

CURE CHARACTERISTICS AND ELECTRICAL
CONDUCTIVITY OF GRAPHENE NANOPATELETS
REINFORCED EPOXIDIZED NATURAL RUBBER: A
CRITICAL REVIEW

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UNIVERSITI TEKNIKAL MALAYSIA MELAKA
2020



**CURE CHARACTERISTICS AND ELECTRICAL CONDUCTIVITY
OF GRAPHENE NANOPATELETS REINFORCED EPOXIDIZED
NATURAL RUBBER: A CRITICAL REVIEW**

Submitted in accordance with the requirement of the Universiti Teknikal Malaysia Melaka
(UTeM) for the Bachelor Degree of Manufacturing Engineering (Hons.)

by

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Tajuk: **CURE CHARACTERISTICS AND ELECTRICAL CONDUCTIVITY OF GRAPHENE NANOPATELETS REINFORCED EPOXIDIZED NATURAL RUBBER: A CRITICAL REVIEW**


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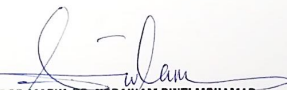
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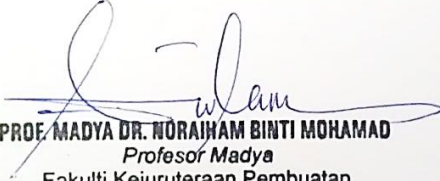
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APPROVAL

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ABSTRAK

Permintaan yang semakin meningkat untuk penggunaan bahan yang lebih lestari atau hijau dalam produk kejuruteraan telah mendorong para saintis dan jurutera untuk mengeluarkan alternatif untuk menggunakan penebat polimer secara meluas dalam aplikasi elektrik. Getah semula jadi yang terepoksida: terbitan daripada sumber semula jadi Malaysia menjadi calon yang berprestasi untuk aplikasi ini kecuali daya tahannya yang rendah terhadap haba dan ozon. Mereka bentuk formula getah semula jadi yang terepoksida dengan pemilihan pengisi yang sesuai merupakan penyelesaian untuk masalah ini. Objektif projek ini adalah untuk mengkaji ciri pematangan dan kekonduksian elektrik komposit getah asli diperkuatkan grafin nanokepingan. Hasil projek telah dibahagikan kepada 2 bahagian yang berdasarkan dapatan makmal dan dapatan postulatif. Pertama, komposit getah semula jadi yang diperkuatkan grafin nanokepingan disediakan dengan pembebanan pengisi 0, 0.5, 1 dan 3% berat menggunakan kaedah penyebatian lebur melalui pengadun dalaman Haake mengikut ASTM D 3192. Setelah dikompaun, sebatian tersebut kemudiannya diuji melalui Rheometer U-CAN Dynatex Inc UR2010 untuk menentukan ciri matang. Komposit kemudian divulkan dengan menggunakan mesin tekan panas pada suhu 150 ° C dan pada masa pengawetan T90 mengikut ASTM D 2084. Walau bagaimanapun, kekonduksian elektrik komposit itu didalilkan melalui tinjauan kritikal terhadap kertas penyelidikan yang mempunyai kaitan dan data kemudiannya dianalisis. Kekonduksian elektrik komposit yang dipostulat kemudian disokong oleh sifat morfologi, termal, komposisi dan struktur bahan dengan menggunakan kemikroskopian elektron imbasan (SEM), kalorimetri imbasan pembezaan (DSC), spektroskopi inframerah transformasi Fourier (FTIR) dan analisis pembelauan sinar X (XRD). Peratusan berat yang berbeza dari pengisian GNPs telah membuktikan bahawa pemuatan GNP mempengaruhi peningkatan ciri pengawetan komposit ENR / GNPs. Pemuatan GNP meningkat, masa *scorch* (T2) dan masa pengawetan (T90) juga meningkat dan mengakibatkan kelewatan proses pemvulkanan. Peningkatan tork minimum (ML) dan tork maksimum (MH) mewakili kelikatan dan kekuatan komposit. Ini

