Investigation on The Characteristics of Magnetorheological Elastomer Using Natural Rubber and Petroleum-Based Oil

by

Marni Zalikha Binti Mohd Zarir
22752

Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
(Mechanical)

January 2020

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CERTIFICATION OF APPROVAL

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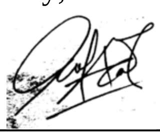

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TRONOH, PERAK

January 2020

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

MARNI ZALIKHA BINTI MOHD ZARIR

ABSTRACT

A comprehensive study on the characteristics of magnetorheological elastomer (MRE) has yet been reported although it is vital prior to employ the MRE in any smart devices. The purpose of this paper therefore is to study the influence of petroleum-based oil (PBO) on the characteristics of MRE using natural rubber as MRE matrix and carbonyl iron particles as magnetizable particles. Three PBO that will be used are naphthenic oil, paraffin oil, and light mineral oil. The compositions were mixed and cured without an applied magnetic field to fabricate isotropic MRE. The fabricated MRE was characterized with respect to magnetic properties, morphology, and thermal behaviour. The results showed that natural rubber-based MRE with light mineral oil had homogeneous dispersion of carbonyl iron particles within the matrix. In the meantime, the MRE with naphthenic oil obtained low retentivity magnetization as a result of carbonyl iron particles as a soft magnetic particles that easily demagnetized. The thermal stability of MRE with naphthenic oil improved as PBO improves the dispersion of carbonyl iron particles thus decrease the permeability of heat to pass through the MRE. The enhanced characteristics of MRE was expected as PBO acts as dispersing agent and improve the dispersibility of carbonyl iron particles within the MRE.

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

ABBREVIATIONS AND NOMENCLATURE

EDX : Energy dispersive X-ray spectroscopy

FESEM : Field-emission scanning electron microscopy

MR : Magnetorheological

MRE : Magnetorheological elastomer

MRF : Magnetorheological fluid

NR-based MRE : Natural rubber-based magnetorheological elastomer

PBO : Petroleum-based oil

TG : Thermal gravimetric

TGA : Thermal gravimetric analysis

VSM : Vibrating-sample magnetometer

CHAPTER 1 : INTRODUCTION

1.1 Background

Magnetorheological (MR) consist a mixture of non-magnetic medium and soft magnetic particles, which can be controlled with magnetic field. Magnetorheological fluid (MRF) is first discovered in 1948 by Rabinow, which is a mixture of oil and ferrous particle that can change its phase from liquid to solid [1]. However, MRF exhibit distinct shortcomings such as the long-term stability caused by sedimentation of particles in MRF [2]. Thus, MRE was developed as an alternative of MRF, which is a solid composite material that consist of rubber, iron particles and additives.

There are two types of MRE comprises of isotropic MRE and anisotropic MRE [3]. Isotropic MRE is cured without an external magnetic field, while anisotropic MRE is cured in the presence of magnetic field [4]. By curing the anisotropic MRE under an applied magnetic field, the particles will form chains in the matrix, thus increase the interaction between the particles [2][4].

MRE has a number of applications in automotive industry particularly for vibration and noise control. The example of the applications in automotive are suspension systems, bushings, vibration absorbers, dampers, etc. [3][5][6]. Besides that, MRE is also applied in medical rehabilitation for the artificial prosthetic knees, whereby the prosthetic provide shock absorption between femur and tibia, thus prevent the knee from bending [5]. MRE has also showed a potential to be utilized in civil infrastructure to mitigate seismic forces caused by shock events [7][8][9][10].

MRE is a smart composite material that provides advantage due to its responsiveness to external magnetic field. Hence, the development of MRE-based devices or technologies could bring a huge achievement and be commercialised in a broad field.

1.2 Problem Statement

MRE is a developing smart composite which has been discovered for more than two decades starting from year 1995. The magnetic, mechanical and rheological properties, and stability of MRE brings a broad application prospects in the intelligent vibration and noise reduction. However, the existence of gaps between material properties of MRE and practical application results to restriction in commercial and real applications [6]. There are lack of feasibility studies on the characteristics of MRE such as the thermal behaviours of MRE and its functional behaviour. Hence, the characteristics of MRE is still in research state and have yet been discovered for various of application. Thus, this study is conducted to analyse the effect of three different PBO as the dispersing aids on the characteristics of MRE by using natural rubber as carrier matrix and carbonyl iron particles as magnetic particles, under the absence and presence of magnetic field. The characteristics of MRE corresponding to magnetic properties, microstructure and thermal behaviour of the MRE will be examined.

1.3 Objectives and Scope of Study

The main objective of the research is to study the influence of PBO on the characteristic of natural rubber-based magnetorheological elastomer (NR-based MRE). In order to achieve the main objective, three sub-objectives are established as follows:

1. To observe the distribution of carbonyl iron particles in MRE through microstructure of NR-based MRE with the influence of three different PBO, consists of paraffin oil, naphthenic oil, and light mineral oil.
2. To analyse the magnetic properties of NR-based MRE using naphthenic oil.
3. To examine the thermal behaviour of NR-based MRE with naphthenic oil as the dispersing aids.

