THE EFFECT OF WEIGHT PERCENTAGES OF CARBONYL IRON PARTICLES (CIP) ON THE PHYSICOCHEMICAL CHARACTERISTICS OF EPOXIDIZED NATURAL RUBBER-50 (ENR-50) BASED MAGNETORHEOLOGICAL ELASTOMERS (MRE)

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MECHANICAL ENGINEERING
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by

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24723

Dissertation submitted in partial fulfilment

of the requirements for the

Bachelor of Mechanical Engineering

with Honours

JANUARY 2020

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the Mechanical Engineering Programme

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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.

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AJIT SINGH A/L AMARPALL SINGH

ABSTRACT

Magnetorheological materials are a type of smart material whose rheological properties can be manipulated within the presence of magnetic field. Magnetorheological Elastomers (MRE) and MR Fluids (MRF) are examples of materials under this class. Recently, MRF has been found to be not as reliable as expected with issues of fluid sedimentation, leakage, oxidation, and hard cake effect. This led to the re-emergence of MRE which does not possess the issues faced in MRF. MRE is a type of smart composite material comprised of an elastomer-based matrix, magnetic particles, and additives. To make MRE a viable alternative to MRF, MRE used solid matrix elastomer which can prevent all the issues that faced by MRF and the properties of magnetic particles such as low remnant, and high saturation magnetization make the MR effect in the rheological part is high. In order to possess those qualities, the magnetic particles must disperse efficiently throughout the matrix of the MRE. This research studied the effects of different Carbonyl Iron Particles (CIP) content in weight percentages of an ENR-50 matrix on its physicochemical properties. Samples underwent FESEM imaging and VSM testing. Analysis of VSM data showed an increase in CIP within the matrix increase the magnetic properties of MRE. However, an increase of CIP within the matrix will affect the microstructure which may result in a reduced elasticity and ductility of MRE. Microstructure analysis of FESEM imaging also showed that a higher CIP content will increase the packing density between CIP and matrix due to reduced spaces between one CIP and another. Magnetic saturation and remnant magnetisation values increased for sample which had 70 wt% CIP compared to 30 wt% CIP but coercivity values decreased with increase of CIP content. Overall, it was found that increasing CIP content will increase the magnetic properties but only up to a point. At 70 wt%, the CIP starts affecting the physical structure of the MRE due to high agglomeration and reduced mechanical properties. Further research should be done to study the rheological behaviour of CIP content on ENR-50 based MRE.

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1. CHAPTER 1: INTRODUCTION

1.1. Background of Study

Magnetorheological Elastomer (MRE) is part of the Magnetorheological (MR) family. MRE is a kind of smart composite material where some basic properties including mechanical, electrical, and magnetic properties are adjustable by external magnetic field. This was first discovered by Thom Rabinow in the year 1948. Since then, the MRF material has been innovated and became commercially viable. The MR materials currently exist include MR Fluid, Elastomers, Foams and Gels. The basic principle of these smart materials is that its rheological properties can be controlled continuously, rapidly, and reversibly through the stimulation of a magnetic field. These materials typically consist of micron-sized particles which are suspended in a non-magnetic matrix.

MR fluids is the most common of the other MR materials in terms of commercial usage. MRF are the commonly used of smart materials that consists of ferromagnetic particles that suspended randomly in a liquid carrier such as hydrocarbon oil. When it is supplied with magnetic field, the viscoelastic properties of the material increase and can be controlled by varying the intensity of the magnetic field. Although MR fluid seems to be the best MR material, it has some limitations that has stunted its growth in commercial feasibility. For example, one study mentioned that a prosthetic knee joint was developed with MR fluid to last for 3,000,000 cycles, however joint stiffness increased due to poor long term effectiveness of MR fluid until eventually the part needed to be replaced after just 1,000,000 cycles (Utami, et al., 2018). Hard cake phenomenon in MR fluid is when the magnetic particles clump together which do not re-disperse due to it still holding on to remnant magnetisation between particles. Besides that, the ferromagnetic particle sediment rate (Settling rate) is also a concern for commercial applications. Other issues include the magnetic particles oxidation as well as leaking of MR fluid over time which will eventually require frequent replacement of the fluids (Sunkara, Root, Ulicny, &

Klingenberg, 2009). The concerns in the stability and durability of the MR fluid has caused the re-emergence of the MRE which can tackle those problems. MRE consists of three basic components: elastomer-based matrix, magnetic particles, and additives. Some types of elastomer matrices which have been used include Natural Rubber (NR), Epoxidized Natural Rubber (ENR), Silicone Rubber (SiR) and Polydimethylsiloxane (PDMS) elastomer (Liu & Xu, 2019).

ENR has been the go-to choice as a matrix for previous researches because it possesses enhanced qualities of an NR such as higher oil resistance, increased glass transition temperature (T_g), improved damping and lower gas permeability (Hoon, 2006). ENR is an enhanced version of NR through chemical modification. It is commercially available in 25% (ENR-25) and 50% (ENR-50) mole epoxidation levels.

For a composite to be categorised as an MRE, it has to have these specific components, an elastomer matrix, magnetic particles, and additives. However, the magnetic particles within the matrix is what gives an MRE the ability to alter its material properties when under a magnetic field. In fabrication of MRE, the preferred properties of the magnetic particles are high permeability, low remnant, and high saturation magnetisation as it will influence the performance of MRE investigation. Various studies have shown the great influence of magnetic particles on MR effect which just goes to show that there is a direct correlation between the role of ferromagnetic particles on the performance of MRE (Yu, Ju, Fu, Liu, & Yang, 2012), (Seidabdi, Mehranpour, & Ohadi, 2016), (Yunus, et al., 2016).

