

**The Study of Magneto-Active Polymer Composite Physiochemical Characteristics Using
Petroleum Based Oil as a Dispersing Aid**

by

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22652

Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Mechanical Engineering
With Honours

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Mechanical Engineering Programme
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in partial fulfilment of the requirements for the
BACHELOR OF ENGINEERING (Hons)
(MECHANICAL)

Approved by,



(Dr. Nurul Azhani Yunus)

UNIVERSITI TEKNOLOGI PETRONAS
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 and persons.



WAN AIZAT BIN MOHAMAD NOOR

Table of Contents

CERTIFICATION OF APPROVAL	ii
CERTIFICATION OF ORIGINALITY	iii
LIST OF FIGURES	v
LIST OF TABLES	v
ABSTRACT	vi
ACKNOWLEDGEMENT	vii
CHAPTER 1	1
INTRODUCTION	1
1.1 Background	1
1.2 Problem Statement	2
1.3 Objectives and Scope of Study	3
CHAPTER 2	4
LITERATURE REVIEW	4
CHAPTER 3	8
METHODOLOGY	8
3.1 MRE Fabrication.....	8
3.2 MRE physicochemical characteristics testing	9
3.3 Gantt Chart.....	12
CHAPTER 4	14
RESULTS	14
4.1 Microstructure of Samples	14
4.1.1 Energy Dispersive X-Ray (EDX)	14
4.1.2 Field Emission Scanning Electron Microscope (FESEM).....	16
4.1.3 Other Physicochemical Characteristic Testing	19
4.2 Discussion	20
CHAPTER 5	21
CONCLUSION	21
5.1 Conclusion	21
5.2 Recommendations	21
REFERENCE	23

LIST OF FIGURES

FIGURE 1: ISOTROPIC MRE AND ANISOTROPIC MRE	6
FIGURE 2: MRE FABRICATION FLOWCHART	9
FIGURE 3: FIELD EMISSION SCANNING ELECTRON MICROSCOPE (FESEM) MACHINE	10
FIGURE 4: THERMOGRAVIMETRIC ANALYSIS (TGA) MACHINE	11
FIGURE 5: EDX GRAPHS OF SAMPLES, S1(A), S2(B), S3(C) AND S4(D)	14
FIGURE 6: MICROSTRUCTURE OBSERVATIONS AT: (A) 70X, (B) 100X, (C) 500X AND (D) 1000X OF MAGNIFICATIONS FOR NR-BASED SAMPLE S1	16
FIGURE 7: MICROSTRUCTURE OBSERVATIONS AT: (A) 70X, (B) 100X, (C) 500X AND (D) 1000X OF MAGNIFICATIONS FOR NR-BASED SAMPLE S2	17
FIGURE 8: MICROSTRUCTURE OBSERVATIONS AT: (A) 70X, (B) 100X, (C) 500X AND (D) 1000X OF MAGNIFICATIONS FOR NR-BASED SAMPLE S3	18
FIGURE 9: MICROSTRUCTURE OBSERVATIONS AT: (A) 70X, (B) 100X, (C) 500X AND (D) 1000X OF MAGNIFICATIONS FOR NR-BASED SAMPLE S4	19

LIST OF TABLES

TABLE 1: FABRICATION COMPOSITION OF SAMPLES	8
TABLE 2: FYP I GANTT CHART	12
TABLE 3: FYP I GANTT CHART	13
TABLE 4: ELEMENTAL RESULT OF NR-BASED MRE SAMPLES	15

ABSTRACT

Rubber is a natural polymer of Isoprene which has elastic properties and it undergoes long range reversible extension. Magnetorheological fluids are known to be used in shock absorbers, dampers as well as isolators. Researchers have also studied the potential of these fluids to be used in various industries. However, the limitation that they faced was due to the substance being formed in a fluid state. It is believed that by forming a solid polymer composite, numerous applications can benefit from the characteristics that it may possess. Rubber is a natural polymer of Isoprene which has elastic properties and it undergoes long range reversible extension. Rubber is also known as an elastomer that inhibits the ability of being magnetised. This is made possible by combining the polymer elastomers with magnetisable elements such as carbonyl iron particulates. In this report, we studied the physicochemical characteristics of magnetorheological elastomers which consists of rubber and carbonyl iron particulates using different types of petroleum based oil as a dispersing aid. In this case, we are using natural rubber (NR) as a matrix as well as carbonyl iron particles as the magnetisable element that would allow the presence of magnetorheological effect. As for the additives, dispersing aids in the form of petroleum based oil will be used in all the fabricated samples. The oil that will be used is paraffin oil. Once the MRE samples have been fabricated, the physicochemical characteristics of the fabricated MRE samples will be examined in order to obtain the composition, thermal behaviour as well as the behaviour when the material is exposed to an external magnetic field. The tests that have been done on the samples are field emission scanning electron microscope (FESEM) and energy-dispersion x-ray (EDX). We found that the ratio used between paraffin oil and aromatic oil during the fabrication process affects the dispersing of iron particles as well as the end condition of the matrix itself. Some agglomeration can also be seen on certain samples.

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CHAPTER 1

INTRODUCTION

1.1 Background

Magneto-active polymers or magnetorheological elastomers (MRE) are smart composites that contains a magnetisable element most commonly in the form of micro-sized carbonyl iron particulates (CIP) which have been embedded in the elastomers through cross-linking process. These particulates can be aligned by applying an external magnetic field hence offering several potential applications including variable stiffness components and damping elements. The magnetisation of the iron particles leads to a phenomena called magnetostriction causing a change in the overall material stiffness and other properties (Liu & Xu, 2019). When the field is taken away, the material will return to the original natural state. There are various types of magnetorheological material available in the current market including magnetorheological fluid and gel.

Magnetorheological effect is termed as the remarkable increase of the rheological properties of the magnetorheological material induced by an external magnetic field (Hao, 2005). The magnetorheological effect was discovered back in 1940s and was in the form of fluids containing micro-sized iron particles suspended in a dense carrier fluid. Magnetorheological fluid has widely been commercialised in numerous industries including from automotive up to human prosthetics. When subjected to a magnetic field, the fluid rapidly increases its viscosity to the point of becoming a viscoelastic solid. Viscoelastic materials exhibit both viscous and elastic characteristics within a single material. Although magnetorheological fluid has been widely used, several limitations of the fluid has led to the research of magnetorheological elastomers as a better material to be used compared to using fluid. This is due to the fact that the CIPs suspended in the fluid tends to sediment and cause a precipitation known as hard cake phenomena. In able for the magnetorheological effect to function properly, the CIPs need to remain dispersed throughout the substance therefore allowing a continues reaction to the external magnetic field.

