

EFFECT OF MILLING TIME ON RHEOLOGICAL PROPERTIES OF PLATE-  
LIKE CARBONYL IRON PARTICLES IN MAGNETORHEOLOGICAL FLUID

SALIHAN BINTI TAN SHILAN

UNIVERSITI TEKNOLOGI MALAYSIA

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SALIHAN BINTI TAN SHILAN

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## DEDICATION

*The dedication of this thesis goes to my supervisor, my parents and all my family members who really supported me during this period. Not forgetting my beloved friends who remained on my side. Thank you very much to all your concerns and kindness.*

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## ABSTRACT

Magnetorheological (MR) fluid is a magnetic field responsive fluid containing polarizable particles and a liquid carrier component. This MR fluid is typically used within the working gap of dampers to provide varied damping forces, depending on magnetic field strength. Generally, maximum yield stress will be increased with the increasing amount of particles in the MR fluid, and consequently enhancing the maximum force output of the device. An additional number of particles also lead to increasing the off-state viscosity and sedimentation of the MR fluid. Researchers have concentrated on improving the properties of particles to overcome the sedimentation problem. Particle shape is a critical factor to enhance the sedimentation stability. Thus, this research aims to synthesize the plate-like carbonyl iron (CI) to enhance the maximum yield stress and stability of the suspension fluids. Several sizes of plate-like CI were produced using the ball milling method, with the variation of milling time. Observation from X-ray Powder Diffraction, Scanning Electron Microscope and Vibrating Sample Magnetometer results showed that milling duration play a crucial role in controlling the plate-like CI's microstructure, morphology, and consequent magnetic properties obtained from the ball-milling route. The crystallography phase of CI particles was conserved after the milling process, and it is flattened, resulting in the enhancement of the particle length and surface area. Accordingly, the plate-like CI obtained a larger contact surface, which builds stronger clusters due to the magnetic field and resulted in higher saturation magnetization than the spherical CI. This study identified that the sedimentation stability of the plate-like CI-based MR fluid increased significantly due to the increase of the friction force resulted from the increase of the surface area of particles. Subsequently, the field-dependent rheological test demonstrated that the yield shear stress of the plate-like CI-based MR fluid increased up to 134% compared to the spherical CI-based MR fluid. In sum, this study found that CI particles which undergo 20 hours of milling has the best parameter to be used in MR fluid.

## ABSTRAK

Bendalir Magnetorheological (MR) adalah cecair responsif medan magnet yang mengandungi medan zarah terpolarisasi dan komponen pembawa cecair. Cecair MR ini biasanya digunakan dalam jurang kerja peranti seperti peredam untuk memberikan daya redaman yang bervariasi, bergantung pada kekuatan medan magnet. Secara amnya, tekanan hasil maksimum akan meningkat dengan peningkatan jumlah zarah di dalam cecair MR dan seterusnya meningkatkan output daya maksimum peranti. Walaupun begitu, sejumlah zarah tambahan juga menyebabkan peningkatan kelikatan dan pemendapan cecair MR di luar keadaan. Kebanyakan penyelidik telah menumpukan perhatian untuk meningkatkan sifat zarah dalam mengatasi masalah pemendapan. Bentuk zarah merupakan factor kritikal untuk meningkatkan kestabilan pemendapan. Oleh itu, idea utama penyelidikan ini adalah meleperkan besi karbonil (CI) untuk peningkatan tekanan hasil maksimum dan kestabilan cecair suspensi. Beberapa ukuran CI leper dihasilkan dengan menggunakan kaedah penggilingan bola, dengan variasi tempoh penggilingan. Hasil pemerhatian dari keputusan *X-ray Powder Diffraction*, *Scanning Electron Microscope* dan *Vibrating Sample Magnetometer* menunjukkan bahawa tempoh penggilingan memainkan peranan penting dalam mengawal struktur mikro, morfologi, dan sifat magnetik CI leper yang diperoleh melalui penggilingan bola. Fasa kristalografi zarah CI masih kekal setelah proses penggilingan dan bentuk leper terhasil dengan peningkatan panjang dan luas permukaan zarah. Sehubungan itu, CI leper mempunyai permukaan sentuhan yang lebih besar yang membentuk gugus yang lebih kuat untuk dibentuk kerana medan magnet, dan menghasilkan magnetisasi tepu yang lebih tinggi berbanding dengan CI sfera. Kajian ini mengenalpasti bahawa kestabilan pemendapan cecair MR berasaskan CI leper meningkat dengan ketara disebabkan oleh peningkatan daya geseran yang terhasil daripada pertambahan luas permukaan zarah. Seterusnya, ujian reologi yang bergantung pada medan menunjukkan bahawa tegangan ricih hasil cecair MR berasaskan CI leper meningkat sehingga 134% dibandingkan dengan cecair MR berasaskan CI sfera. Secara konklusinya, kajian ini mendapati zarah CI yang menjalani penggilingan selama 20 jam mempunyai parameter terbaik untuk digunakan dalam cecair MR.