1.2. Problem Statement

As mentioned above, the reliability of MR fluids has reduced due to several factors. Firstly, the absence of certain additives caused MR fluids to sediment and cause hard cake formation. (Wahid, Ismail, Aid, & Abdul Rahim) This causes the magnetic particles harder to re-disperse due to strong aggregation. On top of that, the particles also do not completely dissipate when magnetic field has been deactivated due to the low remnant magnetization. Temporary solutions have been found, such as the addition of silica nanoparticles as surfactant to avoid particle settling. However, the silica gel particles are rapidly broken down on-state which allows for iron particles to settle down and sediment, again, causing hard cake phenomenon.

Notwithstanding the fact that the MR fluid also must be replaced from time to time due to particles oxidation and fluid leakage. Due to the reasons mentioned above, issues such as hard cake sedimentation may occur in MR Fluids. That will affect the MR Effect, which is the most important trait of any MR material. Therefore, MR Fluids are considered to be not commercially feasible.

Material engineers and researchers then started to find alternatives which could replace this material and eventually Magnetorheological Elastomers (MRE) was developed. MRE does not has the same issue with MR Fluid as it is a solid analogue of MR fluid. Furthermore, MRE could be based on various base solid elastomers such as Natural Rubber (NR), Silicone Rubber (SiR) or Epoxidized Natural Rubber (ENR).

As part of the efforts of the Malaysian Rubber Board (MRB) or locally known as Lembaga Getah Malaysia (LGM) to assist the local rubber industry, they have spurred a sustainable and non-petroleum based resource that has a lower carbon footprint than synthetic rubbers such as Silicone Rubber.

ENR is basically a chemically modified version of NR. This product by MRB, known as EKOPRENA, is Epoxidized Natural Rubber (ENR) which is commercially produced at an epoxidized mole percentage of 25% and 50%, named ENR-25 and ENR-50 respectively (Malaysian Rubber Board, 2019). ENR is usually preferred to NR due to its better heat and chemical resistance especially in environments which

require improved heat and oil resistance or a lower gas permeability (Polymer Database, 2015). ENR is also more preferred compared to silicone rubber due to its matrix is a chemically modified enhanced version of NR. This means the ENR properties possess enhanced properties of NR without its weakness, whereas silicone rubber is an entirely different compound due to it being a synthetic material.

There has been many researches done by various organizations on the effects of CIP on matrixes such as NR, SiR and ENR-25 (Yu, Ju, Fu, Liu, & Yang, 2012), (Seidabdi, Mehranpour, & Ohadi, 2016), (Yunus, et al., 2016). However, there has not been a comprehensive study on the effect of CIP on the performance of an ENR-50 based MRE. Thus, the purpose of this study is to investigate the effect of various weight percentages of CIP on the physicochemical properties of the ENR-50 based MRE.

1.3. Objectives & Scope of Study

1.3.1. Objectives

The main objective of this research is to study the physicochemical properties of MRE with different weight percentages of Carbonyl Iron Particles (CIP). The two sub-objectives stated below will assist in achieving the main objective:

(a) To fabricate an ENR-based MRE with different weight percentages of CIP.
(b) To analyse the effect of different weight percentages of CIP on the physicochemical properties of the ENR-50 based MRE with respect to particle distribution and magnetic properties.

1.3.2. Scope of Study

The scope of study for this research will be focused on the following aspects:

This research will focus on the smart composites known as MRE. The type of rubber used as matrix will be Epoxidized-Natural Rubber (ENR), specifically ENR-50. The type of MRE composite structure will be isotropic. The properties which will be studied in this study is the effect of different weight percentages of CIP on the physicochemical properties of ENR-50.

Physicochemical Analysis of the MRE performance will be evaluated on three aspects, namely the Microstructural Analysis and Magnetic Properties Analysis. The Microstructural Assessment of samples is expected to show the inner structure and dispersion of CIP within ENR-50 matrix. This will be conducted with a Field Emission Scanning Electron Microscope (FESEM). Second aspect of a physicochemical analysis, the Magnetic Properties Analysis is the most important of all. This test will be conducted using a Vibrating Sample Magnetometer (VSM). This test will indicate various magnetic properties such as Saturated Magnetization (M_s), magnetic retentivity (M_r) and coercivity (H_c).

2. CHAPTER 2: LITERATURE REVIEW

2.1. Introduction to MRE

MRE belongs to a class of materials commonly referred to as Smart Composites. They are referred to as that because smart composites possess the ability to change their material properties when under influence of an external stimuli (Brancati, Massa, & Pagano, 2019). These kinds of smart materials can be implemented in devices or incorporated in existing composites to form smart composites, who's the rheological properties can be altered by magnetic field. The working principle of MR materials is that it has magnetically polarisable colloidal particles suspended in a fluid suspension or elastomer matrix. The magnetic particles will be randomly distributed in the matrix when magnetic field is not supplied, but when magnetic field is supplied, it will magnetize and form chain like structure aligned with the magnetic field. MRE is just one of few types of MR materials such as MR Fluids (MRF) and MR Gels. MRF can be considered as the predecessor to MRE. MRF fluids are the most commonly found form of MR materials and the most popular. MRF is a type of ferrofluid which has iron particles which are suspended in a carrier fluid, usually a type of oil. When exposed to a magnetic field, the particles line up and thickens immediately. MRF technology has been applied in various fields of engineering such as dampers valves and brakes.

2.2. Re-emergence of MRE

Recent developments in the MRF field of study has raised some questions into the reliability of this material due to its defects. Issues such as particle deposition, sedimentation, sealing of fluid as well as environmental contamination has discouraged many from utilising this material in vibration control mechanisms (Li, Li, Li, & Du, 2014). One of the MR researchers find that MRF has to face serious issues such as hard cake, clumping effects, fluid particle separation as well as oxidation of ferromagnetic particles within the fluid. Moreover, MRF stability is also questioned

because of the particle sedimentation and settling rates (Wahid, Ismail, Aid, & Abdul Rahim).

