetite with magnetic saturation of 410 kA/m [37].

Generally, bidisperse MR fluid can increase the value of yield stress compared to monodisperse MR fluid, but however there are certain level of nanoparticles concentrations that can be substituted before the yield stress is decreased [30]. From manufacturer’s point of view, bidisperse MR fluid gives advantages in terms of device weight and cost. This is because in bidisperse MR fluid, the concentration of micron-sized particles is reduced and subsequently reduce the weight of the device.

There is also another way to improve the MR fluid stability and at the same time increase the yield stress. Based on the literature, most of the researchers focused on the development of bidisperse MR fluid and there are only a few researches on the use of magnetic nanoparticles as an additive in MR fluid. The usage of magnetic nanoparticle additive is considered as an effective way to enhance both the dispersion stability and MR fluid behaviour. In this type of MR fluid, the magnetic nanoparticle is added to the suspension of MR fluid without reducing the concentration of micron-sized particles. Over the years, there were only 2 researches that have been conducted on the influence of magnetic nanoparticle as an additive in the MR fluid. Park et al. [36] reported the use of iron nanoparticles derived from carbonyl iron with the size of 4 nm, having magnetic saturation of 4.58 emu/g. They added the iron nanoparticles as an additive in the MR fluid with concentration of 0.1 and 1 wt% and found that at high magnetic field, the yield stress of pure MR fluid is 6.8 kPa, MR fluid with 0.1 wt% additive is 7.2 kPa and MR fluid with 1 wt% additive is 9.7 kPa. Later on, Jang et al. reported the use of rod shape maghemite (γ-Fe_2O_3) nanoparticle with size of 500 nm as an additive in the MR fluid. They found that the addition of 1 wt% of rod shape γ-Fe_2O_3 in the MR fluid improved the sedimentation rate and at the same time increased the MR properties. Therefore, the research on the effect of magnetic nanoparticles as an additive in the MR fluid should be increasingly chosen and investigated as it contributes to a better performance of MR fluid.

Most of the researchers investigated the use of magnetite nanoparticle in MR fluid compared to maghemite nanoparticle (γ-Fe_2O_3) because the value of magnetic saturation of magnetite nanoparticle is slightly higher than maghemite nanoparticle [38]. However, maghemite nanoparticle is chemically stable and exhibits higher curie temperature compared to magnetite nanoparticle that is not stable and easy to oxidize [39]. The use of maghemite (γ-Fe_2O_3) nanoparticles in MR fluid would contributes to a better performance of MR fluid in terms of fluid stability and yield stress. Therefore, the development of bidiperse MR fluid and MR fluid added with nanoparticles additive utilizing maghemite (γ-Fe_2O_3) nanoparticles were investigated in this research.

1.2 Problem Statement

MR fluids are known as smart materials due to the rapid changes in MR response when subjected to a magnetic field. In order to produce high maximum yield stress of MR fluid, micron-sized magnetic particles are used instead of nano-sized magnetic particles [35]. However, due to high density of micron-sized magnetic particles, MR fluid is faced with the problem of instability of the suspension caused by high settling rate which makes it a severe drawback towards more generalized applications [40]. The formation of hard and compact sediment over time is due to the gravitational forces and remixing it would be difficult because of the remnant magnetism that keeps them in aggregates [28,33,41]. Hence, the need of finding improved methods is crucial in order to stabilize the MR fluid and at the same time to improve the maximum yield stress. In addition, re-dispersion is one of the biggest challenges in the realization of MR fluid and researchers were focusing on the stability or sedimentation rate of this suspension and not what happened after the particles sedimentation occurs [23]. Over the years, researchers improve the stability by adding non-magnetic particle into the MR fluid. Even though the stability has been improved, the rheological properties of the fluid are affected. The formation of particle chains is hinder due to the presence of non-magnetic particle, thus reduce the MR effect. Besides, the value of yield stress also reduced if lower volume fraction or smaller particle size is used.

