WASTE TIRE RUBBER BASED MAGNETORHEOLOGICAL ELASTOMERS

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A thesis submitted in fulfilment of the
requirements for the award of the degree of
Doctor of Philosophy

Malaysia-Japan International Institute of Technology
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JULY 2016

To my beloved mother, father, wife, children, and relatives

ACKNOWLEDGMENT

Alhamdulillah, I wish to praise the Almighty Allah for his blessings and mercy that have been given to traverse this long journey. I admit my careless and mistakes during this journey, therefore; his forgiveness is the best gift for me. I express my sincere thanks to my advisor, Assoc. Prof. Ir. Dr. Hj. Saiful Amri Mazlan, for all his guidance, support, enthusiasm, and encouragement throughout my graduate studies. His patient, expertise, knowledge, advice, and comments were priceless. To thank Dr. Joko Sutrisno and Dr. Yancheng Li, it is wonderful to have knowledge sharing and friendship. I also express my appreciation to the Malaysia-Japan International Institute of Technology for the MJIIT scholarship.

I express my grateful to Prof. Seung-Bok Choi and Prof. Hyung-Jin Choi, Inha University, for their constructive advices to my scientific manuscripts. I express my appreciation to my family abroad Dr. Fitrian Imaduddin, Burhanuddin, Irfan Bahiuddin, and Muhammad Yani for the warmest daily livings. Sincere thanks are conferred to all colleagues in the Vehicle System Engineering for helping me a lot. I regret that I cannot mention all valuables persons; but I believe that Allah will grant best rewards as for kindness was given to me.

Last but not least, I express my sincere appreciation to my parents, Mr. Sabino and Mrs. Umriyah for their unconditional supports during my studies. They have been replacing responsibility for looking after my family. To my lovely wife, Hasna Nuha Faridah, my sons, Thalhah, and Nuh, my daughter, Ruhmaa, I do ask your forgiveness for almost reducing your rights during this time. I do believe your love, understanding, patient, and prays were the keys for my study completion. To my relatives, Sarroh and Fida, my parents in law, Mr. Mulyoto and Mrs. Suwarni, my grandma Suwarti and my uncle, Arroniri, for their prays and kindness help. Allah would reward their sacrifices.

ABSTRACT

The High-Pressure High-Temperature (HPHT) sintering is an established process for reclaiming Waste Tire Rubber (WTR) into Magnetorheological Elastomers (MREs). Even though the WTR is generally recycled to other products, the usage of WTR as the main matrix of MRE is a new and novel concept. Therefore, this research focuses on studying the physicochemical and viscoelastic properties of the WTR based MRE produced through HPHT process. The WTR, carbonyl iron particles, and additives were mixed and compacted by applying simultaneous temperature and pressure at 200^{o}C and 25 MPa, respectively. Swelling test, morphological examination, infrared spectroscopy, magnetization, and thermal analysis were among the physicochemical properties studied. Meanwhile, the magneto-induced viscoelastic properties were assessed through shear mode test in both steady and dynamic conditions. The highest degree of reclamation based on swelling test, achieved up to 54 % confirming that crosslinking occurred during reclamation process. The dispersion of the magnetic particles were examined through Scanning Electron Microscopy (SEM) and the morphology of the fractured matrix indicated that the WTR blended well without any grain boundaries of uncured WTR. The highest magnetization saturation was achieved at 76.079 emu/g. While, the infrared spectroscopy identified rubber substances including synthetic and natural rubbers based on the band characteristics. Additionally, the thermogram patterns and decomposition rates of the samples also approved the matrix composition. The glass transition temperatures were also measured at -60.6 ±0.5^{o}C showing conformity with the reclaimed pure WTR. The WTR based MRE achieved maximum static stress ranging from 9 to 13 kPa (at 700 mT) with Linear Viscoelastic (LVE) region above 3% strain amplitude. The MRE exhibited MR effect up to 24 % with the range of storage modulus between 0.6 to 0.74 MPa (at 700 mT). Based on the examination results, the WTR based MRE demonstrated acceptable physicochemical characteristics and presented outstanding viscoelastic properties for future potential applications of MREs.

