



**UNIVERSITI TEKNIKAL MALAYSIA MELAKA**

**CHARACTERIZATION OF EPOXIDISED NATURAL RUBBER /  
ETHYLENE PROPYLENE DIENE MONOMER (ENR/EPDM)  
BLEND PREPARED VIA SOLUTION MIXING IN TOLUENE**

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

by

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# UNIVERSITI TEKNIKAL MALAYSIA MELAKA

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**TAJUK:** Characterization of Epoxidised Natural Rubber/Ethylene Propylene Diene Monomer (ENR/EPDM) blends prepared via Solution Mixing in Toluene.

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## **APPROVAL**

This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfillment of the requirements for the degree of Bachelor of Manufacturing Engineering (Engineering Materials) (Hons.). The member of the supervisory is as follow:

.....

(Dr Noraiham Binti Mohamad)

## ABSTRAK

Objektif kajian ini adalah untuk mengkaji kebolehcampuran getah asli terepoksida (ENR) dalam elastomer monomer etilena propilena diena (EPDM). ENR adalah terbitan kimia getah asli dan mempunyai kumpulan epoksida disepanjang tulang belakang rangkaian getah NR. Ciri ENR ini dipercayai akan menyumbang kepada peningkatan sifat rekatan kepada sebarang adunan yang melibatkan getah asli dan EPDM. Dalam kajian ini, campuran ENR dan EPDM telah diadunkan melalui pencampuran larutan di dalam cecair toulena sebagai bahan pelarut. Kesan formulasi dan masa penuaan terhadap tahap penggaulan ENR dengan EPDM dikaji. Kebolehcampuran adunan ENR/EPDM ditentukan melalui sifat fizik, termal, dan komposisi telah diuji melalui penentuan ketumpatan, analisis kebezaan imbasan kalorimetri (DSC) dan analisis spektroskopi penjelmaan inframerah Fourier (FTIR). ENR/EPDM sebagai penserasi telah diadun bersama dengan NR/EPDM menggunakan alat pencampur dalaman dan analisis keserasian dari segi mekanikal dan morfologi telah diuji melalui ujian tegangan dan kemikroskopan elektron imbasan (SEM). Masa pencampuran selama 0, 8 dan 24 jam dengan masa penuaan selama 14, 7 dan 1 hari telah menghasilkan adunan ENR/EPDM dengan kebolehcampuran dan sifat-sifat yang bererti. Campuran ENR/EPDM dengan 8 jam pencampuran dan 7 hari penuaan menunjukkan peratusan kumpulan epoksida tertindak balas yang paling tinggi iaitu 89%. Peratusan epoksida tertindak balas yang tinggi menunjukkan tahap kebolehcampuran yang tinggi antara fasa dan seterusnya menyumbang kepada sifat tegangan yang tinggi dan berupaya mengalami ubahbentuk plastik yang besar sebelum patah. Kesimpulannya, ENR/EPDM menunjukkan potensi yang tinggi untuk meningkatkan kebolehcampuran dan sifat mekanik adunan NR/EPDM.

## ABSTRACT

The objective of this research is to study the miscibility of epoxidized natural rubber (ENR) in elastomer of ethylene propylene diene monomer (EPDM). ENR is a chemical derivative of natural rubber with epoxide groups distributed along the back-bond of natural rubber chains. This characteristic of ENR is believed to contribute to the improvement of adhesion in any blend of NR and EPDM. In this research, mixture of ENR and EPDM were blended together via solution mixing in Toluene as the solvent. The effect of mixing formulation and aging time to the ENR/EPDM blend were studied. The miscibility of ENR in EPDM was determined through physical, thermal, and compositional via density measurement, differential scanning calorimetry (DSC) and Fourier transformation infrared spectroscopy (FTIR). NR/EPDM incorporated with ENR/EPDM as compatibilizer was melt blended by using internal mixer and compatibility analyses in term of mechanical and morphological have been tested via tensile and scanning electron microscopy. Mixing time at 0, 8 and 24 hour with aging time at 14, 7 and 1 day produced ENR/EPDM blend with significant miscibility and properties. ENR/EPDM blend with 8-hours mixing and 7 days aging shows the highest percentage of reacted epoxide which is 89%. This high percentage of reacted epoxide contributes to high degree of miscibility between phases hence resulted in high tensile properties and large plastic deformation before fracture. As the conclusion, ENR/EPDM as compatibility agent shows high potential to increase miscibility and mechanical properties of NR/EPDM blend.