Due to those reasons, the MRF usage has declined recently, which led to the recent spike of interest in MRE. MRE is the solid-state analogue of MRF (Chen L. , et al., 2007). These problems faced in MRF such as particle sedimentation and fluid sealing can be overcome by replacing the fluid matrix with a solid matrix such as an elastomer (rubber). Several studies have observed the MRE is quite analogous to MRF with the main difference being that MRE has controllable, field dependent shear modulus while MRF has a field dependant yield stress (Kallio, 2005).

2.3. Isotropic & Anisotropic MRE

MRE can be categorised into two types, namely isotropic and anisotropic structures. Isotropic structures have similar properties in all directions due to its randomly distributed particles which gives it a homogenous structure. An isotropic MRE does not have magnetic microparticle alignment due to no magnetic field applied to the composite during the crosslinking process. Anisotropic structures however have different properties in varying crystallographic orientation which means it is directionally dependant. Which means, the anisotropic material property may vary according to different direction.

Anisotropic structure is produced by applying a controlled magnetic field during curing phase, where the particles are cured and lock in make sure the magnetic particles looks like a chain-like structure. This will allow the anisotropic structure of MRE to have magnetic microparticle alignment parallel to magnetic field (Gong, Zhang, & Zhang, 2005). The figure below shows the schematic microstructure of the isotropic and anisotropic microstructures.

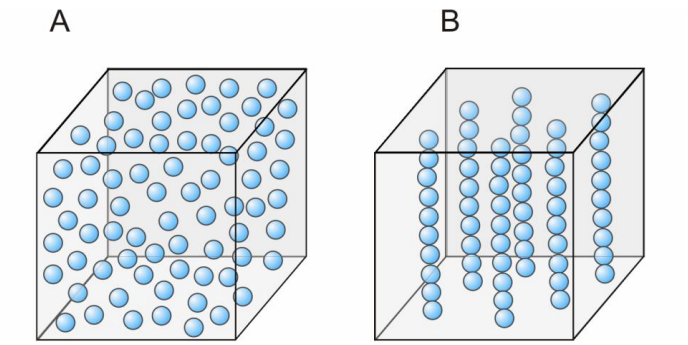

Figure 1: Schematic Microstructure of: (A) Isotropic structure & (B) Anisotropic Structure (Boczkowska & Awietjan, 2012)

2.4. Components in MRE

2.4.1. MRE Matrix Types

Usually when the word elastomer comes to mind, natural rubber, silicon rubber and polyurethane rubber are the material associated with it. However, recently there is a more commercially available type of rubber which is known as Epoxidized Natural Rubber (ENR) which is becoming more popular from them. Reason being that Natural Rubber (NR) applications are limited due to its low resistance to heat and Ultraviolet (UV) radiation (Polymer Database, 2015). Moreover, NR is highly soluble in various solvents and oils. Thus, ENR is introduced due to its more stable structure due to introduction of hydrophilic groups by epoxidation. The hydrophilic group is introduced is peracid (Peroxy Acid), which is formed by the reaction of acetic acid with hydrogen peroxide. This will result in a NR with dispersed epoxide groups along the polymers backbone.

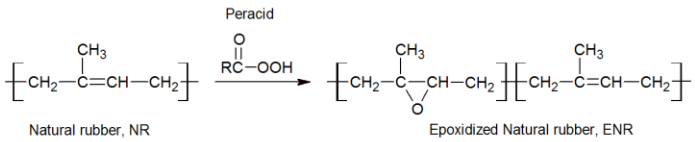

Figure 2: Treatment of NR with Peracid to Form ENR (Polymer Database, 2015).

ENR has been established and found commercial applications in wide range of areas such as applications in acoustic damping devices (Gelling, 1996). Other available forms of matrixes for MRE besides NR and ENR is Silicone Rubber (SiR) which is a form of synthetic elastomer.

2.4.2. Fabrication of MRE

The general fabrication of an MRE involves three basic components: magnetic particles, elastomer as matrix and additives. In mixing stage, the elastomer is placed in-between two rolls rotating opposite directions with different speeds. In this stage the matrix is subjected to a strong extrusion pressure and shear force which causes molecular chains to be broken down. This causes the material to lose its elasticity and begins to become a viscous body gradually. At this stage, crosslinkers and plasticizers can be easily added to matrix (Chen L. , et al., 2007). Ferromagnetic crosslinkers such as Carbonyl Iron Particles (CIP) function as a 3-dimensional crosslink network which will link with matrix to remain its original state when external mechanical loading is applied and proportional to magnetic field intensity (Kallio, 2005). The second stage is the pre-configuration stage (only to achieve a anisotropic structure) where the heating system and magnetic field is turned on so temperature and external magnetic flux density can be set properly. At this point, the particles are magnetised and form chains along the magnetic field. Once it is done, the magnetic field is shut down and the final stage of sulfuration (curing process) can begin. Temperature is raised to above 120˚C and material will undergo sulfuration for specified time (Abd Wahab, et al., 2016).

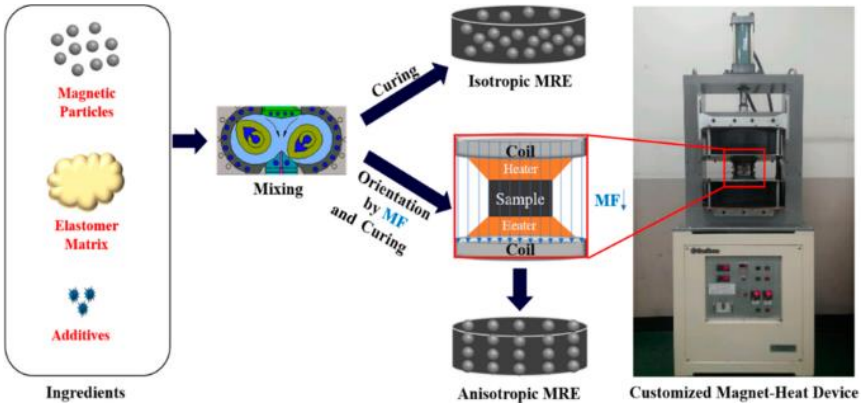

Figure 3: Isotropic and Anisotropic Fabrication Process (Kwon, Lee, & Choi, 2018).