ABSTRAK

Proses pensinteran Tekanan-Tinggi Suhu-Tinggi (HPHT) merupakan proses terkenal untuk tebus guna Sisa Tayar Getah (WTR) menjadi Elastomer Reologi Magnet (MRE). Walaupun WTR secara umumnya dikitar semula kepada pelbagai produk lain, penggunaan WTR sebagai matriks utama di dalam MRE adalah satu idea baru dan novel. Oleh itu, penyelidikan ini memberi tumpuan kepada sifat-sifat kimiafizik dan likat anjal MRE berasaskan WTR yang dihasilkan melalui proses HPHT. Campuran WTR, serbuk besi karbonil, dan bahan tambah telah dicampur dan dipadatkan pada suhu dan tekanan serentak pada 200^{o}C dan 25 MPa. Ujian pembengkakan, pemerhatian morfologi, spektroskopi inframerah, pemagnetan, dan analisis haba adalah antara sifat kimiafizik yang dikaji. Sifat likat anjal magnetik pula dinilai melalui ujian mod ricihan di dalam keadaan statik dan dinamik. Ujian pembengkakan menunjukkan tahap tertinggi tebus guna mencapai 54% yang mengesahkan pautan silang berlaku semasa proses tebus guna. Serakan partikel magnet diperiksa menggunakan Mikroskop Imbasan Elektron dan permukaan patah menunjukkan WTR bercampur baik tanpa wujudnya sempadan antara dua domain dari WTR yang belum ditebusguna. Ketepuan pemagnetan tertinggi telah dicapai pada 76.069 emu/g. Manakala, spektroskopi inframerah telah mengenal pasti getah sintetik dan asli berdasarkan penemuan ciri jalur. Disamping itu, pola termogram dan kadar penguraian sampel juga mengesahkan komposisi matriks. Suhu peralihan kaca juga telah diperolehi pada -60.6 ±0.5 ^{o}C yang mematuhi tebus guna WTR tulen. MRE berasaskan WTR mencapai tegasan statik maksimum pada julat dari 9 kepada 13 kPa (pada 700 mT) dengan kawasan Likat Anjal Lelurus (LVE) melebihi ketegangan amplitud 3%. MRE ini mempamerkan kesan MR setinggi 24% dengan julat modulus simpanan di antara 0.6 kepada 0.74 MPa (pada 700 mT). Berdasarkan keputusan kajian, MRE berasaskan WTR menghasilkan ciri-ciri kimiafizik dan likat anjal yang amat baik untuk aplikasi MRE yang berpotensi pada masa hadapan.

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LIST OF SYMBOLS

d_{MREs} - density of MRE (g/cm^3)
d_W - density of pure reclaimed rubber (g/cm^3)
d_{MP} - density of magnetic powder (g/cm^3)
ø - volume fraction (vol.%)
SF - Soluble fraction (%)
m_1 - Initial mass (*g*)
m_2 - Swollen mass (*g*)
m_3 - Dried mass (*g*)
Q - Swelling degree (%)
v - Crosslink density (mol/cm^3)
V_r - Volume fraction in the swollen sample (vol.%)
V_1 - Molar volume of toluene (cm^3/mol)
χ - Parameter of rubber-solvent interaction
m_e - Mass of dry MRE (*g*)
ρ_e - Density of dry MRE (g/cm^3)
m_t - Mass of absorbed toluene (*g*)
ρ_t - Density of toluene (g/cm^3)
v_0 - Crosslinking density before reclamation (mol/cm^3)
v_1 - Crosslinking density of reclaimed WTR (mol/cm^3)
M - Magnetic moment / magnetization saturation (*emu/g*)
H - Field intensity (*G*)
μ_r - Relative magnetic permeability (*H/m*)
μ - Magnetic permeability (*H/m*)
μ_0 - Magnetic permeability in vacuum space (*H/m*)
B - Flux density (*T*)
τ - Shear stress (*Pa*)

G or G^* - Complex shear modulus (*Pa*)

γ - Shear strain (%)

η - Dynamic viscosity (*Pa s*)

λ - Relaxation time (*s*)

