



**Comparative Study of Natural Rubber and Recycled
Natural Rubber as Toughening Agent in Epoxy Foam**

by

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LIST OF ABBREVIATIONS

EPs	Epoxy resin
NR	Natural rubber
rNR	Recycled natural rubber
LNR	Liquid Natural Rubber
DNR	Depolymerized natural rubber
phr	Per hundred rubber
DGEBA	Diglycidyl ether of bisphenol A
NBR	Acrylonitrile butadiene rubber
DVB	Divinylbenzene
HTBN	Hydroxyl terminated butadiene
CTBN	Carboxyl terminated butadiene-acrylonitrile
NCOPBER	Isocyanate terminated polybutadiene
rpm	Rotation per minute
SEM	Scanning Electron Microscope
T _g	Glass Transition Temperature
K _{IC}	Toughness
MWD	Molecular Weight Distribution
M _w	Molecular Weight

LIST OF SYMBOLS

MPa	Mega Pascal
GPa	Giga Pascal
%	Percentage
C°	Degree Celsius
NaHCO ₃	Sodium bicarbonate (SB)
H ₂ O	Water
NaOH	Sodium hydroxide
CH ₃ COOH	Acetic acid
CO ₂	Carbon dioxide
KHz	Kilo hertz
MHz	Mega hertz
C°/ min	Degree Celsius per minute
vol %	Volume percentage
wt %	Weight percentage
mm	Millimeter
g/cm ³	Gram per cubic centimeter
g/mole	Grams per mole

Kajian Perbandingan Getah Asli dan Getah Asli Dikitarsemula Sebagai Agen Penguatan di Dalam Busa Epoksi

ABSTRAK

Busa epoksi adalah salah satu bahan umum yang digunakan terutamanya sebagai bahan penebat dalam tujuan elektronik dan aplikasi aeroangkasa. Dalam usaha untuk menggunakan manfaat getah asli (NR) dan getah asli dikitar semula (rNR) pada sifat-sifat busa epoksi, getah mesti tersebar sekata di dalam epoksi. Di samping itu, jumlah getah asli dan getah asli yang dikitar semula memainkan peranan yang sangat penting untuk mempengaruhi sifat-sifat mekanikal di dalam busa epoksi. Penambahan agen peniup seperti natrium bikarbonat (NaHCO_3) kepada sistem epoksi dengan NR dan rNR sebagai agen penguatan telah disiasat. Sampel-sampel busa epoksi telah dihasilkan dengan menggunakan teknik pencampuran mekanikal. Natrium bikarbonat telah dipilih sebagai agen peniup disebabkan penimbangan mesra alam dan kos yang rendah. Manakala, getah asli dan getah asli dikitar semula mengawal kesan penguatan kerana busa epoksi adalah rapuh secara semula jadi. Kajian ini terdiri daripada tiga bahagian kajian. Pertama, kesan kandungan natrium bikarbonat, kedua, kesan kandungan getah asli dan terakhir, kesan kandungan getah asli dikitar semula ke atas sifat-sifat busa epoksi. Sifat-sifat mekanikal, fizikal dan morfologi telah dijalankan. Kandungan natrium bikarbonat telah diubah pada 5, 20, 15 dan 20 phr. Kandungan getah asli dan getah asli dikitar semula telah dikaji pada 5, 10, 15, 20 dan 25 peratusan isipadu (vol.%). Keputusan eksperimen menunjukkan kandungan tertinggi agen peniupan yang telah dipilih adalah pada 20 phr dan ia memberikan peratusan keliangan (%) yang tertinggi, tetapi ia menunjukkan nilai yang terendah dalam sifat-sifat mekanikal dan ketumpatan. Morfologi menunjukkan pada 20 phr kandungan natrium bikarbonat telah menghasilkan % struktur liang yang tinggi. Untuk bahagian kajian yang kedua, kandungan getah asli pada 10 isipadu % menunjukkan nilai optima untuk sifat-sifat kelenturan, keliatan patah, ketumpatan, % keliangandan morfologi. Morfologi untuk permukaan kelenturan dan keliatan patah telah dikdii dengan menggunakan Mikroskop Pencirian Elektron (SEM). Keputusan menunjukkan interaksi antara NR dan epoksi adalah lebih baik dibandingkan rNR di mana telah menunjukkan terdapat kelekatan yang lemah diantara epoksi dan rNR. Di sebaliknya, apabila dibandingkan antara NR dan rNR sebagai agen penguatan terhadap sifat-sifat busa epoksi, NR menunjukkan sifat-sifat kelenturan, keliatan patah, ketumpatan dan morfologi yang lebih baik dibandingkan kepada rNR.