Fabricating the magnetorheological material requires a certain key aspect in order to ensure that it performs efficiently. These aspects include that the material needs to be of high permeability and high saturation magnetisation. The reason for the need of a high permeability structure is that it will allow the captivation of magnetic leakages thus allowing the induction of magnetic field throughout the elastomer. FeCo alloys are one of the materials that has the highest saturation magnetisation available today. Another factor that is necessary for the elastomer to be able to return to its original state after removing the external magnetic field is to have the CIPs dispersed and does not coagulate with each other.

In order to prevent the CIPs from coagulating, dispersing aids are used to decrease molecular gaps and help the particulates stay in place. It is desired that each particle should remain dispersed as the surface energy of individual particle is not sufficient to separate the particle hence the interaction will form a large particle. In this project, we will be testing the effect of three different petroleum-based oil including light mineral oil, paraffin oil and naphthenic oil as a dispersing aid for the CIPs will be examined. Dispersing aid will also increase the zeta potential and thus are responsible for avoiding the particle interactions.

1.2 Problem Statement

Magnetorheological Fluid has been widely commercialised in various industries. However, the fluid has several downsides which decreases the reliability and sustainability of the material. One of the disadvantages is the magnetisable element that suspends in the fluid tends to sediment creating a hard cake formation. This affects the magnetism of the fluid to function properly as the magnetic field could not magnetise the magnetic particles present in the fluid hence will not be able to interact with the whole fluid. This sedimentation also causes some difficulties for the particulates to re-disperse and will not be able to return the material's original natural state. Furthermore, magnetorheological fluids contain particles that are primarily micro-sized which is too dense for the Brownian Motion to keep them suspended in the fluid (Tao, 2001). Using fluid or oil as a carrier for the CIPs is also less practical as the fluid tends to thicken after prolonged use hence must be replaced from time to time. This will increase the cost of maintaining the system as high quality fluids are expensive. Upon moving into the future, determining technologies that are reliable and cost effective are part of the aim since the material has potential to be commercialised worldwide.

Researchers have studied and came up with an alternative to replace fluid as a carrier by using elastomers instead. MREs are solids that consists of a polymeric matrix embedded with CIPs. It is expected that this material will not encounter the same limitations as using fluids. However, the dispersion of CIPs throughout the material is still a main concern for the elastomer to work efficiently. Hence, utilisation of dispersion aids might be useful in enhancing the dispersion of particulates while mitigating the particulates' agglomeration. Aromatic oil was formerly used to disperse CIPs in the material particularly using unsaturated rubber such as natural rubber (NR) and epoxidised natural rubber (ENR). In the production of saturated rubber like silicon rubber, silicon oil has been used to disperse the additives during the mixing process. As an alternative, the dispersion effects of using petroleum-based oil will be studied. The petroleum-based oil has a high molecular weight which will be beneficial for the CIPs dispersion. Nevertheless, this claim is still under research and a comprehensive study on the physicochemical characteristics of MRE using petroleum-based oils as a dispersing aid has yet been reported. Therefore, the aim of this project is to study the effect of different petroleum-based oil on the physicochemical characteristics including microstructure, magnetic and thermal properties of MRE.

1.3 Objectives and Scope of Study

The main objective of this study is to investigate the effects of petroleum-based oil which paraffin oil is being used as a dispersing aid on the physiochemical characteristics of the NR-based MRE. The main objective can be divided into several sub-objectives as follows:

- To study the microstructure particularly CIPs distribution within NR-based MRE.
- To analyse the influence of using petroleum-based oil on magnetic properties and thermal behaviour of NR-based MRE.

CHAPTER 2

LITERATURE REVIEW

Magnetorheological elastomers (MRE) belongs to a class of smart material in which rheological properties can rapidly change under the influence of an external magnetic field. This material is becoming today's hot topic and is constantly increasing due to the various potential and possibilities of their applications. Giant deformation effects, tuneable elastic modulus, non-homogenous deformation and quick response to magnetic field open new opportunities for using such material for various applications (Zrinyi, 2014). This phenomenon is called the magnetorheological effect. Basically, micro-sized magnetisable particles are magnetized to produce orderly movement and positioning when an external magnetic field is applied. This movement generating at the beginning of micro particles are magnetized and finishing until reaching a relatively stable state, forming a fix structure (Yiping, Biao & Hongjuan, 2014). These particles are attracted to each other under the force of the external magnetic field forming a chain-like structure. The magnetic particles will be randomly distributed in the material without an external magnetic field. However, once magnetic field is applied, the magnetic particles rapidly polarises and forms an alignment which will alter the rheological properties of the material instantly. Magnetorheological materials are already present in the market today in the form of fluids, foams and gels. The most common type that is widely used in dampers and absorbers is magnetorheological fluids.

Magnetorheological fluids (MRF) can be classified as a ferrofluid. It is a colloidal suspension of single-domain magnetic particles with typically dimensions of about 10nm dispersed in a liquid carrier or more often in oils. The liquid carrier can be polar or nonpolar (Scherer & Neto, 2005). It has the same working principle of the MRE which the magnetic particles create a chain-like structure upon the presence of an external magnetic field. In this case, the fluid thickens causing the alteration of the MRF's rheological properties. However, recent issues of MRF in the industry have brought up the question whether the material is reliable enough to be feasibly commercialised in the market. This is due to the fact that the magnetic particles in the carrier fluid tends to sediment creating a flaw in the system. Moreover, the fluid also has the tendency to cause leakage in the system which discourages developers to inhibit such material in their systems. A container is necessary

to prevent any leakage of the MR fluid, and the magnetic particles in liquid tend to settle down during the service (Wang, Zhang, Oh & Chung, 2015).

Therefore, it is less practical to be utilising magnetorheological fluid for commercial usage as their downsides will affect the performance of the material itself. Hence, these issues have led to the study of magnetorheological elastomers which is believed to be the solution to the problems faced in the usage of magnetorheological fluid. Magnetorheological elastomers are the solid analogous version of the MRF where the carrier oil has been replaced by rubber materials. Wang, Zhang, Oh and Chung (2015) also said that MREs do not have leakage and sedimentation issues, and the shape is flexible, so it is suitable for MREs to be applied in the mechanical engineering field, especially for the automobile industry.