In terms of MR fluid performance, if the yield stress is reduced, lower performance by the MR device is produced. Furthermore, the use of MR fluid in MR devices is limited commercially due to its high manufacturing cost and low output maximum yield stress. If higher output performance need to be produced, the MR device must be equipped with bulkier and heavier coils to provide high magnetic field, thus an additional space is required [28]. Moreover, the weight of the MR device might also increase if higher volume fraction or larger size of magnetic particles is used in the MR fluid [9]. Therefore, the particles sizing and concentration of magnetic particles suspended in the fluid are limited in order to maintain its stability and low off-state viscosity [15]. The use of magnetic nanoparticles as a substitute particles in MR fluid (bidisperse MR fluid) is reported to improve the fluid stability and increase the fluid yield stress, thus the weight of MR device can be reduced due to lower volume fraction of magnetic microparticles is used. On the other hand, the use of magnetic nanoparticles as an additive in MR fluid is also reported to improve the fluid stability and increase yield stress by maintaining the magnetic microparticles concentration. Based on the previous results, the most generalized method is by introducing the magnetic nanoparticle in the MR fluid either by substituting the magnetic nanoparticles in the MR fluid [29,42,43] or by adding the magnetic nanoparticle as an additive [36,44,45].

1.3 Research Objectives

The objective of the study is to formulate a novel MR fluids utilizing superparamagnetic γ-Fe_2O_3 nanoparticles and investigate the rheological properties suitable for MR device system. More specifically the objectives of this research were:

a) To synthesize and modify γ-Fe_2O_3 nanoparticles so as to ensure its suitability to the oil phase.
b) To formulate different compositions of oil-based MR fluid which consist of microparticles, nanoparticles, carrier liquid and additives in order to obtain the most suitable MR fluid based on their rheological characteristics.
c) To analyze physical and rheological properties under influence of magnetic field.
d) To evaluate the performance of MR fluid in terms of damping force using MR valve equipped with hydraulic bypass damper.

1.4 Research Scope

The scopes of the study are as follows:

a) The γ-Fe_2O_3 nanoparticles are synthesized using co-precipitation method and the surface of the nanoparticles are modified using oleic acid.
b) The oil-based MR fluids are formulated using carbonyl iron (CI) as micron-sized particles, γ-Fe_2O_3 as nano-sized particles and hydraulic oil as carrier liquid. γ-Fe_2O_3 nanoparticles was also added as an additive to investigate the influence of the nanoparticles as additive in MR fluid.
c) The physical properties (density, sedimentation rate and re-dispersibility) and rheological properties (apparent viscosity, shear stress and dynamic yield stress) of the formulated MR fluids are evaluated during off and on-state condition.

d) The performance of MR fluid in terms of force versus displacement and force versus velocity was measured using MR valve equipped with a double rod hydraulic cylinder in bypass configuration at different current magnitudes and frequencies.

1.5 Significance of Research

The significance of this research lies in the enhancement of MR fluids especially to answer the demand for high performance fluid with low sedimentation rate and easy to re-disperse. In this study, synthesized super-paramagnetic nano-sized magnetic particles namely maghemite (γ-Fe_2O_3) nanoparticles coated with oleic acid are added to be a part of MR fluids. This research provides knowledge on the effect of γ-Fe_2O_3 nanoparticles in the MR fluids which improves both physical and rheological properties of MR fluids which was never reported. Finally, the novel MR fluid was used in the MR devices which results in the improvement of device performance thus demonstrating its application.

1.6 Outline of Thesis

This thesis is organized in six chapters. The first chapter of this thesis contains an introductory chapter including the research objectives and contributions. Each respective chapter in this thesis ends with a brief summary outlining the achievement and findings that were established in the chapter.

Chapter 2 covers the theoretical background and literature review of the field responsive fluids that undergo rheological changing upon application of external field. This chapter also explains the theoretical background of nanotechnology and MR technology as well as the integration of magnetic nano in micro-particles MR fluids in terms of basic principles and rheological properties.

Chapter 3 elaborates the experimental evaluation of the γ-Fe_2O_3 nanoparticles and MR fluids utilizing γ-Fe_2O_3 nanoparticles including the description of experimental setup and the experimental procedure. The details of the procedure to synthesize and coating γ-Fe_2O_3 nanoparticles, formulation of MR fluids containing γ-Fe_2O_3 nanoparticles, characterization of MR fluid and the evaluation of MR fluid performance using MR device are also been elaborated. This chapter also includes the list of materials used in this research.

Chapter 4 presents the result and discussion of the experiment for both physical and rheological properties of MR fluid during off- and on-state condition, including the analysis of the experimental results with respect to the rheological model. This chapter also discussed the results obtained from the evaluation of MR fluid performance using MR valve equipped with hydraulic bypass damper.

The final chapter 5 is the concluding chapter which highlights the achieved contribution of the research in the relation to the research objectives. The recommendation for future research work is also presented in this chapter.

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