The scopes of study for the research are as below:

1. Natural rubber will be used as the carrier matrix of MRE
2. Three types of PBO are chosen as additives for fabrication of MRE which include paraffin oil, naphthenic oil, and light mineral oil
3. The characteristics of MRE which will only be examined cover magnetic properties, morphology, and thermal behaviour.
4. The thermal and morphological analyses will be measured without the presence of magnetic field.

CHAPTER 2 : LITERATURE REVIEW

MRE was first initiated by T. Shiga of Japan, in 1995 [11]. It is a smart material composite which consist of elastomer, ferromagnetic particles, dispersing aids, and other additives. MRE is a composite that are controllable via external magnetic field [12]. The ability of MRE to possess field-dependent mechanical storage and magnetic properties results in the applicability of MRE in automotive, medical, and civil industry [5][12]. The functionality of MRE that exhibit variable stiffness and damping properties is used to control noise, vibration, and also harshness.

2.1 Matrix in MRE

MRE has been fabricated using three types of material as the matrix, such as saturated elastomer (e.g. silicone rubber), unsaturated elastomer (e.g. natural rubber, synthetic rubber), and thermoset-thermoplastic elastomer (e.g. polyurethane). The matrix in MRE is a hyperelastic matrix that exhibits viscoelasticity behaviour when subjected to external loading [13]. A suitable matrix material should be selected depending on the desirable magnetic field-induced storage modulus and MR effect.

For MRE fabrication using silicone rubber, the MRE provides an increment in the dynamic stiffness, damping, and conductivity [5]. The silicon-based MRE is applied in the magnetically controlled actuators. There are various study in improving the mechanical properties of silicone-based rubber due to its difficulty to blend in the silicone matrix. The study focuses on the additives added to the silicone matrix such as silicone oil [14], methyl tri-methoxysilane [15], silane clear [16], etc. However, the silicon rubber has poor mechanical properties which is not suitable to be used for the bushing in automotive industry [5][17].

Compared to the silicon-based MRE, unsaturated elastomers have a better mechanical properties and heat resistance [5]. The tensile strength and rupture strength of MRE using natural rubber as matrix is ten times higher than using silicon rubber [17]. Synthetic rubber-based MRE has a high resistance to oil and weathering, while natural rubber has a high resistance to tearing at high temperature and good dynamic performance [17][18].

2.2 Ferromagnetic Particles in MRE

Besides the various selection of elastomers, ferromagnetic particles also assist in providing larger MR effect towards MRE. The magnetic particles could be carbonyl iron particles, nickel, cobalt, magnetite, iron sand, etc. Carbonyl iron particles are commonly used in the MRE due to the high permeability, high magnetic saturation and low remanence [5][19][20]. Moreover, the smaller size of magnetic particles will result in increment of interfacial friction among the particles. The particle size of 0 to 10 μm result in high interfacial friction between the particles thus achieve a good damping factor [5].

2.3 Dispersing Aids in MRE

Furthermore, dispersing aids or plasticizers are needed in the fabrication of MRE. Dispersing aids allows the dispersion of ferromagnetic particles within the rubber. In addition, dispersing aids enhances the chains mobility of the elastomer by improving its flexibility and deformability, through decrement of cohesive forces between the chains. The conventional dispersing aids of rubber which is commonly used is aromatic oil. For natural rubber, the compatibles dispersing aids are paraffin oil, naphthenic oil, and aromatic oil [21]. However, aromatic oil have been reported to be carcinogenic[22][23]. There are few studies conducted on the influence of dispersing aids. Hence, these studies of plasticizers on the rubber only focused more on mechanical properties [18][24][25]. The example of dispersing aids that has been explored are epoxidized palm oil, aromatic oil, light mineral oil and naphthenic oil by Aziz et al. [18]. The result showed that epoxidized palm oil has the highest performance among aromatic oil, light mineral oil, and naphthenic oil [18]. Besides that, a study of natural rubber using palm oil and paraffin oil had also been conducted to observe the morphology and mechanical properties [25].

