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**The effect of recycled rubber on the properties of epoxy/ recycled rubber blends filled with carbon black**

**By**

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## LIST OF ABBREVIATIONS

CPC	Conductive Polymer Composites
ICPs	Intrinsically Conductive Polymer
LR	Liquid Rubber
CB	Carbon Black
PLLA	Poly (L-lactide) Acid
PMMA	Polymethyl Methacrylate
HIPS	High Impact Polystyrene
DVB	Divinylbenzene
HTPB	Hydroxyl Terminated Butadiene
CTBN	Carboxyl Terminated Butadiene-acrylonitrile
NCOPBER	Isocyanate Terminated Polybutadiene
EPDM	Ethylene Propylene Diene Monomer
HIPS	High Impact Polystyrene
SEM	Scanning Electron Microscopy
TGA	Thermogravimetric Analysis
ABS	Acrylonitrile-Butadiene-Styrene
UCST	Upper Critical Solution Temperature
CTBN	Carboxyl-terminated butadiene
CTE	Coefficient Thermal Expansion
PMC	Polymer Matrix Composite
PI	Polyimide
PES	Polyethersulfone
PEEK	Polyetheretherketone



PEI	Polyetherimide
PPS	Polyphenyl sulphide
WPE	Weight Per Epoxide Group
Phr	Per Hundred Resin
EP	Epoxy Resin
LEDs	Light Emitting Diodes
DETA	Diethylenetriamine
TETA	Triethylenetetramine
MPDA	M-phenylenediamine
RNH <sub>2</sub>	Primary Amines
DGEBA	Diglycidyl Ether of Bisphenol A
DGEBF	Diglycidyl Ether of Bisphenol F
SEN-T	Static Tensile Loading
RR	Recycled Rubber
ROM	Rules of Mixtures
UTM	Universal Testing Machine

## LIST OF SYMBOLS

$V_f$	Volume fraction of filler
$V_m$	Volume fraction of matrix
$\rho$	Density
$M_f$	Mass of filler
$M_m$	Mass of matrix
$\rho_f$	Density of filler
$\rho_m$	Density of matrix
$\underline{\sigma}_f$	Stress in outer fibres at midpoint, (MPa)
$\epsilon_f$	Strain in the outer surface, (mm/mm)
$E_f$	Flexural Modulus of elasticity,(MPa)
$F$	Load at a given point on the load deflection curve, (N)
$\sigma_f$	Stress in outer fibres at midpoint, (MPa)
$L$	Support span, (mm)
$b$	Width of test beam, (mm)
$d$	Depth of tested beam, (mm)
$D$	Maximum deflection of the centre of the beam, (mm)
$m$	The gradient (i.e., slope) of the initial straight-line portion of the load deflection curve,(P/D), (N/mm)
R	Resistance
A	Cross-sectional area
h	Sample Thickness