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## LIST OF ABBREVIATIONS

<b>BASF</b>	-	Baden Aniline and Soda Factory
CI	-	Carbonyl iron
CIP	-	Carbonyl iron particles
ER	-	Electro-rheological
MR	-	Magnetorheological
SEM	-	Scanning electron microscopy
VSM	-	Vibrating sample magnetometer
XRD	-	X-ray diffractometer

## LIST OF SYMBOLS

$A$	-	Ampere
$a$	-	Diameter
$B$	-	Magnetic flux density
$^{\circ}\text{C}$	-	Degree Celsius
$F_{ij}$	-	force tensor
$H$	-	External magnetic field
$H_c$	-	Coercive Force
$kA/m$	-	Kilo ampere per meter
$kPa$	-	Kilo pascal
$m$	-	Magnetic dipole moment
$mT$	-	Milli-Tesla
$T$	-	Tesla
wt %	-	Weight percentage
%	-	Percentage
$\mu_0$	-	Vacuum permeability
$\mu_f$	-	Specific permeability particles
$\mu_s M_s$	-	Saturation magnetization of particles
$\mu_p$	-	Specific permeability carrier fluid
$\mu m$	-	Micron meter
$\eta$	-	Shear viscosity
$\eta_r$	-	Relative viscosity
$\delta$	-	Phase angle
$\gamma$	-	Shear strain
$\dot{\gamma}$	-	Shear rates
$\tau$	-	Shear stress
$\tau_y$	-	Yield stress
$\varphi$	-	Volume fraction

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# CHAPTER 1

## INTRODUCTION

### 1.1 Introduction

Controllable fluids are composed of components that can react to an external excitation field. When exposed to either an electric or a magnetic field, their rheological behaviour goes through exceptional changes. This type of fluid is known as "smart fluid". Some common names for these smart fluids include magnetorheological (MR) fluids, electrorheological (ER) fluids, and ferrofluids. ER fluids are suspensions of extremely small nonconductive but electrically active particles in an insulating fluid. [1] Ferrofluids are a stable colloidal combination of ferrimagnetic and/or ferromagnetic nanoparticles, while MR fluids are suspensions of magnetizable microparticles dispersed in a carrier fluid. MR fluids are garnering more attention than other forms of smart fluids since they are able to produce the maximum stress and can therefore be utilized in a wide variety of applications. This is one of the reasons why MR fluids are becoming increasingly popular.

A magnetic field-responsive fluid is referred to as an MR fluid. This type of fluid is made up of polarizable particles, a non-magnetic liquid carrier, and an additive. Rabinow discovered this controllable fluid in 1947 [2], and it has since been refined by several researchers. MR fluid has a variety of properties that can be used to evaluate performance. These include field-dependent apparent viscosity, yield stress, reaction time to the magnetic field, sedimentation due to particle-to-carrier liquid density mismatch, and chemical stability. In general, as the magnetic field intensity increases, so does the apparent viscosity of the MR fluid. This is because the suspended magnetic particles form chain-like structures in the direction of the applied magnetic field, restricting fluid flow [3]. In other words, the intensity of the applied magnetic field can be used to control rheological properties of MR fluids such as apparent viscosity and yield stress [4]. As a result, when a magnetic field is applied, MR fluid behaves as a



non-Newtonian fluid, and the field-dependent yield stress is expressed as a function of the magnetic field [5, 6]. The apparent viscosity of MR fluid is primarily determined by shear rate as well as the magnitude and direction of the applied magnetic field [7]. Furthermore, the response time of MR fluid is the most significant advantage in terms of application, as it is known to be dependent on the strength of the applied magnetic field. Aside from that, temperature has little effect on the operational characteristics of MR fluid, which can be controlled over a wide range of viscosity [5]. Because of its fast response to magnetic field strength, MR fluid requires relatively little power to operate, and up to 100 kPa of dynamic yield stress is represented [8]. When compared to other rheological fluids, this unique property of MR fluid has enabled its use in small devices and a wide dynamic range of application [11-13]. As a result, MR fluid is suitable for a wide range of mechanical applications and is one of the fastest electromechanical interfaces.