γ_a - Shear strain amplitude (%)

τ_a - Shear stress amplitude (*Pa*)

δ - Phase angle (*rad*)

G' - Storage modulus (*Pa*)

G'' - Loss modulus (*Pa*)

$\tan\delta$ - Loss factor

LIST OF ABBREVIATIONS

ATR	-	Attenuated total reflectance
$BaFe_{12}O_{19}$	-	Barium ferrite
BIIR	-	Bromobuthyl rubber
CI	-	Carbonyl iron ($Fe(CO)_5$)
CIIR	-	Chlorobutyl rubber
CNTs	-	Carbon nanotubes
CO	-	Carbon monoxide
CSM	-	Chlorosulphonated monomer
CTE	-	Coefficient of thermal expansion
DCP	-	Dicumyl peroxide
DMBH	-	Double methyl hexane
DOP	-	Di-2-ethylhexyl phthalate
DSC	-	Differential scanning calorimetry
DTG	-	Differential thermogravimetric
ESR	-	Electron spin resonance
Fe_3O_4	-	Magnetite
FESEM	-	Field emission scanning electron microscopy
FTIR	-	Fourier transform infrared
HAM	-	High amplitude modulus region
HPHT	-	High-Pressure High-Temperature
HTV	-	High temperature vulcanization
KBr	-	Kalium Bromide
LAM	-	Low amplitude modulus region
LVE	-	Linear viscoelastic
Magpols	-	Magnetic polymers
MAPs	-	Magnetoactive polymers
MGP	-	Magnetic gradient pinch

MR	-	Magnetorheological
MRE	-	Magnetorheological elastomer
MRFs	-	Magnetorheological fluids
MRVEs	-	Magnetorheological visco-elastomers
$Nd_2Fe_{14}B$	-	Neodymium-iron-boron
NH_4HCO_3	-	Ammonium bicarbonate
NO_2	-	Nitrogen dioxide
NR	-	Natural rubber
OOT	-	Oxidative onset temperature
PAH	-	Polyaromatic hydrocarbons
PDF	-	Pressure driven flow
PDMS	-	Polydimethylsiloxanes
PSA	-	Particle size analyzer
PU	-	Polyurethane
RTV	-	Room temperature vulcanization
SBR	-	Styrene butadiene rubber
SEM	-	Scanning electron microscopy
SMEs	-	Soft magnetic elastomers
SO_2	-	Sulfur dioxide
$SrFe_2O_{19}$	-	Strontium ferrite
TGA	-	Thermogravimetric analysis
TMA	-	Thermomechanical analysis
VRTM	-	Vacuum resin transfer molding
VSM	-	Vibrating sample magnetometer
WTR	-	Waste tire rubber

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Magnetorheological (MR) materials have been attracting the attention of researchers for many years. The penetration of this smart material into some applications makes the functionality of the devices becomes more adaptable for user needs [1]. Adaptability to the desired parameter is mainly due to their capability to change properties upon the penetration of magnetic fields inside the materials. MR materials are constructed of at least two different states of components, typically magnetizable filler and non-magnetic carrier. Additional materials are usually incorporated for specific purposes. The state of MR materials depends on the matrix forms such as fluids, viscoplastic, or viscoelastic. Therefore, the name for each MR material is also matched with the matrix state such as MR fluids (MRFs) for fluid based, MR gels/grease/plastomers for viscoplastic, as well as MR elastomers for viscoelastic matrices.

Viscoelastic matrix based MR materials have been studied intensively in last twenty years. Researchers have developed specific terminologies for various features. The term magnetoactive elastomers have been used by several research groups [2–6]. Other researchers have coined the terms ferrogels [7–9], magneto-active polymers (MAPs) [10], elastomer-ferromagnetic composites [11], magnetorheological visco-elastomers (MRVEs) [12], soft magnetic elastomers (SMEs) [13–15], ferromagnetic elastomer composites [11], and magnet filler-polymer composites or Magpols [16]. Most researchers use the term magnetorheological elastomer (MRE) referred to their functional behavior that is rheological properties. Therefore, the term “MRE” will be used as a reference name in this thesis.