Comparative Study of Natural Rubber and Recycled Natural Rubber as Toughening Agent in Epoxy Foam

ABSTRACT

Epoxy foam is one of the common materials used, which being applied especially as the insulator material in electronic purposes and aerospace applications. In order to maximize the potential of natural rubber and recycled natural rubber to be acted as effective toughening agent epoxy foam, the rubber phase must be distributed and dispersed well in the epoxy. In addition, the amount of natural rubber (NR) and recycled natural rubber (rNR) play very important role in influencing the mechanical properties of the epoxy foam. Addition of blowing agent: sodium bicarbonate (NaHCO_3) to the epoxy system with NR and rNR as toughening agent have been investigated. The epoxy foam samples were fabricated using mechanical mixing technique. Sodium bicarbonate was selected as blowing agent due to the environmental friendly and low cost concern. While natural rubber and recycled natural rubber controls the toughening effect because epoxy foam is brittle in nature. This research consists of three parts of studies. First, the effect of sodium bicarbonate content, secondly, the effect of natural rubber content and lastly, the effect of recycled natural rubber content on the properties of epoxy foam. Mechanical, physical and morphology properties were done. Sodium bicarbonate content was varied at 5, 10, 15 and 20 phr, respectively. Natural rubber and recycled natural rubber content were varied at 5, 10, 15, 20 and 25 volume percentage (vol %), respectively. The experimental results showed the maximum content of the blowing agent was selected at 20 phr and it gave the highest porosity percentage (%), however it revealed the lowest value in mechanical and density properties. The morphology shows that, at 20 phr sodium bicarbonate content produced the high % of porous structure. As for the second part of study, for natural rubber loading, 10 vol % revealed the optimum value of flexural, fracture toughness, density, porosity % and morphology properties. For third part of the study at 15 vol % of rNR showed optimum value of flexural, fracture toughness, density, porosity % and morphology properties. The morphology of flexural and fracture surface were observed by Scanning Electron Microscopy (SEM). The results showed the interaction between NR and epoxy matrix was better compared to rNR which showed there was poor adhesion between epoxy and rNR. On the other hand, when compared between NR and rNR as toughening agent on the properties of epoxy foam, NR toughened epoxy showed better of flexural, fracture toughness, density, and morphology properties compared to rNR.

CHAPTER 1

INTRODUCTION

1.1. Research Background

Epoxy resin was introduced in a commercial quantity in 1950 and their usage increased by about 20% per year in the first five decades (Bagheri et al., 2009). Epoxy resins are one of the most popular and useful thermoset polymers which exist in the form prepolymers or oligomers. They have been utilized expansively as high achievement adhesive composite materials owing to their exceptional thermal and mechanical properties for instance tensile strength, high modulus, high thermal stability, high glass transition temperature, low creep, excellent corrosion and weather resistance (Ratna & Banthia, 2004; Ben Saleh et al., 2009). They are being selected in this study because they are widely applied as the insulation material in microelectronic devices and component for packaging of computer chips and circuit board.

Thermoset polymers, as a whole, and epoxy resin, specifically, are well known as brittle and rigid polymeric materials. Even though strength and rigidity are required for numerous technical applications, lack of crack growth resistance or brittleness eradicates broader usage of epoxy resins. Thus, strengthening of epoxy has become a requirement to assure the practicability of these materials used for practical applications (Seng et al., 2011). Certain toughening modifier used for epoxy, more specially, blends of epoxy resins and toughening polymers are used for adhesives, coatings, especially as primers and electrical applications such as castings, coatings, encapsulants, compounds of potting and solder masking, and applications of laminates and construction such as civil engineering, flooring, concrete repair and consolidation, secondary containment of tankage, grouts, sealants, and polymer concrete, and structural composites, and tooling (Masse et al., 1995).

To date, there has been great progress in the making of foam product and generally for the use of cushioning to produce mattress, house whole used, others. In addition of these applications, foam similarly possesses a good need for engineering applications for example thermal insulations for buildings, packaging product, and also in automotive industries. The key advantage of foam above other kind of materials is it has low density which causes generating lightweight product (Ariff et al., 2008).