In the early stages of development, MR fluids have received less attention than electrorheological (ER) suspensions due to the limitations in applications [7]. Because of this, the enhancement of MR fluid characteristics has been the subject of a lot of research. In general, the maximum yield stress of MR fluid will be enhanced if the particle concentration is increased or if a larger particle size is used [11]. Consequently, an increase in the device's capacity to generate its maximum force can be accomplished. However, the use of larger particle sizes and the rise in particle concentration will raise the off-state viscosity, resulting in poor fluid stability [7, 12, 15, and 16]. Several methods for improving the poor stability of MR fluid have been investigated, including the use of thixotropic additives, electrostatic additives, non-spherical particles, and nanometer-sized particles. The particle shape was discovered to be the critical factor in improving the MR fluid sedimentation stability [7]. Details about the significance of particle shape in MR fluid performance are explained in the next chapter.

## 1.2 Motivation of Study

The conventional MR fluid has some difficulty finding widespread commercial use due to high manufacturing costs and low yield stress [7]. To increase the output of MR devices, the coils must occupy more space in order to generate a stronger magnetic field, and the increased amount of MR fluid may cause the devices to become heavier. To maintain its low off-state viscosity [13, 14], the number of particles suspended in MR fluid is limited, resulting in a low yield stress. Researchers [11] say that the most important things about MR fluid are a high yield stress, a low viscosity in the off-state, and a low sedimentation rate.

The development of MR fluids with a high yield stress has been intensively studied by researchers in order to improve the application efficiency of MR fluids. The high yield stress of MR fluid was obtained by increasing the volume fraction of particles and employing larger particle sizes [15–17]. Nonetheless, the use of larger particle sizes and a greater volume fraction of particles has increased the MR fluid's off-state viscosity and sedimentation rate [18].

To address the sedimentation issue, researchers have focused on enhancing particle properties. Utilizing thixotropic additives [19,20], electrostatic additives [21], the addition of non-spherical particles [7,22], and nanometer-sized particles [23] have all been used to combat the sedimentation issue of MR fluid. On the other hand, the yield stress has decreased as a direct consequence of the incorporation of these particles. Consequently, this varies according on the composition and the particle size [10].

Later, researchers discovered that particle shape significantly influenced MRF properties [7]. Upadhyay et al. [15] reported that flaky-shaped pure iron decreased sedimentation without impairing magnetic properties. Ido et al. [24] investigated the impact of particle shape on damping force. It demonstrates that nickel-iron with an irregular shape achieves a greater damping force than nickel-iron with a spherical shape. Bell et al. [12] reported the effect of suspensions containing magnetic wires on the enhancement of yield stress. However, the most cost effective particle to be use is

carbonyl iron. In sum, plate-like shape particles promoted the increasing of magnetisation, enhancing the effective permittivity more than that of the spheres or needles shape particles due to the polarization resulting from the large plate diameter.

Quite a few of researchers have addressed the impact of plate-like CI on MR fluid properties. Though, no researchers have thoroughly investigated the relation of aspect ratio of plate-like CI towards MR performance. Most of the researcher, only be used one type or size of the plate-like particles produced by a company and did not discuss the manufacturing (milling) procedure. Therefore, it is necessary to investigate the essential properties of plate-like CI, such as particle size and magnetic saturation.

### **1.3 Research Objectives**

This research aims to investigate the effect of milling time towards the MR fluid base plate-like CI performance. The specific objectives of this study are as follows:

- (a) To investigate the characteristics of plate-like CI with different milling time.
- (b) To analyse the sedimentation rate of plate-like CI produced with different milling time.
- (c) To evaluate the effects of plate-like CI size on the field-dependent rheological properties of MR fluid.

