OPTIMIZATION OF CATHODE WASTE MATERIALS ON EPOXY BASED CONDUCTIVE PAINT PROPERTIES

by

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CERTIFICATION OF APPROVAL

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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

MOHD SAHRUL BIN MD JAEDAN

ABSTRACT

Battery waste of as filler was mixed with Epoxy Resin and Polyamide at the preparation of electrically conductive paint coating, which led to enhancement of their electrical-mechanical properties. In this study, only the optimized mixing ratio and particle size was used in the preparation which determined from experiments of mixing ratio and particle distribution analysis. The grinding process, particle analysis distribution, electrical resistivity, macrostructure and adhesion of the conductive paint test have been carried out using Grinder, Sieve Machine, Multimeter and Revetest Scratch Tester. Optimized particle size of filler was used in the study is 150 microns and optimum mixing ratio between Epoxy Resin and Polyamide is 1:1. The mixing ratio choose because has fast curing time and good mechanical properties. The electrical resistivity decreased as the concentration of filler increased, from the result the maximum value was 24.13 x $10^6\,\Omega$ at 40% of carbon electrode (CE) filler. The adhesion properties of the paint decreased as concentration of filler increased, while the dispersion of filler in the conductive paint became better with the increased of filler. Between the three fillers CE, CW and MIX used in the study, CE filler has the best mechanical and electrical properties.

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TABLE OF CONTENTS

CHAPTER 1: INTRODUCTION

CHAPTER 2: LITERATURE REVIEW

CHAPTER 3: METHODOLOGY

LIST OF FIGURES

CHAPTER 2:

CHAPTER 3:

CHAPTER 4:

LIST OF TABLES

CHAPTER 2:

CHAPTER 3:

CHAPTER 4:

CHAPTER 1

INTRODUCTION

1.1 Paint

According to Lakshmanan (1998) paint is a type of colored liquid applied to any surface. Generally, paint is used as a protective layer on surface area and adding color to the surface with additions of pigment in the paint liquid solution. In overall, paint can be applied on all surfaces whether for art creation, industrial uses and corrosion resistance from water element. The used of the paint is depends on its application and certain performance. As an example, paint that used as protective agent from the environment, such as climate changes and chemical attacks, the paint should be a good resistance for this purpose. So, the factor of finishing like shining finish surface and attraction become second factor [1].

Usually the performance of the paint depends on the formulation or recipe. For that reason, it is important to know the final characteristic of the paint and the selection of the main ingredient of the paint to ensure the desired characteristic will be achieved. The main ingredients are pigment, binder, and additives.

In couple of years before, mostly composite contains particle dispersion in their matrices. This type of composite was examined to be used as thermostat and pressure gauge. Conductor particle inside the matrix will be interacting between one and each other to become an electrical conductor.

1.2 Application of Conductive Paint

Wikipedia website says that there are many conductive composite polymers which prepared using metal, graphite and conductive ceramic as the filler. Conductive coating function is to prevent the creation of sparks from static charge. For now, aerospace industry still does not have organic coating that can sustain direct emission from the sun. The used of organic coating will be affected the absorption and reflection of electromagnetic radiation. Natural disruption (electric storm) and synthetic (radiation source) happens at the outside (EMI / RFI) will caused failure of equipment, devices loss the memory, disruption in receiving radio signal and flight radar [2].

From Wikipedia website, Electromagnetic interference (EMI), also called radio frequency interference (RFI) is a usually undesirable disturbance that affects an electrical circuit due to electromagnetic radiation emitted from an external source. The disturbance may interrupt, obstruct, or otherwise degrade or limit the effective performance of the circuit. The source may be any object, artificial or natural, that carries rapidly changing electrical currents, such as an electrical circuit, the Sun or the Northern Lights [2].

EMI can be induced intentionally for radio jamming, as in some forms of electronic warfare, or unintentionally, as a result of spurious emissions and responses, intermodulation products, and others. It frequently affects the reception of AM radio in urban areas. It can also affect cell phone, FM radio and television reception, although to a lesser extent.

1.3 Protective Coating Components

1.3.1 Binder

Binders include synthetic or natural resins such as acrylics, polyurethanes, polyesters, melamine resins, epoxy, or oils. Some binder needs addition of pigment for surface painting purpose and it combined in the paint solution.

The binder, or resin, is the actual film forming component of paint. It is the only component that must be present; other components are optionally, depending on the desired properties of the cured film [2].

Paint is a binder which formulated with a complex mixture and intensive observation during the analysis in the laboratory. This is because to get and indentified every behavior of each components. Usual composition is contain one polymer or resin whether neutral or synthetic and others such as extra solvent, binder, stabilizer, pigment, and filler. Everything is based on the performance of the product that will be produced.

1.3.2 Pigment

Pigments are granular solids incorporated into the paint to contribute colour, toughness or simply to reduce the cost of the paint. Alternatively, some paints contain dyes instead of or in combination with pigments. Other paints contain no pigment at all. Hiding pigments, in making paint opaque, also protect the substrate from the harmful effects of ultraviolet light. Hiding pigments include titanium dioxide, phthalo blue, red iron oxide, and many others.

Vengadaesvaran (2003) says that pigment gives color to the paint. In addition, pigment also has others function that similar to the filler such as increase the thickness and strength of the coating. Other functions of the pigment are controls the level of glaze in paint, corrosion resistance and improve the sustainability to the weather. Pigment also provides strength and stiffness to the binder [3].

Lakshmanan (1998) also says that pigments can be classified as either natural or synthetic types. Natural pigments include various clays, calcium carbonate, mica, silicas, and talcs. Synthetics would include engineered molecules, calcined clays, blanc fix, precipitated calcium carbonate, and synthetic silicas [1].

1.3.3 Filler

According to Vengadaesvaran (2003) filler sometimes acts to increase the thickness of the coating, support the structure and increase the viscosity of the paint. Not all paint was filled with the filler because sometimes pigment can become a filler, gives color and at the same time acting as strengthener [3].

Besides the three main categories of ingredients, paint can have a wide variety of miscellaneous additives, which are usually added in very small amounts and yet give a very significant effect on the product. Some examples include additives to modify surface tension, improve flow properties, improve the finished appearance, increase wet edge, improve pigment stability, impart antifreeze properties, control foaming and control skinning.

1.3.4 Solvent

According to Wikipedia website, the main purpose of the vehicle or solvent is to adjust the viscosity of the paint. Solvent is the liquid that carries the solid components of paint. Solvent must be present in the paint to allow the paint's viscosity in correct consistency for application by brush, roller, spray or dipping. Solvent is volatile and does not become part of the paint film because after the paint applied on the substrate the solvent evaporates and leave a solid dry film on the substrate [2].

Wikipedia website also says that solvent-based, sometimes called oil-based, paints can have various combinations of solvents as the vehicle, including aliphatics, aromatics, alcohols, and ketones. These include organic solvents such as petroleum distillate, esters, glycol ethers, and the like. Sometimes volatile low-molecular weight synthetic resins also serve as diluents. Such solvents are used when water resistance, grease resistance, or similar properties are desired [2].

1.4 Background of Study

In this study, battery waste materials is chosen as the filler particles and the binder is consists of epoxy resin. Battery waste materials was used because the cost is relatively low, it also has a low density and it allow conduction by having a conductive network depending on the rheology, post-processing morphology of the cathode waste materials, wettability rates of solidification after mixing and the surface activity of polymer (matrix) that is being used.

The other advantage of over metallic particle is the metallic particles have large contact points that may causes the conduction network that is formed is unstable. Just like other normal material, epoxy resin provide flexibility, good mechanical properties, low melting temperature that allow processing to be perform in low temperature and having relatively low cost and low electrical conductivity.

1.4.1 Significance of Study

Whether new or used, batteries contain heavy metals and when improperly handled, stored, or disposed, they can be dangerous to public health and damaging to the environment. Batteries of all types should never be mishandled as they may explode and/or release harmful toxins into the air, ground, and water. Those released toxins then have the potential to contaminate food sources and water supplies and even harm wildlife. Recycling the batteries will help to reduce the pollution. This study will use part of the batteries to produce or create another product which is conductive paint.

The widespread use of batteries has created many environmental concerns, such as toxic metal pollution. Battery manufacture consumes resources and often involves hazardous chemicals. Used batteries also contribute to electronic waste. Some areas now have battery recycling services available to recover some of the materials from used batteries. Batteries may be harmful or fatal if swallowed.