MREs are divided into two base types which are isotropic magnetorheological elastomers (i-MREs) and anisotropic magnetorheological elastomers (a-MREs) according to the arrangement of the magnetic particles in a matrix. The i-MREs and a-MREs have a uniform and aligned distribution of magnetisable particles respectively (Yunus, Mazlan, Ubaidillah, Aziz, Shilan & Wahab, 2019). As can be observed from Figure 1 below, the difference between the two types of configurations are achieved during the fabrication of the elastomer itself. The i-MREs develop uniform distribution of magnetic particles due to the absence of an external magnetic field during fabrication whilst a-MREs are fabricated with the presence of an external magnetic field which cures and locks the chain structures resulting in the aligned distribution of magnetic particles in the elastomer. This will allow the anisotropic structure of MRE to have magnetic particles alignment parallel to the magnetic field (Gong, Zhang & Zhang, 2005). The structure of the MREs affects their mechanical behaviour and magnetorheological properties directly hence is necessary to understand the relationship between the fabrication process and structure change which in this case is fabricating with and without the presence of an external magnetic field.

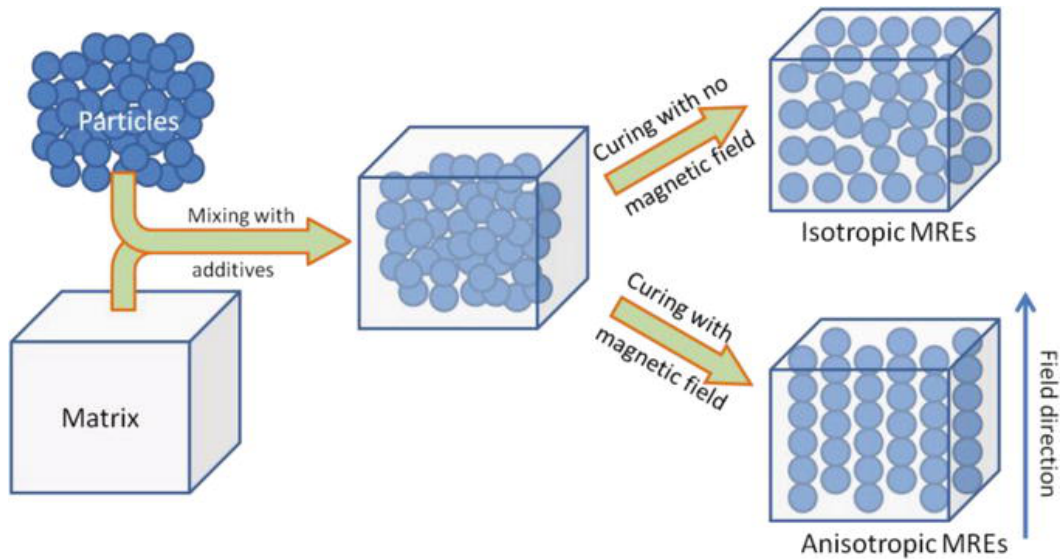


Figure 1: Isotropic MRE and Anisotropic MRE

MRE composites comprises of the matrix, filler and additives such as cross-linking agents, antioxidants and mixing aids may be used. Rubber is commonly used as the MRE matrix in order to overcome the downsides of using fluid as the matrix. There are two types of unsaturated rubber that are used in the fabrication of MREs today which are Natural Rubber (NR) and Epoxidised Natural Rubber (ENR). Natural rubber has a very good mechanical properties, flexibility and processing performances which are suitable for wide applications. However, ENR is quite ahead against NR due to the low performance to overcome damping and high solubility in hydrocarbons and oils of NR. ENR on the other hand has a much stable structure compared to NR because of the epoxidisation process whereby hydrophilic groups is introduced along the isoprene backbone of rubber (Mascia, Russo, Verdolotti, Clarke, Lavorgna & Acierno, 2015). ENR has much improved heat and chemical resistance because the oxirane ring increases the hydrophilicity and reduces the number of double bonds in the backbone.

Magnetic particles are the most important component in fabricating the MRE as the particles are the reason which the material's properties can be altered using magnetic field. The properties of MRE depends on the selection of matrix and magnetisable particles. Several types of elements have been used previously in the fabrication of MR materials including carbonyl iron particles (CIP), Nickel and also Cobalt. Filler particles should be homogenous and well distributed into the elastomer to produce a good network connection (Rajhan, Hamid, Azmi & Ismail, 2014). The sizes and shapes of these particles also play

an important role that may enhance or reduce the MR effect of the MRE. The most common magnetic filler being used throughout researches is iron particles because of their high permeability, low permanent magnetisation and high saturation magnetisation which will provide a huge interaction between particles hence, a greater MR effect will be produced.

In order to facilitate the fabrication process of the MRE, additives are being used for various different purposes. Additives such as cross-linking agents, reinforcing agents and dispersing aids are widely used in aiding the fabrication process as well as enhancing the mechanical properties of the MRE. In the rubber industry, petroleum-based oil is widely used as additives in reducing the viscosity, enhancing the elastomeric performance resulting in a better magnetisable particle dispersion. Addition of processing oil causes lowering of the cohesive forces between the polymer chains and hence increases the chain mobility (Li, Tan, Liu, Hoch & Zhao, 2016). The effects of different petroleum-based oil on the dispersion of magnetisable particles in ENR-based MRE is still being studied.

CHAPTER 3

METHODOLOGY

This segment will be divided into two parts which are the fabrication process and the testing and analysis of the physicochemical characteristics of the fabricated NR-based MRE. In fabricating the MRE, several parameters need to be determined such as the specific quantity of ingredients and ratios of substances to work with. The MR effect of MRE depends on the substances being used hence it is vital to select the best material to be used in fabricating the MRE. As for the testing and analysis phase of the fabricated MRE, characteristics of the MRE will be studied to observe on the effects of using such materials on the microstructure and physicochemical properties of the MRE.

3.1 MRE Fabrication

In fabricating the MRE samples, paraffin oil (PO) with the combination of aromatic oil (AO) will be used. All of these additives will be formulated with multiple ratios from 100% to 30 % of paraffin oil. The fabricating formula is shown in the table below:

The samples are then labelled as S1, S2, S3 and S4 with the ratio of 100:0, 70:30, 50:50 and 30:70 respectively.