MRE is a type of magnetically-actuated smart material in which their rheological properties such as storage and loss moduli, loss tangent (loss factor/damping factor) as well as physical properties such as thermal conductivity, electrical capacitance, sound absorption properties, magnetostriction, and resistivity, can be altered in a fraction of milliseconds by applying magnetic fields [17]. Both rheological and physical responses of the MRE are referred to in the foundation of devices design and development. For instance, the tunable rheological properties are relevant for vibration absorber since the field dependent modulus affects both stiffness and damping characteristics. Meanwhile, the physical properties are applicable for sensory development according to the change of resistance, capacitance and thermal reluctance. Implementations of MRE in vibration isolation, sensors, as well as actuators have been patented and disseminated in several papers. Typical applications in vibration absorber include variable stiffness bushing [18,19]; propeller shaft [20]; variable spring rate [21] and prosthetic leg [22]. MRE applications for sensory use are tire pressure control [23] and MEMS [24]. Meanwhile, solicitation in the active actuator was releasable attachment [25] and active morphing composites [16,26].

A growing amount of research on MREs has addressed the enhancement of rheological properties such as the magneto-induced stiffness and damping. The MRE property enhancements could be manifested in the following ways: a) controlling the particle types, size, morphology, and composition; b) modifying crosslink density through additives and processing conditions, and c) manipulating the matrix either by using a single type of matrix or a hybrid matrix. The mentioned strategies have been highlighted from the previous studies based on the fact that the MRE properties are mainly influenced by the matrices, magnetizable particles, and some other parameters.

Magnetizable particles as fillers play an important role, since this part directly interacts with external magnetic fields. Therefore, the magnetizable particles employed in MRE should follow some criteria such as high magnetization saturation and low remnant magnetization. The structure of magnetizable particles inside the MRE influences performance. According to the type of particle dispersion, MREs can be grouped into isotropic type and anisotropic type. The isotropic MREs have random distribution inside the matrix, and it can be provided by mixing the matrix and magnetizable particles and then curing the blended materials without magnetization treatment. Meanwhile, the anisotropic MREs require treatment during crosslinking of the materials so that the particles cluster can be formed and locked at the fixed position.

Among the various type of ferrous particles, the carbonyl iron (CI) particles are the most interesting of the soft magnetic materials, compared to iron sand [27,28] and magnetite [29]. A higher level of CI contents, for instance, 90 wt% [30], was proposed to increase the MR effect; however, this effort was limited to the problem that the higher particle content makes the more brittle MRE. Another approach to achieve the better MRE properties is modifying the particle surfaces such as particles surface grafting or coating [27,31,32]. Insertion of hard magnetic materials ($BaFe_{12}O_{19}$, $SrFe_2O_{19}$, $Nd_2Fe_{14}B$) [33,34] and bimodal particles [35,36] are also considered as alternative ways for better MRE response to external magnetic fields. Moreover, to attain a reasonable MR effect with a lower content of magnetizable particle, 20 wt% ferromagnetic $FeCo_3$ nanoparticles [37] was introduced. Locking magnetic particles within the MRE matrix is also believed to boost the MR effect up to about 700% relative MR effect [38]. Playing with particle contents and clustering particles are the most influence factor for particles optimization in MREs. However, the optimum particle content inside the MREs has been reported about 30 vol% [39]. Besides, the clustering particles would be effective only for a liquid-based matrix (such as silicone rubber).

The other key role in enhancing the MRE performances is improving crosslink quality through additive manipulation. The additives can enhance magneto-induced not only mechanical but also electrical properties. For instance, introducing graphite [40,41] and other conductive metals such as nickel [42] improves electrical properties. Addition of silicon carbide [43] and carbon black [44,45] have proven the chemical property augmentation of the matrix, thus improving the mechanical performances of the MRE. Modifying sulfur content as a curing agent [46] and plasticizer [47] could improve the crosslink density. Hence, the MR effect was getting better at a particular content. The obtained improvements, especially MR effect and thermal stability, were reasonable but less significant. In industrial applications, such efforts have not made substantive progress until now as a result of comprehensive bad performance [48].