Recycling or proper disposal prevents dangerous elements (such as lead, mercury, and cadmium) found in some types of batteries from entering the environment.

1.5 Problem Statement

Conductive paint is an organic coating that conducts electrical current. Most of the conductive paint in the market is using silver and platinum as filler to get more conductive than the carbon paint products. The silver and platinum paints, especially the platinum product, are expensive, and their cost may be prohibitive for certain applications.

Using battery waste materials as filler in the conductive paint form a new type of conductive paint. Usually cathode waste and carbon electrode can be found in the dry cell battery. There are many type of battery which using different type of cathode and electrode. By recycling the cathode and electrode from the battery also can help to reduce pollution to the environment.

For now, there is no specific value that shows the quantity and the particle size of the cathode waste material and the carbon electrode used in the epoxy based conductive paint. As results, it is difficult to determine the quantity and the particle size of cathode waste materials that can be utilizes to get the optimum conductivity without losing the mechanical properties such as scratch resistance of the paint.

1.6 Objective

1. Perform mixing ratio analysis to determine the optimum mixing ratio of epoxy resin and polyamide.
2. Perform particle size analysis to determine the suitable carbon electrode and cathode waste materials size to be used as filler material.
3. Perform resistivity test to determine the optimum value of resistivity for each type of filler.
4. Perform scratch test analysis to determine the mechanical properties.

1.7 Scope of Study

This study focuses on optimization of cathode waste materials and carbon electrode on epoxy based conductive paint properties. In the study, cathode waste material and carbon electrode which commonly used in the dry cell battery will be recycled and act as filler in the paint. There are three types of battery waste materials that will be used which are cathode waste, carbon electrode and combination of cathode waste and carbon electrode.

The battery waste materials will be optimized in term of particle size and quantity (weight percentage). The optimum epoxy mixing ratio will be selected from mixing ratio experiment. The sample taken will undergo resistivity test to determine the optimum resistivity value in term of battery waste material type, optimum particle size and the optimum quantity of battery waste material.

CHAPTER 2
LITERATURE REVIEW

2.1 Conductive Coating

A.C. Hart (1989) says paints are inherently non-conducting that would provide no shielding effect and to produce a paint, which is conductive, it is necessary to incorporate conductive pigments into the system [4]. J.A. Saunders (1977), D.M. Bigs (1986) and B.A. Baudler (1982) say that electrically conductive coatings are required for a variety of applications such as static charge dissipation and electromagnetic/radio frequency interference (EMI/RFI) shielding. They are also used in the production of antistatic coatings, space heating, and in various electrical applications [5 – 7].

According to Lighting Technologies Inc website, the amount of dc conductivity required is dependent upon the specific application. Tribo electric charge buildup by dielectric substrates, such as fiberglass structures in frictional contact with other materials, can result in very large static voltages that may lead to dangerous discharge sparks. The amount of surface resistance required effectively to bleed off this charge and prevent sparking, is usually rather low 10^{-6} to 10^{-9} Ωcm^2. At the other extreme, near metal like conductivity is required for directing large current such as those experienced in lighting strikes on composite aircraft structures. Currently, there are no organic coatings enough to provide complete lighting protection, but some can reduce or minimize damage to local areas [8].

1. Utilize conductive polymers as the continuous matrix.
2. Incorporate conductive pigments as sufficient pigment volume concentration.
3. Combination of both methods.

2.2 Composition of Paints

Paints are made of numerous components, depending on the method of application, the desired properties, the substrate to be coated, and ecological and economic constraints. The most important component of a paint formulation is the binder. Binders essentially determine the application method, drying and hardening behavior, quality of adhesion to the substrate, mechanical properties, chemical resistance, and resistance to weathering.

Wikipedia Website says that paint components can be classified as volatile and non-volatile. Volatile paint components include organic solvents, water, and coalescing agents. Non-volatile components include binders, resins, plasticizers, paint additives, dyes, pigments, and extenders. In some type of binders, chemical hardening can lead to condensation products such as water, alcohols, and aldehydes or their acetyls, which are released into the atmosphere, thus being regarded as volatile components [2].

2.2.1 Binders and Resins

Chew Kuew Wai (2003) says that binders are macromolecular products with a molecular mass between 500 and ca. 30 000. Today, most binders are synthetic resins such as alkyd or epoxy resins. The natural resin most commonly used as a binder today is rosin, which often tailored by chemical modifications to suit specific applications. Also, many synthetic hard resins mainly based on cyclohexanone, acetophenone, or aldehydes, are used in the paint industry. Hard resin binders increase the solids content, accelerate drying, and improve surface hardness, luster and adhesion [9].

Stoye D. and Freitag W (1998) says that most of synthetic binders are softer and more flexible than hard resins and consequently, impart good elasticity, high impact resistance, and improved adhesion, even to critical undercoats, as well as offering adequate resistance to weathering and chemicals. These binders are produced with a property profile tailored to suit particular application methods and to comply with a range of technical requirements, including environmental protection, low toxicity, and suitability for recycling and disposal [10].

2.2.2 Pigments and Extenders

Chew Kuew Wai (2003) defines that a pigment is a substrate that is insoluble in the application medium (e.g. paint) and is used as a colorant or because of its corrosion inhabiting or magnetic properties. A dye is defined as a colorant that is soluble in the application medium. Extenders are sometimes difficult to distinguish from pigments; they are defined as substances that are insoluble in the application medium and used to increase the volume, obtain certain technical properties, and/or improve optical properties [9].

Several different pigments may be used within the same coating. All of them contributing to the coating general characteristics and to perform important functions such as providing:

1. Colour
2. Protection to resin binder
3. Corrosion inhibition
4. Corrosion resistance
5. Film reinforcement
6. Non-skid properties
7. Sag control
8. Increased coverage
9. Hide and gloss control
10. Adhesion

For optimization of these effects, the colorants must be mixed in a suitable manner and in appropriate amounts with the relevant binder. The distribution of the primary pigment particles in the polymer matrix should be as uniform as possible.

2.2.3 Additives

In addition to resins, solvents, and pigments, paints also contain additives. The additives content is typically between 0.01 and 1% [11]. Paint additives are used to prevent defects in the coating (e.g., foam bubbles, poor leveling, flocculation sedimentation) or to impart specific properties to the paint (e.g., better slip, flame retardance, UV stability) that are otherwise difficult to achieve.

Such products were often only used to correct a paint batch that did not comply with the required specifications and showed some defects. Nowadays additives are already taken into consideration when a new paint formulation is created. They form an essential constituent of the coatings. In view of the increasingly stringent quality and environmental requirements for the production and use of coatings, high quality paint systems are almost always formulated with additives, and specific technologies (e.g., aqueous system, powder coatings) require specific additive developments. Changes in legislation (reduction of aromatic solvents, VOC regulations, replacement of alkyphenolethoxylates) also made new additives necessary.

Additive may be classified in the following groups:

1. Defoamers
2. Wetting and dispersing additives
3. Surface additives
4. Rheology additives
5. Driers and catalyst
6. Preservatives
7. Light stability
8. Corrosion inhibitors

Although being applied in small quantities, additives are important constituents of coating materials (paints). Additives for coating materials significantly affect their quality, economy and properties. Paint additives are used to thicken paints, improve the dispersion of pigments, decrease foam formation and inclusion of air and improve wetting and adhesion to the substrate. They are also to give the coating a mat appearance, adjust flow and surface properties, assist the coalescence of the polymer particles in layer of polymer dispersion and accelerate the solidification and curing of liquid coating layers. Special paint additives are used for preventing skin formation of paints in containers, for absorbing destructive UV radiation, to inhibit corrosion, suppress the biological deterioration of coating materials, retard ignition, suppress flammability and initiate the curing by photochemical mechanisms [12].

2.2.4 Solvents

Most coatings are made with multiple solvents and rarely with a single solvent. The choice of solvents influence viscosity, flow properties, drying rate, spraying or brushing characteristics, and gloss. There is no universal solvent for protective coatings. The best solvent in one system may be often impractical for the other. Asphalts, for example, can be readily dissolved by hydrocarbons but are insoluble in alcohols. One of the most serious problems associated with coatings is the wrong choice of solvent since it can severely affect the curing and adhesion characteristic of the final coating [9].