As for elastomer, the task to produce foam is assisting to its tedious formulations and great viscosity. Formulations play a very significant role in changing the whole properties of the produced foam and from time to time not compatible or mistakable formulations will result in cell collapse or no foam production completely. Altered amount and kind of blowing agent utilized will have an impact on the total cell growth as well as distributions of the cell growth of the foam. There are researchers who study the rubber foam however most of them employed mixture of two blowing agent, generally between sodium bicarbonate and azodicarbonamide (Ariff et al., 2008). The use of sodium bicarbonate by reason of volume of carbon dioxide release in the course of the decomposition and it decomposes at a relatively low temperature (145°C–150°C) and often results in an open-cell structure, which is suitable for use with polymer (Fauzi et al., 2015; Najib et al., 2009). Thus, in this specific study, sodium bicarbonate was used in order to assess its ability to promote expansion to the epoxy compound.

In recent years, toughening of epoxy resins has been the focus of deep investigations all over the world. The epoxy resins are largely successfully toughened through integrating rubbery filler as a separate stage of microscopic particles (Saleh et al., 2014). This can be accomplished in two methods: a) blending by functionalized liquid rubber possessing limited solubility which is miscible in the beginning, with the epoxy hardener mixture and experience phase separation at a particular stage of the curing reaction resulting to two phase microstructure, b) by dispersing preformed rubbery particles openly in the matrix of epoxy with no need of experiencing phase separation (Ratna & Banthia, 2004).

Elastomers such as natural rubber and recycled natural rubbers have been chosen as toughened agent in porous epoxy. Natural Rubber is an elastomer that was initially originated from latex. An incision prepared into the bark of the plant and the milk

coloured latex is composed and developed into a functional rubber. The sanitized form of natural rubber is chemical polyisoprene. Natural rubber is utilized in various products and applications. It is very flexible and stretchy and water proof. The purpose of the process is to alter the brittleness of epoxy matrix by increasing discrete rubbery phases to develop the toughness (Yishi et al., 1987).

In this study, recycled natural rubber is introduced as toughening agent into epoxy resin with sodium bicarbonate as blowing agent to improve the crack growth resistance of epoxy. The scrap rubber is rubber that not meets the product and processing specification, defective rubber product and leftover rubber from manufacturing products. Generally, the scrap rubbers are discharged and waste which can cause the environmental problems because they degrade rapidly and easily. To overcome and reduce pollution, the scrap rubber need to recycled (Baharin, et. al, 1997).

1.2. Problem Statement

Although these porous materials are both useful and successful in a number of applications, certain structural reinforcement applications in the electronics and automotive industry require a material having improved foaming properties (Michael et al., 2003). Porous materials are essential component to contribute products for the industrial markets such as filtration, aeration, sorbents, structural material, and sensor (Jansen et al., 1999). The percentage of porosity will influence the mechanical also dielectric of epoxy foam. Porosity was initiated to epoxy matrix because the complex dielectric constant is not required for moving the signal among electronic components.

This study has chosen sodium bicarbonate as blowing agent which is a food grade material and not harmful to the human and environmental friendly. Beside that, it is also a low cost material. The toughened agent loading is the critical parameter to investigate because different amounts of toughening agent will affect the presence properties of the epoxy porous material. Epoxy is brittle and has poor resistance to crack propagation. In order to improve toughness properties, different quantity of NR and rNR were used in this study. rNR was chosen as toughening agent in this study is due to

the abundant supply of material from waste glove. The effect of rNR as toughening agent was compared to NR. Acetic acid was added in order to promote the reaction rate of sodium bicarbonate (Wan Hamad et al., 2013).

1.3. Objectives of study

The objectives of this research are:

- 1) To study the effect of blowing agent loadings on the properties of epoxy foam.
- 2) To investigate the effect of natural rubber as toughening agent on properties of epoxy foam.
- 3) To analyze the effect of recycled natural rubber as toughening agent on the properties of epoxy foam.