# KESAN GETAH DIKITAR SEMULA ATAS SIFAT-SIFAT ADUNAN EPOKSI/GETAH DIKITAR SEMULA TERISI KARBON HITAM.

## ABSTRAK

Dalam kajian ini, kesan karbon hitam (CB) dan pembebanan getah dikitar semula (RR) ke atas sifat-sifat dan pencirian epoksi / kitar semula getah / karbon hitam telah dikaji. Pada bahagian pertama, kesan kandungan karbon hitam yang berbeza telah disiasat. Komposit ini disediakan dengan menggunakan pengacau mekanikal pada kelajuan 600rpm pada suhu bilik. Kesan karbon hitam (CB) pemuatan epoksi/CB pada ketumpatan, sifat kekuatan lenturan, keliatan pematahan, sifat morfologi, elektrik dan haba telah disiasat. Karbon hitam yang telah digunakan sebagai pengisi yang telah diuji pada 5, 10, 15 dan 20 vol.% pembebanan. Ketumpatan epoksi/CB meningkat apabila kandungan CB meningkat. Tidak seperti ketumpatan, sifat-sifat lenturan, dengan setiap penambahan CB, sifat lenturan menunjukkan arah yang menurun. Pada keliatan pematahan, hasilnya menunjukkan bahawa, dengan setiap peningkatan kandungan CB, ketahanan epoksi/CB menurun. Pada bahagian pertama, kandungan 15vol% daripada CB telah dapat mencapai puncak tertinggi kekonduksian. Dengan penambahan CB pada epoksi komposit dapat meningkatkan kestabilan terma epoksi/CB komposit berbanding dengan epoksi kawalan. Sementara itu dalam bahagian kedua, yang 15vol.% kandungan daripada karbon hitam telah digunakan untuk menentukan kesan penambahan berbeza kandungan getah kitar semula terhadap epoksi/getah dikitar semula/karbon hitam. Kaedah pemprosesan yang sama telah digunakan seperti bahagian yang pertama dan kesan penambahan berbeza memuatkan getah kitar semula pada 0, 2, 4, 6, 8 dan 10phr pada ketumpatan, sifat lenturan, keliatan pematahan, morfologi, sifat elektrik dan kestabilan terma disiasat. Dengan penambahan 15 vol.% daripada CB untuk epoksi/RR gabungan, meningkatkan sifat-sifat ketumpatan. Dalam ujian lenturan, penurunan yang seragam dalam kekuatan lenturan epoksi/RR dari 0-8phr. Kehadiran zarah getah di dalam epoksi komposit akan mengurangkan ketegaran yang epoksi/RR dan epoksi/RR/CB pada modulus lenturan. Walau bagaimanapun, modulus lenturan untuk epoksi/CB/RR adalah lebih tinggi menunjukkan bahawa komposit menjadi lebih kaku apabila CB diperkenalkan. Modulus lenturan tertinggi adalah pada 4phr pembebanan RR untuk kedua-dua epoksi/RR dan epoksi/CB/RR. Dari SEM micrograph untuk epoksi/RR, ia boleh diramalkan bahawa apabila CB adalah memperkenalkan dalam epoksi/RR, ia lebih cenderung untuk mengelilingi zarah RR dan seterusnya membentuk gumpalan dan menyebabkan menurunkan sifat-sifat lenturan. Manakala dalam keliatan pematahan, untuk epoksi/RR/CB nilai optimum dapat dilihat dengan jelas pada 6phr untuk kedua-dua epoksi/RR dan epoksi/RR/CB komposit. SEM micrograph untuk epoksi/6 phr RR/CB menunjukkan bahawa retak tersebut bergerak dari arah sisi ini menyumbang dalam meningkatkan sifat-sifat kekuatan pematahan. Kekonduksian elektrik untuk epoksi /CB 15vol% kandungan CB adalah  $(3.80 \times 10^{-7} \text{-cm})$  dan pada 6 phr daripada pembebanan RR untuk epoksi/RR/CB adalah  $(3.27 \times 10^{-9} \text{-cm})$ , ini menunjukkan bahawa kekonduksian elektrik telah meningkat sebanyak 2 langkah magnitud. Tidak ada peningkatan dalam kestabilan terma kecuali bahawa ia boleh menyebabkan kestabilan terma yang lebih rendah daripada bahan epoksi kemas. Penambahan pembebanan RR hanya menambahkan penguatan. Sebaliknya dengan epoksi/6 RR/CB, apabila CB ditambahkan, telah meningkatkan lagi kestabilan terma komposit.