Table 1: Fabrication Composition of Samples

Compound Ingredients	Ratio of PO : AO			
	100:0	70:30	50:50	30:70
	Amount (phr)			
Natural Rubber (NR)	100	100	100	100
Carbon Black	19	19	19	19
Zinc Oxide	5	5	5	5
Stearic Acid	2	2	2	2
Sulfur	2.3	2.3	2.3	2.3
CBS	0.8	0.8	0.8	0.8
Paraffin Oil	10	7	5	3
Aromatic Oil	0	3	5	7

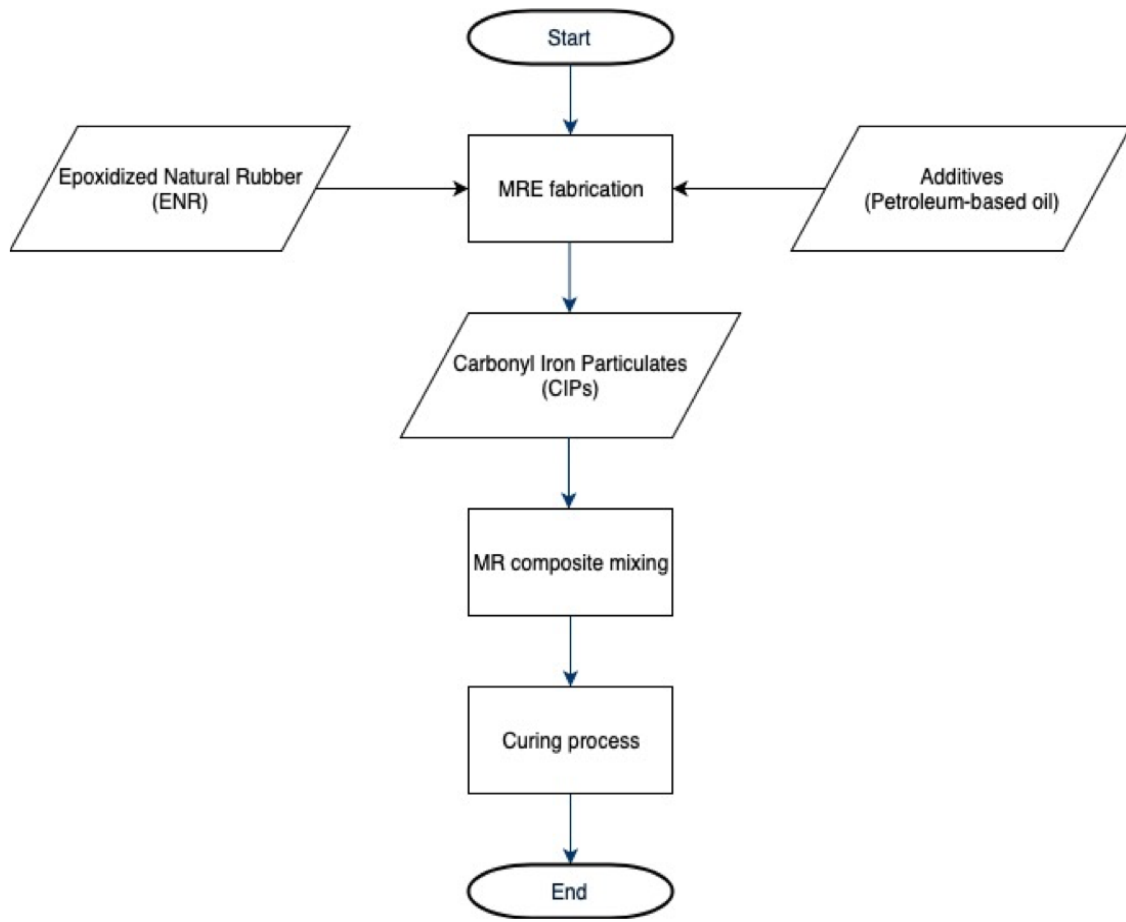


Figure 2: MRE Fabrication Flowchart

3.2 MRE physicochemical characteristics testing

In this part of the methodology, the fabricated MRE samples will be tested and the physicochemical characteristics will be analysed. The performance and MR effect of the different PBO used in each MRE composite will be compared. Among the tests that are planned to be done include:

- i. Field emission scanning electron microscope (FESEM)
- ii. Energy dispersive x-ray (EDX)
- iii. Thermogravimetric Analysis (TGA)
- iv. Vibrating sample magnetometer (VSM)

Field emission scanning electron microscope (FESEM)

FESEM provides topographical and elemental information at magnifications of 10x up to 300,000x with virtually unlimited depth of field. FESEM produces clearer, less electrostatically distorted images with spatial resolution down to 1.5 nanometres compared to conventional SEM. In this study, the samples have been studied using the magnifications of 70x, 100x, 500x and 1000x.



Figure 3: Field Emission Scanning Electron Microscope (FESEM) Machine

A field-emission cathode in the electron gun of a scanning electron microscope supplies narrower probing beams at low as well as high electron energy, resulting in both improved spatial resolution and minimised sample charging and damage. For applications that demand the highest magnification possible, in-lens FESEM is possible to produce ultra-magnification.

Energy dispersive x-ray (EDX)

EDX identifies the elemental composition of materials imaged in a scanning electron microscope for all elements with an atomic number greater than boron. The application of EDX includes material and product research, troubleshooting and de-formulation.

As the electron beam of the SEM is scanned across the sample surface, it generates x-ray fluorescence from the atoms in its path. The energy of each x-ray photon is characteristic of

the element that produced it. The EDX microanalysis system collects the x-rays, sorts and plots them by energy and automatically identifies and labels the elements responsible for the peaks in this energy distribution.

Thermogravimetric Analysis (TGA)

TGA measures the weight changes in a material as a function of temperature and time under a controlled atmosphere. Its principle usage includes measurement of a material's thermal stability, filler content in polymers, moisture and solvent content and the percent composition of components in a compound.



Figure 4: Thermogravimetric Analysis (TGA) Machine

A TGA analysis is performed by gradually raising the temperature of a sample in a furnace as its weight is measured on an analytical balance that remains outside of the furnace. In TGA, mass loss is observed if a thermal event involves loss of a volatile component. Chemical reactions such as combustion involves mass losses whereas physical changes such as melting do not. The weight of the sample is plotted against temperature or time to illustrate thermal transitions in the material such as loss of solvent and decomposition of the material upon being exposed to heat.

Vibrating sample magnetometer (VSM)

VSM is used to measure the magnetic properties of solids and liquids. The magnetometer measures magnetic moment as a function of an applied magnetic field. A unique feature of this system is the ability to measure magnetostriction using special sample holder that strains the sample during the measurement.

A VSM operates on Faraday's Law of Induction which informs that a changing magnetic field will produce an electric field. This electric field can be measured and provides us with information regarding the changing magnetic field.