membuktikan bahawa penggabungan pengisi GNP meningkatkan ciri penyembuhan, kebolehprosesan sehingga menghasilkan kekuatan tervulkan yang baik. Julat pemuatan pengisi yang sesuai untuk komposit ENR / GNP sebagai penebat elektrik yang baik didalilkan dalam julat 0.2wt% hingga 0.5wt%, ini adalah ambang perkolasi postulatif komposit ENR / GNPs. Komposit ENR / GNP pada julat ini akan kekal sebagai penebat. Peningkatan kekonduksian elektrik membawa kepada peningkatan sifat pelepasan haba. Penguatan nanoplatelet graphene akan meningkatkan ketahanan haba komposit getah semula jadi yang teroksidasi melalui mekanisme pelepasan terma tanpa membahayakan terlalu banyak sifat penebat elektrik mereka. Penemuan ini akan bermanfaat bagi industri perkakas elektrik semasa.

ABSTRACT

Increasing demand for the use of more sustainable or green materials in engineering products has urged the scientist and engineers to come out with alternative for widely use polymeric insulators in electrical application. Epoxidized natural rubber: a Malaysian natural resource derivative seems to be a promising candidate for this application except for its low resistance towards heat and ozone. Designing the formula of epoxidized natural rubber with suitable selection of fillers would be the solution to this problem. The aim of this project is to investigate the cure characteristics and electrical conductivity of graphene nanoplatelets reinforced epoxidized natural rubber. The project result has been separated into 2 parts which are based on laboratory results and postulated results. Firstly, the graphene nanoplatelets reinforced epoxidized natural rubber composites were prepared with filler loading of 0, 0.5, 1 and 3 wt.% using a melt compounding method through a Haake internal mixer according to ASTM D 3192. Once compounded, the compound was then tested through a Rheometer U-CAN Dynatex Inc UR2010 to determine the cure characteristics. The composites were then vulcanized using a hot press machine at 150°C and at cure time of T90 following ASTM D 2084. However, the electrical conductivity of the composites was postulated through a critical review of closely related previous research papers and the data were then been analysed. The postulated electrical conductivity of the composites were further supported by the morphological, thermal, compositional and structural properties of the material by using existing scanning electron microscopy (SEM), differential scanning calorimetry (DSC), fourier transform infrared (FTIR) spectroscopy and X-ray diffraction (XRD) analyses data. Different weight percentage of the GNPs filler loading has proven that the GNPs loading affects improved the cure characteristics of ENR/GNPs composite. GNPs loading increase, the scorch time (T2) and cure time (T90) hence increase and result in vulcanisation process delay. The increase in minimum torque (ML) and maximum torque (MH) represent the viscosity and strength of the composites. It proven that incorporation of GNPs filler improved the cure characteristics, processability hence resulted in good vulcanized strength.

Suitable range of filler loading for ENR/GNPs composite as a good electrical insulator is postulated to be in the range of 0.2wt% to 0.5wt%, it is the postulated percolation threshold of ENR/GNPs composites. The ENR/GNPs composites at this range will remain as insulator. Increment in the electrical conductivity leads to the improvement in heat dissipation properties. The graphene nanoplatelets reinforcement would increase the thermal resistance of the epoxidized natural rubber composites via thermal dissipation mechanism without jeopardizing too much of their electrical insulation properties. This finding would be benefitted to current electrical appliance industries.

DEDICATION

This report is dedicated to my beloved parents,
who educated me and enable me to reach this level.

To my honoured supervisor,

Prof. Madya Dr. Noraiham Binti Mohamad

for her advices, support and patience during completion of this project

and to all staffs & technicians,

for their advices and cooperation to complete this project.