Apart from the magnetic particles and additives, the researchers have also widely investigated different non-magnetic matrices for MREs such as silicone, rubber (natural and synthetic), and other synthetic elastomers. Based on the material mapping of MREs, silicone room temperature vulcanization (RTV)-based MREs have accounted for more than 50% of the studies and development due to the flexibility and ease of both isotropic and anisotropic fabrication. Likewise, the use of thermoplastic and thermoset elastomers has also been great interest due to easier early stage process. The required modulus of the MRE at off-state condition may be realized by changing the raw material ingredients. However, the fabrication cost of thermoplastic/thermoset based MREs is relatively higher than other matrix types [49]. Meanwhile, unsaturated elastomer (natural and synthetic rubbers) based MREs have generated less interest because of the harder mixing of the raw materials and the pre-structuring (magnetization) process compared to the mentioned matrix previously. Several researchers have proposed single type unsaturated elastomers for MREs, such as natural rubber [45,50–53], synthetic rubber [54–56], as well as hybrid rubbers [57–59] according to the desired properties such as good mechanical and aging properties and sources availability. Various types of the elastomeric matrix can be selected for MRE fabrication compared to the magnetizable particles. Therefore, the opportunity to develop a new class MRE from the matrix point of view is always widely opened.

1.2 Motivation of Study

In last two decades, researchers have developed the MRE in a wide variety of matrices due to the influence of matrix types on the viscoelastic properties. Note that the expansion of MRE fabrication based on the type of matrix remains a great opportunity due to the tremendous types of elastomeric matrix such as silicone rubber, natural rubber, synthetic rubber, as well as other thermoplastic polymers. Nevertheless, the development of MRE materials is currently less considering environmental aspect. This smart rubber development would be another potential contributor for increasing number of waste rubber. Since, the establishment of rubber based devices always concerns on the environmental issues [60], the research in MREs should consider another point of view to meet the green technology development issues. Along with the advanced technology on waste rubber reclamation, the use of waste rubber in MREs is a respectful reason for future investigation. It is due to the fact that the number of waste rubber has been growing in a big number nowadays [60].

The amount of waste rubber growth cannot be exactly determined. However, it can be approached from the number of tire rubber production, since the major contributor of waste rubber sources from the waste tire rubber [61–63]. The demand for tires increases approximately 4.2% annually through 2015 [64]. For instance, in Indonesia, the amount of waste tire rubber (WTR) is seriously increasing due to the fast growing vehicle sales year by year as can be seen in Figure 1.1. The abraded rubber from the tire is less than approximately 1% [61], therefore, the number of discarded tire is nearly equal to the produced tires. So far, WTR has been recycled for tire retreading, recovering functional elements such as zinc, activated carbon, carbon nanotubes, and heavy metals [65], functional composites (particle board, asphalt and concrete mixtures), as well as energy recovery through clinker burning [66] or pyrolysis for liquefied fuel [67]. However, these strategies have other negative effects, such as a higher rate of road accidents caused by retreaded tires [68] or community health and environmental degradation (air pollution) triggered by the release of toxic gasses such as CO, SO_2, and NO_2 as well as mono- and polyaromatic hydrocarbons (PAH) [69,70]. Besides, waste rubber is usually utilized as a secondary material that does not enhance the functional value of the recycled articles.