2.4.3. Dispersing Aids Influence in MRE

A good magnetic particle-elastomer interaction will allow an additional crosslink in the network which will inadvertently influence the dynamic response of material. The great dispersion of magnetic particles within the smart composite, the better the MR effect of MRE (Abdul Aziz, et al., 2018).The common types of dispersing aids additives used are Petroleum Based Oil (PBO) and Silicon Based Oil (SO). A comparative study on different dispersing aids in MRE observed that dispersing aids helps to enhance chain mobility by lowering cohesive forces between elastomer chains.

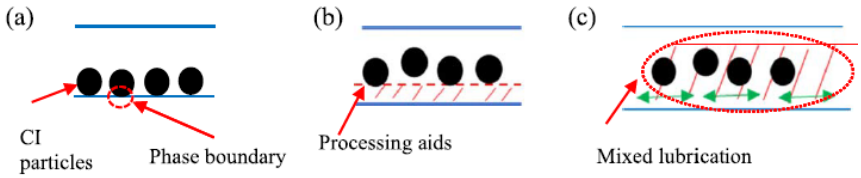

Figure 4: Mechanism of CIP Dispersion with & without Dispersing Aids. (Abdul Aziz, et al., 2018)

Some types of PBO include paraffin, naphthenic and aromatic oils (AO). Generally, AO provide best processability but may have harmful effects on staining, colour stability, ageing resistance and also. AO also has cytogenic which can cause cancer. AO is also known to be the most common processing oil in the rubber industry (Rahmah, Wan Zain Norazira, Shafie Nur Ashyikin, & Mohd Nurazzi Norizan, 2013). Another research also investigated the effect of using an alternative plasticiser, sucrose

acetate isobutyrate (SAIB) to study its effect on the MRE performance (Ahmad Khairi, et al., 2016). The usage of SAIB increased the MR effect of sample from 10% to 37% when SAIB content was increased from 0 wt% to 10 wt%.

2.4.4. Magnetic Particles Influence in MRE

Magnetic particles within the MRE matrix is assumed as the most important element of an MRE because it is the magnetic properties of the ferromagnetic particles which allows the altering of the physical and mechanical properties of the matrix, thus becoming an MRE. Carbonyl Iron Particles (CIP) are the most widely used ferromagnetic materials in MRE but there is some research which also used iron sand as fillers instead. CIP is used widely as magnetic filler in MRE is due to its high permeability, high magnetic saturation and low magnetic remanence which inadvertently increases the materials MR effect when introduced to an MRE (Abdul Aziz, et al., 2018). The high permeability will easily attract magnetic leakages within field and cause maximum induction of MR effect. A low remnant magnetization however is good because the particles will not stick together and will return to place after demagnetisation. That helps in making this a reversible MR effect possible (Cantera, Behrooz, Gibson, & Gordaninejad, 2017).

A study on the influence of CIP on the MR effect and damping properties of an isotropic Silicone Rubber (SR) based MRE concluded that CIP plays a vital role in influencing the shear storage modulus, damping property (loss factor) and MR effect under magnetic field (Yu, Ju, Fu, Liu, & Yang, 2012). Another study which focused on the comparison of isotropic and structured (anisotropic) Natural Rubber (NR) based MRE with different particle size of CIP indicated that the presence of CIP even though in varying sizes does affect the damping properties of MRE sample (Seidabdi, Mehranpour, & Ohadi, 2016). Additionally, another research on the effect of different CIP weight percentages on commercially available ENR-25 concluded that CIP was identified as the most important parameter which affects the rheological properties of an MRE (Yunus, et al., 2016).

The literature review done shows that in previous research, there has been no studies on the impact of different weight percentages of CIP on the performance of ENR-50 based MRE. This will be the novelty and prime focus of this study.

3. CHAPTER 3: METHODOLOGY & PROJECT WORK

3.1. Introduction

Methodology will be divided into two sections, namely the MRE Fabrication and the Physicochemical testing methodology of specimen. In MRE Fabrication, the fabrication formula will be explained, and the flowchart of the project will be shown. In the physicochemical testing, the material physicochemical testing will be described in detailed. The types of equipment and tests to be done will be described. At the end, the Gantt Chart will be attached to show work progress with key milestones and Final Year Project (FYP) milestones.

3.2. MRE Fabrication

The fabrication of the MRE sample with varying weight percentages of CIP will be fabricated using conventional double roll mill machine. The formula of fabrication and the research flowchart is shown on the following pages:

Table 1: Formulation of ENR-50 Based MRE for Varying CIP Content

Compounding Ingredients	**Batch Weights**		
	0 wt% CIP	**30 wt% CIP**	**70 wt% CIP**
	Amount in phr (parts per hundred rubber)		
ENR-25 (phr)	100	100	100
Carbon Black (phr)	19	19	19
Aromatic Oil (phr)	5	5	5
Zinc Oxide (phr)	5	5	5
Stearic Acid (phr)	2	2	2
Sulphur (phr)	2.3	2.3	2.3
CBS (phr)	0.8	0.8	0.8
Calcium Stearic (phr)	5	5	5
CIP (wt%)	0	30	70
MRE Structure Type	Isotropic	Isotropic	Isotropic