2.4 Fabrication of MRE

The fabrication of MRE will undergoes two stages starting with mixing, and crosslinking or curing. For mixing process, rubber, ferromagnetic particles, dispersing aids and other additives are mixed together. The mixing of natural rubber to fabricate the natural rubber-based MRE is performed with additives (i.e. zinc oxide and stearic acid as activators), plasticizers (i.e. aromatic oil, PBO, etc.) and vulcanization agents (i.e. sulphur). The mixing process can be done through conventional rubber mixing using double roll mixing. After mixing process complete, crosslinking or curing process will be performed. The curing process can be in two types which is isotropic curing and anisotropic curing. Isotropic MRE is cured without an external magnetic field, while anisotropic MRE is cured in the presence of magnetic field [4]. The curing process will involve chain formation alignment between ferromagnetic particles and rubber. Figure 2.1 below shows the general procedure in fabricating MRE:

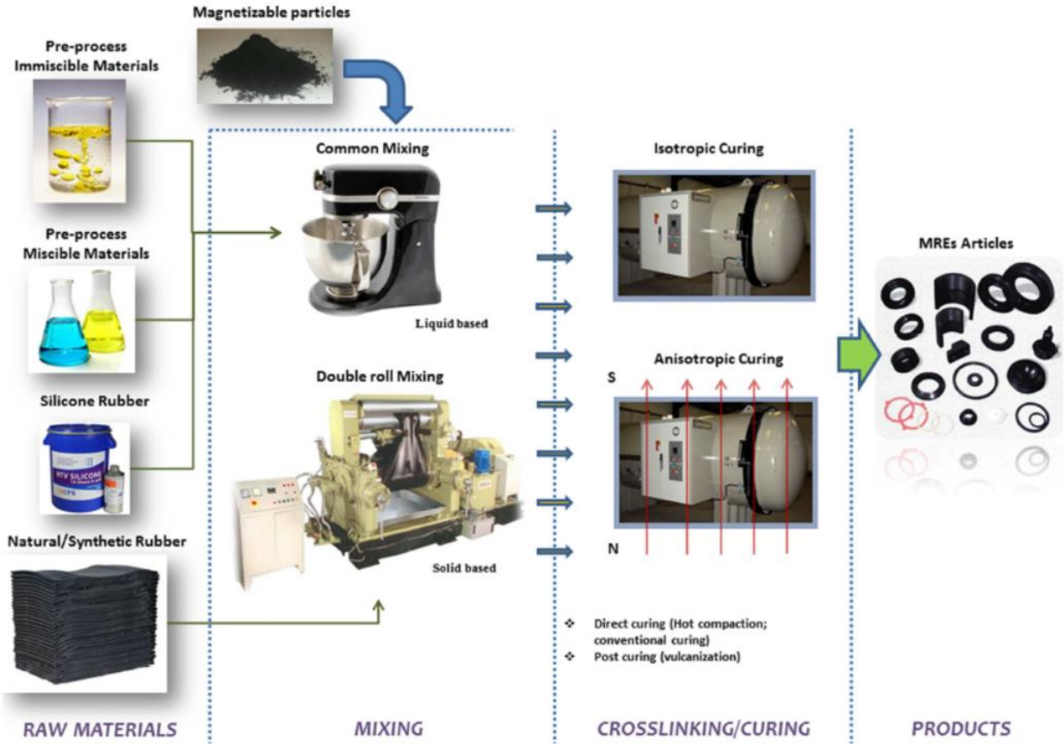


Figure 2.1 General procedure of MRE preparation [5]

CHAPTER 3 : METHODOLOGY

The methods in conducting the study are through literature review and lab experimental. This study will first start with evaluating various methods in the fabrication of MRE, by making a comparison of the methods along with its characteristic effects. Based on the comparison and knowledges, lab experiments will be conducted to identify the characteristic effects of MRE using natural rubber and carbonyl iron particles, with different types of PBO as dispersing aids. The dispersing aids in this study are naphthenic oil, paraffin oil, and light mineral oil.

The stages to fabricate MRE starts with mixing of natural rubber, carbonyl iron particles (60 wt%), with each of the three different PBO. The composition chart is shown in Table 1 for the fabrication of NR-based MRE samples, with three different types of PBO:

Table 3.1 Composition chart of MRE samples using three types of PBO

Compound Ingredients	Amount (phr)
Natural rubber	100
Carbon black	19
Zinc Oxide	5
Stearic Acid	2
Sulfur	2.3
CBS	0.8
PBO	10

The MRE will then be cured to form chain particles between natural rubber and carbonyl iron particles. The curing process will be conducted without external magnetic field, for 15 minutes at a temperature of 150ºC. The characteristics effect of MRE for these three types of PBO will then be examined to analyse the influence of PBO. Magnetic properties and thermal behaviour will be examined only for MRE using naphthenic oil. Morphology will be conducted for MRE with naphthenic oil, paraffin oil, and light mineral oil to determine the characteristic of MRE.