# THE EFFECT OF RECYCLED RUBBER (RR) ON THE PROPERTIES OF EPOXY/RECYCLED RUBBER BLENDS FILLED WITH CARBON BLACK.

## ABSTRACT

In this study, the effect of carbon black (CB) and recycled rubber (RR) loading on properties and characterization of epoxy/recycled rubber/carbon black were studied. In the first part, the effect of different carbon black loading was investigated. The composites were prepared by using a mechanical stirrer at speed of 600rpm at room temperature. The effect of carbon black (CB) loading of epoxy/CB on density, flexural properties, fracture strength, morphological, electrical and thermal properties were investigated. The carbon black that were used as a filler at 5, 10, 15 and 20 vol.% of loading. Density of the epoxy/CB increased as CB loading increased. Unlike density, the flexural properties, with every increase in CB loading the flexural properties show a declining trend. For fracture toughness, the result shows that, with every increase of CB filler loading, the toughness of epoxy/CB decreased. On the first part, a 15vol% of CB was able to reach the percolation. With addition of CB to epoxy composite was able to improve the thermal stabilities of epoxy/CB composite if compared to neat epoxy. Meanwhile in the second part, the 15vol.% of carbon black was used to determine the effect of different recycled rubber loading of epoxy/recycled rubber/carbon black. The same processing method was use and the effect of different loading of recycled rubber loading at 0, 2, 4, 6, 8 and 10phr was investigated on density, flexural properties, fracture toughness, morphology, electrical properties and thermal stability was investigated. With addition of 15 vol.% of CB to epoxy/RR blend, increases the density properties. In flexural testing, a uniform decrease in flexural strength of epoxy/RR blends from 0-8phr. The presence of rubber particle in epoxy composite should reduce rigidity of the epoxy/RR and epoxy/RR/CB in flexural modulus. However, the flexural modulus for epoxy/CB/RR were higher showing that the composites becomes more rigid when CB was introduced. The highest flexural modulus was observed at 4phr of RR loading for both epoxy/RR and epoxy/CB/RR. From SEM micrograph for epoxy/RR, it can be predicted that when CB is introduced in epoxy/RR, it tend to go surround the RR particles and thus form agglomerates and result in lowering the flexural properties. Whereas in fracture toughness, for epoxy/RR/CB optimum value can be clearly seen at 6phr for both epoxy/RR and epoxy/RR/CB composite. SEM micrograph for epoxy/6 phr RR/CB shows that the crack was initiated from the poles contribute in increasing the fracture toughness properties. The electrical conductivity of epoxy/CB at 15vol% of CB loading ( $3.80 \times 10^{-7} \Omega\text{-cm}$ ) and at 6 phr of RR loading epoxy/RR/CB ( $3.27 \times 10^{-9} \Omega\text{-cm}$ ), it showed that the electrical conductivity was improved by 2 order of magnitude. There was no improvement in thermal stabilities except that it could resulted in lower thermal stability than neat epoxy material in spite of more toughening behavior observation. Contrarily with epoxy/6 RR/CB, upon addition of CB, improves the thermal stability of the composites.

# CHAPTER 1

## INTRODUCTION

### 1.1 Background History

Conductive polymeric composite (CPC) are the functional composite consisting of insulating organic polymeric material and several conductive substances. Electrically conductive polymers are mainly fractionate into two categories that is intrinsically conductive polymers (ICPs), in which the electronic structure (Simon, 2003) of these polymers is responsible for their conductivity, and CPC, in which the inclusion of conductive fillers to the polymer imparts the composites with conductivity. CPCs are better in processing, low cost and the electrical properties are adjustable (Pang et al., 2014). The common type of conductive fillers used are carbon black, carbon fibres, metal particles and conducting polymers such as polyaniline (Feller, Linossier, & Levesque, 2002). Significantly, elastomers and plastics are insulators (dielectrics) to which conductivity are transferred by the addition of a finely divided or colloidal filler of high intrinsic conductivity, such as carbon black (Nasr et al., 1997).

The brittleness of the highly cross-linked epoxy resins have made it one of the most useful and popular thermoset polymers and possesses many excellent properties including high strength, excellent creep resistance, high thermal, chemical and environmental stability. Thus, the epoxy resin posses the highest fracture energy among crosslinked glassy polymer and this be one of the reason for their widespread use in the field of composite material and structural resin (Levita, De Petris & Marchetti, 1991).

Epoxy resin are used extensively in the composite materials for a variety of demanding structure requirements (Reinhart, 1987). As in accordingly, of their high cross-link densities, structural epoxy materials are innately brittle with poor resistance to crack initiation and growth. When exposed to mechanical stresses it can fail prematurely (Kinloch, 1989).

Over the years, research has shown that it is possible to toughen a material by using different additives and techniques, one of it by introducing rubber into the resin. The rubber will utilize the presence of inhomogeneity in epoxy matrix and consequently, resulted in promoting the toughening of the resin (Sue et al., 1996). Novoa et al, (2006) proved that the addition of rubber particles improved toughness of material and this was achieved by separation of rubber particles from the solution which result in the formation of a second phase at the materials interfaces. The formation of the second phase promote and effective in toughening effect. Other factor such as the type of rubber, particles size and dispersion also can greatly affect the toughening. Such that, in the past three decades, an effective and frequently used method using a reactive liquid rubber (LRs) forming a 1-10  $\mu\text{m}$  of particles during cure are used in order for improving toughnes of epoxy composites (Kinloch et al., 2008; Gabr et al., 2010).