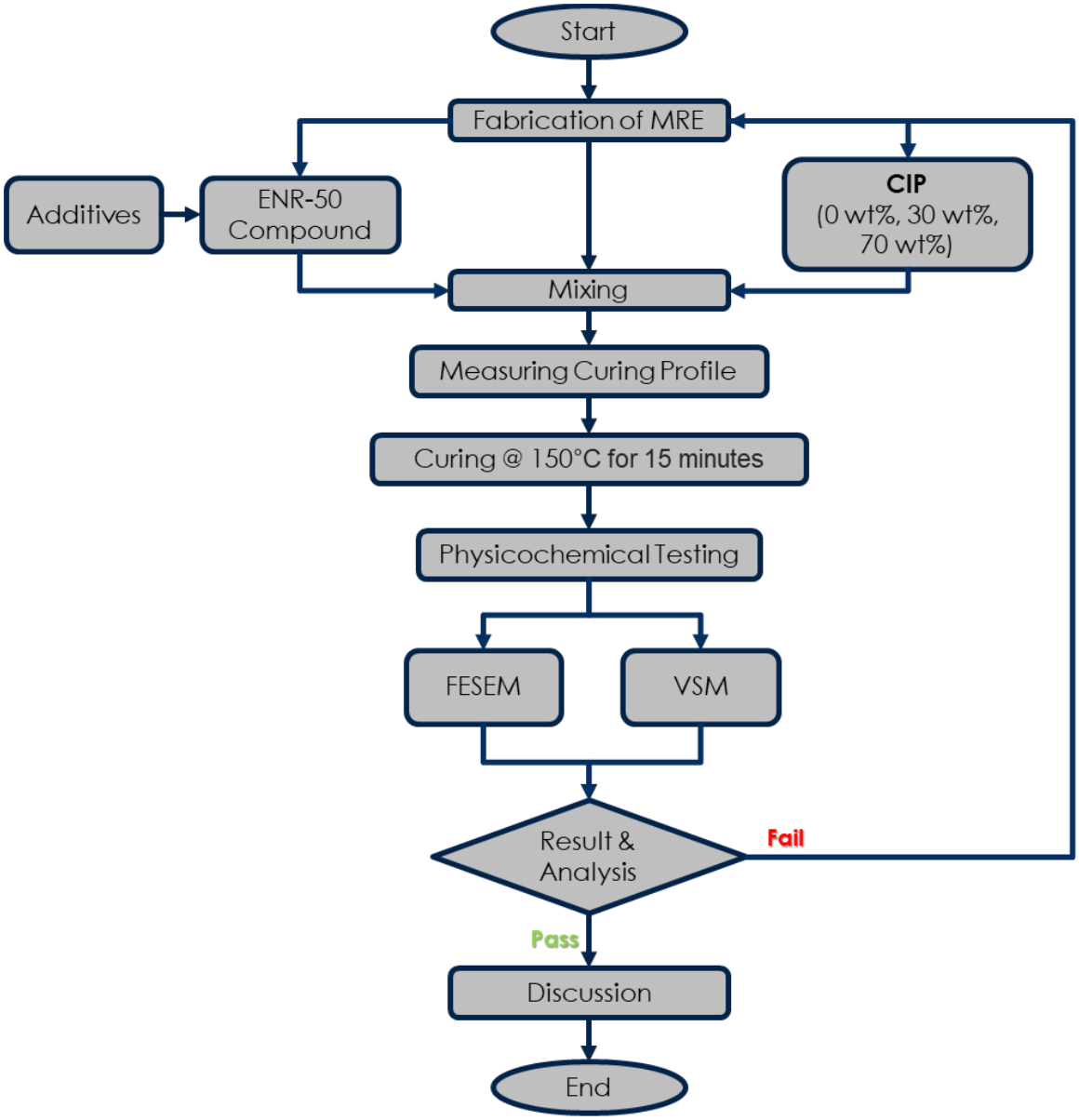

Figure 5: Flowchart of Research Procedures

3.3. MRE Physicochemical Testing

The methodology in this study will focus on the analysis and testing methods on the physicochemical properties of MRE with different weight percentages of CIP in the MRE smart composite. Physicochemical Testing will consist of multiple analysis such as the microstructural analysis and magnetic properties analysis.

Microstructural analysis will allow the observation and assessment of the sample's inner microstructure and dispersion of CIP within its matrix. This testing will be done with a Field Emission Scanning Electron Microscope (FESEM). The FESEM used is located in the Centralised Analytical Laboratory (CAL) of Universiti Teknologi PETRONAS (UTP).

Figure 6: FESEM Model Used: Zeiss Supra 55 VP (Universiti Teknologi PETRONAS, n.d.).

Finally, the magnetic property analysis, which is the most important aspect when analysing an MRE. The measurement of magnetic properties of the MRE will be conducted with a Vibrating Sample Magnetometer (VSM). Through this testing, the magnetic saturation, retentivity and coercivity of the CIP within ENR-50 matrix can be identified. The magnetic moment (M) vs magnetic field intensity (H) data obtained via the VSM test can be used to determine crucial magnetic properties for an MRE smart composite such as coercivity (H_c), retentivity (M_r) and saturated magnetisation (M_s). The VSM testing will was conducted at room temperature for all samples under a magnetic field of up to 0.8 Tesla or 8000 Gauss.

The model of VSM used is *Lake Shore 7400 Series VSM System*. This VSM is located in the Nanotechnology & Catalysis Research Centre (NANOCAT) at the Institute of Advanced Studies, University of Malaya, Kuala Lumpur.

Figure 7: VSM Model: Lake Shore 7400 Series VSM System (University of Malaya, n.d.).

3.4. Gantt Chart

Table 2: FYP I Gantt Chart

FYP I - September 2019 Semester														
Task	**Academic Week**													
	1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**
Weekly Meetings with FYP Supervisor to update on progress.	◎													
Data Collection & Literature Review				◎										
Submission of Draft Literature Review			◎											
Extended Proposal Report Submission & Progress Assessment 1						▟								
Proposal Defence Mock Presentation with FYP Supervisor														
Proposal Defence								▟						
Progress Assessment 2 Submission									▟					
Preparation & Submission of Draft Interim Report FYP I											◎			
Submission of Final Interim Report FYP I														▟

Table 3: FYP II Gantt Chart

FYP II - January 2020 Semester														
Task	**Academic Week**													
	1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**
Continuous Literature Review to Assist Analysis						◎								
Fabrication & Collection of MRE Samples from MRB.						◎								
Conduct Physicochemical Testing of Samples									◎					
Submission of Progress Report 1							▟							
Analysis of Data from Physicochemical Testing										◎				
Submission of Progress Report 2														▟
Submission of Draft Dissertation														▟
Viva Session														▟
Final Submission of Project Dissertation														▟

◎ Key Milestones

▟ FYP Milestones

4. CHAPTER 4: RESULTS & DISCUSSION

There were three samples of ENR-50 based MRE sent for physicochemical testing. The samples vary in weight percent content of CIP of 0 wt%, 30 wt% and 70 wt%.