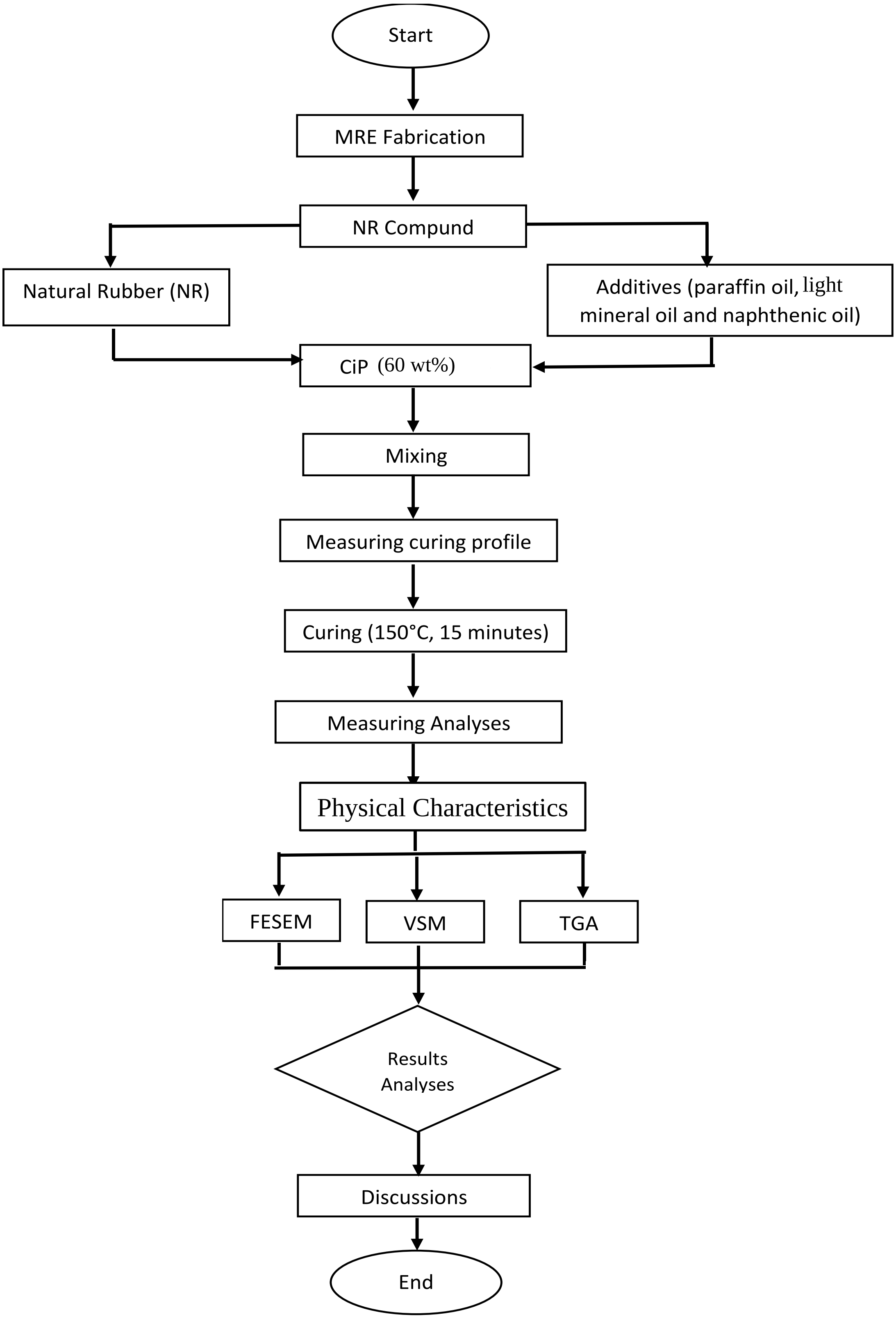


Figure 3.1 Flow of Experimental Work

3.1 Morphology

The morphology will be examined through field-emission scanning electron microscopy (FESEM) with energy dispersive X-ray (EDX) analysis; ZEISS SUPRA™ 55 VP to observe the distribution of carbonyl iron particles within MRE. The scanning electron resolution is 10 µm, with accelerating voltage of 5 kV at working distance of 4.2 mm. The sample is coated with a thin layer of gold before the morphology test to avoid charging. Maximum size of the sample is within 1 mm. FESEM images of MRE cross-section was observed for the overall micrograph, and the dispersion of CI particles in the matrix with magnification of 70x, 100x, 300x, 500x, 1000x, 3000x, and 5000x.

3.2 Magnetic Properties

Magnetic properties of the MRE will be measured using vibrating-sample magnetometer (VSM); Lake Shore 7400 Series. The magnetic properties are determined by measuring the magnetic moment of MRE against the magnetic field. VSM test is conducted by applying magnetic field in both parallel and perpendicular to the direction of the particle chains [5]. By conducting VSM test, the magnetic moment of the rubber samples can be quantified. The sample is measured under field intensity up to 8000 Gauss.