3.3 Gantt Chart

Table 2: FYP I Gantt Chart

Task	Academic Week													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Understanding and study the general view about the project title														
Collecting data and information for the project														
Studying the suitable material composition														
Extended proposal report submission														
Proposal defence														
Fabrication of magnetorheological elastomer (MRE)														
Preparing Interim draft report														
Submission of Interim report														

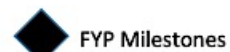
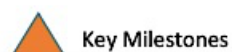


Table 3: FYP I Gantt Chart

Task	Academic Week													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Data gathering	■	■												
FESEM Testing		▲	■	■										
VSM Testing				▲	■	■								
TGA Testing						▲	■	■						
Analysis of gathered data								▲	■	■				
Validation and documentation											■	■	■	
Preparation on Project Dissertation												■	■	■
Submission of Project Dissertation														◆

 Key Milestones

 FYP Milestones

CHAPTER 4

RESULTS

4.1 Microstructure of Samples

The structure of MREs affects their mechanical and magnetorheological properties directly hence it is necessary to understand the relationship between the fabrication process and structural change.

4.1.1 Energy Dispersive X-Ray (EDX)

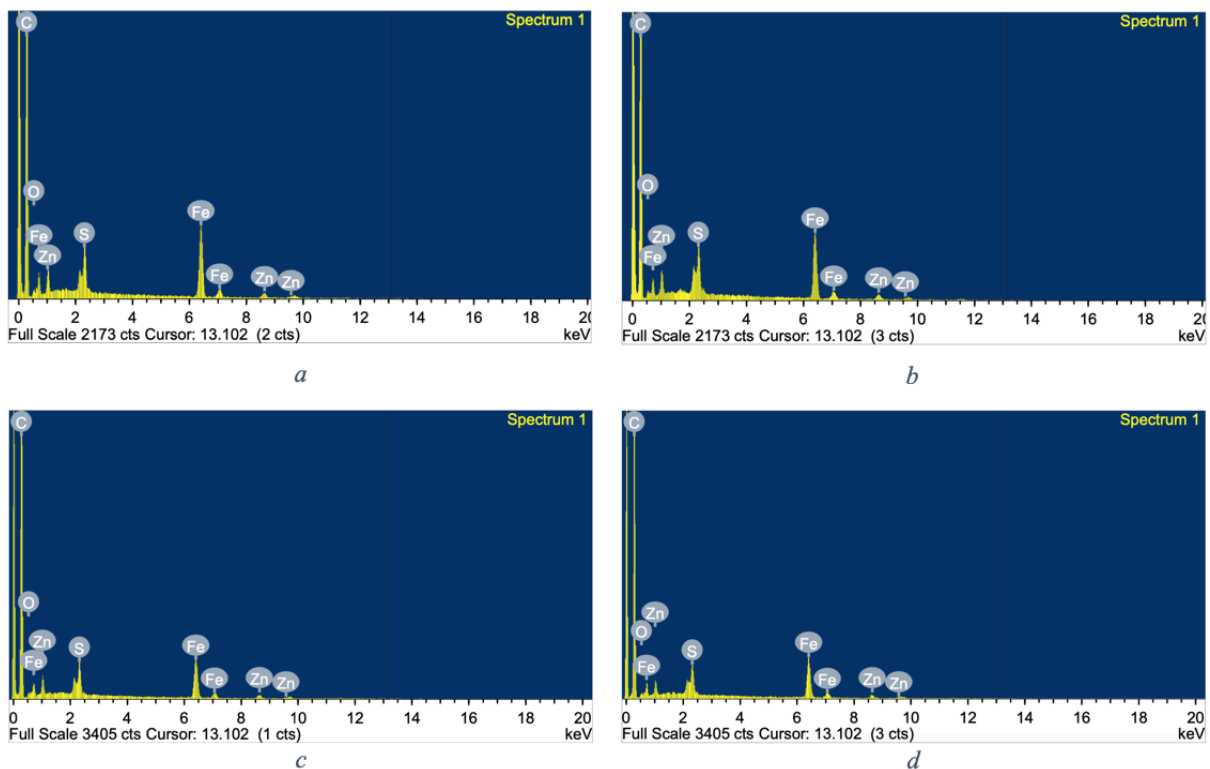


Figure 5: EDX Graphs of Samples, S1(a), S2(b), S3(c) and S4(d)

Figure 5a-d depicts the energy dispersive X-ray spectroscopy (EDX) graphs while Table 4 below details the elemental results of the NR-based MRE for all the samples with various ratios of paraffin oil to aromatic oil. Comparing all the EDX graphs, Fe elements peak the highest on sample S1 which has the ratio of 100:0 of paraffin oil to aromatic oil. The Fe elements correspond to the CIPs that are embedded in the matrix during fabrication process as CIPs

contains approximately more than 97% Fe element. Meanwhile, several other peaks can be seen in the EDX graphs namely carbon (C), oxygen (O), sulphur (S) and zinc (Zn) that exists in the form of additives.

Table 4: Elemental Result of NR-based MRE Samples

Sample (PO:AO)	Element (Weight %)				
	Fe	C	S	Zn	O
S1 100:0	14.25	78.42	1.85	2.40	3.08
S2 70:30	12.86	79.66	1.75	2.42	3.31
S3 50:50	10.29	83.11	1.71	2.17	2.72
S4 30:70	12.49	80.33	1.89	2.32	2.97

Based on the table of NR-based MRE elemental results, the weight percentage of Fe is the highest with S1 while the rest ranges from 10.29-12.86 weight %. Similar to the EDX results, carbon, sulphur, zinc and oxygen elements were detected in all the samples. Comparing all the elements, it is observed that carbon takes up a higher content in the samples. This can be confirmed due to the presence of carbon black as additives during the fabrication process in order to produce a better bond between the NR matrix and CIPs. It is clear that the results of the EDX graphs and elemental results of the samples correspond to each other.