4.1. Microstructural Analysis of FESEM Imaging

FESEM imaging was done at various magnifications from 70x up to 10,000x. For result and discussion purposes the clearest magnification. This is to allow for best visibility of microstructure analysis of each sample will be selected to allow more thorough.

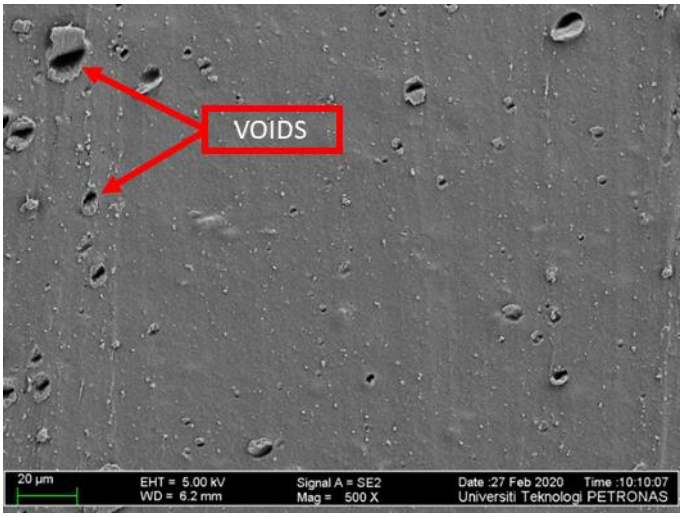

Figure 8: Microstructure of ENR-50 with 0 wt % CIP.

Figure 7 shows the result of FESEM imaging on ENR-50 sample with 0 wt% CIP content. From initial observation of the microstructure, most of the surface of sample seems to be a dark surface which is something attributed to the effect of carbon black on the colour of ENR matrix (Shuib & Pickering, 2016). There are no visible metal particles within the current matrix which is in line with the fabrication of 0 wt% of CIP. However, there is some void within the matrix as observed in the darker spots. This voids could be porosity could be due to a chemical reaction between additives and the ENR or due to inhomogeneous mixture during mixing process (Puente-Córdova, et al., 2018). Finally, this sample cannot be considered to be an MRE due to

absence of ferromagnetic particles within matrix, which is essential in determining MR effect.

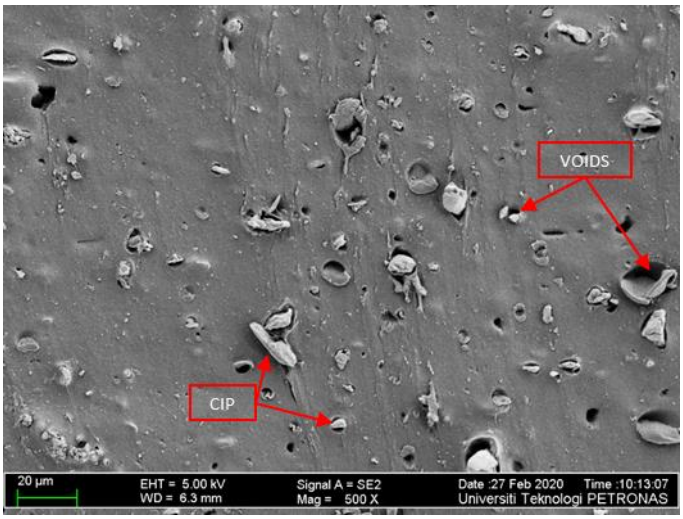

Figure 9: Microstructure of ENR-50 with 30 wt % CIP.

The figure above shows the microstructure of 30 wt% CIP MRE. CIP is seen to be present in the matrix in the elliptical or irregular shapes. One important observation to note is the scattered dispersion of the CIP which shows this is an isotropic MRE. An anisotropic MRE will have more columnar-like structure arrangement due to external magnetic field influence during curing process. Figure 8 also shows the presence of various locations of void on the surface of sample. This may be caused by irregular shape of CIP which will not allow ENR matrix to fill the gaps subsequently causing the empty pockets to be replaced by air. This is not a desired phenomenon as once enough air replaces the ENR matrix, Critical Pigment Volume Concentration (CPVC) value will indicate a point where there is no enough matrix to fill voids between the irregular-shaped CIP (Kallio, 2005). As a result, the mechanical properties of the specimen will be greatly diminished.

Besides that, microstructural assessment of the FESEM imaging in figure 8 suggests minimal agglomeration between the ferromagnetic particles in the matrix. However, this is because said matrix just contains a 30 wt% of CIP, which allows the CIP to be scattered further from each other.

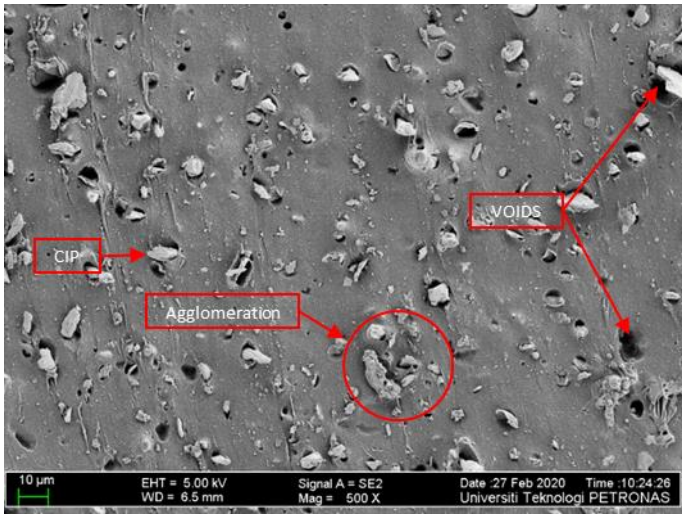

Figure 10: Microstructure of ENR-50 with 70 wt % CIP.

