Faculty of Mechanical and Manufacturing Engineering Technology

MECHANICAL DISPERSION OF CARBON NANOTUBE FOR CHLOROPRENE RUBBER COMPOUNDING

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in fulfilment of requirement for the degree of Bachelor of Manufacturing Engineering Technology (Process & Technology) with Honours.

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DECLARATION

I hereby, declared this report entitled "Mechanical Dispersion of Carbon Nanotube for Chloroprene Rubber Compounding" is the results of my own research except as cited in references.

Signature	:	………………………………………….
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APPROVAL

This report is submitted to the Faculty of Mechanical and Manufacturing Engineering Technology of UTeM as a partial fulfillment of the requirements for the degree of Bachelor of Manufacturing Engineering Technology (Process & Technology) with Honours. The member of the supervisory is as follow:

………………………………

(Dr. Lau Kok Tee)

Main Supervisor

DEDICATION

This report is especially dedicated to my family, and to my lecturers (Dr. Lau Kok Tee and Mr. Hairul Effendy Bin Ab. Maulod which have been my source for me to finish this project. Thanks for all your support and give me strength until I completed this project.

ABSTRACT

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Synthetic rubber is the one of the most common types of material used in Malaysia due to their ease of use. Synthetic rubber have different type properties and manufacturers and suppliers are producing different range of rubber products which are used for commercial and industrial purpose. Nanocomposite technology is a new era of rubber material, using a carbon nanotube (CNT) called nanofiller mixed with chloroprene rubber (CR) to improve mechanical properties, heat, dimensions and barriers. CNT loss during chloroprene rubber compounding. This is due to CNT is light weight, thus easily loss to surrounding. Consequently, it is difficult to control amount of CNT added during the compounding. The CNT reinforced has very high rigidity good gas barrier properties and fire resistance. CNT plays a role to enhancing mechanical properties and also reducing the impact properties of CR. This research is to investigate effect of CNT dispersion concentration on CR compounding rheology behavior and to characterize tensile strength and hardness of the CNT-reinforced CR composite after curing. CNT particles were dispersed into an intermediate medium by mixing functionalized the CNT into CR matrix. The mixing method reduced CNT loss to environment because the CNT particles are bound by attractive forces generated by interaction between the CNT particles and liquid medium. CNT's functionalization is enable good coupling between the CNT particles and the rubber matrix. The functionalization changes the chemical and mechanical properties of CNT reinforced CR.

ABSTRAK

Getah sintetik adalah salah satu jenis bahan yang paling biasa digunakan di Malaysia kerana kemudahan penggunaannya. Getah sintetik mempunyai ciri-ciri jenis yang berbeza dan pengeluar dan pembekal menghasilkan pelbagai jenis produk getah yang digunakan untuk tujuan komersil dan perindustrian. Teknologi Nanokomposit adalah era baru bahan getah, menggunakan nanotube karbon (CNT) yang dipanggil nanofiller bercampur dengan getah chloroprene (CR) untuk memperbaiki sifat mekanikal, haba, dimensi dan halangan. Kerugian CNT semasa penyebatian getah sintetik. Oleh kerana CNT adalah ringan, ia mudah hilang ke sekitarnya. Oleh itu, adalah sukar untuk mengawal jumlah CNT yang ditambahkan semasa penyebatian CR yang diperkuat dengan CNT. Nanokomposit getah sintetik yang diperkuat dengan CNT mempunyai nisbah ketegaran tinggi, sifat penghalang gas dan ketahanan api yang baik. CNT memainkan peranan untuk menguatkan sifat mekanikal dan juga mengurangkan sifat hentaman. Kajian ini adalah untuk mengkaji kesan kepekatan serakan CNT ke atas sifat reologi dan mencirikan kekuatan tegangan dan kekerasan komposit CR bertetulang CNT selepas mengawetan. Zarah diserakkan ke dalam medium perantaraan dengan mencampurkan CNT yang difungsikan ke dalam CR matriks. Kaedah pencampuran ini mengurangkan kehilangan CNT terhadap persekitaran kerana zarah CNT terikat dengan daya tarikan yang dihasilkan oleh interaksi antara zarah CNT dan cecair pengantaraan. Fungsian CNT membolehkan gandingan yang baik antara zarah CNT dan matriks getah. Pengfungsian ini mengubah sifat kimia dan mekanikal komposit CR bertetulang CNT.