3.3 Thermal Behaviour

Thermal behaviour of the MRE will be quantify using thermal gravimetric analysis (TGA). The mass of the sample is measured over time, against the changes of temperature. From the analysis, thermal stability of MRE can be evaluate to determine its temperature range [26:262]. ASTM D6370-99R03 Standard Test Method for Rubber – Compositional Analysis by Thermogravimetry is referred [27]. A sample of MRE with naphthenic oil was placed into the platinum pan of a Thermogravimetric Analyzer. Nitrogen purge is applied as a reagent for TGA. The sample is heated with a temperature range of 30.19 ºC to 901.1 ºC. From the TGA, the percent mass loss of each components in MRE is recorded and a thermogram of weight percentage against temperature is produced.

Table 3.2 FYP I Gantt Chart

Task	Academic Week													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Study the general overview of MRE and its application	●	■												
Conduct literature review to gather information of the project title		■	■	■	■	■	■	■	■	■	■	■		
Submission of Progress Assessment 1						★								
Submission of extended proposal						■	■							
Proposal defence								★						
Submission of Progress Assessment 2											★			
Collect findings from literature review and compare each of the research paper											■	●		
Submission of Draft Interim Report												■	■	
Submission of Interim Report														★
Preparation of sample fabrication														●

● Key Milestone ★ FYP 1 Milestone

Table 3.3 FYP 2 Gantt Chart

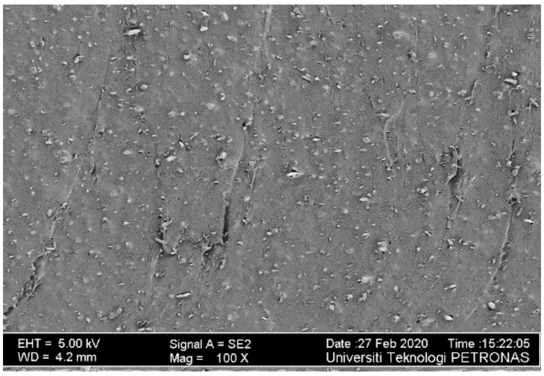
Task	Academic Week														
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Sample preparation						●									
Check lab availability			●												
Submission of Progress Assessment 1							★								
Sample Testing											●				
Data and Result Gathering												●			
Submission of Draft Final Report													●		
Submission of Dissertation (Soft Bound)														★	
Video Presentation and Viva															★
Submission of Progress Assessment 2															★

● Key Milestone ★ FYP 2 Milestone

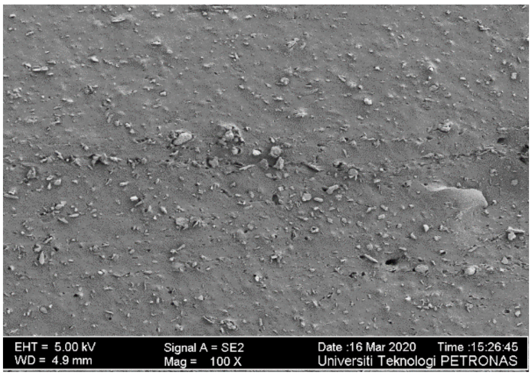
CHAPTER 4 : RESULTS AND DISCUSSION

4.1 Morphology

The microstructure for all the three samples of NR-based MRE with different types of PBO were tested through FESEM-EDX analysis. Figure 4.2 display the FESEM images of NR-based MRE using three different PBO at magnification of 100x. All of the sample exhibit random dispersion of carbonyl iron particles within the matrix. The MREs depict isotropic types MRE as the curing process of the sample was conducted without magnetic field. By curing without an external magnetic field, none of chain formation alignment between ferromagnetic particles and rubber is exist. Therefore, carbonyl iron particles is randomly disperse within the rubber matrix.



(a)



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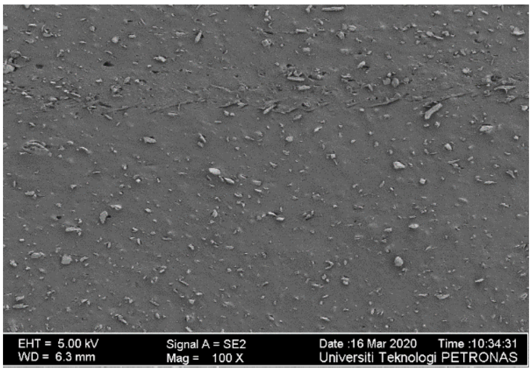
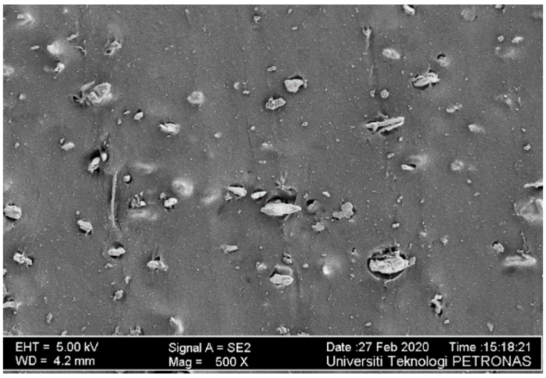