In order to preserved mechanical properties of the composites with a suitable electrical conductivity, the lowest CB content was used. Theoretically, the most effective approach is to selectively localize the CB particles between polymer powders or at the interface of an immiscible polymer blend (Feng & Chan, 1998). But firstly it is important to understand that, according to (Ling, Ng, & Yeoh, 2011), a critical concentration of filler beyond which the polymer composite becomes conductive are

referred to as the percolation threshold. In recent years, immiscible blends was used to reduce the percolation threshold and also to enhance the composite conductivity at low CB content (Thongrungsri et al., 2002; Cui et al., 2007; Gubbles et al., 1995). This can be attributed to a ternary system of CB with two immiscible polymers. In this system, CB will selectively disperse in one of the two phases or accumulate at the interface because CB was found to have different affinity to each polymer in the blend (Mamunya, 1999). The percolation in such blends was determined by the percolation of the CB rich phase and the continuity of this phase in the blend. The selective localization of the filler at the interface provides a composite with the lowest filler loading if the interfacial region was continuous. This provide a better mixture processability and avoid poor mechanical properties caused by high-filler loading (Huang, 2002; Gubbles et al., 1995).

There are many research had been done, among these are from Buys et al,(2010), which experimented with carbon filler introduced in poly(L-lactide) (PLLA) /poly(methyl methacrylate) (PMMA) to reduce the carbon black content in polymer CPCs. Likewise with Pötschke, Bhattacharyya, & Janke, (2004), which stated that conductive polymer blend was a class of materials that syndicates two tasks at the same time in order to get conductive at very low carbon black loadings by creating co-continuous blend structures using the double percolation concept and to use the benefit of polymer blends concerning the improvement of mechanical or other properties. Besides the factor of immiscibility, another factor such that the electrical conductivity of a composite material with a polymeric matrix should be taken into consideration as been mentioned in (Scarisbarick, 1973; Pierre et al., 1990; Wessling, 1988). Hence, in this work, the mechanical, electrical and thermal stability properties of epoxy resin toughened with recycled latex rubber filled with carbon black was investigated.

## 1.2 Problem Statement

It was mentioned earlier that thermosetting polymers, in general, and epoxy resins, in particular, are identified as rigid and brittle polymeric materials. While rigidity and strength are desired for many engineering applications, brittleness or lack of crack growth resistance eliminates practical usage of epoxies. Therefore, addition of a second phase, either soft or rigid, can be a solution to this problem by improving the fracture toughness of brittle epoxies (Garg & Mai, 1988). The addition of second phase or the modification can be done by adding a rubber particle to add toughness to the epoxy. But when it comes to modify the brittle nature of epoxy, an increase in toughness without any reduction in the other important properties is required, such as modulus, thermal properties and environmental corrosion resistance.

According to Smith, (1965), the first article concerning rubber as the toughening agent in polymer was published in 1956 by Merz et al, (1996) which addressed a rubber-toughening mechanism for a high impact polystyrene (HIPS) system. Since then, many researchers have studied the mechanism of rubber as the toughening agent in plastics (Riew & Smith, 1989). The poly(ethylene terephthalate) toughened by natural rubber had been discussed in (Sheir et al., 1994). The dispersion of the rubber particles in the matrix is one of the main factors that affect the mechanical properties of the material. Previous research had been done in (Kambour, 1973), whereby by giving a rubbery content to the uncured epoxy resins had improve toughness and this also had been



proved in many studies related on modification of epoxy resin with liquid rubber, for example, the liquid rubber modified by divinylbenzene (DVB), hydroxyl terminated butadiene (HTPB), carboxyl terminated butadiene-acrylonitrile (CTBN), or isocyanate terminated polybutadiene (NCOPBER) (Broutman & McGarry, 1965; Kramer, 1983; Bucknall & Smith, 1965)

It has been known that the electro-conductive carbon black has excellent conductivity, but it also had some drawback which it is limited by issues of hardness and dispersibility (Chiu et al., 2011). Moreover, Premamoy Ghosh, (2000) had mentioned that, by increasing carbon black loading produce a monotonic increase in density and hardness and also in tensile strength with a equalizing of trend for carbon black filler loading that is more than 40 phr. Therefore in this research, a study are focused on a new material using recycled latex rubber as the modifier to increase toughness. The loading effect of carbon black (CB) filler on the Epoxy/Recycled latex rubber has also been studied on the conductive mechanism, electrical conductivity and mechanical properties epoxy/Recycled latex rubber/CB.