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LIST OF ABBREVIATIONS, SYMBOLS AND NOMENCLATURE

CNT	-	Carbon nanotube
SEM	-	Scanning Electron microscope
OM	-	Optical Microscope
CR	-	Chloroprene Rubber
EPM	-	Ethylene-propylene rubbers
Fr	-	Friction force
Fn	-	Normal force.
ρ	-	Density
T	-	Thickness
LO	-	Length overall
L	-	Length of narrow section
ΔV	-	Volume difference
Ts	-	Scorch Time
Ml	-	Minimum Torque
Mh	-	Highest Torque
Tc	-	Cure time
S'	-	Elastic Torque curve
TAN	-	Tan Delta value
g	-	Grams
CRI	-	Cure Rate Index
MPa	-	Mega Pascal
Si-69	-	Silane 69

μm	-	Micrometer
N550	-	Carbon Black
MGO	-	Magnesium Oxide
ZXO	-	Zinc Oxide
ETU-80	-	Ethylene Thiourea 80
CBS	-	Cyclohexyl-2-Benzothiazole Sulphenamide
ST Acid	-	Stearic Acid
C-resin (G90)	-	Coumarone Resin (G90)
Phr	-	Per Hundred Rubber
SWCNT	-	Single Wall Carbon Nanotube

CHAPTER 1

INTRODUCTION

This chapter will explain the overview of the study and the main purpose of this study. This chapter includes the background of the study, problem statement, objectives that is expected to be achieved and the scope of the study that is going to be conducted.

1.1 Background of Study

Synthetic rubber is the one of the most common types of material used in Malaysia due to their ease of use. Synthetic rubber have different type properties and manufacturers and suppliers are producing different range of rubber products which are used for commercial and industrial purpose.

Rubber in its native form is basically useless. When certain chemicals are added, the varied rubber products of rubber can be produced. Synthetic rubber is used as a substitute for natural rubber in many cases. The synthetic rubber can be as hard as a bowling ball or as resilient as a rubber band or as soft as a sponge, it is depending on the chemicals added and the properties.

The characteristics of rubber like flexing endurance withstanding greater distortion without injury. Meanwhile, the mechanical properties of rubber for the tensile strength is much larger than the proportionality limit which is greater degree of stretch adjustment that can change the rubber from soft rubber to hard rubber and the higher capacity to absorb energy.

1.2 Problem Statement

Chloroprene Rubber possesses have high resistance to aging and ozone, good chemical resistance, low risk of glazing, better wear resistance than natural rubber. Chloroprene elastomers are rigid and elastically deformable under load. After stress, elastomers return to their initial shape and usually chloroprene in soft form. When heated, elastomers do not become plastic (and are not deformable), and will not melt. At high temperatures, then decomposition takes place and the material is destroyed. The elasticity of an elastomer material can be defined vulcanization or curing by the degree of cross-linking.

Challenge of these approach is the introduction of new materials into the compounding. The excessive amount of the suspension medium may deteriorate the mechanical properties of the rubber composite. Thus, investigation of the approach in terms of parameters optimization and the mechanical properties of the related cured composite are required. This is to validate whether the approach is effective to control the CNT loss during compounding.

During blending operation, we found carbon nanotube (CNT) loss during synthetic rubber compounding. This is due to CNT is light weight, thus easily loss flying to surrounding. Consequently, it is difficult to control amount of CNT added into the compounding. CNT could be dispersed into an intermediate medium, for example liquid suspension medium using functionalising agent, and then dried before added into the matrix. The mixing avoid loss caused by the air flow from environment because the CNT particles are bound by attractive forces generated through interaction between the functionalising groups on CNT particles. Furthermore, CNT's functionalization is required to enable good coupling between the particles and the rubber matrix. The functionalization changes the chemical properties of CNT, resulting in different dispersion behaviour in suspension and interaction between CNT particles with rubber matrix during compounding.

The hydrophobic can be describes the fact that nonpolar substances cannot be combine with water molecules. Water is a polar molecule that is carries a partial charge between its atoms. The electronegative atom is oxygen and draws the electrons of each bond closer to its core and more negative charge have been created. Beside, any materials with a charge, be it negative or positive can dissolve by interact with water molecules. On the other hand, hydrophilic is molecules can dissolve in water. The molecules have a charge (positive or negative) in order to interact with water molecules in polar form. The molecule has a partially at both side (positive and negative).