Figure 9 above shows the microscopic imaging of EN-50 based MRE with 70 wt% CIP content. CIP shape irregularity is similar to the one observed in figure 8 due to both samples use CIP from the same source, thus the uniformly-irregular shape. Dispersion in this sample is seen to be affected due to the high concentration of CIP within the matrix causing the spaces within the CIP being greatly reduced. With reference to figure 8 sample (30 wt%), figure 9 shows that a higher CIP concentration within the matrix will cause smaller gaps within CIP.

As a result, various clusters of agglomeration of CIP can be observed in the microstructural assessment. This behaviour has also been observed in another study which also used 70 wt% CIP but with an ENR-25 matrix (Yunus, et al., 2016). Thus, it can be assumed that agglomeration will occur with the introduction of higher weight percentages of ferromagnetic particles within an MRE matrix. Moreover, a higher

concentration of CIP in the matrix caused poor attachment of CIP to the matrix in some locations as well as voids in other locations.

On the other hand, the smaller gaps between CIP in this matrix may allow for better flow of magnetic flux along the MRE, thus, increasing its MR effect. However, the materials mechanical properties such as tensile and elasticity/ductility is expected to be reduced due to CIP induced increase in stiffness.

4.2. Magnetic Properties Analysis of VSM Testing

VSM testing and analysis is essential in determine the magnetic properties of an MRE such as its magnetic permeability. Two samples are prepared in the VSM one at a time and its hysteresis loops were measured between -0.8 Tesla to 0.8 Tesla or in Gauss units, -8000G to 8000G.

The figure below only shows the M-H curves for 30 wt% and 70 wt% samples. The sample with 0% CIP does not need to undergo VSM testing as it does not contain any ferromagnetic particles, which would negate the purpose of the test. Besides, previous research indicated then when it conducted VSM testing on 0 wt% samples, the results were too small, which is negligible (Yunus, et al., 2016). The minor magnetic properties observed in the 0% sample was attributed to the presence of carbon black in the matrix.

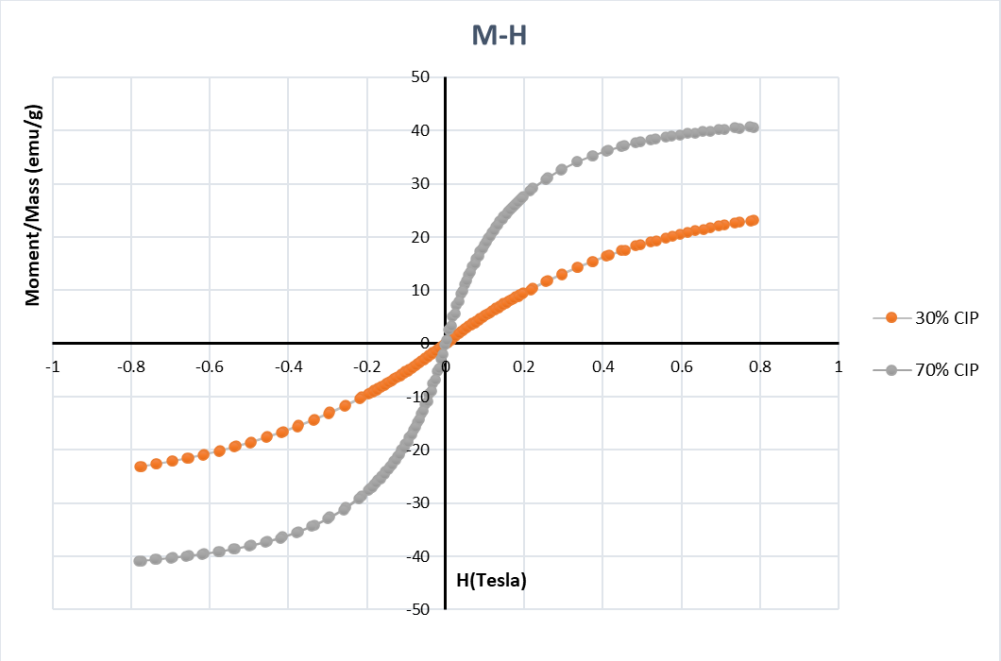

Figure 11: Hysteresis M-H Curves of Samples With 30% CIP & 70% CIP

Initial observations of the hysteresis loops in the M-H curve above, it is obvious that the CIP withing the ENR-50 matrix were excited/induced when exposed to an external magnetic field by the VSM. Both samples also are seen to possess narrow hysteresis loops which is an indication of a soft ferromagnetic particles, which

means the materials can magnetise and demagnetise easily (Ivaneyko, Toshchevikov, Saphiannikova, & Heinrich, 2014), which consequently can rise up the values of MR effect in the rheological sections.

The 30 wt% curve in figure 10 shows the sample to possess paramagnetic characteristics. This is due to the materials ability to be weakly attracted to an induced magnetic field by the VSM (Helmenstine, 2019). In the case of the 70 wt% sample however, it is observed that the sample is a superparamagnetic ENR-50 based MRE. This is because the of the sample's narrow shape of hysteresis cycles which shows it is more magnetically susceptible as compared to the other sample (30 wt%). From here, we can assume that an increase in CIP content in the MRE matrix will allow the material to transition from a paramagnetic behaviour to superparamagnetic behaviour.