2.3 Epoxy Resin

Epoxy products are used for paints and other surface coatings, molded and reinforced plastics, electronic component potting resins, and adhesives ranging from spray foams to dental cement. They are often used in jobs where tough, durable coatings or adhesives are needed. Epoxy resin systems are made up of an epoxy resin and a curing agent (also called a hardener or catalyst).

When epoxy resin systems are used, single molecules (monomers) of the epoxy resin chemical and the curing agent combine to form long chains of molecules (polymers). As the mixture cures, it becomes a hard polymer. Some epoxies cure in a few minutes at room temperature. Others need additional time or heat to harden. The characteristics of hardened epoxies (such as whether they are firm or flexible, or resistant to heat or chemicals) depend on which epoxy monomers, curing agents, solvents, and fillers are added [13].

The hardened, finished polymers are almost non-toxic; it is exposure to the uncured resin components that can be harmful. In a two-component epoxy product, the epoxy resin and the curing agent are packaged separately and must be mixed together just before being used. Each component can be hazardous. In a single-component product, the resin and the curing agent are supplied in a pre-mixed form. Single-component systems are usually safer, because the hazardous chemicals are already partly combined into less toxic polymers and because they do not evaporate into the air as easily [13].

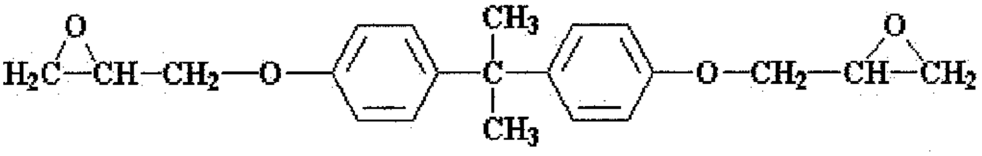


Figure 2.1: Chemical structure of Epoxy Resin [13]

2.4 Battery

In electronics, a battery or voltaic cell is a combination of one or more electrochemical Galvanic cells which store chemical energy that can be converted into electric potential energy, creating electricity. The battery has become a common power source for many household and industrial applications, and a multi-billion dollar industry.

Universal wastes are hazardous wastes in United State of America. Universal waste generally poses a lower threat relative to other hazardous wastes. The waste became a threat because they are ubiquitous and produced in very large quantities by a large number of generators. Some of the most common universal wastes are fluorescent light bulbs, batteries, cathode ray tubes, and mercury-containing devices. Universal wastes must still be disposed of properly to avoid pollution.

2.4.1 Battery Construction

A battery is a device that converts chemical energy directly to electrical energy. It consists of one or more voltaic cells. Each voltaic cell consists of two half-cells connected in series by a conductive electrolyte. One half-cell is the negative electrode (the cathode) and the other is the positive electrode (the anode). In the redox reaction that powers the battery, reduction occurs in the cathode, while oxidation occurs in the anode. The electrodes do not touch each other but are electrically connected by the electrolyte, which can be either solid or liquid. In many cells, the materials are enclosed in a container, and a separator, which is porous to the electrolyte, which prevents the electrodes from coming into contact.

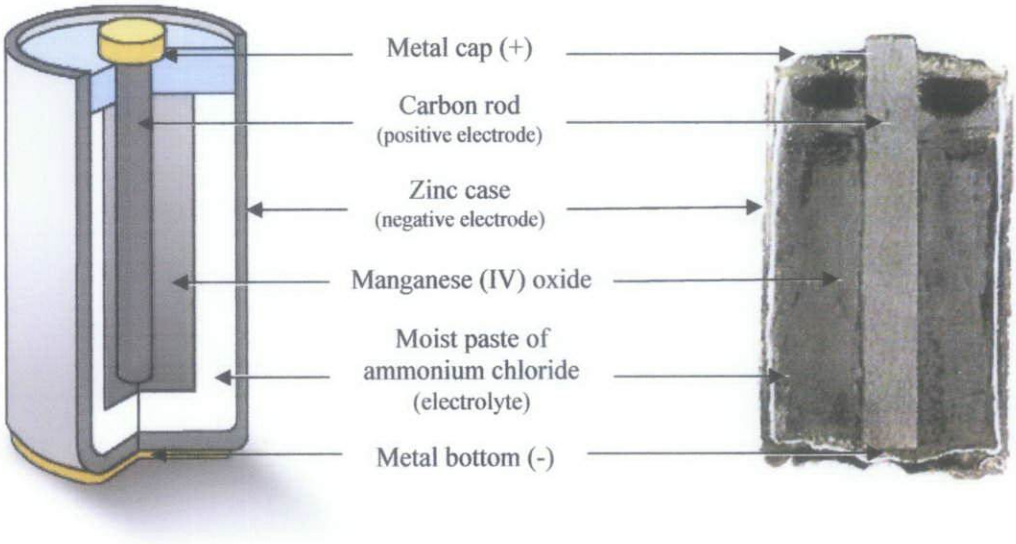


Figure 2.2: Cross-section of a zinc-carbon battery. [14]

The container of the zinc-carbon dry cell is a zinc can (Figure 2.2). This contains a layer of NH_4Cl with $ZnCl_2$ aqueous paste separated by a paper layer from a mixture of powdered carbon & manganese (IV) oxide (MnO_2) which is packed around a carbon rod.

2.4.2 Battery Compositions

In Energizer Manganese Dioxide the cathode used are a mixture of MnO_2 and conductor. The carbon zinc battery uses a manganese dioxide cathode, and an electrolyte of ammonium chloride and or zinc chloride dissolved in water. Powdered carbon is used in the cathode mix, usually in the form of carbon black to improve conductivity of the mix and for moisture retention [14].

Table 2.1: Details of materials used in Energizer Manganese Dioxide battery. [14]

MATERIAL OR INGREDIENT	%/wt.
Graphite (CAS# 7782-42-5)	2 - 6
Manganese Dioxide (CAS# 1313-13-9)	30 - 45
Potassium Hydroxide (CAS# 1310-58-3)	4 - 8
Zinc (CAS# 7440-66-6)	12 - 25

Wikipedia.org says that a Zinc-carbon dry cell or battery is packaged in a zinc can that serves as both a container and anode. The cathode is a mixture of manganese dioxide and carbon powder. The electrolyte is a paste of zinc chloride and ammonium chloride dissolved in water [15].

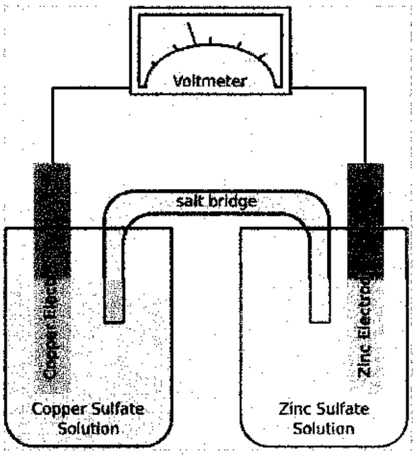


Figure 2.3: The two half-cells are linked by a salt bridge separator that permits the transfer of ions [15].

In a dry cell, the outer zinc container is the anode (-). The zinc is oxidized according to the following half-equation.

$$Zn(s) \rightarrow Zn^{2+}(aq) + 2\ e^-$$

A graphite rod surrounded by a powder containing manganese (IV) oxide is the cathode (+). The manganese dioxide is mixed with carbon powder to increase the conductivity of the cathode mixture [15]. The cathode reaction is as follows:

$$2MnO_2(s) + 2\,H^+(aq) + 2\,e^- \rightarrow Mn_2O_3(s) + H_2O(l)$$

The H^+ comes from the $NH_4^+(aq)$:

$$NH_4^+(aq) \rightarrow H^+(aq) + NH_3(aq)$$

and the NH_3 combines with the Zn^{2+}.