Overall, this research provides a significant basis in understanding more about the preparation and characterization of the CR rubber dough nanoparticle. The most important problem of nanoparticle is that synthetic rubber is lost in the compounding, this causes the working nanoparticle to be concerned with the control of the matrix interface and the filler in producing the ultimate properties of superior mechanics, morphology, functional properties, mechanical performance and thermal stability of nanoparticle for compatibility with liquid before being incorporated into the mixer, further understanding of the interface matrix interfaces and filler structures and properties of the edible nanoparticle mixtures, can be translated more clear and comprehensive.

1.3 Objectives

The objective of the project is to:-

i) To investigate effect of Silane functionalizing agent concentration on CNT particles dispersion behaviour.

ii) To assess the effect of functionalizes CNT filler's concentration on Chloroprene rubber (CR) compounding in term of rheological behaviour.

iii) To evaluate the mechanical properties of the functionalized CNT-CR composite in term of tensile strength and hardness.

1.4 Scope

The study on this topic can be benefit for certain circumstances. This research is "Mechanical dispersion of carbon nanotube for Chloroprene rubber Compounding" through the mixing material using internal mixer. The scopes of this study is focusing on CNT-reinforced with Chloroprene rubber is prepared using treated with sedimentation test method concentration of CNT and more on mechanical properties of Chloroprene rubber and carbon nanotube through the melt compounding method and its properties effect during mixing.

1.5 Organization of Research Study

This research study is has five chapters that discuss the analytical and experimental research performed. This dissertation shows the carbon nanotube used as a reinforcement. The effects of different loading of carbon nanotube have been studied, in search of improvements on their physical and mechanical properties. The organization of this research study is as follows. This dissertation has been organized into five chapter. The first chapter begins with an introduction about the research study and also brief about the objectives, problem statement, significant of study and the thesis overview.

Chapter two begins on the literature background of this study. It discusses on the history of synthetic rubber, carbon nanotube (CNT) types of composites and matrices. The important element that included in this chapter is about the mechanical properties and used of carbon nanotube, synthetic rubber and the wear behavior of polymer composite.

Chapter three provides information detail and explanations on the methodology used for overall research work, raw materials, formulation, and procedure property analysis that had been done.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

In this chapter, it clarify about presentation of mechanical dispersion of carbon nanotube (CNT) and synthetic rubber used to compounding. This chapter contains main discoveries from literature reviews research, which derived from the article, journal, book and internet that has the related topic to this study.

In the production this project, all theory and information of material substance used, production and testing in relation this project has been described to achieve objective of the project those implemented

2.2 Synthetic Rubbers

Synthetic rubber is a type of elastomer or rubber which is produced in manufacturing plants by manufacture it from petroleum and other mineral. The different types of rubber in synthetic as crude are the principle of raw material. Synthetic rubber is basically an artificial polymer. The property of synthetic rubber is undergoing deformation under stress but can also return to its previous size without permanent deformation and is its native from is basically useless it is only when certain chemicals are added. The synthetic rubber are produces to make varied rubber product. Compare to natural rubber where there is only one chemical type, different almost 20% different chemical type of synthetic rubber, there are different type and grades. The different types of synthetic rubber have their specific properties and advantages (Foster, 2018).

2.2.1 Properties of Synthetic Rubber

Synthetic rubbers are the most often produced by means of polyreactions of the chain or gradual character. The chain of polyreactions are:

i) Ionic

ii) Radical

The most Coordination polymerization and copolymerization often used:

i) Solution

ii) Emulsion

iii) Suspension

2.2.2 Structure & Type of Synthetic Rubber

2.2.2.1 Chloroprene

Chlorobutadiene rubber or Chloroprene Rubber (CR), is a diene-based elastomer. The name Neoprene is also same meaning of chloroprene and came from Elastomers. The 1,4-polychloroprene is commercial grades are mostly trans-produced by free-radical emulsion polymerization of 2-chloro-1,3-butadiene The oxidizing agents can be reduces the reactivity chlorine in the polymer (Klingender, 2015).