1.4. Scope of study

One of the purposes of this study is to examine the effect of blowing agent loadings then the effect of toughening agent over the physical and mechanical properties. The first part of the study is to study the effect of blowing agent loadings. Blowing agent content were varied at 0, 5, 10, 15, and 20 phr. The second part of study is to investigate the effect of natural rubber content at 5, 10, 15, 20 and 25 vol %. Then, the effect of recycled natural rubber content at 5, 10, 15, 20 and 25 vol % on physical and mechanical properties which the processing were done at room temperature. The influence of the amount of toughened agent, to the viscosity, porosity, density, and mechanical properties of the epoxy foam were investigated.

CHAPTER 2

LITERATURE REVIEW

2.1. Thermoset materials

Polymer is one of the most significant materials in human life, with its synthesis technology that had been advanced since the early 19th century. Polymer is used in many applications, which affect every aspect of our lives. Plastic materials can be classified into two main categories, thermoplastics and thermosetting, depending upon the response of the polymer to cyclic heating. The difference can be traced back to the nature of the monomer, the polymerization reaction, the polymeric molecular structure and the bonding systems within the polymer (Pascault et al., 2002). A thermoplastic acts like a fluid which is above a particular temperature level, however the heating of a thermoplastic causes its degradation without a need to go through a fluid state. Typical thermoplastics are polystyrene (PS), polyethylene (PE), polyvinyl chloride (PVC) and polypropylene (PP). The thermoplastic or thermosetting feature of a plastic depends on the possibility of reaching to a fluid state by heating. This state can be reached only if single macromolecules is separated to produce flow (Ratna & Banthia, 2004).

Thermosets, plastic produced via curing of liquid monomers, signify approximately 18% of the overall plastic market. Clear examples of thermosetting plastics are urea formaldehyde resins, phenolic and epoxy resins, and unsaturated polyesters. Among them, epoxy polymer is the most famous plastic used for many applications. It is giant macromolecules that include covalently bonded repeating units made among the polymerization reaction. Its nature of a polymer is as a result of existence of a crosslink made by covalent bonds. Besides, thermoset polymers are ordinarily noncrystalline as there is no possibility to order portions of the network structure because of the restrictions enforced by the occurrence of crosslink (Pascault, 2002). The thermoset resin is significant since it has high modulus and comparatively

high range of suitable temperatures. Thermosets have high modulus because of their dense crosslinking. Not like thermoplastic polymers as the processing of thermosets consist of the curing reactions. Cure of thermosets is presented in Figure 2.1 meant for a polymer with co-reactive monomers for instance an epoxy-diamine phase. For the reaction of a difunctional with a trifunctional monomer is considered. Reaction in the first steps of cure (a) to (b) yields bigger and branched molecules, increasing in molecular weight accelerates, bringing about an rise in viscosity and reduces the entire number of molecules (Prime, 2005).