## **DEDICATION**

To my father; Azamam Bin Hassan, my mother; Jamilah Binti Che Hamid, my siblings and friends. Your love is my driving force.

To my supervisor Dr. Noraiham Binti Mohamad, to my mentors Mazlin Aida, Nor Nadiah Hamid, Nur Sharafina, Juliana and all staffs in UTeM.

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# LIST OF ABBREVIATIONS, SYMBOLS AND NOMENCLATURE

ASTM	-	America standard test method
CNT	-	Carbon nano tube
C <sub>p</sub>	-	Heat capacity
DCA	-	Dichloroacetic acid
DRC	-	Dry rubber content
ENR	-	Epoxidation of Natural Rubber
EPDM	-	Ethylene Propylene Diene Monomer
EV	-	Efficient vulcanisation
IISR	-	International institute of synthetic rubber
IR	-	Infrared
IR	-	Isoprene rubber
MA	-	Maleic anhydride
MFI	-	melt flow index
MPa	-	mega pascal
NR	-	Natural rubber
PTFE	-	Polytetrafluoroethylene
PVC	-	Polyvinyl chloride
SBR	-	Styrene butadiene rubber
Semi-EV	-	Semi-efficient vulcanisation
SEI	-	Secondary Electron Image
TEM	-	Transmission Electron Microscopy
TOR	-	Trans-polyoctylene
TPE	-	Thermoplastic elastomer
TSE	-	Thermoset elastomer
TCE	-	Trichloroethylene
$\rho$	-	Density



# CHAPTER 1

## INTRODUCTION

### 1.1 Background

Polymer blends have received much attention since blending is a simple, effective approach to develop new materials exhibiting combinations of properties that cannot be obtained by individual polymers. Blending two or more elastomers is carried out for particular objectives such as enhancement of technical properties, improvement of ageing resistance and also processing characteristics (Alex *et al.*, 2003). It is most common for compatibilization to be succeeded by addition of a third component (compatibilizer) or by inducing in situ chemical reaction between blend components (reactive blending) leading to modification of the polymer interfaces in two-phase blends, and thereby for tailoring of the phase structure and properties (Hussain *et al.*, 2010).

Natural rubber (NR) is normally blended with ethylene propylene diene rubber (EPDM) to improve ageing resistance of the former without losing its good mechanical properties. Due to the difference in unsaturation level between these components, a mutual incompatibility can exist, which contributes for a decreasing of mechanical performance.

EPDM is the fastest growing elastomer among the synthetic rubbers since its introduction in 1963 it represents 7% of the world rubber consumption and it is the most widely used non-tire rubber (Hussain *et al.*, 2010). ENR have found commercial applications in a wide range of areas, e.g. their hysteresis properties are utilized in both

mechanical and acoustic damping devices and in specialty shoe soles to give high wet grip. ENR also are used in the construction of composites conveyor belts because of their adhesives properties. The combination of high strength and low resilience of ENR based conveyor belts are advantageous under severe service conditions (Gelling, 1991)

## **1.2 Problem statement**

Nowadays, rubber-rubber mixture has attracted number of researcher around the world due to its potential to combine the attractive properties of both the constituents in the blend. One of the famous types of rubber-rubber mixtures is NR/EPDM .The NR/EPDM blend is one most used blend in the market nowadays. The application of NR/EPDM is used especially to make the automotive weather-strips, engine mounts, washing machine gaskets and grommets.

In the application of NR/EPDM blend, it has been shown that most of the blend are immiscible and incompatible thus resulting in poor mechanical properties. They have coarse phase morphology with sharp interface. Furthermore, the adhesion between both blend phases is poor, so that these blends are useless without being compatibilized. Although there are many compatibilizer agents such as maleic anhydride (MA) and trans-polyoctylene (TOR) can be incorporated into this blend to increase the compatibility, it has their own drawback due to expensive price.

EPDM usually imparts good aging properties, good weathering oxidation and chemical resistance. NR also exhibits unique physical and chemical properties such as elasticity, stickiness, resilience and etc. However, most application of NR are limited to the low stability to heat, oxygen, sunlight and high solubility in most hydrocarbon/hydrophobic solvents including oils. Those limitations resulted from NR chemical structure (Cis 1, 4-polyisoprene), which contain double bond and hydrocarbon atoms. The chemical modification at double bonds and introduction of hydrophilic groups along NR backbone are alternative strategies to improve the stability of NR and to provide widespread

application. Epoxidised natural rubber (ENR) is a best material for the present work since the introduction of epoxide groups not only reduces the number of double bond but also increase the hydrophilicity of NR (Yoksan, 2008); which will be very critical in the production of any NR/EPDM based composites.