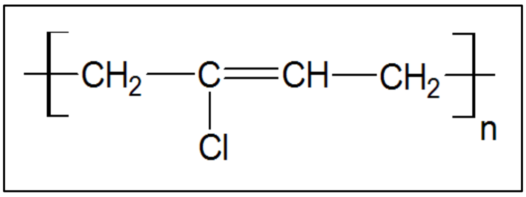

Figure 2.1: Chloroprene chain (Barlow, 2014)

Chloroprene rubber are only moderate or poor resistance to acids, solvents, and fuels, but chloroprene good resistance to ozone cracking, heat aging and to chemical attack, good resistance to many chlorofluorocarbons, aliphatic hydrocarbons, mineral oils, greases and ozone (Barlow, 2014).

Chloroprene rubbers (CR) is types of synthetic rubbers (Salleh, 2016). More radical emulsion polymerization of chloroprene can be produced. The most frequently used for initiation and mercaptanes (W-CR) or sulfur (G-CR) are used for regulation of molecular weights is Potassium or ammonium peroxodisulphate. Sulfur behaves during polymerization of chloroprene as co-monomer and it creates sulfur bridges in polymer chains with 2 – 6 sulfur atoms that reduce temperature resistance and improve their flexibility. The W – CR does not contain sulfur bridges and macromolecules can be terminated with the rest of transfer agent molecules (in presence of n-dodecyl mercaptane $- SR \equiv - S\ (CH_2)_{11}\ CH_3$). Chloroprene are used for rubber industry purposes is normally produced at temperature around 30 – 40 °C, it depending of what material to produce. They are less flexible, but more resistive against temperature (Weidinger, 2016).

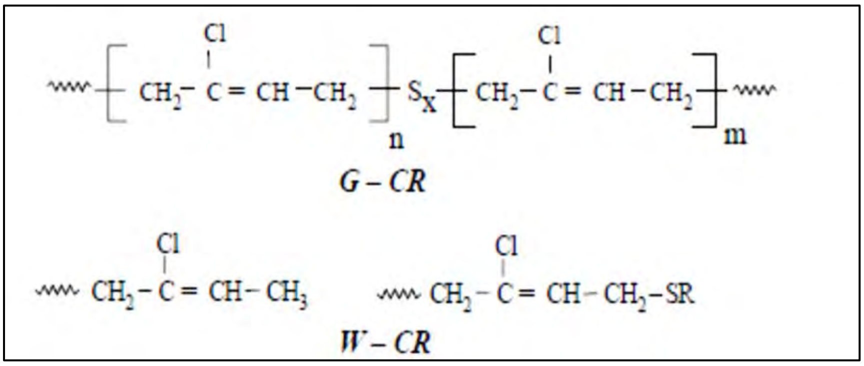

Figure 2.2: Chloroprene are bonded in polymer chain (Weidinger, 2016).

Based on Figure 2.2, the structural units of chloroprene have bonded in polymer chain mostly in form of 1.4-trans structural units.

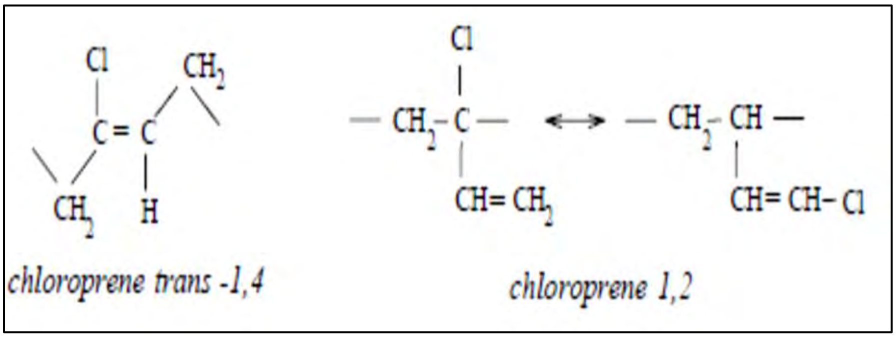

Figure 2.3: Structural units of CR rubbers (Weidinger, 2016).

Table 2.1: The Chloroprene Rubber Influent Type on Crystallization (Sercer, 2018)

Type of chloroprene	**Crystallization ability**	**Crystallization half-time** $\tau_{1/2}$
A – CR	High	minutes to hours
W – CR	Middle	months to years
G – CR	Low	months to years
Chloroprene copolymers	Not crystallize	-