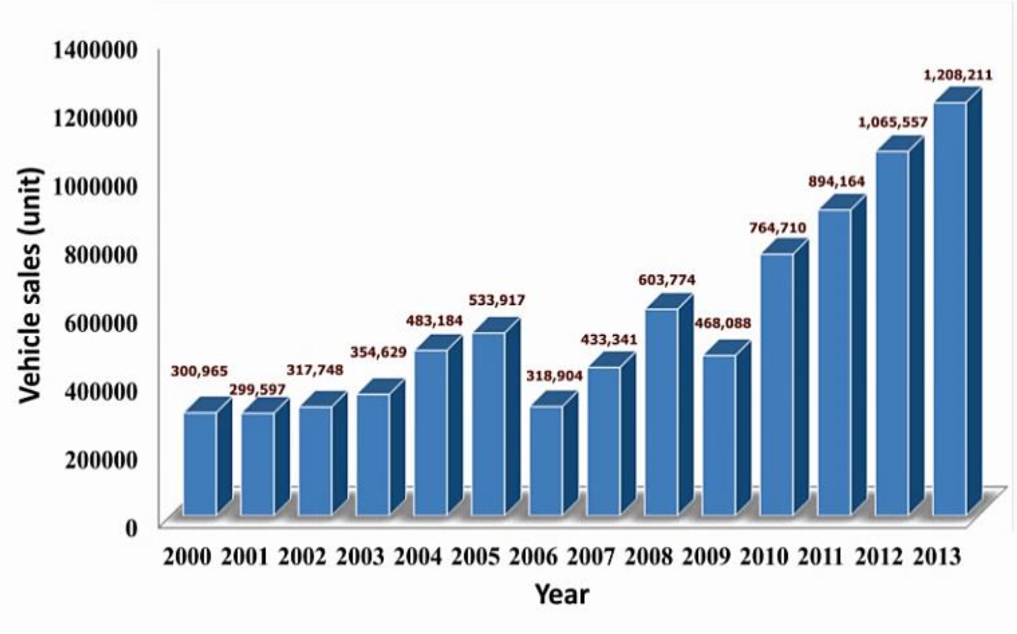

Figure 1.1 Annual vehicle sales in Indonesia (2000-2013).

The idea of using WTR as MRE matrix would be a strategic way to enhance the functional value of the recycled product. Due to the different state of polymer, the common manufacturing method of MREs cannot handle the curing of WTR. According to the literature in [17], fabrication for virgin rubber based MREs is normally conducted in the room to moderate curing temperature. For saturated elastomer such as silicones and polyurethanes in which the uncured state is in viscous form, the curing process takes the temperature process below 100^{o}C and without applying a particular pressure. Meanwhile, the unsaturated elastomers such as natural and synthetic rubbers, the vulcanization mechanism took place under applying curing temperature and pressure, whereas, the curing temperature and time depends on the type of rubber and amount of additives.

The latest finding related to the vulcanizing issue was about self-crosslinking between two different compounds. Wang et al. [48] fabricated a new class of MREs by blending polychloroprene rubber with epoxidised natural rubber without curing agents, so-called self-crosslinking MRE. This work indeed provided a breakthrough in the preparation of MRE since there was no additive involved in the vulcanization process. The curing process of the MRE also conducted under the equal routine. ENR bears epoxy groups, which have high reactivity, while polychloroprene rubber (CR) also has reactive chlorine groups. They can crosslink together at high temperature by themselves without a crosslinking agent. Additionally, CR/ENR self-crosslinking blends are made of two constituents, which are beneficial to the molecular design of new materials. Therefore, the vulcanization mechanism of virgin rubber based MREs is not a serious issue as long as the curing process follows the common procedures.

Differing from virgin rubber-based MREs, the incorporation of WTR as the main MRE matrix requires specific curing treatment according to the built three-dimension chemical network beforehand. Reclamation of WTR is usually performed by blending WTR with virgin rubber or by revulcanizing the dead rubber by treating it in physical and chemical ways [71]. For the case of rubber reclamation without incorporation of virgin rubber, the most appropriate methods were developed by Morin et al. [72] and Tripathy et al. [73]. This group proposed reclamation method to reclaim tire rubber based on supporting research by Tobolsky et al. [74–77]. Departing from fundamental theory of rubber scission and reformation, Morin et al. [72] discovered that HPHT sintering successfully reclaims WTR without the incorporation of virgin rubber. In this process, the reclaimed rubber yields about 35 to 40% recovery of the original rubber. The mechanical properties of that process were improved by Tripathy et al. [73] by incorporating phthalimide, raising the recovery yield to about 75% of the original rubber. Based on the successful of High-Pressure High-Temperature (HPHT) sintering, the idea of utilizing WTR as a primary matrix of MRE seems promising for further development.