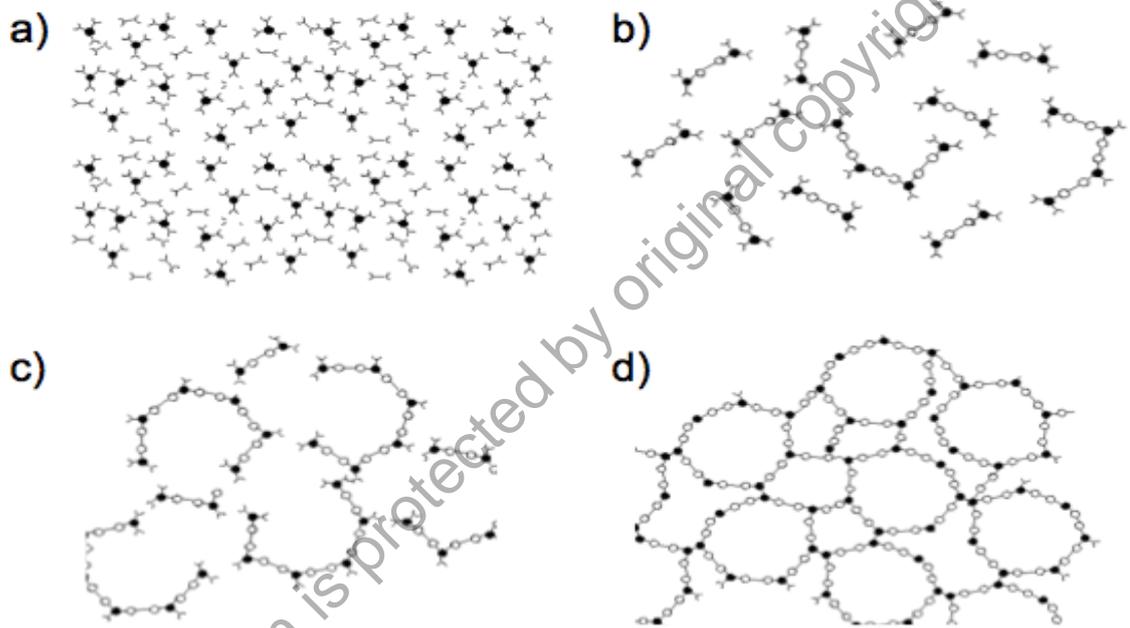


Figure 2.1: Cure of thermosets (Prime, 2005).

2.1.1. Epoxy

Epoxy resins form one of the main classes of industrial thermoset resins. They are the mostly used in structural (structural adhesives) and electronic (microencapsulation) applications. They are very well suited for electronic encapsulation owing to their good electrical resistance. Epoxies are described by a three-membered ring identified as the epoxy, oxirane, epoxide, or ethoxline group, as shown in Figure 2.2 (Ahmetli et al., 2015).

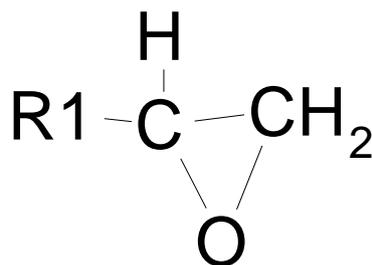


Figure 2.2: Functional group of epoxide (Ahmetli et al., 2015).

Epoxy resin is prepared through the reaction of compounds comprising an active hydrogen group with epichlorohydrin and subsequently by dehydrohalogenation with existence of a stoichiometric amount of NaOH as shown in Figure 2.3. Since both the chlorine and the epoxy group in epichlorine are reactive toward the hydroxyl group in bisphenol A, a linear polymer is produced. (Carrillo et al., 2011), reported that some epoxide of various kinds were prepared from epichlorohydrin and diphenylolpropane (bisphenol A) and identified as novalac resin (Carrillo-Castillo & Osuna-Alarcón, 2011).

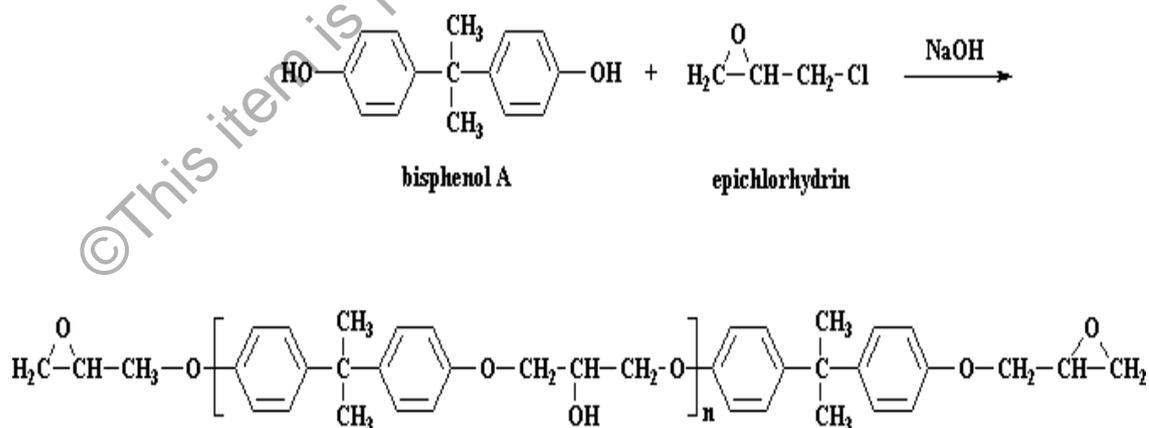


Figure 2.3: Reaction between bisphenol A and epichlorohydrin (Carrillo, 2011).

The epoxide resin was cured with several compounds, for example metaphenylenediamine and diaminodiphenylmethane. The highest adhesive strength is acquired when the stoichiometric quantity of curing agent and high temperature of

curing are utilized. Epoxy can be crosslinked by co-reacting an amine or an acid anhydride to give tough, dimensionally stable products.

