RHEOLOGICAL AND ELECTRICAL PROPERTIES OF COBALT-BASED MAGNETORHEOLOGICAL ELASTOMER

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DEDICATION

Dear self,

One day, if you are given a long life, do revisit this page once in a while so it's become a reminder of where you begin.

Please remember how strong Emak and Almarhum Abah supported and du'a for you, how Mawar stands by you, how Kakak, Azim, Adik and Baby are always there for one another, how many people around you helped you to get up, remember that you were never alone. Never let go of this one religion and be steadfast in your deeds, even if you do not achieve what you want in this world, at least you try to excel in His eyes. Be humble, never be arrogant and forgetting where you come from and never look down or disrespect those who are behind you. We are all khalifah and has our own responsibilities dictated by Him.

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ABSTRACT

In most studies, carbonyl iron particles (CIP) were merged with carbon-based particles such as graphite (Gr) or carbon black (CB) particles as fillers to enhance the electrical properties of magnetorheological elastomers (MRE). Although the electrical properties were improved, excessive implementation of particle in MRE led to brittle phase which caused decrement of properties such as elasticity. Hence, this study examined a single material, cobalt particles, as a filler to enhance the rheological and electrical properties in MRE. The selection of cobalt particles is due to its dual properties – magnetic and electrical. A total of three MREs containing 53, 60 and 67 wt% of cobalt were fabricated through mixing and curing processes. Characterization related to physicochemical properties of MRE samples was analysed by using X-Ray diffraction (XRD), energy-dispersive x-ray spectroscopy (EDX), field emission scanning electron microscopy (FESEM) and vibrating sample magnetometer (VSM). Then, the rheological properties of the MRE in various strengths of magnetic field intensity between 0 to 0.8 T were conducted by using a rheometer. Subsequently, the effect of the cobalt on the electrical properties was investigated and compared with different applied forces towards the MRE. The physicochemical properties indicate the presence of cobalt has influenced the rheology and electrical properties of the MRE. Both properties were enhanced with the increase of cobalt content that embedded in the silicone matrix. Even though, the initial storage modulus of MRE increased from 0.28 to 0.52 MPa, the magnetorheological (MR) effect has enhanced from 57.14% to 82.69%. On the contrary, the MRE resistance decreased when increasing the applied force from 1 to 10 kg. In sum, the findings show a higher cobalt content in MRE contributed to a higher MR effect, and simultaneously lower the electrical resistance. The finding suggests the potential of cobalt particles as a filler in the MRE fabrication for future sensing applications.

ABSTRAK

Dalam kebanyakan kajian, partikel besi karbon (CIP) digabungkan dengan zarah berasaskan karbon seperti zarah grafit (Gr) atau karbon hitam (CB) sebagai pengisi untuk meningkatkan sifat elektrik pada Elastomer Reologi Magnet (MRE). Walaupun sifat elektrik ditambah baik, zarah berlebihan di dalam MRE membawa kepada fasa rapuh yang menyebabkan penurunan sifat seperti keanjalan. Oleh itu, kajian ini mengkaji bahan tunggal, iaitu zarah kobalt, sebagai pengisi untuk meningkatkan sifat reologi dan elektrik di dalam MRE. Pemilihan zarah kobalt adalah kerana dwi-sifatnya – magnet dan elektrik. Sebanyak tiga MRE yang mengandungi 53, 60, dan 67 wt% kobalt dibuat melalui kaedah proses pencampuran dan pengawetan. Pencirian yang berkaitan dengan sifat fizikokimia, MRE dianalisis dengan menggunakan difraksi sinar-X (XRD), spektroskopi sinar-X penyebaran tenaga (EDX), mikroskop elektron pengimbasan pelepasan medan (FESEM) dan magnetometer sampel bergetar (VSM). Kemudian, sifat reologi MRE dalam pelbagai kekuatan intensiti medan magnet antara 0 hingga 0.8 T dilakukan dengan menggunakan rheometer. Seterusnya, kesan kobalt pada sifat elektrik disiasat dan dibandingkan dengan daya yang berlainan terhadap MRE. Sifat fizikokimia membuktikan bahawa kehadiran kobalt telah mempengaruhi sifat reologi dan elektrik MRE. Kedua-dua sifat telah dipertingkatkan dengan peningkatan kandungan kobalt yang terdapat di dalam matriks silikon. Walaupun modulus penyimpanan awal MRE dilihat menaik dari 0.28 MPa hingga 0.52 MPa, kesan MR meningkat dari 57.14% kepada 82.69%. Sebaliknya, nilai rintangan berkurang apabila daya yang dikenakan meningkat daripada 1 kg hingga 10 kg. Oleh yang demikian, kandungan kobalt yang tinggi dalam MRE menyumbang kepada kesan magnetorheologi (MR) yang lebih tinggi dan sekaligus merendahkan nilai rintangan elektrik. Penemuan ini telah mencadangkan potensi zarah kobalt sebagai pengisi dalam fabrikasi MRE untuk aplikasi penderiaan pada masa akan datang.