Thus far, based on ENR capability, the introduction of ENR/EPDM blend as a compatibilizer agent to the NR/EPDM will improve the compatibility and increase the interaction between immiscible phases.

### **1.3 Objective**

The main objectives on this research are:

- a) To synthesis ENR/EPDM blend through solution mixing
- b) To study the effect of formulation, mixing and aging time of ENR/EPDM blend.
- c) To characterize the physical and thermal properties of ENR/EPDM as a potential compatibilizer agent to the NR/EPDM blend

### **1.4 Scope**

This research is focusing on the characterization of ENR/EPDM blend compatibilizer and its effect in NR/EPDM. In this research, ENR/EPDM blend is prepared via solution mixing with help of mechanical stirrer with addition of solvent and then it is dried to become a film. Analysis such as thermal and morphology are performed by using DSC and SEM to analyze the thermal and morphological properties of the blend. Fourier transformation infrared spectroscopy is used to characterize the chemical composition of the blend while mechanical properties were performed by using tensile.

## 1.5 Chapter review

There are three chapters in this report.

- a) Chapter 1 is the introduction of the research. This chapter consists of background, problem statement, objectives of the report, scope and chapter review of the report.
- b) Chapter 2 is the literature review. This chapter presents the published literatures that are relevant to particular topic of this research, demonstrating the information of any previous work and awareness of related theories and discussions.
- c) Chapter 3 is the methodology of the report. This chapter discusses the review of the methodology carried out in order to produce the desired product or outcome of the project.

## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Polymer

According to Hatsuo (2006) the word "polymer" consists of "poly; many", and "mer; unit", and indicates a large molecule with many repeating units. Other words, "high polymer" and "macromolecule" are often used synonymously. However, macromolecule strictly means a large molecule and does not necessarily indicate a repetitive structure. Thus, macromolecule is more general than polymer. The repeating unit of the polymer is called, "chemical repeat unit," and the original small molecule that leads to the repeat unit is called, "monomer," meaning a single unit. Typically the molecular weight of a monomer is from several tens to several hundred whereby that of a polymer is tens of thousands to several millions. An example of the relationship between the monomer and polymer is shown in Figure 2.1.

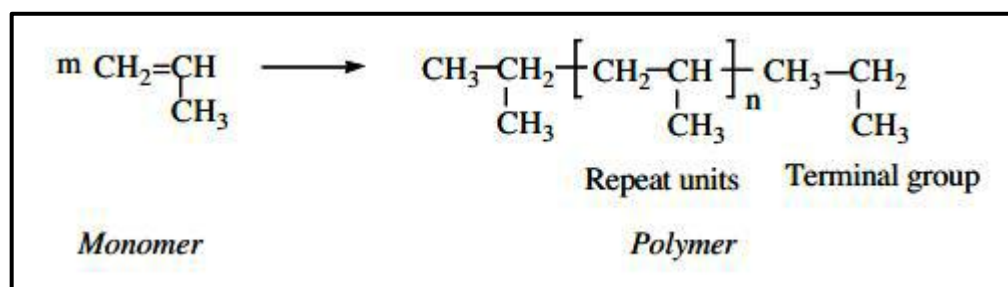


Figure 2.1: Polymer formation (Hatsuo, 2006)

In many polymers, only one monomer is used. In others, two or three different monomers may be combined. Polymers are classified by the characteristics of the reactions by which they are formed. If all atoms in the monomers are incorporated into the polymer, the polymer is called an addition polymer. If some of the atoms of the monomers are released into small molecules, such as water, the polymer is called a condensation polymer. Most addition polymers are made from monomers containing a double bond between carbon atoms. Such monomers are called olefins, and most commercial addition polymers are polyolefins. Condensation polymers are made from monomers that have two different groups of atoms which can join together to form, for example, ester or amide links. The most common ways of classifying the polymer is to separate it into three large group which is thermoplastic, thermoset and elastomer.