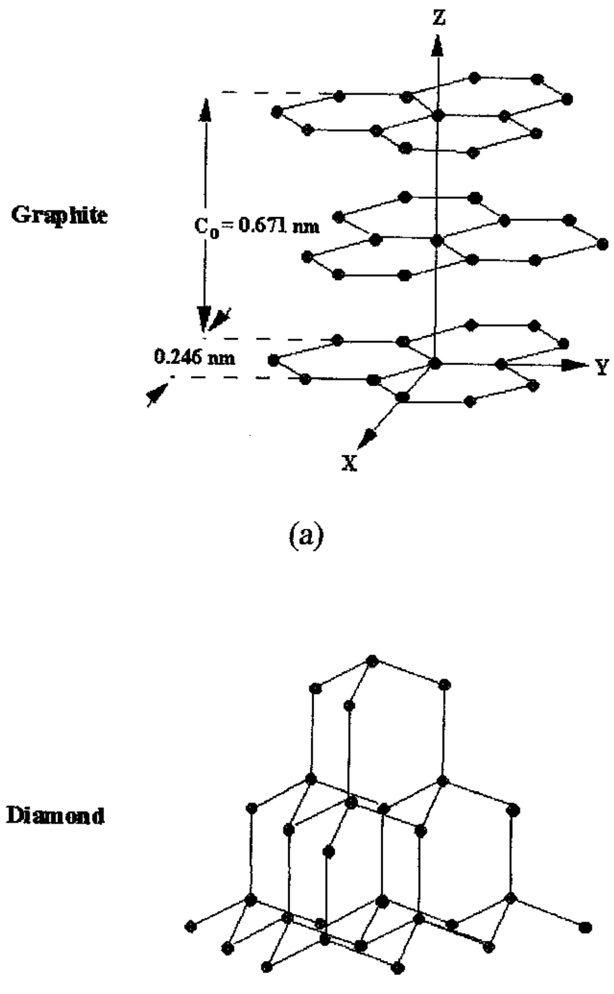
In this half-reaction, the manganese is reduced from an oxidation state of (+4) to (+3) [15]. There are other possible side-reactions, but the overall reaction in a zinc-carbon cell can be represented as:

$$Zn(s) + 2MnO_2(s) + 2NH_4^+(aq) \rightarrow Mn_2O_3(s) + Zn(NH_3)_2^{2+}(aq) + H_2O(l)$$

2.5 Carbon

Carbon is one of the most abundant elements found on earth. It occurs freely in crystalline forms such as diamond and graphite. The crystal structures of these carbons are shown in Figure 2.4. The figure clearly shows that these carbons have two distinctly different structures.

The diamond crystal is cubic, with the atoms arranged in a tetrahedral configuration. This arrangement of carbon atoms produces a solid that is the hardest known substance. Consequently, it is used as an industrial abrasive. In addition, diamond has a very high refractive index; hence it produces brilliant cut gems. Graphite, on the other hand, is soft, has a hexagonal structure, with the carbon atoms arranged in layer planes. The spacing between the layer planes in graphite is 0.3354 nm (nm = billionth of a meter) [16].



(a)

(b)

Figure 2.4: Figure (a) and (b) - Crystal structures of carbon [16]

Kim Kinoshita (2001) says that when the dimensions of the layer planes are small and the separation between the layer planes becomes large, the carbon is referred to as amorphous carbon (for example, charcoal, coke, and soot). Because of their difference in structures, diamond is an electrical insulator, whereas graphite is a good electrical conductor. The high conductivity of graphite and its good chemical stability are attractive features for its use in electrochemistry [16].

Desirable properties of carbon and graphite for electrochemical applications:

1. Good electrical conductivity
2. Acceptable corrosion resistance
3. Availability in high purity

4. Low cost
5. High thermal conductivity
6. Dimensional and mechanical stability
7. Light in weight and ease of handling
8. Availability in a variety of physical structures
9. Ease of fabrication into composite structures

2.6 Reviews on previous study

2.6.1 Studies on graphite based conductive paint coatings [17].

The study is done by S. Syed Azim∗, A. Satheesh, K.K. Ramu, S. Ramu, and G. Venkatachari. The objective was to determine the optimal graphite/carbon black combination, in terms of conductivity, in a conductive paint and to suggest an explanation of the improvement in conductivity achieved over the graphite loaded paint.

The binder used in the study was, a poly sulphide modified epoxy resin having 100% solids and was cured with a polyethylene polyamine. The graphite filler used was lamellar structured graphite which has carbon content of 98–99% and typical particle size is up to $50\mu m$ and its density is 2.26 g/cc. The graphite used was a conductive grade. Carbon black used was a commercial one.

The resistivity of the formulated paints was measured using the four point probe resistance method. From the resistivity measured, the specific volumetric electrical resistance (ΩM) is calculated as per ASTM D-4496-87. The coating composition, which had the minimum specific volumetric electrical resistance, was concluded as the optimized composition. The paint was applied over a solvent cleaned glass plate at different thickness. The surface resistivity of films has been made for:

1. Optimization of solid content (Vs);
2. Optimization of pigment content (PVC);
3. Optimization of mix ratio between graphite and carbon black.

Optimization of solid content

The paint was varied from 30 to 100% and the PVC was kept at a constant volume, i.e. 55%. The particle to particle contact of the pigments would be increased when the solid content of the paint was further reduced. Hence, the resistivity would be reduced at the low solid content.

Table 2.2: Solid content with the resistivity test result

Solid content (%)	**Resistivity (ΩM)**
100%	3.7×10^{-2} ΩM
55%	1.3×10^{-3} ΩM
50%	8.6×10^{-4} ΩM
40%	6.3×10^{-5} ΩM

Below 40% poor hiding problem occurs due to the low binder content in the paint. Thus, at 40% of solids the particle to particle contact was at maximum

Optimization of pigment volume concentration (PVC)

The PVC was varied from 30 to 60%. The PVC was optimized at the optimized binder volume, i.e. 40%. Upon increasing the pigment volume, the resistivity was found to reduce. Above 55% the dispersion of pigment was too difficult.

Table 2.3: Pigment volume concentration (PVC) with resistivity test result

Pigment volume concentration (PVC) (%)	**Resistivity (ΩM)**
25%	3.3×10^{-2} ΩM
30%	1.6×10^{-2} ΩM
40%	9.1×10^{-3} ΩM
50%	6.7×10^{-4} ΩM
55%	6.0×10^{-5} ΩM

Optimization of graphite and carbon black mix ratio

In order to reduce the resistivity further, carbon black is added as the additional pigment. The minimum resistivity value (2×10^{-5} ΩM) was observed at 85:15 mix ratios. The ratio 85:15, which has the low resistivity value is considered as the optimized mix ratio. From the resistivity values observed it is found that 20% of carbon black inclusion gives the minimum resistivity. The graphite and carbon black, the carbon black mix ratio was further narrowed in between from 10 to 20%, as 10, 13, 15, 17 and 20%. From the resistivity values it is found that 15% mix ratio of the carbon black has the minimum resistivity.

Table 2.4: Effect of mix ratio to the resistivity value

Graphite and carbon black mix ratio	**Resistivity (ΩM)**	**Remarks**
90:10	4.0×10^{-5} ΩM	The resistivity is reduced with increasing the carbon black ratio
87:13		
85:15	2.0×10^{-5} ΩM (minimum resistivity)	
83:17		When black content is increased above 15% there is no decrease in resistivity.
80:20		
75:25		
60:40		

2.6.2 Improving the electrical and mechanical behavior of electrically conductive paint by partial replacement of silver by carbon black [18].

This study was done by CHIA-KEN LEONG and D.D.L. CHUNG from Composite Materials Research Laboratory, University at Buffalo, State University of New York, Buffalo, NY 14260-4400.

The primary objective is to investigate the effectiveness of carbon black as a partial replacement for silver in electrically conductive paint for improving both the electrical and mechanical performance. The secondary objective is to compare the performance of paints obtained in this work with that of commercial silver paints.

The silver (99.9%, metal basis) with sizes ranging from 0.5 μm to 1.0 μm. The carbon black was Vulcan XC72R GP-3820 from Cabot Corp. The vehicle used was silane (Dow Corning Corp., Midland, MI, Z-6020, H2NCH2CH2NHCH2CH2CH2 Si (OCH3)3). Two commercial silver paints, provided by Ernest F. Fullam (Latham, NY, Product 14,810) and Metech (a Lord Company, Elverson, PA, Product 6103A). These commercial silver paints do not contain carbon black and commercial alumina substrate (96% Al2O3) cut to a size of 25 mm × 7 mm × 0.6 mm.