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LIST OF ABBREVIATIONS

CB	-	Carbon Black
CIP	-	Carbonyl iron particle
EDX	-	Energy-dispersive X-ray spectroscopy
ENR	-	Epoxidized natural rubber
Fcc	-	Face-centered cubic
FESEM	-	Field emission scanning electron microscope
Gr	-	Graphite
Hcp	-	Hexagonal-closed packed
JCPDS	-	Joint Committee on Powder Diffraction Standards
LVE	-	linear viscoelastic region
MR	-	Magnetorheological
MRE	-	Magnetorheological elastomer
MRF	-	Magnetorheological fluid
MRG	-	Magnetorheological grease
MRP	-	Magnetorheological plastomer
NR	-	Natural rubber
PDMS	-	Polydimethylsiloxanes
PU	-	Polyurathane
RTV	-	Room temperature vulcanized
SR	-	Silicone rubber
VSM	-	Vibrating sample magnetometer
XRD	-	X-ray diffraction

LIST OF SYMBOLS

A	-	Ampere
B	-	Flux density
emu	-	Electromagnetic unit
Fe	-	Iron
G’	-	Storage modulus
G"	-	Loss modulus
G*	-	Complex modulus
G_0	-	Initial modulus
G_{max}	-	Maximum modulus
g	-	gram
Hc	-	Coercivity
M	-	Magnetization
MPa	-	Megapascal
Mr	-	Remanence
Ms	-	Magnetic saturation
NdFb	-	Neodymium
Ni	-	Nickel
Oe	-	Oersted
wt%	-	Weight percentage
μ_r	-	Relative magnetic permeability
μ	-	Magnetic permeability
δ	-	Phase angle
ΔG	-	Absolute MR effect
tan δ	-	Loss factor
σ	-	stress
ε	-	strain
ω	-	Viscoelastic materials
˚C	-	Degree celcius

CHAPTER 1

INTRODUCTION

1.1 Introduction

Magnetorheological (MR) materials fall under the class of smart materials due to their controllable rheological properties. Their rheological properties can be changed continuously, quickly and reversibly with the presence of a magnetic field, which makes this material of great interest due to its fast real-time response. Recently MR materials such as MR fluids (MRF) [1–3], MR grease (MRG) [4–6], MR foam [7–9], MR plastomer (MRP) [10–12], and MR elastomer (MRE) [13–16] have been widely researched in both industrial and academic areas as potentials materials for vibration/impact management or position control [17,18].

In recent years, MRE has attracted the most interest among those MR materials, since MRE shows a desirable performance due to the stability of magnetizable particles in the matrix and simpler material handling such as no leakage and no sedimentation [19]. MRE is a kind of composites material that mainly consists of rubber matrix and magnetic particles. Elastomer such as rubber has been used as a matrix to mitigate shock and vibration in structures, vehicles, and other types of machinery due to its high and reversible deformability. Few types of rubber such as natural rubber (NR) [20–23], silicon rubber (SR) [24,25], thermoplastic [26,27], and even hybrid matrix [28] have been used in the manufacture of MRE. It is believed that rubber's modulus properties are constant throughout any applied magnetic field due to the non-magnetic nature of rubber. The interaction of the particles will therefore be more effective and, the MR effect will be greater. The MR effect of soft rubber-like SR based on MRE, for example, can reach as high as 500 %, while the thermoplastic MR effect is only 70 % [29,30].

