UNIVERSITI PUTRA MALAYSIA

CHARACTERIZATION AND PROPERTIES OF GAMMA IRRADIATED EPOXIDIZED NATURAL RUBBER LATEX

CHAI CHEE KEONG

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By

CHAI CHEE KEONG

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

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DEDICATION

This thesis is dedicated to my beloved parents, wife and son.

Chai Chee Keong
October 2020

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

CHARACTERIZATION AND PROPERTIES OF GAMMA IRRADIATED EPOXIDIZED NATURAL RUBBER LATEX

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CHAI CHEE KEONG

October 2020

Chairman : Professor Luqman Chuah Abdullah, PhD
Faculty : Engineering

Epoxidized natural rubber latex (ENRL) is the liquid form of epoxidized natural rubber (ENR), which possesses many enhanced properties that are lacking in its unmodified origin, i.e. natural rubber latex. Despite extensive literature on the studies and applications of ENR, very limited work done on ENRL to understand and exploit commercial applications of this material. In relation to this, it is important to test the hypothesis that properties of ENRL can be improved by means of gamma irradiation. Hence, a study was conducted to determine the effect of gamma irradiation on ENRL, the effect of sensitizer on ENRL during gamma irradiation and the effect of antioxidant on gamma irradiated ENRL. The novelty of this study is the utilization of gamma radiation for crosslinking of ENRL. ENRL with 25 mol% (ENRL-25) and 50 mol% (ENRL-50) epoxidation level was used in this study. The sensitizers were n-butyl acrylate (n-BA), 1,6-hexanediol diacrylate (HDDA) and trimethylolpropane triacrylate (TMPTA). The antioxidant was 4,6-bis (octylthiomethyl)-o-cresol (Irganox 1520). Gamma radiation ranged from 0 to 160 kGy. Cast films were prepared from the gamma irradiated ENRL and used for characterization via tensile test, gel fraction test, aging test, Fourier transform infrared analysis (FTIR), dynamic mechanical analysis (DMA) and thermogravimetric analysis (TGA). Results revealed that gamma irradiation is capable of inducing crosslinks in ENRL-25 and ENRL-50, with an optimum dose and tensile strength of 80 kGy and 5.4 – 6 MPa respectively. The effect of gamma irradiation is more prominent on ENRL-25 than ENRL-50 due to higher quantity of unreacted carbon-carbon double bonds (C=C) in the former. Dynamic mechanical properties of gamma irradiated ENRL showed significant positive shift in glass transition temperature (T_g) of tan delta and loss modulus. Effect of gamma irradiation on thermal properties of ENRL-25 is negligible, whereas its effect on ENRL-50 is positive but marginal. The presence of sensitizer gives positive impact on gamma irradiation of ENRL. The impact increases in the order of n-BA < HDDA < TMPTA. The effect of sensitizer on ENRL-25 is higher than ENRL-50 due to the presence of more C=C in ENRL-25. The tensile strength of

gamma irradiated ENRL with sensitizer are lower than those without sensitizer, suggesting that physical chain entanglement prevails chemical crosslink in the former. The presence of sensitizer in gamma irradiated ENRL enhances its thermal stability. Their effect on thermal stability increases in the order of TMPTA < HDDA < n-BA. Irganox 1520 is effective in enhancing the resistance of gamma irradiated ENRL against thermo-oxidative degradation. However, it inhibits the efficiency of radiation-induced crosslinking due to radical scavenging effect. ENRL-50 shows better heat resistance than ENRL-25 due to the presence of more epoxide groups in the former that resulted more ether crosslinks in the former. Based on experimental findings, ENRL-25 and ENRL-50 are crosslinkable by gamma radiation, thus, their mechanical, dynamical and thermal aging properties can be improved by means of gamma irradiation.