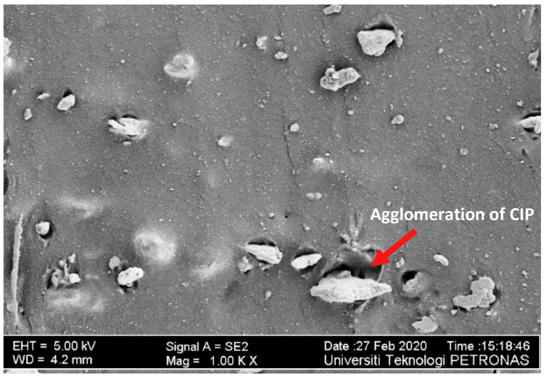
(c)

Figure 4.1 FESEM images of MRE using (a) naphthenic oil, (b) paraffin oil, and (c) light mineral oil at 100x magnification

Figure 4.3 shows all of the samples at 500x and 1000x magnification to observe the effect of different types of PBO on the dispersion of carbonyl iron particles within the matrix. Small agglomeration of carbonyl iron particles are seen on all of the samples. The sample with light mineral oil as PBO has the least agglomeration shown in Figure 4.3 (c) and Figure 4.3 (f). The sample also depicts homogenous dispersion of carbonyl iron particles within the rubber matrix in Figure 4.2 (c). The compatibility of PBO plays an important role to lubricate and enhance the dispersion of carbonyl iron particles in the matrix. Hence, light mineral oil exhibits good performance in improving the dispersion of magnetic particles in the MRE. Meanwhile, MRE using naphthenic oil and paraffin oil display in Figure 4.3 (a), (b), (d), and (e) show few agglomerations in the distribution of carbonyl iron particles particles within the rubber matrix.



(a)



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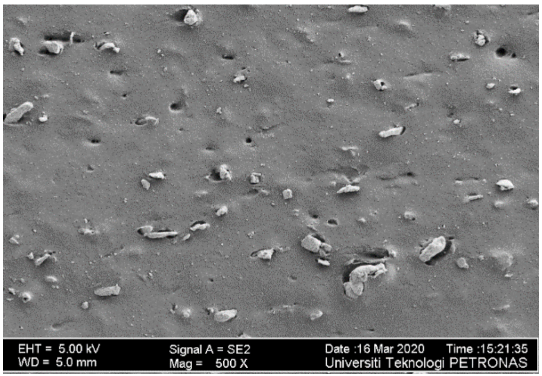

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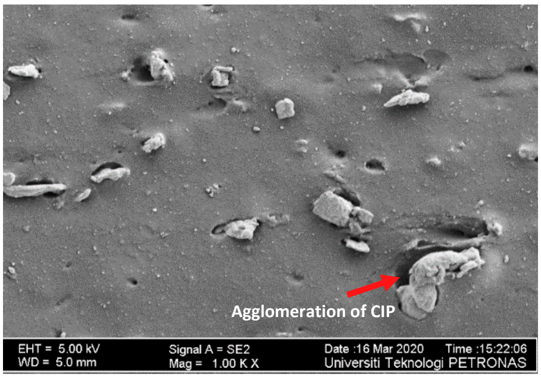

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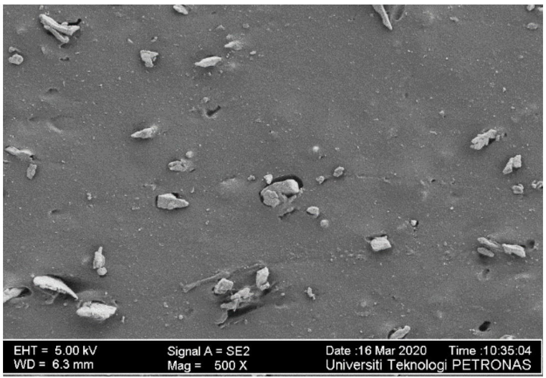

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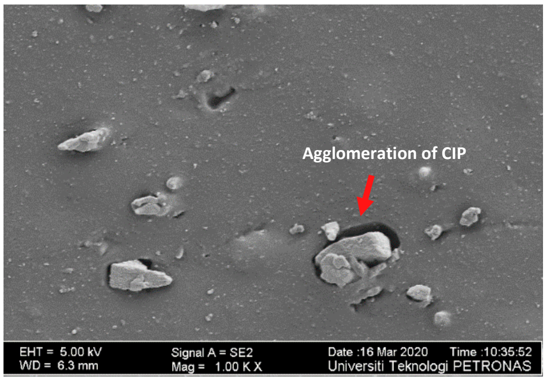

(f)

Figure 4.2 FESEM images of MRE using (a) naphthenic oil, (b) paraffin oil, (c) light mineral oil at 500x magnification; (d) naphthenic oil, (e) paraffin oil, (f) light mineral oil at 1000x magnification

Table 4.2 tabulated EDX results obtained from the FESEM images for all three samples. The samples comprise five elements consist of carbon (C), oxygen (O), sulfur (S), iron (Fe), and zinc (Zn) due to addition of additives for MRE fabrication. The existence of carbonyl iron particles is presented in the form of iron (Fe). All of the samples comprise of 60 wt% of carbonyl iron particles. Hence, Table 4.2 depicts Fe element in the range of 10 – 13 wt%.