Epoxy are identified for their outstanding adhesion, good-to-excellent mechanical properties, chemical heat resistance, and very good electrical insulating properties as it protects electrical components from dust, short circuiting and moisture (Raghavachar et al., 1999). Their good adhesion characteristics with glass, aramide, carbon and inorganic fibers have resulted a successful matrix for fiber composites (Nikhil & Samuel, 2014). Epoxy resin is mostly used in coating, surface protection and electrical and also electronic application. The traditional used of epoxy is in electrical insulation, cast high voltage insulators, and electrical laminates. Despite these required properties, the main disadvantages are their poor crack resistance, low toughness and brittle nature at room temperature (Najib et al., 2009).

Glassy, extremely crosslinked epoxy resins are well-known as brittle thermoset which require to be toughened for several applications were the first to display that the fracture toughness of epoxy can be developed by the introduction of a distributed rubber phase (Jansen et al., 1999). The limitation of epoxy polymers in industrial application owing to its brittleness properties, low impact resistance and processing fracture energy lesser than industrial thermoplastics and metal (Seng et al., 2011). This fundamental brittleness result in the bad damage tolerance to effect of the composite prepared from epoxy and bad shear strength and peeling of epoxy created adhesive (Ratna & Banthia, 2004).

2.2. Curing agent (Hardener)

The neat epoxy resin is a viscous fluid and do not has any practical value. Crosslinking agents were used to convert the neat epoxy resin to solid. Crosslinking of epoxy resins with hardeners or curing agents exhibits three-dimensional infusible and insoluble crosslinked. Epoxy resin can be treated with a wide range of hardeners. The option of curing agents or hardeners is contingent on the essential chemical and physical properties, processing techniques and curing states (Yuhana et al., 2012). There are including amines, polyamides, phenolic resin, anhydrides, isocyanates and

polymercaptans. Triethylenetetramine (TETA) is one of the examples of highly reactive primary aliphatic hardener of polyamine monomer. When the component mixed with each other, the amine group will act in response with epoxy group to create a covalent bond and thus form polymer that heavily crosslinked, rigid and strong chemical structure. Triethylenetetramine (TETA) structure is shown in Figure 2.4.

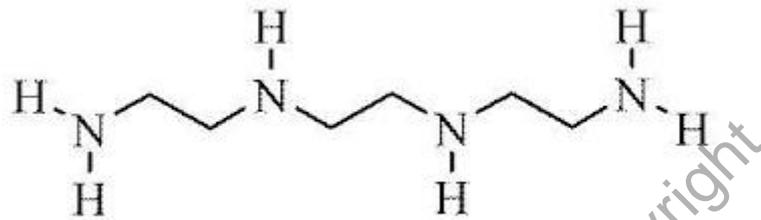


Figure 2.4 : Triethylenetetramine.(Weinmann, Dangayach, & Smith, 1996).

The glass transition temperature of cured systems and the cure kinetics are reliant on the molecular structure of the curing agent. Applying variable kinds and quantity of curing agent which be likely to control crosslink density contrast the structure. Polyamide is also another type of hardener which when reacts with epoxy group and form covalent bonding and the resulting of heavily crosslinking, rigid and strong polymer. Figures 2.5 and 2.6 show the chemical reaction between diamine and diepoxy, amine and epoxy, polyamide and epoxy, respectively. Figure 2.7 shows the chemical reaction between epoxy and polyamide.

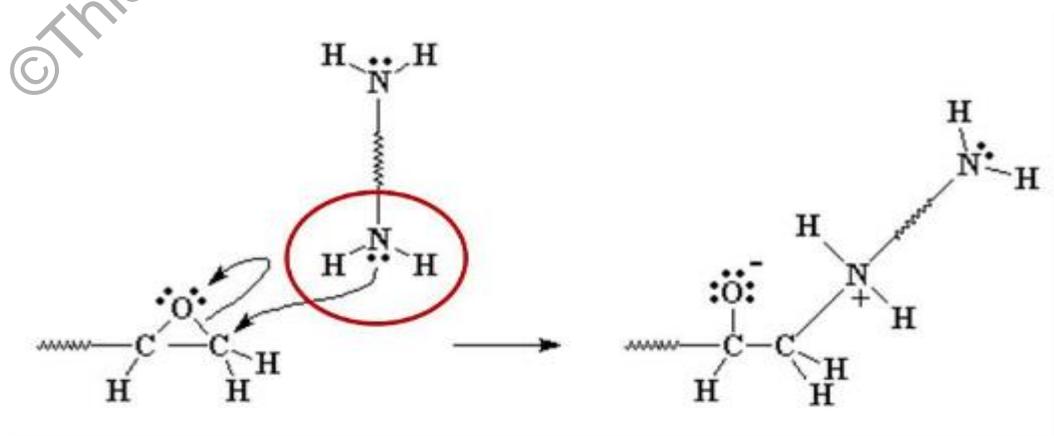


Figure 2.5: The chemical reaction between diamine and diepoxy (Lee, 2002)