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PENCIRIAN DAN SIFAT-SIFAT LATEKS GETAH ASLI TEREPOKSIDA YANG DISINAR DENGAN SINARAN GAMA

Oleh

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Lateks getah asli terperoksida (ENRL) merupakan getah asli terperoksida (ENR) dalam bentuk cecair. Ia memiliki banyak sifat tertingkat yang tiada pada sumber asal tanpa modifikasi, iaitu lateks getah asli. Sungguhpun terdapat banyak maklumat tentang kajian dan aplikasi ENR, kajian ke atas ENRL untuk memahami dan mengeksploitasi aplikasi komersil bahan ini adalah amat terhad. Sehubungan ini, adalah penting untuk menguji hipotesis sifat ENRL dapat dipertingkatkan dengan kaedah penyinaran gama. Justeru itu, satu kajian telah dijalankan untuk menentukan kesan penyinaran gama ke atas ENRL, kesan bahan pemeka ke atas ENRL semasa proses penyinaran gama dan kesan bahan antioksida (Irganox 1520) ke atas ENRL yang sinarkan dengan sinaran gama. Kebaharuan dalam kajian ini ialah penggunaan sinaran gama untuk taut silang ENRL. ENRL yang digunakan dalam kajian ini ialah ENRL dengan tahap pengepoksidaan 25 mol% (ENRL-25) dan 50 mol% (ENRL-50). Bahan pemeka ialah n-butil akrilat (n-BA), 1,6-heksanadiol diakrilat (HDDA dan trimetilolpropana triakrilat (TMPTA). Bahan antioksida ialah 4,6-bis(oktiltiometil)-o-kresol (Irganox 1520). Julat sinaran gama ialah 0 hingga 160 kGy. Filem-filem tuangan telah disediakan daripada ENRL tersinar dengan sinaran gama dan seterusnya digunakan untuk pencirian melalui ujian tegangan, ujian pecahan gel, ujian penuaan, analisis infra-merah transfom Fourier (FTIR), analisis dinamik mekanika (DMA) dan analisi termogravimetri (TGA). Keputusan menunjukkan penyinaran gama mampu mencetuskan taut silang dalam ENRL-25 dan ENRL-50, dengan dos dan kekuatan tegangan optimum pada 80 kGy dan 5.4 – 6 MPa masing-masing. Kesan penyinaran gama adalah lebih menonjol pada ENRL-25 berbandingan dengan ENRL-50. Ini disebabkan oleh kuantiti ikatan dubel tidak bertindakbalas (C=C) yan lebih tinggi pada ENRL-25. Sifat dinamik mekanika ENRL tersinar dengan sinaran gama menunjukkan peralihan positif pada suhu transisi kaca (T_g) pada tan delta dan modulus kehilangan. Kesan penyinaran gama ke atas sifat terma ENRL-25 boleh diabaikan manakala kesan ke atas ENRL-50 adalah positif tetapi kecil. Kehadiran bahan pemeka memberikan impak positif ke atas penyinaran gama ENRL. Impak tersebut meningkat dalam urutan n-BA < HDDA < TMPTA. Kesan bahan pemeka ke atas ENRL-25 adalah lebih tinggi berbanding dengan ENRL-50. Ini disebabkan oleh kehadiran lebih banyak C=C dalam ENRL-25. Kekuatan tegangan ENRL tersinar dengan sinaran gama dalam kehadiran bahan pemeka adalah lebih rendah berbanding dengan ENRL tanpa bahan pemeka. Ini mencadangkan kekusutan rantaian secara fizikal mengatasi taut silang kimia dalam ENRL yang mengandungi bahan pemeka. Kehadiran bahan pemeka dalam ENRL tersinar dengan sinaran gama meningkatkan kestabilan termanya. Kesan bahan pemeka ke atas kestabilan terma meningkat dalam urutan TMPTA < HDDA < n-BA. Irganox 1520 adalah berkesan dalam meningkatkan ketahanan ENRL tersinar dengan sinaran gama terhadap degradasi terma-oksidatif. Sungguhpun begitu, ia menghalang keberkesanan taut silang yang dicetuskan oleh sinaran. Ini disebabkan oleh kesan menangkap radikal. ENRL-50 menunjukkan ketahanan haba yang lebih baik berbanding ENRL-25. Ini disebabkan oleh kehadiran lebih banyak kumpulan epoksida yang mampu menghasilkan lebih banyak taut silang eter dalam ENRL-50. Berdasarkan hasil kajian ini, ENRL-25 dan ENRL-50 tersinar dengan sinaran gama adalah bahan salutan berpotensi untuk produk getah. Berdasarkan dapatan experiment ini, ENRL-25 dan ENRL-50 dapat ditaut silang dengan sinaran gama, oleh itu sifat mekanikal, dinamik dan penuaan haba dapat dipertingkat dengan kaedah penyinaran gama.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Phiosophy. The members of the Supervisory Committee were as followed:

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS

C=C	Olefinic carbon-carbon double bonds
T_g	Glass transition temperature
σ_1	Tensile stress
F_1	Applied tensile force
A_o	Cross-sectional area of the material
ε_1	Tensile strain
l_o	Initial length of the material
l_1	Length of stretched material
k	Rate of extension
t	Time taken to reach length l_1
ε	Elongation at break
A	Absorbance
T	Transmittance
ε	Molar absorptivity
c	Concentration
l	Pathlength
E^*	Complex modulus
E'	Storage modulus
E''	Loss modulus
δ	Phase angle
σ^o	Maximum stress
ε^o	Strain at maximum stress
τ^o	Maximum shear stress
γ^o	Shear strain at maximum shear stress

*G**	Shear complex modulus
G'	Shear storage modulus
G"	Shear loss modulus
C-O-C	ether

LIST OF ABBREVIATIONS

NRL	Natural rubber latex
RVNRL	Radiation vulcanization of natural rubber latex
IAEA	International Atomic Energy Agency
NR	Natural rubber
ENR	Epoxdized natural rubber latex
TPE	Thermoplastic elastomer
ENRL	Epoxidized natural rubber latex
NBR	Acrylonitrile butadiene rubber
DMA	Dynamic mechanical analysis
TGA	Thermogravimetric analysis
FTIR	Fourier transform infrared
IR	Isoprene rubber
SBR	Styrene-butadiene rubber
BR	Butadiene rubber
CR	Chloroprene rubber
n-BA	n-butyl acrylate
HDDA	1,6-hexanediol diacrylate
TMPTA	Trimethylolpropane triacrylate
PVC	Poly(vinyl chloride)
IPPD	N-isoproply-N'-pheny-*p*-phenylenediamine
DSC	Differential scanning calorimetry
DTG	Differential thermogravimetry
TG	Thermogravimetry
TSC	Total solid content

THF	Tetrahydrofuran
ATR	Attenuated total reflectance
M300	Modulus at 300% elongation
TS	Tensile strength
EB	Elongation at break
GF	Gel fraction
TS_{max}	Tensile strength maximum
M700	Modulus at 700% elongation

CHAPTER 1

INTRODUCTION

1.1 Introduction

Epoxidized natural rubber latex (ENRL) is the preceded product of ENR in the production process of ENR. It is a chemically modified natural rubber latex (NRL) by the addition of oxygen atom to the carbon-carbon double bonds of rubber molecule chains, thereby converting them to oxirane (epoxide) rings. The need for natural rubber (NR) modification is driven by the fact that NR, despite its superior elasticity, has low resistance towards oil, chemical and solvent compared to nitrile butadiene rubber and relatively lower abrasion resistance than styrene butadiene rubber. And this subsequently has limited its application in the related fields and industries. This situation has changed with the development in epoxidation of NR.

Epoxidation of NR can be achieved by treating NRL with preformed preoxyacetic or *in-situ* generated peroxyformic acid (Ng and Gan, 1981). The epoxidation level can achieves over 75 mole% without the formation of secondary ring-opened structures if performed under controlled conditions (Gelling, 1991). ENR is claimed to exhibit improved oil resistance, gas impermeability and good wet grip comparable to synthetic rubber (Baker et al., 1985). As a preceded product of ENR, ENRL is expected to possess such enhanced properties and should be a good raw material form manufacturing of thin rubber articles based on latex dipping process

Compared to ENR, ENRL has gained far less interest from researchers. Various studies has been conducted on ENR which include curing (Luo et al., 2012; Liau, 1999; Sadequl et al.,1999; Sadequl et al., 1998; Nasir et al., 1989), thermal oxidation (Poh and Lee, 1994), reversion (Poh et al., 1995) and gas permeability (Barrie et al., 1992). Most studies on ENR that involves vulcanization are based on sulfur vulcanization. Some works on electron beam irradiation of ENR has been reported and found evidence of irradiation-induced crosslinking in ENR (Ratnam et al., 2001a; Ratnam et al., 2000).