Based on the aforementioned facts, fabrication of MRE based on WTR would be interesting from many aspects. Firstly, waste rubber is usually difficult to be vulcanized without presentation of virgin rubber. By taking advantage of current rubber reclaiming technology, waste rubber could be converted into MRE. Secondly, it is convinced that the utilization of WTR in MRE is a useful idea for waste tire management as well as another way to discover environmental friendly smart materials. Furthermore, it is predicted that this breakthrough will enhance the economic value of the recycled product. Since nowadays, eco-friendly rubber products are encouraged to minimize the waste rubber problem as well as economically reduce the production cost [60,78]. Therefore, comprehensive assessment on the MRE based on WTR as the main matrix would be an interesting step in realizing this pioneer notion.

1.3 Research Objectives

The aim of this study is to discover a new class MRE by introducing WTR as the main matrix. This research embarks on the following intentions, accordingly:

(a) To fabricate the MRE based on WTR as the main matrix through rubber reclamation process namely High-Pressure High-Temperature (HPHT) sintering using readily conventional equipment.
(b) To characterize the physicochemical properties of the MRE, including vulcanization achievement, morphological, magnetic, spectroscopy, and thermal properties.
(c) To evaluate the stress – strain responses of the MRE to the magnetic fields under steady state loadings.
(d) To analyze the rheological properties of MRE under dynamic shear loading in absence and presence of magnetic fields, various strain amplitudes, and excitation frequencies.

1.4 Research Scope

The scope of research is specified on the experimental investigation of WTR based MRE fabrication as well as a fundamental characterization to confirm the feasibility of using WTR as the main matrix without virgin elastomers. The scope includes:

(a) The ingredients of the WTR based MRE especially the amount of additives were determined based on the common process of waste rubber reclamation [61]. Therefore, the optimum composition of raw materials is not the main focus of this study.

(b) The parameters of fabrication those are temperature and compression force followed the optimum decision from previous literature [72,73].

(c) The physicochemical properties of the MRE including swelling test through immersion technique, morphological observation within the specimen, infrared spectroscopy, and thermal analysis were conducted in the absence of magnetic fields.

(d) The viscoelastic properties of the MRE are analyzed only in shear mode and under ambient temperature.

(e) The rheological experiment in shear mode is studied under steady state and oscillatory or harmonic loadings. The stress-strain relationship obtained from the steady state test is considered as static properties of the MRE. Meanwhile, the storage modulus, loss modulus, and loss factor have been studied under several conditions: ramp magnetic fields, ramp frequency, and ramp strain. The dynamic/oscillatory test excluded the reverse treatment usually conducted for hysteresis properties of MREs [79].

1.5 Dissertation Outlines

This thesis consists of six chapters. Each chapter provides the highlights of related information and ends with a summary of achievement and findings. The outline of the chapters is accordingly:

Chapter two : Chapter two is a literature review related to previous research in last two decades on to the development of MREs. The literature searches cover MRE materials, preparations, characteristics, and applications. The materials used to date are mapped and marked based on the level of interest. Several fabrication methods are highlighted. Potential applications that have been patented or published scientifically are summarized into three classifications: vibration absorber or tunable vibration absorber, sensory system, and actuator system.

Chapter three : Chapter three contains the experimental section. In this chapter, a research scenario is presented to give a brief explanation about step by step activities during project completion to achieve the objectives. This chapter also describes the materials and detail fabrication through HPHT sintering. The experimental setup involved in the MRE characterization including physicochemical and rheological examinations are also clearly explained, including details of the facilities.

Chapter four : Chapter four describes the physicochemical characterization and discussion. It covers swelling analysis, morphological observation, infrared spectroscopy, magnetic properties, and thermal analysis covering thermogravimetric analysis, differential scanning calorimetry, and thermomechanical analysis. Discussion encompasses the compatibility of reclamation between WTR and magnetizable particle based on the physical and chemical characteristics.

Chapter five : Chapter five discusses the rheological examination results including discussion on the viscoelastic properties of the WTR based MRE in shear working mode. The discussion features both the quasi-steady and quasi-dynamic loadings. The stress-strain relationship of the MRE under static loading is explained. The viscoelastic properties of the MRE under off and on states from the shear dynamic test are discussed specifically.

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