The largest improvement occurs at 0.00357 weight fraction carbon black for both the electrical and mechanical behavior. Further replacement of silver up to 0.00714 weight fraction carbon black gives incremental improvement. However, still further replacement of silver beyond 0.00714 weight fraction carbon black causes the resistivity to increase, though the scratch resistance remains unchanged. Thus, the optimum carbon black content is the intermediate weight fraction of 0.00714, i.e., 0.0558 of the total filler volume, or 0.0100 of the total filler weight.

The Fullam silver paint is superior to the Metech silver paint in both the electrical and mechanical properties. However, the silver-carbon paint with 0.00714 weight fraction carbon black is much superior to both commercial paints in the scratch resistance, though the resistivity is between the values of the Fullam and Metech products. The modification of the Fullam silver paint by addition of carbon black by up to 0.00755 volume fraction increases resistivity slightly, due to the low resistivity of the unmodified Fullam silver paint. Nevertheless, the carbon black addition decreases the scratch width. The higher is the carbon black content, the less is the extent of plowing.

Partial replacement of silver by carbon black in electrically conductive paint results in a decrease in the volume electrical resistivity and improvement of the scratch resistance, provided that the carbon black content is not excessive. Effective carbon black content is 0.055 of the total filler volume, or 0.010 of the total filler weight. Beyond this carbon black content, the resistivity increases slightly, though the scratch resistance does not change. The positive effect of carbon black is probably due to the compressibility of the carbon black and the consequent improved connectivity of silver particles. However, excessive use of carbon black increases the resistivity, due to the low conductivity of carbon compared to silver and the reduction in workability of the paint.

By using a silver volume fraction of 0.01861, a carbon black volume fraction of 0.0108 and a total solid weight fraction of 0.7163 (i.e., carbon black amounting to 0.0549 of the total filler volume, or 0.0099 of the total filler weight), a paint that gives a film with resistivity $(1.88 \pm 0.25) \times 10^{-3}$ Ω·cm and thickness 47 µm ± 3 µm has been attained. This film is more scratch resistant than films made from two commercial silver paints.

The vehicle in the paint compositions is a solution of silane and 1-methoxy-2-propanol (1:1 weight ratio). The use of silane without the propanol greatly increases the resistivity, in addition to degrading the scratch resistance. The propanol helps because it causes the formation of a siloxane backbone. The addition of carbon black (up to 0.008 volume fraction) to a commercial silver paint of low resistivity and poor scratch resistance increases the resistivity slightly, but improves the scratch resistance. This is because carbon black enhances the connectivity between silver particles, though it decreases the workability.

2.7 Scratch Test

Scratch testers are dedicated instruments for characterizing the surface mechanical properties of thin films and coatings, e.g. adhesion, fracture and deformation. The scratch tester's ability to characterize the film-substrate system and to quantify parameters such as friction and adhesive strength, using a variety of complementary methods, makes it an invaluable tool for research, development and quality control [19].

This technique involves generating a controlled scratch with a sharp tip on a selected area. The tip material (commonly diamond or hard metal (WC)) is drawn across the coated surface under constant, incremental or progressive load.

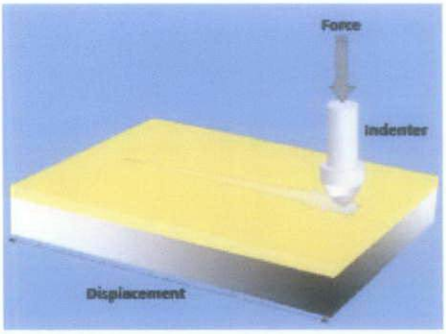


Figure 2.5: Scratch working principle [19].

At a certain critical load the coating will start to fail. The critical loads are very precisely detected by means of an acoustic sensor attached to the load arm but can also be confirmed and collated with observations from a built-in optical microscope. The critical load data is used to quantify the adhesive properties of different film - substrate combinations. In addition to acoustic emission, the Scratch Testers measure the applied normal force, the tangential (friction) force and the penetration depth. These parameters, together with the acoustic emission data, provide the mechanical signature of the coating system [19].

CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter deals with the method of preparation of Epoxy Resin – Polyamide, Carbon Electrode and Cathode Waste. Sample preparation is very important to be precisely done because will affected the experiments result. The important test that will be conducted in the study is resistivity test. The progress of the project can be viewed in Appendix A.

3.2 Materials

Materials that will be used are:

1. Epoxy Resin (Euro Chemo-Pharma Sdn. Bhd.)
2. Polyamide, hardener (Euro Chemo-Pharma Sdn. Bhd.)
3. Carbon electrode from battery (Eveready Heavy Duty, size D)
4. Cathode waste from battery (Eveready Heavy Duty, size D)
5. Aluminum plate (70mm x 35mm)

Epoxy resin acts as a binder while to harden the structure of the binder, polyamide was used to acts as hardener. The materials safety data sheet (MSDS) of epoxy resin and polyamide will be explained more in the Appendix B and Appendix C respectively.

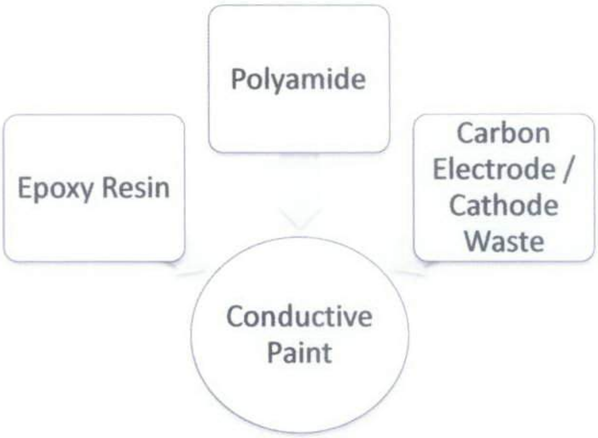


Figure 3.1: Basic composition of conductive paint

3.3 Filler Grinding

Filler must be grinded first especially for carbon electrode because the initial shape of carbon rod is big such as in Figure 3.2 (a).

(a) (b)

Figure 3.2: Carbon electrode before and after grinding process as shown in figure (a) and (b) respectively.

3.4 Preliminary Examination

Before the sample can be prepared, some examination must be done to determined best mixing ratio between binder and hardener and suitable particle size. This experiment is to ensure the sample prepared afterward was optimized in term of mixing ratio and particle size.

3.3.1 Mixing Ratio

From the early research in the part 1 (FYP I), found that there is no specific mixing ratio manual provided during the purchase of Epoxy Resin and Polyamide. Because of that, this experiment is important to determine the best mixing ratio. The mixing ratio between binder and hardener used in the test are 1:1, 2:1 and 3:2.

3.3.2 Particle Distribution Analysis of Filler

There are two type of filler used in the study, which are Carbon Electrode (CE) and Cathode Waste (CW). Initially, CE was in solid state or in rod shape and grinding this material is needed to achieve the suitable size for particle distribution analysis while CW was already in powder state.

Figure 3.2: Filter for sieving process

The fillers were sieve using sieve machine using four sizes of filters, which are 150 microns, 300 microns, 425 microns, and 600 microns. The weight of each size of filler recorded for analysis.

3.5 Sample Preparation

A formula of the paint is very important because this will deliver the desired properties of the paint. From the result of preliminary examination of the mixing ratio and particle size and distribution, the result showed that best mixing ratio is 1:1 and best particle size is less than 150 microns. Ratio of the filler is described in the table below:

Table 3.1: Composition of conductive paint for Carbon Electrode (CE) as filler

Sample Name	Epoxy Resin (g)	Polyamide (g)	Carbon Electrode (±0.2g)	Carbon Electrode (%)
CE0	10	10	0	0
CE5	10	10	1.05	5
CE10	10	10	2.15	10
CE15	10	10	3.06	15
CE20	10	10	4.11	20
CE25	10	10	5.05	25
CE30	10	10	6.17	30
CE35	10	10	7.02	35
CE40	10	10	8.06	40

Table 3.2: Composition of conductive paint for Cathode Waste (CW) as filler

Sample Name	Epoxy Resin (g)	Polyamide (g)	Cathode Waste (±0.2g)	Cathode Waste (%)
CW0	10	10	0	0
CW5	10	10	1.12	5
CW10	10	10	2.13	10
CW15	10	10	3.08	15
CW20	10	10	4.04	20
CW25	10	10	5.20	25
CW30	10	10	6.03	30
CW35	10	10	7.04	35
CW40	10	10	8.11	40

Table 3.3: Composition of conductive paint for Carbon Electrode plus Cathode waste (MIX) as filler

Sample Name	Epoxy Resin (g)	Polyamide (g)	Carbon Electrode (±0.1g)	Cathode Waste (±0.1g)	Carbon Electrode + Cathode waste (%)
MIX0	10	10	0	0	0
MIX 5	10	10	0.55	0.51	5
MIX10	10	10	1.05	1.03	10
MIX15	10	10	1.53	1.55	15
MIX20	10	10	2.04	2.10	20
MIX25	10	10	2.59	2.51	25
MIX30	10	10	3.00	3.02	30
MIX35	10	10	3.51	3.52	35
MIX40	10	10	4.06	4.04	40

Aluminum plate size of 70mm x 35mm was cut for the scratch resistance test. The paint prepared earlier applied on the plate and wait until dried in room temperature between 25° C to 27° C.