Works on ENR blending with natural or synthetic polymers have been reported. These include blending of ENR with natural rubber (Poh et al., 2002; Poh et al., 2001; Ismail and Poh, 2000) and synthetic rubber such as styrene-butadiene rubber (Ismail and Leong, 2001; Ismail and Suzaimah, 2000; Nasir and Choo, 1989). ENR has also been utilized for preparation of thermoplastic elastomer (TPE) where it is blended with a thermoplastic to form a polymer blend suitable for manufacturing processes without sacrificing the elasticity of rubber. Related studies of ENR incorporated TPE include blending of ENR with poly(vinyl chloride) (Egharevba et al., 2011; Ratnam, 2001; Ratnam et al., 2001b; Ratnam et al., 2001c; Ratnam et al., 2001d; Ratnam and Zaman, 1999a; Ramesh and De, 1993), ethylene vinyl acetate (EVA) (Zurina et al., 2008; Zurina et al., 2006a) and polypropylene (Nakason et al., 2008; Nakason et al., 2006). ENR were also used as main matrix in preparation of composite for various applications (Raju et al., 2020; Roy et al., 2020).

All works mentioned above are based on ENR. There are relatively very few studies on ENRL, i.e. the latex form of ENR. In view of the potential of utilizing ENRL in production of thin rubber articles, Darji and Md Said (2010) has conducted a study to investigate sulfur vulcanization and coagulant dipping of ENRL. They found that rubber film can be obtained from compounded ENRL by coagulant dipping method, and postvulcanizaiton of ENR-50 films is more effective than prevulcanization of ENR-50 in increasing crosslink density of ENR.

Rubber needs to be vulcanized prior to use in commercial rubber product manufacturing process. Rubber vulcanization can be achieved by using sulfur or non-sulfur vulcanization system. Sulfur vulcanization which discovered by Charles Goodyear (Sjothun and Alliger, 1978), i.e. the conventional method, has been widely used by related rubber industries. Despite its usefulness, this method has some downsides due to residue chemical additives of either sulfur origin or other aiding chemical ingredients used together with sulfur. These chemical residues remain in manufactured articles expose end users to health problems, e.g. type IV allergenic reaction and carcinogenic nitrosamines (Makuuchi, 2003; Tesiorawski, 2003; Gonlag, 2000; Free, 1998; Pollard and Layon, 1996). Environmental problem, which mainly related to discharge of high zinc content effluent to the environment, also arises upon manufacturing of these rubber articles (Anotai et al., 2007; Nordin and Zaid, 1997). One of the promising solutions to these issues is non-sulfur vulcanization approach, i.e. radiation or peroxide vulcanization. In non-sulfur vulcanization, crosslinks are established by free radical mechanisms induced by high energy radiation or peroxide (Bhattacharya and Ray, 2009).

Intensive and systematic studies on radiation vulcanization of natural rubber latex (RVNRL) have been carried out under the coordination of International Atomic Energy Agency (IAEA) in early 80s. Since then, with positive findings and outcomes from the studies, this technology has been developed into pilot plant scale for promotion and commercialization purpose. Radiation vulcanization is claimed to be more user and environmental friendly than sulfur vulcanization due to the absence of sulfur and accelerators. However, the mechanical properties of RVNRL are inferior to sulfur vulcanized NRL (Makuuchi, 2003).