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LIST OF ABBREVIATIONS

CB	-	Carbon Black
GNPs	-	Graphene nanoplatelets
NR	-	Natural rubber
ENR	-	Epoxidized natural rubber
GE	-	Graphene
SBS	-	Styrene-butadiene-styrene tri-block copolymer
ASTM	-	American Society for Testing and Materials
AC	-	Alternative current
XRD	-	X-ray diffraction
SEM	-	Scanning electron microscopy
FTIR	-	Fourier transform infrared
DSC	-	Differential scanning calorimetry
C	-	Carbon
V	-	Measured voltage
I	-	Constant current
R	-	Resistance
E	-	dielectric strength
VBD	-	Voltage breakdown value
t	-	Thickness of sample
Phr	-	Parts per hundred rubber
MBTS	-	2,20-dithiobis (benzothiazole)
TMTD	-	Tetramethylthiuram disulfide
6PPD	-	N-(1,3-Dimethylbutyl)-N'-phenyl-p phenylenediamine

LIST OF SYMBOLS

wt. %	-	weight percentage
°C	-	Degree Celsius
rpm	-	Revolutions per minute
MPa	-	Mega Pascal

CHAPTER 1

INTRODUCTION

1.1 Research Background

Nowadays, natural rubber (NR) known as cis-1,4-poly(isoprene) compounds are commonly use in the electrical field and automotive field industry due to their mechanical and physical properties such as high elasticity, tensile strength, elongation at break, good insulation and fatigue resistance (Bokobza, 2018; Phetarporn et al., 2019). Natural rubber is non-polar polymer (Mohamad et al., 2008). However natural rubber also has limitation such as low corrosion resistance, degradation due to the heat and oxidation and poor in thermal and ozone properties (Phetarporn et al., 2019).

Therefore, epoxidized natural rubber (ENR) is used which is a modification of natural rubber by epoxidation process. The process of epoxidation has changed the non-polar polymer natural rubber into polar polymer epoxidized natural rubber. This made the rubber more compatible to polar filler due to the existing of polar epoxide group in the epoxidized natural rubber (Sarkawi et al., 2017; Mohamad et al., 2008). Moreover, epoxidized natural rubber has being improve by develops the epoxidized natural rubber composite to overcome the limitation of the conventional natural rubber. Epoxidized natural rubber composite is a combination of epoxidized natural rubber matrix and filler as a reinforcement. Different kind of the reinforcement that added to fabricate natural rubber composite will result in different improvement of mechanical, and physical properties of the composite (Fan et al., 2019).

Graphene (GE) has been discovered in 21st century due to it extraordinary mechanical, electrical and thermal properties there are many researchers interest on this material (Kuila et al., 2012). In currently, the development of graphene nanoplatelets (GNPs)

on natural rubber nanocomposites have a significant breakthrough in the area of nanoscience and technology (Krueger, 2010). Graphene nanoplatelets are used as a reinforcement for epoxidized natural rubber composite. Graphene nanoplatelet is a short stacks of individual layer of graphite which has good mechanical properties such as high mechanical strength, elasticity, electrical and thermal conductivity (Anwar et al., 2015). Graphene nanoplatelet was in nano dimension, it provides a good dispersion of reinforcement with epoxidized natural rubber matrix which improve the mechanical and tribology, thermal stability, dielectric performance and electrical and thermal conductivity of the composite (Sadasivuni et al., 2014).

However the researchers have state the graphene nanoplatelets material show significantly improve in the barrier of electrical and thermal conductivity when it was reinforced in the epoxidized natural rubber composite (He et al., 2015; Anwar et al., 2015; Sadasivuni et al., 2014; Sebok et al., 2001). Reinforcing composites with graphene nanoplatelets increase electrical and thermal conductivity, it introduces the heat diffusion pathway of small molecules and improve the heat dissipation properties (Zhang et al., 2019). Because of this motivation, a series of experiments and analyses are carried out to see the effect of graphene nanoplatelet on the mechanical, physical, electrical, and thermal characteristics when it was added to a certain percentage in the epoxidized natural rubber composite.