4.1.2 Field Emission Scanning Electron Microscope (FESEM)

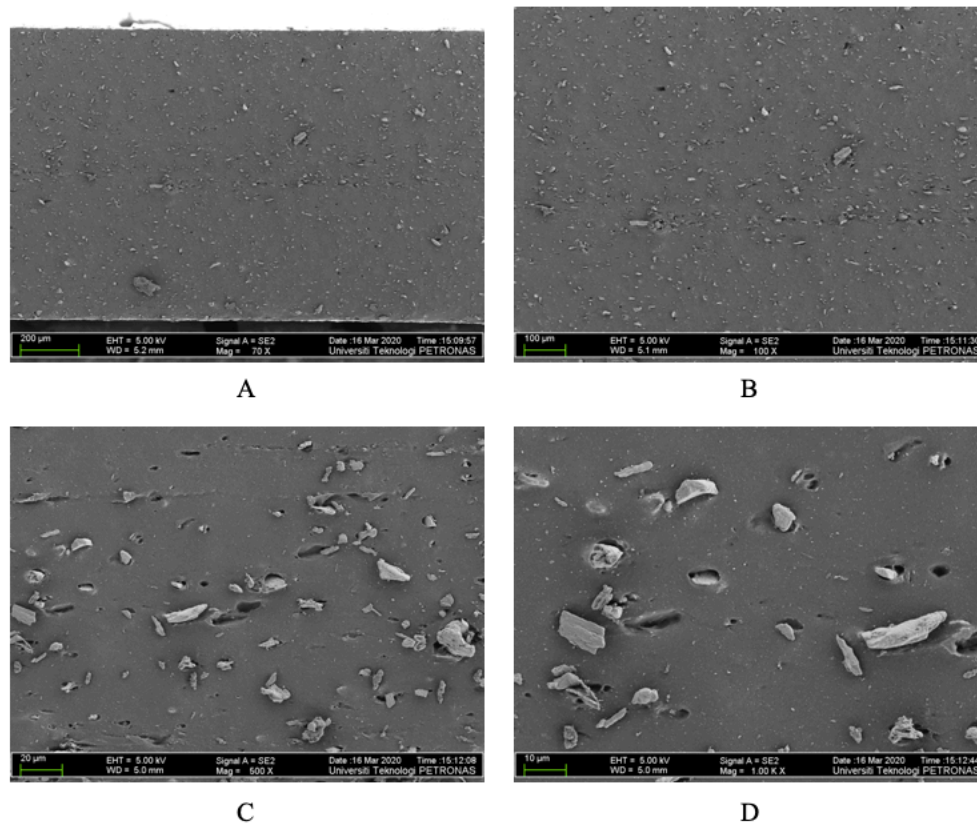


Figure 6: Microstructure Observations at: (A) 70x, (B) 100x, (C) 500x and (D) 1000x of Magnifications for NR-based Sample S1

Figure 6(A)-(D) above illustrates the microstructure of NR-based Sample S1 which has the ratio of 100:0 of paraffin oil to aromatic oil at magnifications of 70x, 100x, 500x and 1000x respectively. The shards that can be seen on the FESEM images are CIPs that were imbedded during the fabrication process. To differentiate between the elastomer and CIPs, the appearance of the CIPs is greyish white and the NR-based matrix can be seen as the black in colour region. The scattering of the embedded CIPs confirms of the MRE produced is an isotropic MRE as no magnetic field was induced during the curing process. The isotropic distribution of iron particles shows adherence to the network structure of the NR-based matrix used. From Figure 6(A) and (B), the CIPs are scattered randomly throughout the NR-based matrix. As we look closer and increase the magnifications such in Figure 6(C) and (D), the CIPs can be seen with irregular shapes and sizes. There are also presence of voids and poorly attached particles. The bonds between particles and matrix will influence the mechanical behaviour of the MRE.

The FESEM microstructure images of Sample S2 with the ratio of 70:30 are illustrated in Figure 7(A)-(D) at different magnifications. At 70x and 100x magnifications from Figure 7(A) and (B), the iron particles look similar to of Sample S1. However, the particles appear to be scattered more evenly but with a lower content compared to Sample S1. This can be confirmed from the previous elemental results where Sample S2 contains less Fe element weight % of 12.86% where Sample S1 with 14.25%. By increasing the magnification to 500x and 1000x, it is observed that the matrix is porous as it exhibits more voids. Some settling of iron particles can also be seen in the polymer composite.

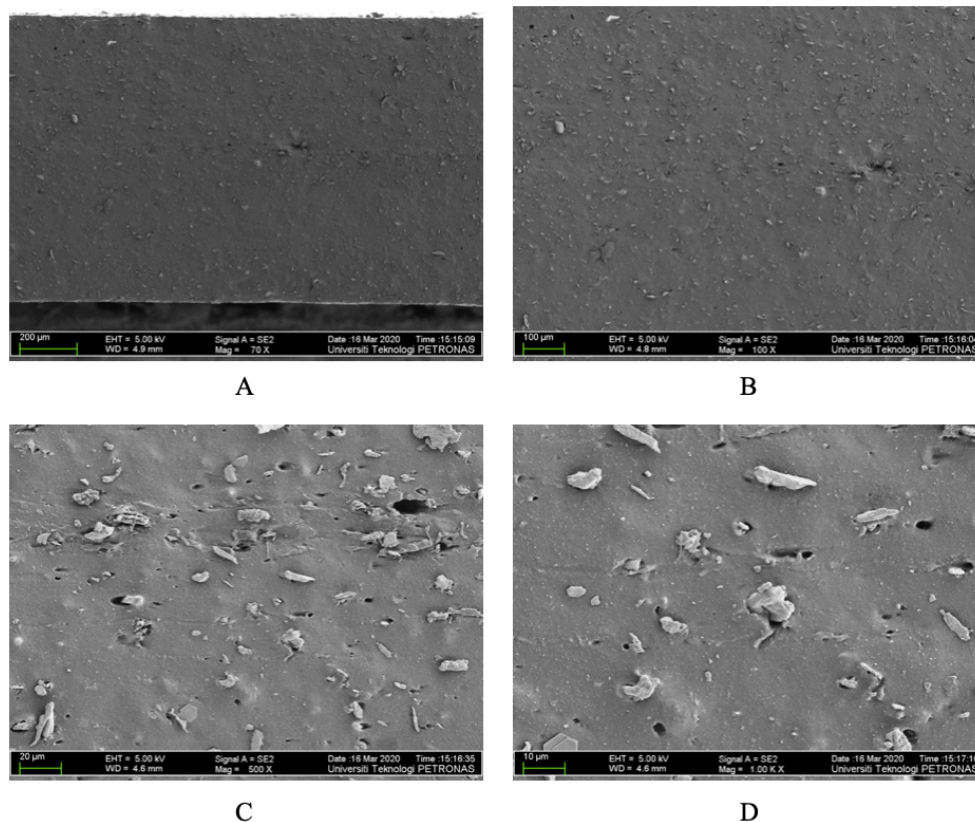


Figure 7: Microstructure Observations at: (A) 70x, (B) 100x, (C) 500x and (D) 1000x of Magnifications for NR-based Sample S2

Samples S3 inhibits the ratio of 50:50 of paraffin oil to aromatic oil. From Figure 8(A) and (B), the carbonyl iron particles disperse randomly and evenly throughout the matrix. This can be very useful as better packing of the particles allows a better pathway for the whole matrix to be magnetised. Further magnification in Figure 8(D) illustrates a larger void despite the iron particles embed better with the matrix. Voids in the matrix are not acceptable as it may affect the mechanical performance of the MRE. No agglomeration can be seen in the matrix.