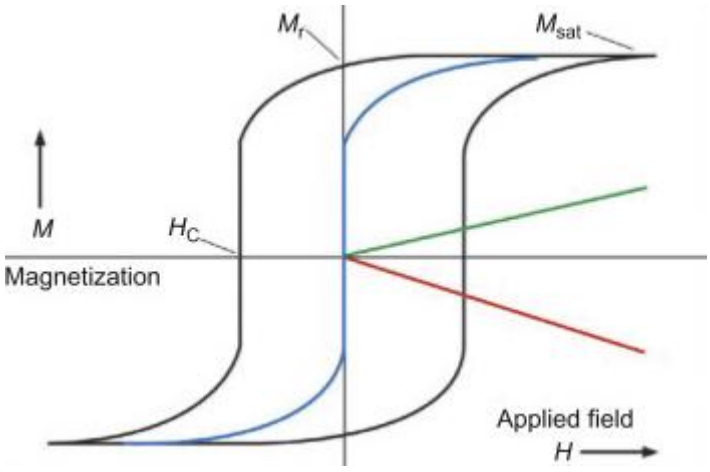

Figure 12: Types of Magnetisation M-H Curves (Chee, Leo, & Lai, 2018).

The M-H curve for both samples have similar trends on the horizontal axis, where both curves become steeper drastically at around -0.2 Tesla (or 2000 Gauss) and returns back to normal around 0.2 Tesla. The curves decrease in gradient steepness once the CIP content in the respective specimens have dispersed to reach its optimum saturated magnetisation, M_S. Magnetic Saturation point is when the magnetic particles within the matrix have completely dispersed according to the magnetic field direction induced by the VSM. The table below show the values of the magnetic properties of specimens:

Table 4: Magnetic Properties of ENR-50 MRE with Different CIP Content

Sample (wt % CIP)	M_s (emu/g)	M_r (emu/g)	H_c (emu/g)
0%	N/A	N/A	N/A
30%	23. 170	0.10030	17.532
70%	40.803	0.39592	14.733

The saturated magnetisation, M_S value is higher in sample with 70 wt % CIP as compared to 30 wt%. This higher content of CIP resulted in a more densely packed CIP-ENR matrix which means that sample will possess a stronger and larger magnetic field strength (Magnetic Materials: Properties , n.d.).

Magnetic retentivity, M_r, value can also be obtained from the M-H curves in figure 10. Retentivity is the ability of a material to retain magnetism after induction of magnetic field is stopped, also known as the ability of the metal to demagnetise. From the M-H curve, we can learn that an increase in CIP content will cause an increase in retentivity of the material from 0.10030 emu/g (30 wt%) to 0.39592 emu/g (70 wt%).The low and very close changes of coercivity values are indicative of a soft magnetic material (Yunus, et al., 2016).

Another important magnetic property observed in a VSM test of an MRE is the coercivity (H_c) of its magnetic particles within the matrix. Coercivity, H_c, is the force required to reverse magnetization after it has achieved its optimum saturated magnetisation (M_S) point. Table 4 describes that as content of magnetic particles in MRE is increased, the coercivity values decrease, from 17.532 G to 14.733 G. Although this difference is not much, the difference could be an indicator of the characteristic of the soft magnetic property of sample with different CIP content due to the phenomenon of transition of paramagnetism to superparamagnetism as mentioned earlier.

From the physicochemical analysis via FESEM microstructural imaging and VSM testing, it can be concluded that increasing the CIP content in a ENR-50 based MRE will increase its magnetic performance. It is also found that the superparamagnetic behaviour of the 70 wt% sample is in line with expected properties from an MRE.

5. CONCLUSION & RECOMMENDATION

5.1. Conclusion

This study involved the fabrication of various ENR-50 based MRE samples with varying weight percentages of CIP content. Physicochemical testing of was then done to conduct a microstructure analysis and magnetic property analysis of MRE sample. Microstructural analysis of different CIP contents in MRE showed that an increase in the CIP content increased the chances of porosity and void occurrences due to chemical reaction with ENR during curing at high temperatures. However, the same sample with high CIP content possessed much higher magnetic properties compared to the one with lower magnetic properties.

The superparamagnetic behaviour of the 70 wt% sample was an ideal desired property in an MRE, however, the presence of such a large percentage of CIP is bound to affect the physical aspect of the material, as seen in the microstructural analysis.

This means that increasing the CIP content within the matrix will help increase the magnetic properties. However, this is only up to a certain point until it begins affecting the physical aspect of the MRE such as a much-reduced ductility and elasticity due to high concentration of CIP which will increase the stiffness of the smart composite.

In conclusion, the objectives of this research to fabricate and conduct physicochemical analysis on the samples were successfully carried out. Further research should be done to identify the right balance of CIP content for the matrix. The next section will describe some recommendations which may be useful in conducting further research in this field.

5.2. Recommendations

This study focused on studying the effect of different weight percentages of CIp on the physicochemical properties of ENR-50 based MRE. To further understand this class of MRE, a thorough study of the rheological properties has to be done to reveal the MR effect of the material, which is essential in determining the strength of any MR material. Furthermore, understanding the rheology of this material will allow material engineers to develop more industry-stable versions to enable mass production for widespread application in the industry.

Another recommendation for future studies is to study the effects of different ratios of dispersing aids to further reduce the effects such as mediocre dispersion of CIP and help reduce porosities and voids in the matrix due to it being a solid-state analogue smart composite material. Previous literature review showed some positive result with using sucrose acetate isobutyrate (SAIB) plasticiser to help with dispersion of CIP within the ENR-50 MRE matrix (Ahmad Khairi, et al., 2016). Since that research was also done with ENR-50 matrix, a study on the combination effects of different CIP content together with SAIB as plasticizer should be done to observe the effect of both these factors.

This study focused on an isotropic structure MRE. Future studies should be recommended to analyse the effect of varying CIP contents in an anisotropic structure. The results are expected to be different due to the fact that anisotropic MRE undergo treatment to alight the magnetic particles to be parallel to a magnetic field line. This is expected to improve their function as an MRE.

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