### **2.1.1 Thermoplastic**

Thermoplastics are polymers that solidify as they are cooled, no longer allowing the long molecules to move freely. They also become softer when heated (Callister, 2008). In overall, thermoplastic polymer can be divided into two sub classes which are amorphous and semi-crystalline as shown in Figure 2.2.

Amorphous thermoplastic are those with molecules that remain in disorder as they cool, leading to material with a fairly random molecular structure (Oswald *et al.*, 2003). An amorphous polymer also solidifies, or vitrifies, as it cooled below its glass transition material. The examples of amorphous polymer are polystyrene, polycarbonate, plasticized polyvinyl chloride.

Semi-crystalline thermoplastic solidifies with a certain order in their molecular structure. So, when they are cooled, they harden when the molecules begin to arrange in a regular order below what is usually referred to as melting temperature (Oswald *et al.*, 2003). The molecules in semi-crystalline that are not transformed into ordered region remain as small amorphous regions. These amorphous regions within the semi-crystalline domains

lose their flow ability below their glass transition temperature. Most semi-crystalline polymer has a glass transition temperature at subzero so it behaving at room temperature as rubbery or leathery material (Callister, 2008). The example of semi-crystalline material is high density polyethylene, polypropylene, poly amide and polytetrafluoroethylene.

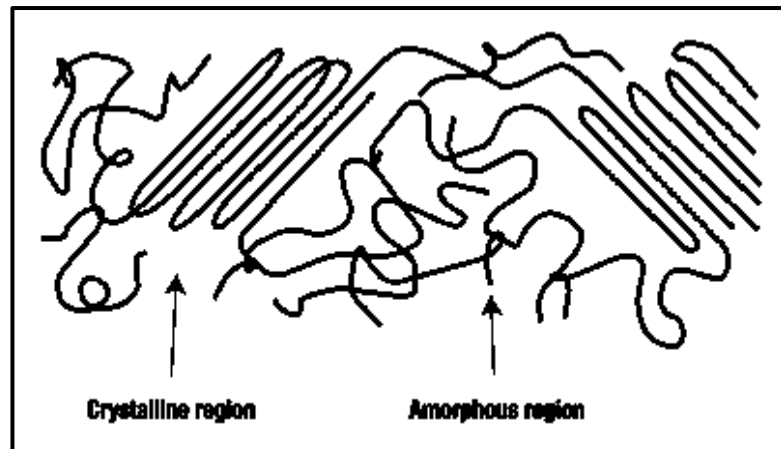


Figure 2.2: Polymer chains in amorphous and crystalline regions (Rosner, 2001).

### 2.1.2 Thermoset

According to Prime (1997), thermosets are network-forming polymers. Thermoset material solidifies when it is chemically cured. The long macromolecules cross link with each other resulting in a network of molecules that cannot slide past each other (Oswald *et al.*, 2003). The formation of these networks causes the material to lose the ability flow after reheating. The high densities of cross linking between the molecules make thermosetting material stiff and brittle.

Thermoset also exhibits a glass transition temperature which is sometimes near or above thermal degradation temperatures (Oswald *et al.*, 2003). The example of thermoset polymer is epoxy, melamine, phenolic and unsaturated polyester.

### 2.1.3 Elastomer

Elastomers are based on polymers which have the property of elasticity. They are made up of long chains of atoms, mainly carbon, hydrogen and oxygen, which have a degree of cross-linking with their neighboring chains. It is these crosslinking bonds that pull the elastomer back into shape when the deforming force is removed. However, there are two type of elastomer used today which is thermoplastic elastomer (TPE) and thermoset elastomer (TSE).

#### 2.1.3.1 Classification of elastomer

Elastomer can be divided into thermoplastic elastomer, thermoset elastomer, and rubber. Thermoplastic elastomers are materials which combine the easy process ability of thermoplastics and elastic behavior of rubbers. Amin *et al.*, (2011) stated that the unique properties of both materials exist because TPE material is created only by physical mixing of a thermoplastic and elastomer and no chemical or covalent bonding exist between the two. This behavior has opened a new field of polymer science.

Thermoplastic elastomer has become a significant part of the polymer industry. They are used in many applications like adhesives, footwear, medical devices, automobile parts house hold goods, etc. Holden (2000) documented that the thermoplastic elastomer can be classified into seven groups:

- a) Styrene block copolymer
- b) Crystalline multi-block copolymer
- c) Miscellaneous block copolymers
- d) Hard polymer/elastomer combination
- e) Hard polymer/elastomer graft copolymer