3.6 Resistivity Test

The resistivity test will be done based on standard procedure ASTM F 1896 – 98 (reapproved 2004). This test method is for determining the electrical resistivity of a printed conductive material.

3.6.1 Apparatus

1. Resistance measuring device, (that is, ohm meter) equipped with test leads and probes. The device should be capable of measuring resistance up to 100 MΩ with and accuracy of greater than 1.5% of full scale reading. Test probes should have tops that are 25 to 250 % of the width (W) of the printed conductor test pattern.

2. Test Surface, to be flat, smooth, unyielding and larger than switch under test.
3. Thickness Measuring Device, capable of measuring to the nearest 0.00005 in. (1.25 μm).
4. Dimensional Measuring Device, capable of measuring to the nearest 0.001 in. (25μm).

3.6.2 Procedure

Pre-Test Setup

1. Secure switch/test pattern (that is printed and cured conductive material) on the test surface.
2. Measure the geometry of the test pattern as follows:
    a. Measure the length (L) of the printed test pattern.
    b. Measure the width (W) of the printed test pattern.
    c. Divide the length (L) by the width (W) to calculate the number of square of the printed test pattern. Should be 50 squares or more.

In-Process Test:

1. Using the resistance measuring device (that is, ohm meter), measure the resistance of the printed test pattern. Place probes at ends of measured length of the test pattern
2. Record resistance in ohms.
3. Using the thickness measuring device, measure the thickness (t) of the printed test pattern in mils, measure in a minimum of three locations across the test pattern.
4. Record average thickness (t) in mils (1 mil = 25 μm).

Calculations:

1. Determine the number of squares of the printed test pattern by dividing the length (L) by the width (W). Record the number of squares.
2. Determine the ohms per square by dividing the measured resistance (in ohms) by the number of square. Record the ohms per square.
3. Determine the resistivity (ohms per square per mil) of the conductive material by multiplying the ohms per square by the measured thickness (t) in mils. Record the resistivity of the conductive material (ohms per square per mil).
4. Examples:

   Resistance Measurement: 3.0 Ω

   Thickness (t) Measurement: 12.5 μm

   Number of Squares: 100 squares

   Ohms per square = 3.0 Ω/100 squares

   Ohms per square = 0.03 Ω per square

   Resistivity (ohms per square per inch) = 0.03 Ω per square x 0.0005 in.

   Resistivity (ohms per square per inch) = 0.000015

   Resistivity (ohms per square per mil) = 0.000015 x 1000 mils per inch

   Resistivity (ohms per square per mil) = 0.015

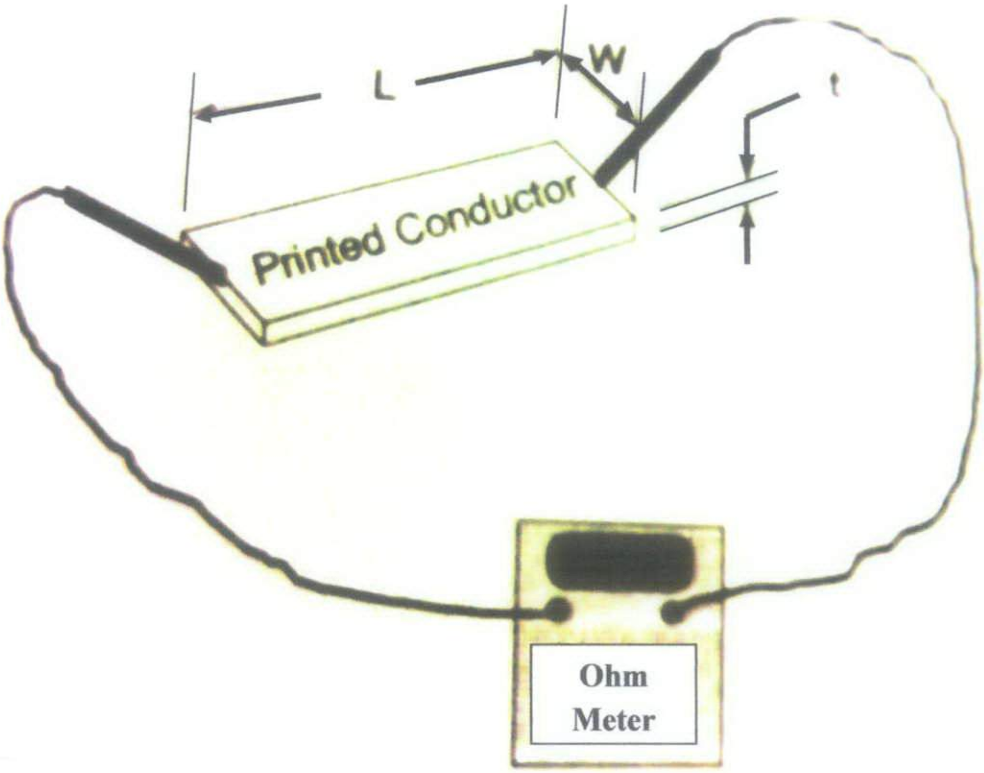


Figure 3.3: Resistance Measurement Test Set-up

3.7 Curing Time

A paint or coating is considered cured if upon touching finger marks were not visible on the coating. Recognized stages of drying were described in the American Society for Testing Materials (ASTMD154-52), which was developed along with hundreds of other highly technical and exacting specifications by Committee D-1 of ASTM. Drying tests, in general, can be split into five stages:

1. Dust free stage
2. Tack-free stage
3. Through-free stage
4. Full hardness
5. Recoat time

Banov (1982) explains that time was measured for each of the five stages. However the definition of each stage of the stages can vary from one tester to another. Testing laboratories and many paint companies have instrument that automatically and continuously apply sand, or lint, or some sort of indenting devices to the surface. The ability if the sand to stick the drying surface at certain intervals and the depth of the indenting device indicate rates of drying [20].

3.8 Scratch Resistance Test

To observe the mechanical properties of the conductive paint, scratch test will be used to determine the scratch resistance of the coating. The carbon electrode and cathode waste were varies from 5% to 40% in the conductive paint and the scratch test will determine the effect of variation of the filler materials. The machine used was from CSM Instruments, Revetest Scratch Tester.

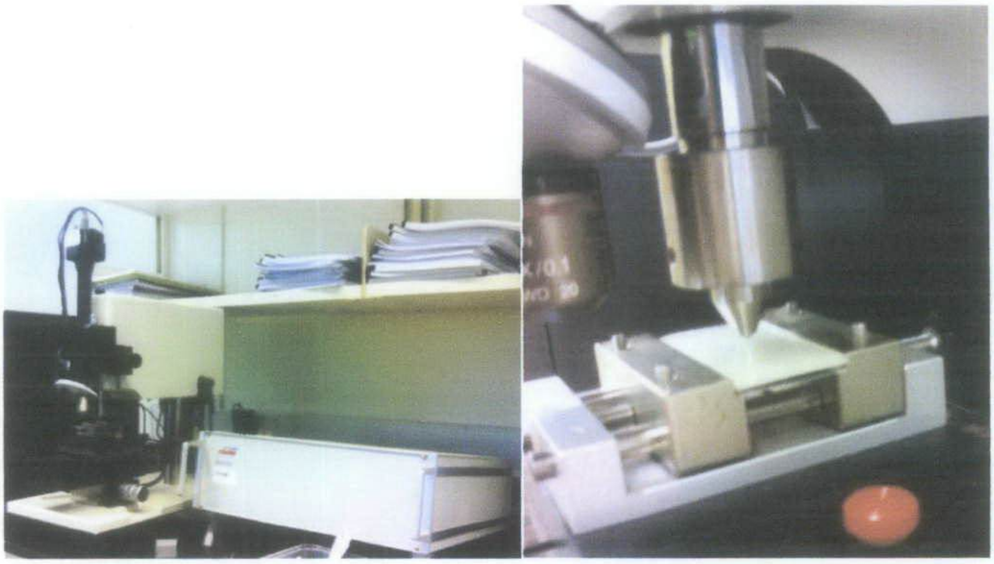


Figure 3.4: Example of scratch test machine in Universiti Teknologi PETRONAS

CHAPTER 4

RESULT AND DISCUSSION

4.1 Introduction

In this chapter the result of each experiment described in the chapter 3 will be presented and discussed. The experiments done are to determine the suitable mixing ratio, optimize particle size, particle distribution, suitable composition of filler to generate lowest resistivity and have good mechanical properties.