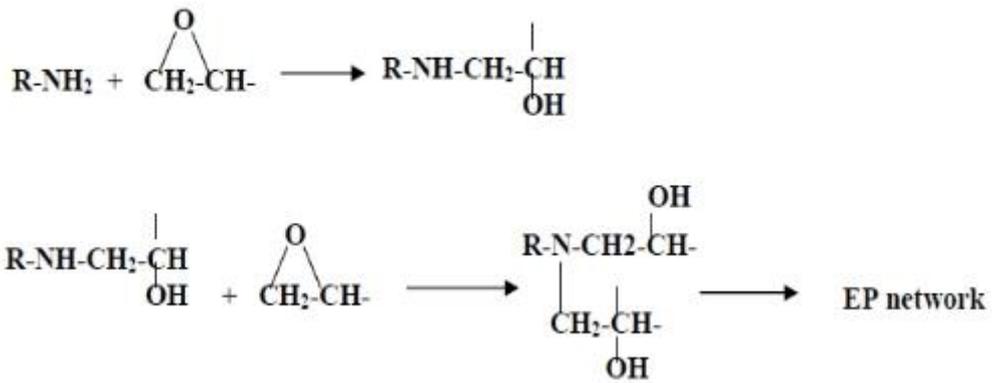


Figure 2.6: Curing reaction between amine and epoxy (Weinmann, 1996).

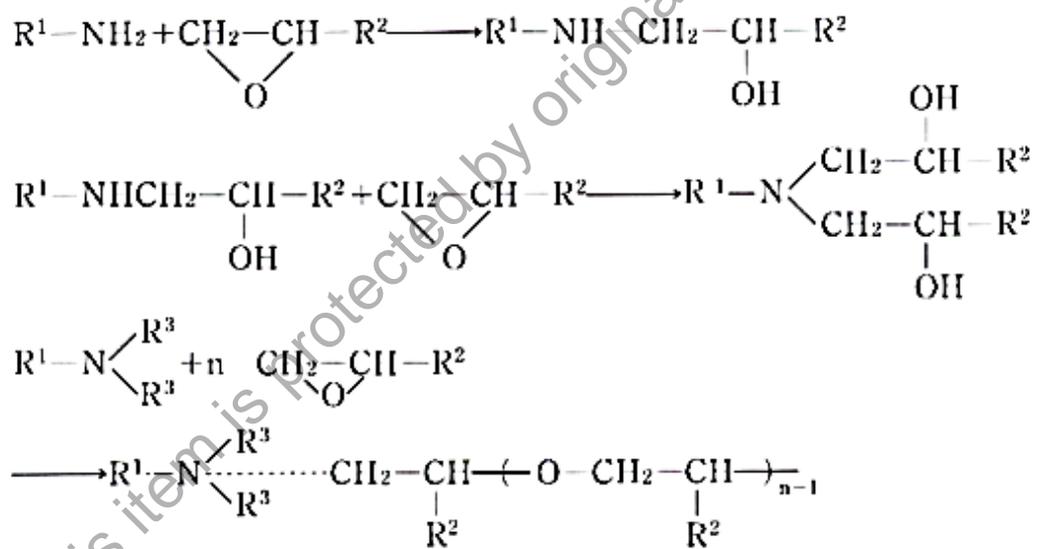


Figure 2.7: Chemical reaction of polyamide and epoxy (Bilyeu, 2000).

Numerous of improved amines have been traded commercially. The reaction between of the amine and a mono or poly-functional glycidyl material is utilized in higher dosage in order for the errors in measuring the curing agent to be reduced extensively. Resinous adducts are produced by reacting spare of diamine with epoxy resin. The great molecular weight of the adduct gives a more needed resin to curing agent proportion and develops processibility of an epoxy composite. Overall, hardener