Carbonyl iron particles (CIP) [31–34] have been the most widely used magnetic particles in MRE fabrication to date because they have high magnetic permeability and saturation, but low in magnetic remanence. Because of these advantages, the MR group, including MRE, has become actively tuned and quick to respond to the applied magnetic field. The amount of magnetic particles used in MRE fabrication also had a significant impact on the MRE's final performance; particularly related to the MR effect. Previous studies have reported that the use of magnetic particles is typically in the range of 10-90% by weight. Even though higher particle loading is known to lead to a greater storage modulus and MR effects, however, some drawbacks like brittle issues [35], will occur which limit the use of MRE in some potential applications like in sensors, automotive and rehabilitation [36–38]. Therefore, several attempts also have been made in order to enhance the mechanical, rheological as well as electrical properties of MRE by the introduction of additives [39–42]. Additive helps in property enhancement, cost reduction, reinforcement and processing improvement [43–50]. A variety of additives including graphite (Gr) [51], graphene [52], silica [53] and carbon black (CB) [54,55] have been utilized and the final performance of MRE, on the other hand, was found to be directly dependent on several factors, including matrix-particle interaction, particle-particle interaction, matrix and particle types, and particle distribution within the MRE.

In addition, MRE can be classified into two groups which are, isotropic MRE and anisotropic MRE. The difference between isotropic and anisotropic MRE is the distribution of the magnetic particle in the MRE. For anisotropic MRE, it has a chain-like structure in a matrix due to the curing condition under an applied magnetic field. As for the isotropic MRE, the curing condition is without the presence of a magnetic field, which resulted in random particle distribution in the matrix. It has been known that these groups have different rheological properties of MRE [56–58]. Due to this fact, anisotropic MRE has closer gaps between the magnetic particle, as compared to the isotropic MRE. Thus, a much higher MR effect can be obtained, which resulted from strong particle interactions. The primary parameters describing the rheological properties of MREs are the shear storage modulus (G') and loss factor or tan δ. The energy storage capacity of the viscoelastic material is referred to as G'. The loss factor, meanwhile, refers to the damping property of the material due to the dissipation of energy in the MRE materials.

1.2 Motivation of Study

The previous studies thus far have contributed to the knowledge concerning the unique functions of MRE that enable them to have sensitive mechanical and electrical properties, which can be changed under external stimuli, such as magnetic field and mechanical pressure. Ghafoorianfar et al. [59] investigated the electrical properties and obtained that the resistivity value of MRE ranging from 4.41 X 10^{29} to 1.7 X 10^{14} Ω mm by varying particle volume fractions under different compression mechanical loads. In order to enhance the electrical properties of MRE, a common approach is to combine carbon-based particles such as Gr, graphene, and carbon black with the CIP. Schûmann et al. [60] analysed an electroconductive MRE (mixed CIP with carbon black) that exhibited a highly complex resistive behavior. The results revealed that the resistance declined exponentially from 2300 kΩ to 1200 kΩ within the region of linear elasticity. In another study, Tian et al. [61] utilized various weight fractions of Gr powder (0-5g) and mixed with CIP in fabricating MRE, so-called Gr-MRE to achieve electrical conductivity. It has been observed that a high concentration of the Gr filler led to an improvement in the MR effect. The conductivity of the Gr-MRE increased with the increment of applied force, which was up to 1 kg. Later, Shabdin et al. [35] continued to investigate the electrical properties of isotropic and anisotropic MRE by adding 33 wt% Gr powder as a filler with 20 wt% of CIP. The changes of Gr-MRE conductivity had a relationship with the external force up to 10 kg in certain magnetic field intensity. However, although the conductivity of MRE could be improved by increasing the Gr particles, excessive use of particles or fillers, in MRE often led to decrement of some other properties such as elasticity. Moreover, the distribution of particles was more challenging, which led to a brittle phase and decrement of the field-dependent modulus of MRE [35,61].