Results from the experiments recorded and analyzed carefully. The data were presented in term of figures, graphs and tables depend on the needs. The discussion of the results also included in this chapter to explain the findings from the data. Hopefully, all the data satisfied and meet the objective of this study.

4.2 Mixing Ratio Test Result

Three combinations of mixing ratio were used in the experiments that are 1:1, 2:1 and 3:2 (Epoxy Resin: Hardener). The epoxy resin was mixed with the hardener using weight ratio. The epoxy resin and hardener were poured into different paper cup and weighted. After that, the epoxy resin and hardener were poured into same paper cup and stirred to get evenly mixture of epoxy resin and hardener. Then the mixture was applied on the aluminum plate and observed.

The sample cured in the room with temperature between 25° C to 27° C. Every each hour the samples were tested with tack-free stage to determine the curing time. In order to test the adhesion properties of the sample on the aluminum plate, scratch test was carried out.

4.2.1 Curing Time

In order to be of use in paints the blends should have a short curing time. Table 4.1 will show the curing time for various blend of mixing ratio and found that mixing ratio of 1:1 is the fastest to cure.

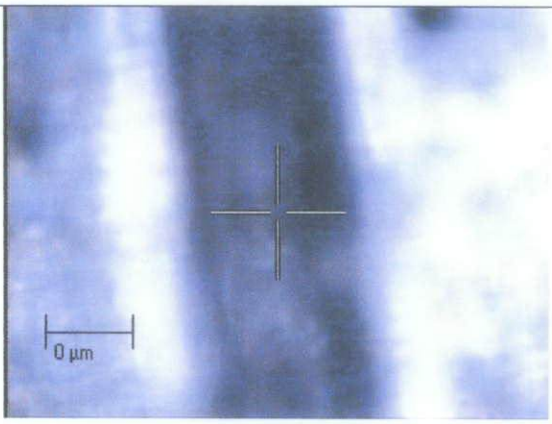
Table 4.1: Curing time of Epoxy resin and Polyamide

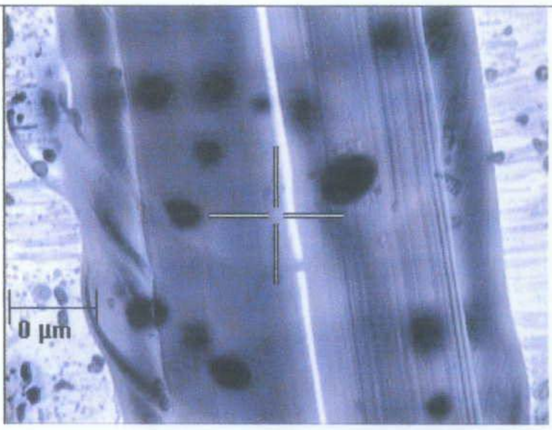
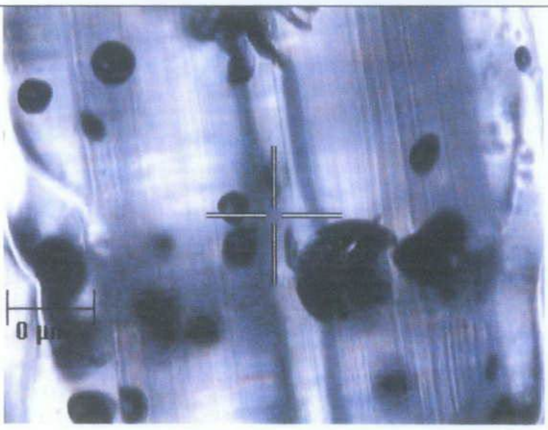
Mixing Ratio Epoxy : Hardener	**Curing Time Tack Free Stage** (hours)
1 : 1	3
2 : 1	6
3 : 2	8

4.2.2 Scratch Resistance

The epoxy mixture sample taken from previous experiment was undergone a constant scratch test at 80N. In the experiment, Standard Operating Procedure was followed to get accurate result. Table 4.2 below show the results from the scratch test.

Table 4.2: Scratch Test Result

Mixing Ratio	Size of Scratch	Scratch Picture at 5x
1:1	Smallest	

2:1	Larger than 1:1 mixing ratio	
3:2	Larger than 2:1 mixing ratio	

The test proved the theory that mixing ratio 1:1 of epoxy resin to hardener was the best mixing ratio in term of mechanical properties. During the test, sample for 2:1 and 3:2 was "peeling off" from the plate but the 1:1 sample was stick well on the plate.

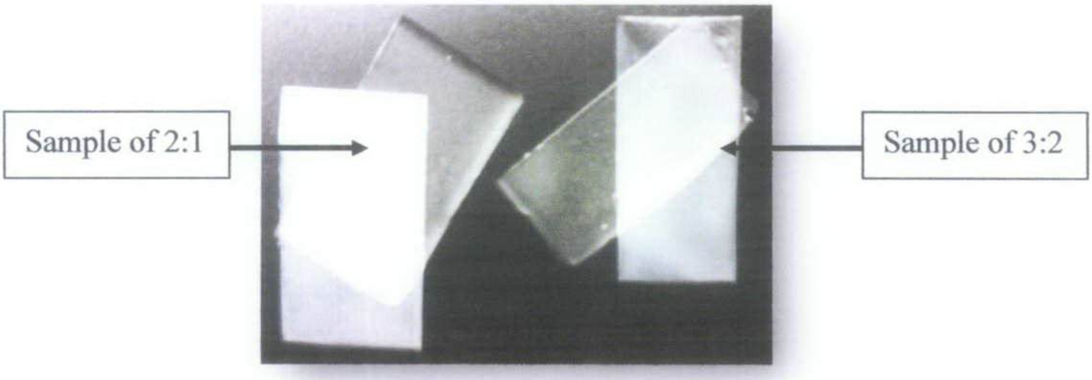


Figure 4.1: Samples that "peel off" from the plate

4.3 Particle Distribution Analysis

The size distribution is often of critical importance to the way the material performs in use. A sieve analysis can be performed on any type of non-organic or organic granular materials including sands, crushed rock, clays, granite, feldspars, coal, soil, a wide range of manufactured powders, grain and seeds, down to a minimum size depending on the exact method.