### **1.3 Objectives**

In this research, objectives has been focused on the determination of filler (carbon black) content to reached the percolation threshold. The used of recycled latex rubber as a new materials, is an approach to introduced a second phase material which was added to impart toughening to the composites material. As the second phase material are introduced, double percolation, the combination of percolation of two

polymer phases (recycled rubber latex and epoxy) and the percolation of carbon black need to be achieved. Therefore, the objectives of this study are:

- i. To study the effect of CB content on the flexural, fracture toughness, thermal and conductivity properties of the composites.
- ii. To study the effect of ratio of epoxy/RR on the properties of epoxy/Recycled rubber/CB composites.

#### **1.4 Scope of Study**

The development of this research based on the electrically conductive polymer are led by the drawbacks on the properties of polymer itself. Polymer are an insulating material which have a low charge carrier that make it non-conductive. Therefore in this research study, the properties of electrically conductive composite of epoxy/RR/CB on the mechanical, conductivity and thermal properties are investigated. There were two stages that are involved, firstly, the determination on the ratio of epoxy/CB on the composite. The percolation concentration ratio for epoxy/CB are achieved at about 15 vol. %. With respect to this, a conductive composite of epoxy/RR/CB are made and tested.

The epoxy/RR/CB composites are prepared by using mechanical mixing. The solution is then transferred to a (12cm x 12cm 0.5cm) size mold which was then undergo a degasification process before cured in an oven. The final product will be cut and polish for measurement. The density of the composites are tested by using the pycnometer. To study the mechanical properties of epoxy/CB and epoxy/RR/CB, flexural and fracture

toughness are tested using an instron. The filler dispersion and also the surface fracture morphology of flexural and fracture toughness for epoxy/CB and epoxy/RR/CB are observed and studied using a Scanning Electron Microscopy (SEM). To determine the amount or content of filler on epoxy/CB, electrical resistivity and conductivity test are conducted. For thermal properties, Thermogravimetric analysis (TGA) are were done using PerkinElmer TGA7.

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## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Polymer Blends

Polymer blend is macroscopically homogeneous mixture that comprise of two or more different species of polymer (Whisson, 1987). In many instances, the reason for this is to produce tough polymers and frequently this involves blending a glassy amorphous thermoplastic with a rubbery material as, for example, with toughened polystyrene, a blend of glassy polystyrene with rubbery polybutadiene, and the acrylonitrile-butadiene-styrene (ABS) plastics (Hoa, 2009). For examples, a 20% blend of acrylonitrile butadiene-styrene copolymer and 80% polycarbonate generated a material that balances the best of that displayed excellent resistance to deformation, high impact strength, and improved stress crack resistance relation to the unblended polycarbonate.

When mixing two polymers, the resulting material may be categorized as either a miscible or immiscible blend. Miscible blends happened when the mixing conditions and the polymer chemistry of the components allows the two polymers to completely mixed at a molecular level. In most of the systems, such as epoxy resins, the rubber

toughened may be soluble in the other phase, so the phase separation will be occurred during cure. This is an example of phase separation during reactive processing.

The achievement of enhanced toughness in a brittle polymer such as an epoxy resin is vital in applications for which peel strength and resistance to interfacial de-bonding through crack propagation are important criteria for performance. This may be achieved by incorporation of elastomeric or other phase-separated particles into the network. These may be present before reaction or they may form during the process of crosslinking and three-dimensional network formation. In the latter process it has been known that there may be three possibilities (Fouassier & Jan, 1993).

- A dispersion of elastomer particles in the matrix material.
- A phase-inverted dispersion of the matrix as particles in an elastomer-rich continuous phase.
- Two co-continuous phases.

The process of phase separation during cure arises from the change in the phase diagram as the cure reaction of the epoxy resin progresses. This is shown schematically in figure 2.1 (Fouassier & Jan, 1993) for a system with an upper critical solution temperature (UCST) in which the lower curve denotes the system miscible at room temperature, with the fraction ( $\phi$ ) of elastomer corresponding to the initial composition of the rubber - epoxy resin system before any cure reaction has taken place.