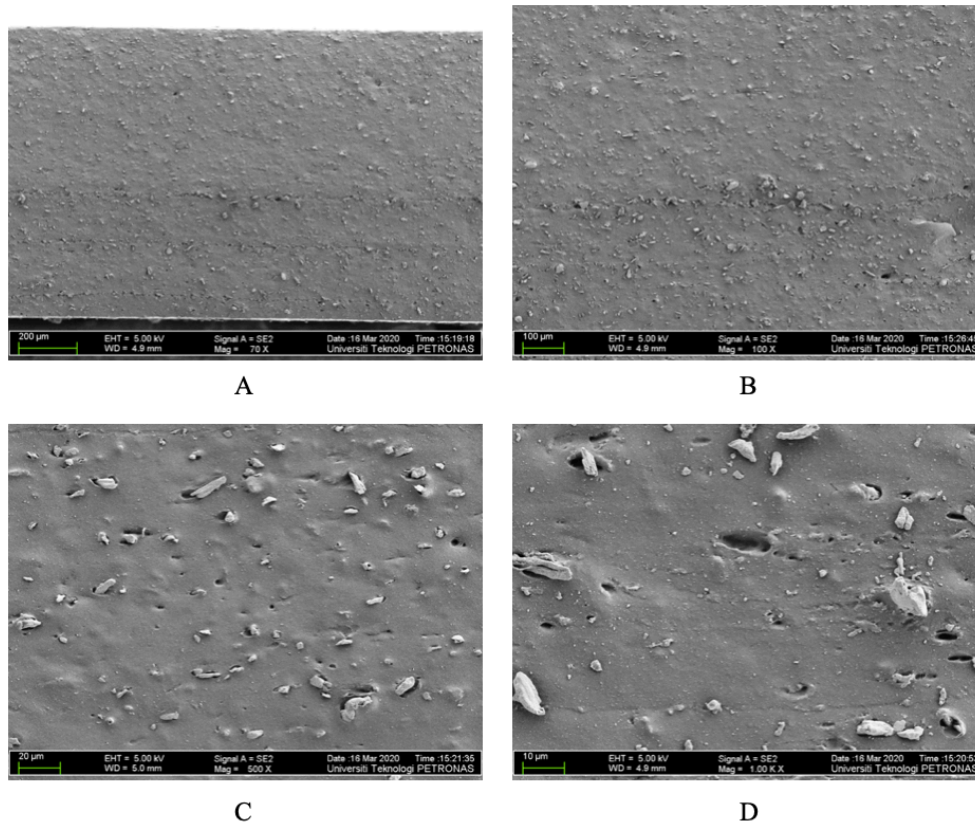


Figure 8: Microstructure Observations at: (A) 70x, (B) 100x, (C) 500x and (D) 1000x of Magnifications for NR-based Sample S3

Figure 9(A)-(D) illustrates Sample S4 which was fabricated with the ratio of 30:70 of paraffin oil to aromatic oil. The FESEM microstructure images at 70x and 100x magnification from Figure 9(A) and (B) respectively shows that the carbonyl iron particles are randomly dispersed throughout the NR-based matrix. However, increasing the magnification to 500x in Figure 9(C), the matrix appears to be very porous and the bond between particles and the matrix is weaker compared to samples S1, S2 and S3. It is clearer in Figure 9(D) with a higher magnification of 1000x whereby the pores are larger than other samples hence not preferable to achieve high MR effect. Poorly attached particles towards the NR-based matrix can also be observed closely from the images which will affect the mechanical behaviour of the MRE as a whole.

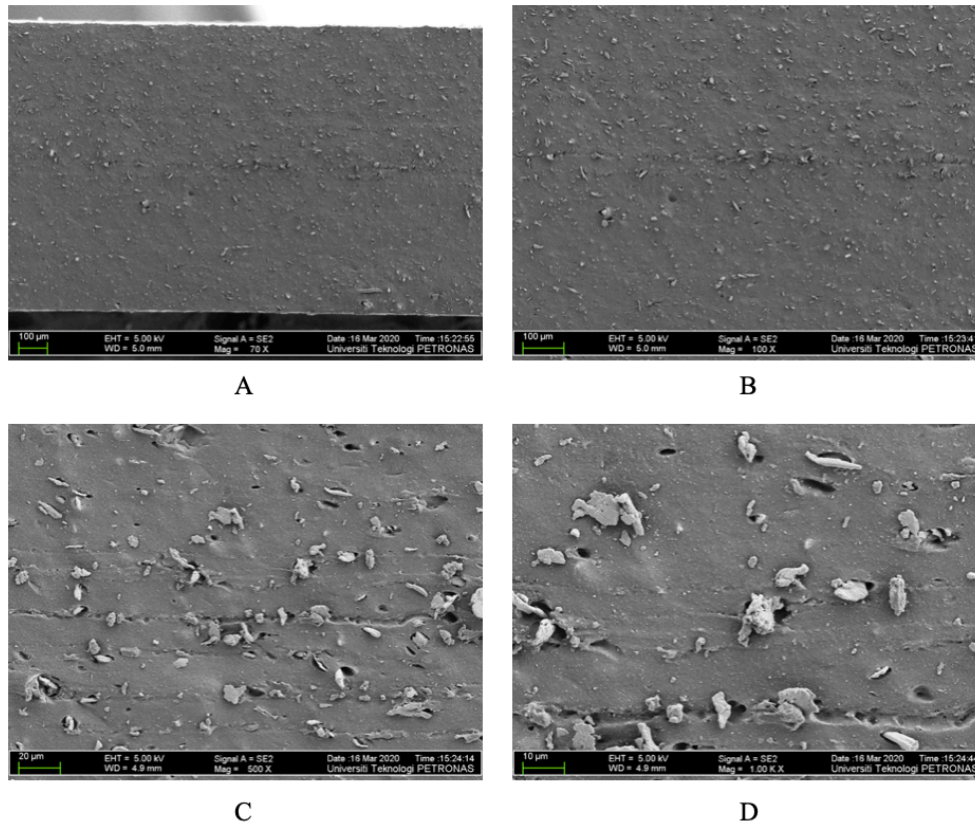


Figure 9: Microstructure Observations at: (A) 70x, (B) 100x, (C) 500x and (D) 1000x of Magnifications for NR-based Sample S4

4.1.3 Other Physicochemical Characteristic Testing

Due to the COVID-19 outbreak, the other planned tests namely thermogravimetric analysis (TGA) and vibrating sample magnetometer (VSM) was unable to be completed as the government ordered all institutes to be shut down.