1.2 Problem Statement

In view of the enhanced properties such as oil resistance, gas impermeability, wet grip and high damping characteristics of ENR, its potential in producing thin rubber articles using ENRL should be exploited in order to benefit more industrial areas such as glove manufacturing. Although some of the above mentioned properties such as oil resistance and gas impermeability are also available in some synthetic rubbers such as butyl rubber and acrylonitrile butadiene rubber (NBR), these synthetic rubbers are based on a non-renewable resource, i.e. petroleum, as opposed to ENR which is produced from natural and renewable origin. Furthermore, such synthetic materials often require sulfur vulcanization treatment prior to use, which has caused environmental and health problem. Sulfur vulcanization system requires zinc oxide as an activator and accelerators such as thiurams, thiazoles and dithiocarbamates for optimal effect and yield (Kruželák et al., 2016). It is common that effluent from latex dipped product manufacturers utilizing sulfur vulcanized latex in their production contains high level of zinc. Two major sources of such effluent are from latex compounding process and leaching tank (Nordin and Zaid, 1997). Despite the fact that zinc is one of the important trace elements for most animals, many aquatic species are actually sensitive to high concentration level of zinc in water system (Häggström, 1997). The presence of accelerators as chemical residue in latex products has caused allergy Type IV (delayed type hypersensitivity) among end users such as health care workers (Noble, 2005). Radiation vulcanization of ENRL can be one of the promising solutions to these problems. However, at present, this seems to be a difficult task as human understanding on ENRL is very limited. There is relatively fewer data published on properties of ENRL compares to its solid state counterpart, and no work has been done on the radiation vulcanization of ENRL, which includes the effect of gamma radiation, sensitizer and antioxidant on properties of ENRL. The findings of this research may aid the effort to stimulate and diversify the application of ENRL in latex dipped product industries.

1.3 Objectives

This research work is mainly aimed to study the effects of gamma radiation on properties of ENRL. The main objectives of this study are as followed:

- To investigate the effect of gamma radiation on ENRL with regards to epoxidation level, i.e. 25 mol% and 50 mol%, and radiation dose.
- To investigate the effect of sensitizer on irradiation of ENRL with regards to type of type of sensitizer and epoxidation level.
- To investigate the effect of antioxidant on irradiated ENRL with regards to epoxidation level.

Due to the non-commercial nature and limited supply of ENRL, there are only 2 grades of ENRL available for this study, i.e. ENRL with 25 mol% and 50 mol% epoxidation level.

For each of the objectives mentioned above, ENRL properties such as tensile properties, gel fraction, dynamic mechanical properties (DMA) and thermal stability (TGA and DSC), and changes in molecular structure (FTIR) as well as accelerated aging resistance are determined in order to study the effect of gamma radiation on ENRL. All the works mentioned may increase the level of understanding on gamma irradiation of ENRL, contribute to the pool of knowledge in the related field and provide insight to the application of ENRL especially in thin rubber article manufacturing and rubber coating industry.

1.4 Organization of the Thesis

This thesis consists of six chapters as followed:

- Chapter 1 contains an introduction to the overall study of this thesis. It covers a brief research background, problem statement and objectives of the study.
- Chapter 2 contains a comprehensive review of the literature related to the subject matter of this thesis, i.e. gamma irradiation of ENRL. It covers an overview of radiation vulcanization of NRL, ENR and characterization of rubber.
- Chapter 3 outlines the materials used and methods applied in all experimental works on gamma irradiation of ENRL.
- Chapter 4 contains experimental results and discussion on gamma irradiation on ENRL of two different epoxidation levels, i.e. 25 mol% and 50 mol%. The results and discussion cover effects of gamma radiation at various doses on tensile, gel, morphological, dynamic mechanical and thermal properties of ENRL.
- Chapter 5 contains experimental results and discussion on gamma irradiation on ENRL with the presence of sensitizer. The results and discussion cover effects of the sensitizer on gamma irradiated ENRL on tensile, gel, dynamic mechanical and thermal properties of ENRL.
- Chapter 6 contains experimental results and discussion on aging resistance of gamma irradiated ENRL. The results and discussion cover effects of antioxidant in gamma irradiated ENRL in the aspects of tensile, gel, dynamic mechanical and thermal properties as well as effectiveness of the antioxidant in enhancing aging resistance of the ENRL.
- Chapter 7 contains an overall conclusion based on findings mentioned and discussed in the previous chapter, and an evaluation on the achievement of the objectives of this study. It also contains suggestions for further research of this study.

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