Nevertheless, several studies have introduced cobalt as magnetic particles in MRE. The uniqueness of cobalt, which offered dual properties; magnetic and electrical properties are believed can supersede the conventional combination of CIP and Gr, as a filler. At such, Tong et al. [62] studied the interaction that occurred in MRE with different shapes of cobalt filler; spherical and flower-like. The result demonstrated that the flower-like shape cobalt particles exhibited a higher magnetic up to 0.1 T and enhanced the rheological performance as compared to the spherical shape. This phenomenon happened due to particle shape that led to a higher crosslink density. However, their work had focused on rheology properties and a lack of supporting data towards the electrical properties. Therefore, a comprehensive investigation to analyse the magnetic, MR effect and electrical behavior of various compositions of cobalt filler is worth investigating.

1.3 Problem Statement

From the previous studies, CIP was merged with carbon-based particles such as Gr or CB as a filler to provide electrical conductivity to the MRE. Although this combination of fillers improves the conductivity, excessive used of particles often results in loss of other properties such as elasticity. Furthermore, it resulted in a decrement in MRE's field-dependent modulus. In fact, the relationship between a variety of properties, including rheological and electrical properties had not been thoroughly investigated. Therefore, some other magnetic particles that exhibit dual properties; magnetic and electrical like cobalt particles can be used to further enhance the properties of MRE.

1.4 Research Objectives

The main aim of this research is to investigate the MRE performance by introducing cobalt as a magnetic particle. Therefore, the objectives of the research are:

I. To characterize the physicochemical properties of the MRE in terms of structural observation and magnetic properties of MRE.
II. To analyse the rheological and electrical properties of MRE with cobalt at off- and on-state conditions.
III. To evaluate the effects of magnetic field and particles content on rheology and electrical behaviour of MRE.

1.5 Research Scope

The scope of this study is specified on the experimental investigation on the rheological and electrical properties of MRE embedded with cobalt. The scope of this study includes:

I. The fabrication of MRE using various compositions 53, 60 and 67 wt% of cobalt particles.
II. The microstructural, elemental analysis and magnetic performance of MRE are performed using FESEM, XRD, EDX and VSM.
III. The rheological test of MRE in the absence and presence of a magnetic field was examined under oscillatory shear mode. The tests are included input parameters of strain sweep, frequency sweep and magnetic field sweep.
IV. The correlation between change in MRE resistance at different applied forces under off- and on-state conditions was analysed using a custom made test-rig.

1.6 Thesis Content

The layout of this thesis is as follows:

Chapter 1 introduces the research idea by providing the background of research, motivation and problem statement that clearly identifies the research gap, research objectives and research scope.

Chapter 2 contains a literature review related to the research work of MR materials, on what has been investigated before and the evidence on how the research is generally conducted in this area. Generally, this chapter summarizes relevant outcomes to the research area by covering a brief history of morphological observation, magnetic measurement, rheological and electrical properties based on several previous studies.

Chapter 3 described step-by-step research activities to achieve the objective. This chapter also describes the materials and details of MRE fabrication. The experimental setup involved in the MRE characterization is described in detail.

Chapter 4 discusses the experimental results and analysis from the physicochemical, rheology and electrical properties of MRE. The physicochemical analysis is conducted by referring to the morphological observations and magnetic properties of MRE. Meanwhile, results of the rheological properties are discussed under oscillatory shear modes test, respective to strain amplitude, frequency and magnetic field sweep. Furthermore, the electrical properties are discussed based on the effect of the applied load on the MRE resistance under the absence and presence of the magnetic field.

Chapter 5 concludes the research work and highlights the contribution of this research as well as the recommendation for potential future research work.

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LIST OF PUBLICATIONS

Journal with Impact Factors

1. **Zainudin, A.A.**, Yunus, N.A., Mazlan, S.A., Shabdin, M.K., Abdul Aziz, S.A., Nordin, N.A., Nazmi, N. and Abdul Rahman, M.A., 2020. Rheological and resistance properties of magnetorheological elastomer with cobalt for sensor application. *Applied Sciences*, *10*(5),1638 **(Q2, IF: 2.474)**

2. **Zainudin, A.A.**, Mazlan, S.A., Shabdin, M.K., Aziz, S.A.A., Homma, K., Nazmi, N., Nordin, N.A., Noordin, A.F. and Rambat, S., 2021. Effects of magnetic field and particles content on rheology and resistivity behavior of magnetorheological elastomer with embedded cobalt particles. *Smart Materials and Structures*, *30*(5), 055002. **(Q1, IF: 3.613)**

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