4.2 Discussion

As observed from part 4.1, the FESEM microstructure of the samples, it is confirmed that the ratio of the additives used, paraffin oil to aromatic oil affects the dispersion of carbonyl iron particulates. The dispersion is key to producing high MR effect MREs. The adhesion between particles and matrix is also important as it holds major responsibility of the mechanical behaviour of fabricated MRE. Samal et. Al (2018) claimed that better adhesion and dispersion of filler particles within the composite matrix will produce higher MR effect and better mechanical behaviour.

In terms of dispersion, Sample S3 exhibits the best particle dispersion throughout the NR-based matrix. The dispersion of particles is important as it creates a pathway for the MRE to be magnetised by an external magnetic field thus enhancing the mechanical performance. The iron particles attached very well with the matrix as compared to the other samples. With better attachment, the bond between iron particles and the matrix is stronger creating an effective medium to be controlled by the magnetic field.

However, in sample S4, the NR-based matrix is very porous and has large voids which will create a disconnection between the particles upon magnetisation hence would be inefficient in working properly. The matrix plays an important role to provide a medium for the interconnection of particle magnetisation.

Therefore, further studies on the mechanical performance must be done to fully understand the effects of the dispersing aids used as well as the suitable ratio in the fabricating process.

CHAPTER 5

CONCLUSION

5.1 Conclusion

In this study, natural rubber (NR) was employed as the MRE matrix. Four different samples were successfully prepared with different ratios of dispersing aid which are paraffin oil and aromatic oil at a ratio of 100:0, 70:30, 50:50 and 30:70. The microstructure of the samples are studied and is crucial to determine the MR effect of the fabricated MRE. The microstructure of the NR-based MREs shown that the CIPs embedded during the fabrication process were dispersed randomly in the NR matrix which produces the isotropic MRE structure of the samples. Apart from the structural analysis, other physicochemical characteristics testing must be done to study thoroughly and to determine the MR properties and MR effects of the samples. Hence, due to the virus outbreak, the tests could not be completed and the mechanical behaviour of the fabricated MRE could not be studied further. Without the VSM tests results, it is unlikely to determine the effectivity of the fabricated samples towards being magnetised and exhibiting the MR effects which is the main concern of the study. In conclusion, the findings gained in this study can be useful and considered for further studies in the future.

5.2 Recommendations

From the obtained results, it is insufficient to determine the best ratio of additives to be used as the mechanical behaviour of the samples could not be tested. In order to study the MR effects of the fabricated samples, the behaviour of the samples upon the presence of an external magnetic field must be studied. This is because the aim of MREs are to work side by side with the presence of magnetic field. The magnetisation ability of the elastomer will determine the effectiveness of the material to be commercialised and to find purpose in industrial usage such as dampers and isolators.

In the future, I would recommend that the samples be tested further to understand the full capability and mechanical behaviour of the material. Physicochemical testing such as vibrating sample magnetometer (VSM) should be done at different magnitudes to identify the material behaviour upon the presence of an external magnetic field. Another test that can be done is the

thermogravimetric analysis (TGA). This is to study the side effects of the material on the presence of high heat. The material should be able to withstand a huge amount of heat and to not lose mass when effectively working in the industry.

REFERENCE

- Gong, X., Zhang, X., & Zhang, P. (2005). Fabrication and characterization of isotropic magnetorheological elastomers. *Polymer Testing*, 24(5), 669–676. doi: 10.1016/j.polymertesting.2005.03.015
- Khairi, M. H. A., Mazlan, S. A., Ubaidillah, Ahmad, K. Z. K., Aziz, S. A. A., & Yunus, N. A. (2016). Effect of sucrose acetate isobutyrate ester on the epoxidised natural rubber based magnetorheological elastomers. *Journal of Physics: Conference Series*, 776, 012034. doi: 10.1088/1742-6596/776/1/012034
- Li, X., Tan, S., Liu, G., Hoch, M., & Zhao, S. (2016). The Effect of Paraffinic Oil and Aromatic Oil on the Crosslinks and Physical Properties of Butyl Rubber. *Journal of Macromolecular Science, Part B*, 55(5), 494–502. doi: 10.1080/00222348.2016.1169576
- Liu, T., & Xu, Y. (2019). Magnetorheological Elastomers: Materials and Applications. *Smart and Functional Soft Materials*. doi: 10.5772/intechopen.85083
- Mascia, L., Russo, P., Verdolotti, L., Clarke, J., Lavorgna, M., & Acierno, D. (2015). Probing The Postgelation Reactions Of Epoxidized Natural Rubber Cross-Linked With Dodecenyl Succinic Anhydride. *Rubber Chemistry and Technology*, 88(4), 560–573. doi: 10.5254/rct.15.84888
- Rajhan, N. H., Hamid, H. A., Azmi, I., & Ismail, R. (2014). Magnetorheological Elastomers: A Review. *Applied Mechanics and Materials*, 695, 255–259. doi: 10.4028/www.scientific.net/amm.695.255
- Scherer, C., & Neto, A. M. F. (2005). Ferrofluids: properties and applications. *Brazilian Journal of Physics*, 35(3a), 718–727. doi: 10.1590/s0103-97332005000400018
- Tao, R. (2001). Super-strong magnetorheological fluids. *Journal of Physics: Condensed Matter*, 13(50). doi: 10.1088/0953-8984/13/50/202
- The positive, negative, photo-ER, and electromagnetorheological (EMR) effects. (2005). *Studies in Interface Science Electrorheological Fluids - The Non-Aqueous Suspensions*, 83–113. doi: 10.1016/s1383-7303(05)80018-3

- Wang, Y., Zhang, X., Oh, J., & Chung, K. (2015). Fabrication and properties of magnetorheological elastomers based on CR/ENR self-crosslinking blends. *Smart Materials and Structures*, 24(9), 095006. doi: 10.1088/0964-1726/24/9/095006
- Yiping, L., Biao, X., & Hongjuan, R. (2014). Summarization of Magneto-Rheological Fluid and Its Application in Engineering. *American Journal of Mechanics and Applications*, 2(1), 1. doi: 10.11648/j.ajma.20140201.11
- Yunus, N., Mazlan, S., Ubaidillah, Aziz, S. A., Shilan, S. T., & Wahab, N. A. (2019). Thermal Stability and Rheological Properties of Epoxidized Natural Rubber-Based Magnetorheological Elastomer. *International Journal of Molecular Sciences*, 20(3), 746. doi: 10.3390/ijms20030746
- Zrinyi, M. (2014). Magnetically responsive polymer gels and elastomers: properties, synthesis and applications. *Smart Polymers and Their Applications*, 134–165. doi: 10.1533/9780857097026.1.134