Table 4.1 Elements percentage in the MRE

Samples	**Element**	**Weight (%)**
MRE (Naphthenic Oil)	C	80.92
	O	2.53
	S	2.27
	Fe	11.70
	Zn	2.58
MRE (Paraffin Oil)	C	83.11
	O	2.72
	S	1.71
	Fe	10.29
	Zn	2.17
MRE (Light Mineral Oil)	C	79.01
	O	4.08
	S	1.98
	Fe	12.81
	Zn	2.11

4.2 Magnetic Properties

Magnetic properties were determined using VSM for NR-based MRE using naphthenic oil. Figure 4.1 below shows the result of magnetic moment against field intensity which form hysteresis loop depending on the soft magnetic characteristics of the sample. The sample were tested from -8000 Gauss up to 8000 Gauss. Furthermore, the magnetic properties of NR-based MRE using naphthenic oil measured at room temperature are depicted in Table 4.1.

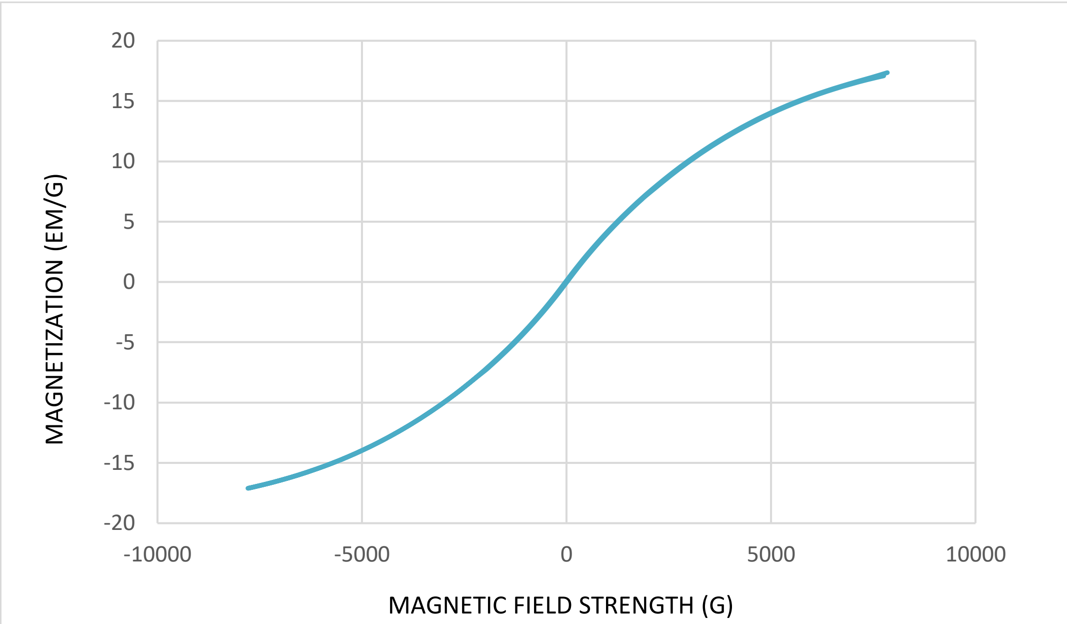

Figure 4.3 Magnetization curves of MRE using naphthenic oil

Table 4.2 Magnetic properties of MRE using naphthenic oil

Sample	M_s (emu/g)	M_r (emu/g)	H_c (G)
MRE (Naphthenic Oil)	17.23	0.075377	16.776

As the magnetic field strength applied increases, the magnetic particles within the matrix tend to align until perfect alignment reaches and maximum magnetization produced. Hence, the sample reached a magnetic saturation of 17.23 emu/g as depicted from Table 4.1 whereas increases in magnetic field strength will bring little or no effect onto the magnetization (M_s) of magnetic particles. Meanwhile, the value of retentivity magnetization, M_r of the sample is relatively low. The MRE sample has low retentivity magnetization as the magnetic particles within the matrix is soft magnetic materials. When the applied magnetic field strength was removed, the magnetic particles easily looses its magnetization and demagnetized. Furthermore, the coercive force, H_c of the sample is 16.776 emu/g. During demagnetization of magnetic particles, negative magnetic field strength was applied to reduce the magnetization to zero, known as coercive force. The sample depicts high coercive force in order to reduce the magnetization. The results had also reported from previous research by Aziz et al., whereas NR-based MRE using naphthenic oil has lower retentivity magnetization and higher coercive force [18].