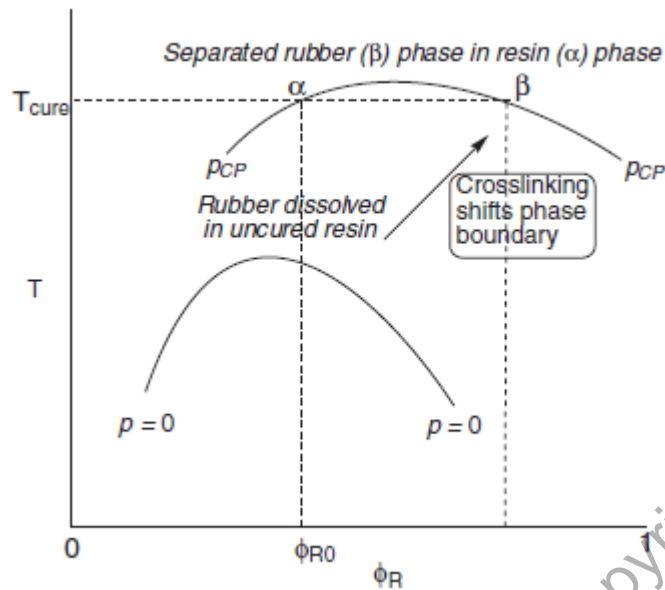


Figure 2.1: Change in the phase boundary (UCST) during the cure reaction for a system of a rubber (e.g. Carboxyl-terminated butadiene, CTBN) dissolved in an epoxy resin. Toughening requires that phase separation be achieved during the cure cycle (Fouassier & Jan, 1993).

The main parameter that it is crucial to control in the reactive phase separation is the diameter of the elastomer particle. This is because the toughness of the resulting network is controlled by the energy-absorbing mechanisms such as particle cavitation and rubber bridging of cracks. Another importance is the limitation of the effect of the rubber dispersed phase on the critical properties of the cured epoxy resin such as the stiffness and glass transition temperature. This will be affected by the extent to which the rubber dissolves in the matrix-rich phase (Fouassier & Jan, 1993).

## 2.2 Polymer Composites

Composite materials are the combination of different types of materials and can be called as multiphase material in order to gained properties that an individual component cannot attained. If they are formed naturally by a reactions, phase transformation or other types of phenomenone, they cannot be called as multiphase

materials. One the example for composite materials are the carbon reinforced polymer. Moreover, the composite materials can be suited for various properties by choosing their components appropriately, their proportions, their distributions, their morphologies, their degrees of crystallinity, their crystallographic textures, plus the structure and composition of the interface between components. Due to this strong tolerability towards processing, composite materials can be designed to fulfill the needs of technologies relating application in the area of aerospace, automobile, electronics, construction, energy, biomedical and other industries. Hence, composite materials is suited to be the most commercialize engineering materials.

The technology and cost of the composite materials can be determined through the processability of how the composites are combined to form a product. The ability of the components to merge and formed a cohesive material largely determined it is processability. This processing is always involved the use of high temperature and pressure. The type and properties of matrix can influence time, pressure and temperature that is used in a process. Using a high temperature for the bonding process between filler and matrix may cause some drawbacks such as bond weakening and even debonding during the cooling process due to the difference in thermal contraction (relating to the coefficient thermal expansion, CTE) between the filler and matrix themselves. Consequently, the filler will be less effective to be used as a reinforcement automatically, the mechanical properties of the composite is reduced.

### **2.2.1 Polymer Matrix Composites (PMC)**

Classification of polymer matrix composites is based on type of matrix whether it is thermoset or thermoplastic. Polymeric composites are much easier to fabricate compared to metal-matrix, carbon-matrix and ceramic-matrix type of composites. The used of comparatively low temperature to fabricate polymer matrix composites is one of the reason. For thermosets, such as epoxy, phenolic, and furfuryl resin, the processing temperature typically ranges from room temperature to about 200°C for thermoplastic polymers, such as polyimide (PI), polyethersulfone (PES), polyetheretherketone (PEEK), polyetherimide (PEI), and polyphenyl sulfide (PPS), the processing temperature typically ranges from 300 to 400°C. For thermosets, have been used for a long time in the production of carbon fiber composites. Throughout curing, usually done in the presence of heat and pressure, a thermoset resin hardens slowly or can be said as curing, due to the completion of polymerization and the associated crosslinking of the polymer molecules (Chung, 2010).

### **2.2.2 Epoxy Resin**

Epoxy resins were introduced as commercial, petroleum derived products in the early 1950s. Since then, they have grown to an annual usage in the United States of over 300,000,000 pounds per year. The major member of the epoxy class of resins is the bisphenol-epichlorohydrin family. While low viscosity liquid resins account for the major share of the chemically resistant mortars and grouts, some solid resins are also used in solvent solution as surface coatings to provide protection to steel and concrete surfaces from corrosion. About 10,000,000 pounds of epoxy are used annually in