1.2 Problem Statement

In the traditional polymeric insulating material, the thermal and electrical conductivity was relatively low. When it is using under high voltage power electrical devices or application, the polymeric insulating material will be generating heat. The polymeric insulating material may lead to failure or breakdown. This situation will occur because of the heat generated from the electrical current is absorbed by the insulative material and cannot be dissipated out. Hence, the molecular chain of the polymeric insulting material will start to degrade, and gripping effect loosen and the strength of the insulative material reduced (Ohtake, 2007). Natural rubber was used as the insulating material in the electrical field industry due to the to it mechanical and physical properties such as high elasticity, tensile

strength, elongation, good insulator and fatigue resistance (Bokobza, 2018; Phetarporn et al., 2019).

Natural rubber insulating material was a material that internal electric charge does not flow freely and make it as a good insulator material. But the use of natural rubber as insulator material for longer period, natural rubber will start to age and become stiffer and damping capability decreases (BoQiao, 2015). The aging process was causing by the heat generation in the rubber compound due to hysteresis loss. Therefore it will be led to the service lifetime of the natural rubber insulator decreases when aging process occur (Banic et al., 2012). Another limitation is electrical stress in the breakdown when apply voltage is exceeding certain value of the insulative material that can hold. Electrical stress in the insulator can be improve by the conduction of graphene (Jain et al., 2013).

Moreover, epoxidized natural rubber is used to alter the conventional natural rubber as the insulator material due to high modulus, tensile strength, and lower plasticity retention index (Harun and Chan, 2015).

The graphene nanoplatelets reinforcement improves the performance and the mechanical properties of the epoxidized natural rubber in term of tensile strength, stiffness, abrasion resistance, tear strength while after epoxidized natural rubber composite vulcanize, especially is thermal and electrical conductivity (Sebok et al., 2001; Anwar et al., 2015). Thermal and electrical conductivity of the graphene nanoplatelets reinforcement was improved when a certain phr is added into the epoxidized natural rubber. In the same time, the aging process in the rubber composite will be slow down due to the improved in thermal and electrical conductivity (Zhang et al., 2019).

The heat can be dissipated equally to the surface of the insulator uniformly. So, in this study the incorporation of graphene nanoplatelets is hypothesized to improve the heat dissipation of epoxidized natural rubber based composite hence increase the potential of their utilization as insulator material. This is due to the increment in the electrical properties of the epoxidized natural rubber composite will leading to the improvement in heat dissipation properties.

1.3 Objective

The objectives are as follows:

1. To characterize the cure characteristics of ENR/GNPs composites prepared via melt compounding for the effect of filler loading at 0 wt.%, 0.5 wt.%, 1 wt.% and 3 wt.%.
2. To postulate the electrical conductivity of ENR/GNPs composites for the effect of filler loading via critical review.
3. To correlate the postulated electrical conductivity of ENR/GNPs composites with their morphological, thermal, compositional, and structural properties review data.

1.4 Scopes of the Research

The scopes of research are as follows:

- a) Graphene nanoplatelets are used as reinforcement and the epoxidized natural rubber is used as polymer matrix. 0 wt.%, 0.5 wt.%, 1 wt.% and 3 wt.% of GNPs of reinforcement is added into the epoxidized natural rubber matrix to produce the composites.
- b) To prepare graphene nanoplatelets reinforced epoxidized natural rubber composite compounds, the formulation of the natural rubber matrix and the graphene nanoplatelets reinforcement mix through melt compounding according to ASTM D 3192 by using Haake internal mixer 70°C, 50 rpm rotor speed. Then, the graphene nanoplatelets reinforced epoxidized natural rubber compounds were vulcanized by using a hot press at 150°C and the time determined T90 from cure characteristics by using Rheometer U-CAN Dynatex Inc UR2010 following ASTM D 2084.