4.3 Thermal Behaviour

The thermal behaviour of NR-based MRE using naphthenic oil was tested via TGA in a nitrogen atmosphere. Figure 4.4 illustrate the thermal gravimetric (TG) curve of the MRE. At the initial decomposition, a slight weight gain is observed from the TG curve. This might be either due to oxidation reaction of the surface material with the gaseous contaminants within the purge nitrogen gas or due to buoyancy effect caused by the density of the atmosphere in the balance decrease with increasing temperature. It is apparent that there are two stage of thermal decomposition of the MRE. The first stage began at the temperature of 256.53 °C while second stage started at 336.24 °C and ended at the temperature of 481.21 °C. The first thermal decomposition was attributed to the decomposition of volatile additives in MRE compound. The second stage of thermal decomposition was due to the decomposition of rubber matrix and contributed to higher weight loss of about 68.45 wt% as compared to the first stage. With the help of PBO in improving the chain formation between natural rubber and carbonyl iron particles, the carbonyl iron particles were randomly distributed as referred to Figure 4.1 (a) and decrease the permeability of heat to pass through the MRE. Therefore, a longer time is required for the thermal decomposition of MRE which led to improvement of its thermal stability.

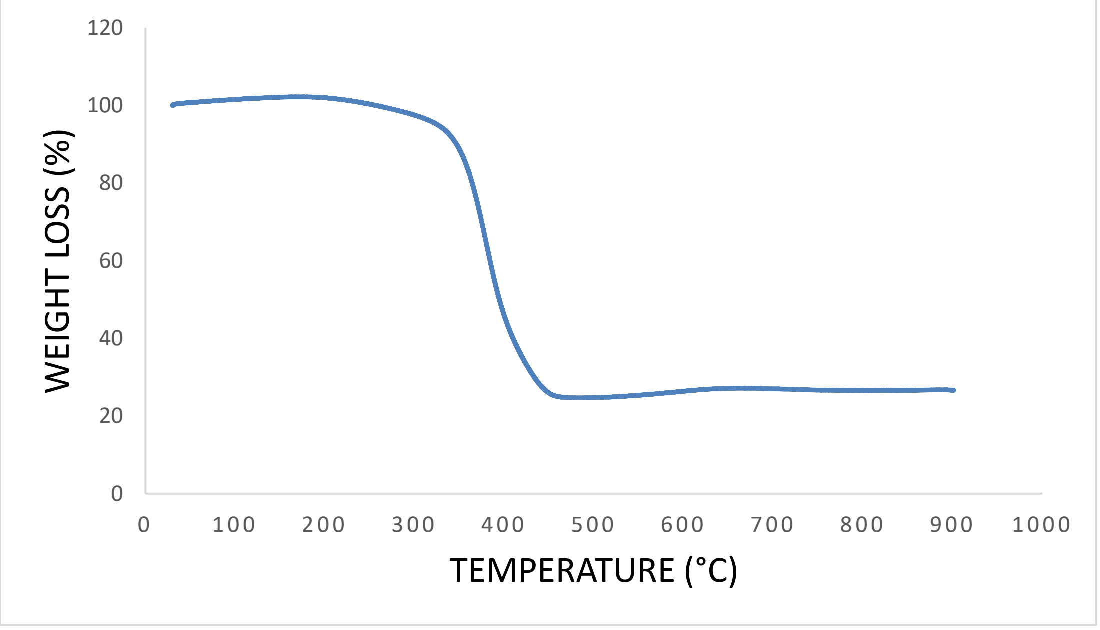

Figure 4.4 TG curve of MR with naphthenic oil

CHAPTER 5 : CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The influence of different PBO on the characteristics of NR-based MRE were studied by analysing its magnetic properties, morphology, and thermal behaviour. The morphology of the MREs depict random dispersion of carbonyl iron particles within the matrix, known as isotropic MRE. MRE with light mineral oil indicates least agglomeration and homogenous dispersion of carbonyl iron particles inside the rubber matrix. The magnetic properties of MRE with naphthenic oil provide low retentivity magnetization at 0.075 emu/g, which result in fast demagnetization of carbonyl iron particles within the matrix . Hence, high saturation magnetization and low retentivity of MRE is desirable for MRE applications in terms of intelligent vibration and noise reduction. The thermal stability of MRE with naphthenic oil was improved with the aid of PBO in improving the distribution of carbonyl iron particles within the rubber matrix. The random distribution of carbonyl iron particles contributed to decrement in the permeability of heat to pass through the MRE and delays the thermal decomposition process thus increase the thermal stability of MRE.

5.2 Recommendations

The study revealed the importance of PBO on the characteristics of MRE, which acts as lubricant in improving the dispersion of carbonyl iron particles within the matrix. Therefore, a comprehensive studies on the magnetic properties and thermal behaviour of MRE using paraffin oil and light mineral oil should be further conducted to enhance the characteristics of NR-based MRE.

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