### **1.4 Research Scope**

The scope of the research is specified on the experimental investigation of the new produced plate-like CI-based MR fluid and comprehensive characterization to confirm the enhancement of yield stress without increasing the sedimentation rate for better applications performance. This includes:

- (a) The plate-like shape of CI particles is fabricated by using the ball milling process. Several sizes of the plate-like CI will be produced to identify the significance of particles aspect ratio on MR fluid properties.
- (b) The characterization of plate-like CI such as particle size and shape morphology, crystallography, and magnetic properties (on-state) are investigated.
- (c) The sedimentation rate of plate-like CI-based MR fluid is identified by placing the MR fluid in a vertical cylindrical container at room temperature.
- (d) The rheological characterisation is analysed in the rotational and oscillatory mode under off and on-state conditions at room temperature. This testing is performed to understand the correlation between CI particle shapes towards yield stress in MR fluid.

## **1.5 Thesis Outline**

This thesis is organised into five segments. For the benefit of readers who are not previously familiar with the topic, this thesis included an introductory chapter that provides a concise overview of MR fluids as well as their behaviour when subjected to the presence of magnetic fields. Additionally, this chapter described the findings of prior research and how they led to the aims of this study. At the end of each chapter in this thesis is a summary that outlines the accomplishments and results that were established in that particular chapter. The remaining portions of this thesis are structured in the following manner:

Chapter 2 presents the theoretical basis and a literature review concerning previous works on MR fluid materials. This includes recent developments in magnetic materials, compositions, particle shape, the rheology of suspensions, and the stability of MR fluid.

Chapter 3 discusses the experimental approaches that were utilised to construct the plate-like CI with different milling time, combined with an examination of the characteristics of the particles created.

Chapter 4 provides a comparative analysis of plate-like CI generated with varied milling times. The behaviour of various CI particle types is investigated and extensively described. In addition, the effect of different milling times on the sedimentation rate and rheological characteristics of MR fluid are investigated in detail.

Chapter 5 summarises and concludes the overall findings from the experimental analysis. The contribution study that corresponds to the objectives is outlined. For the continuation of the study, further research recommendations are given.

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## Appendix A

### LIST OF PUBLICATIONS

#### Journal

1. Nurul Azhani Yunus, Saiful Amri Mazlan, Ubaidillah, Siti Aishah Abdul Aziz, **Salihah Tan Shilan** and Nurul Ain Abdul Wahab, Thermal Stability and Rheological Properties of Epoxidized Natural Rubber-Based Magnetorheological Elastomer, *Int. J. Mol. Sci.* 2019, 20(3), 746. (Q1, IF:4.556)
2. Muntaz Hana Ahmad Khairi, Saiful Amri Mazlan, Siti Aishah Abdul Aziz, Ubaidillah and **Salihah Tan Shilan**, Performance of Magnetorheological Elastomer Based Green Epoxidized Natural Rubber/Sucrose Acetate Isobutyrate Hybrid Matrix, *IOP Conf. Series: Materials Science and Engineering*, 342, 012034, 2018.
3. Abdollah Hajalilou, Saiful Amri Mazlan, **Salihah Tan Shilan**, Ebrahim Abouzari-Lotf, Enhanced magnetorheology of soft magnetic carbonyl iron suspension with binary mixture of Ni-Zn ferrite and Fe<sub>3</sub>O<sub>4</sub> nanoparticle additive, *J. Colloid and Polymer Science*, Issue 9(2017). (Q3, IF:1.447)
4. **Salihah Tan Shilan**, Saiful Amri Mazlan, Yasushi Ido, Abdollah Hajalilou, Balachandran Jeyadevan, Seung-Bok Choi and Nurul Azhani Yunus, A Comparison of Field-Dependent Rheological Properties Between Spherical and Plate-Like Carbonyl Iron Particles-Based Magnetorheological Fluids, *Smart Material Structure*, Vol. 25, (2016) 095025. (Q1, IF:2.909)
5. **S. T. Shilan**, S. A. Mazlan, M. H. A. Khairi and Ubaidillah, Properties of Plate-Like Carbonyl Iron Particle for Magnetorheological Fluid, *Journal of Physics: Conference Series*, 776 (2016) 012033. (IF:0.5)
6. N.A.Yunus, S.A.Mazlan, Ubaidillah, S.A.A.Aziz, M.H.Ahmad Khairi, N.A.A.Wahab and **S.T.Shilan**, Investigation on Magnetic Field Dependent Modulus of Epoxidized Natural Rubber Based Magnetorheological Elastomer, *Journal of Physics: Conference Series*, 776 (2016) 012024. (IF:0.5)