Table 4.3: Size distribution of Cathode Waste (CW)

Particle Size (microns)	Weight (grams)	Percentage of Distribution
600	53.90	32.67
425	20.87	12.65
300	17.67	10.71
150	10.44	6.33
< 150	62.11	37.64
TOTAL	**164.99**	**100.00**

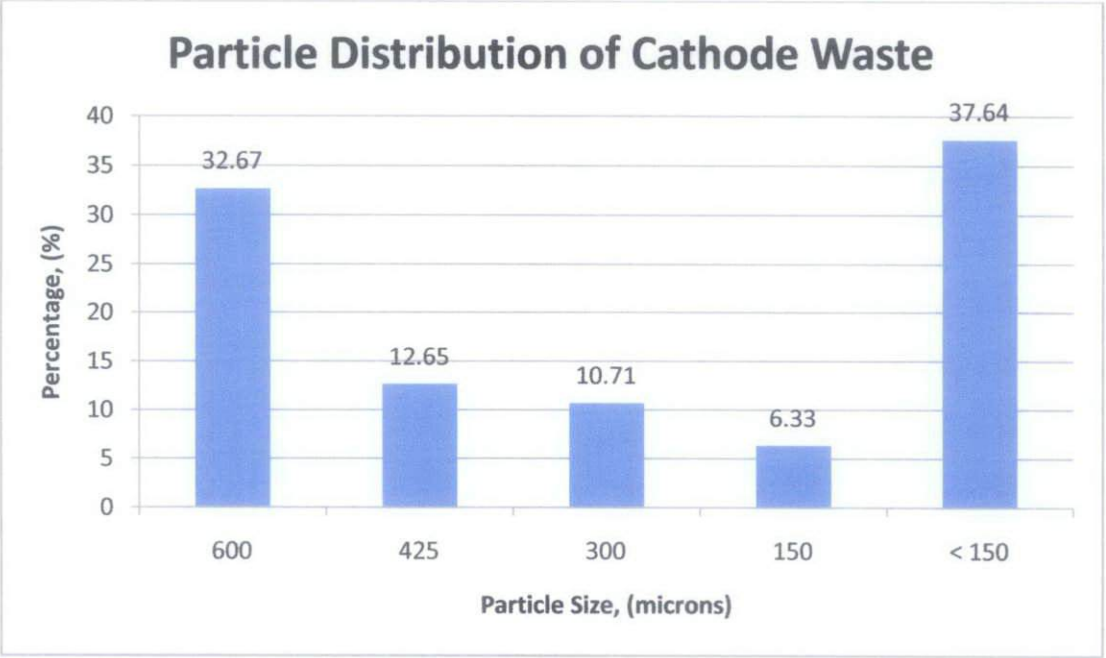


Figure 4.2: Particle distribution of Cathode Waste (CW)

Table 4.3 showed the total weight of CW used in the analysis which about 164.99 grams and the percentage of each particle size. Figure 4.2 clearly showed the particle mostly distributed at the size less than 150 microns.

Table 4.4: Size distribution of Carbon Electrode (CE)

Particle Size (microns)	**Weight (grams)**	**Percentage of Distribution**
600	7.54	14.53
425	4.89	9.42
300	3.30	6.36
150	7.70	14.84
< 150	28.47	54.86
TOTAL	**51.9**	**100.00**

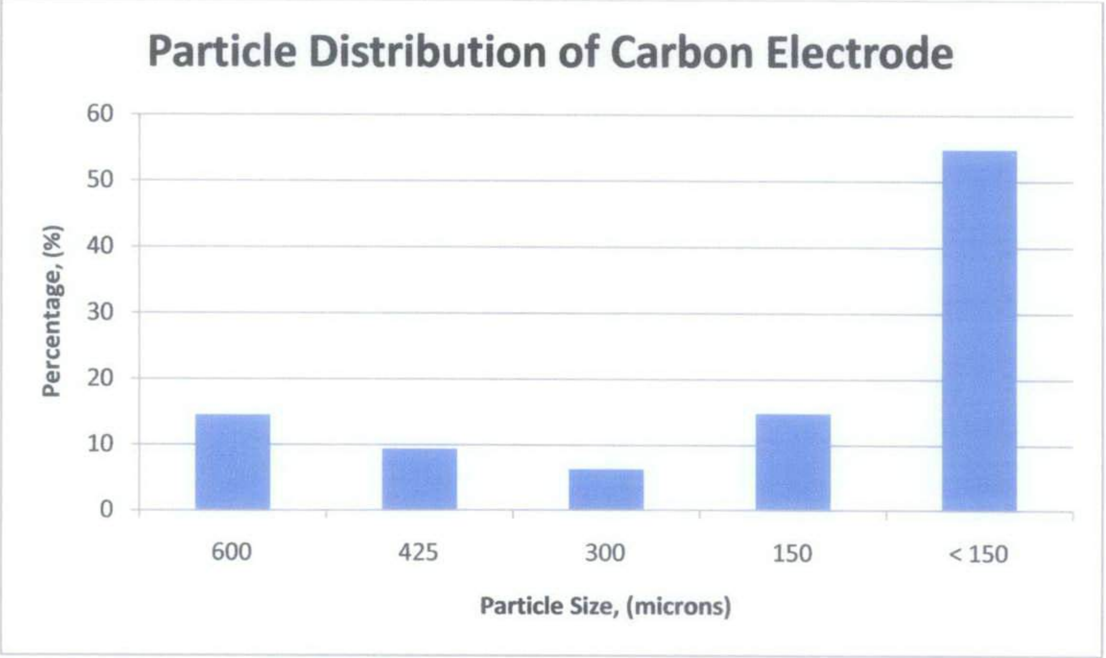
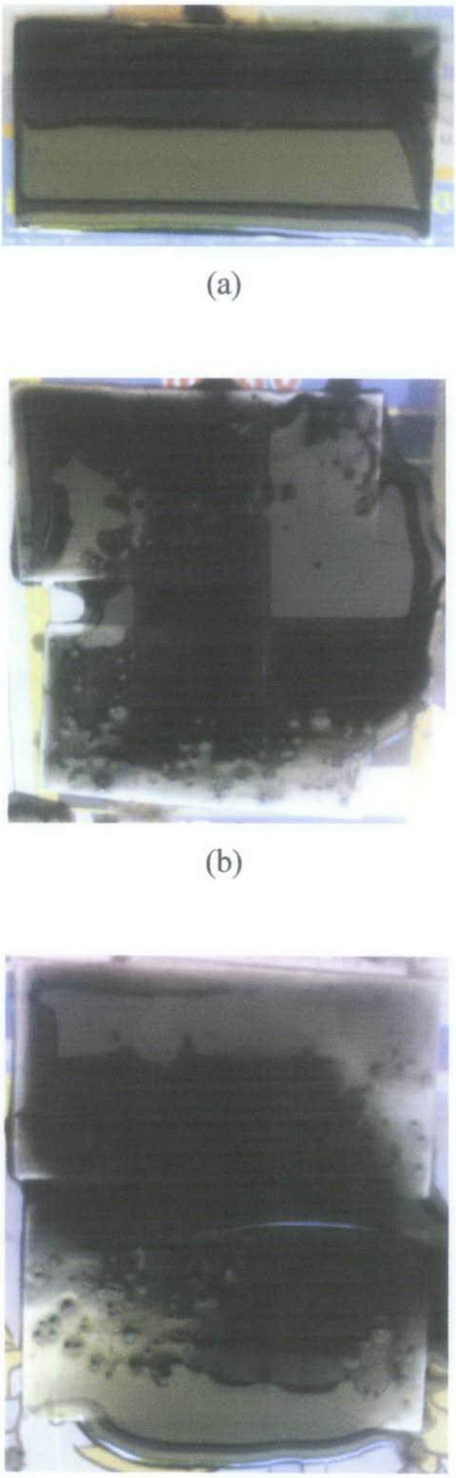


Figure 4.3: Particle distribution of Carbon Electrode (CE)

For particle distribution of CE, the same scenario happened which most of the particle are in less than 150 microns size. This concludes that this particle size is very suitable in term of availability and quantity for sample preparation.

4.4 Sample Preparation Result

Before the sample prepared using characteristic described in Table 4.5, the paint initially prepared using different size of particle. Figure below concluded the result.



(a)

(b)

(c)

Figure 4.4: Preliminary test result for particle size (a) 150 microns (b) 300 microns (c) 600 microns

The objective of the test is to see the effect of the particle size in the sample preparation. The result showed 300 and 600 microns size of particle is not suitable for the sample preparation because the particle did not bind and mix well in the paint solution. This happen because the binder did not have enough strength to bind the filler together since the size of the filler is too big.

The previous experiments result was taken into account when preparing the sample to get the optimize value of resistivity. Table 4.5 below show the characteristic of the sample prepared.

Table 4.5: Characteristics of Samples Prepared

Characteristics	Value	Remark
Mixing Ratio	1:1	Good curing time, good adhesion on the plate
Particle Size	Less than 150 microns	A lot in term of quantity, better particle dispersion
Filler	3 types	Carbon electrode (CE), Cathode waste (CW) and combination of both 1:1 ratio (MIX) 5 % increment up to 40 %

4.5 Resistivity Test

Electrical resistivity is a measure of how strongly a material opposes the flow of electric current. A low resistivity indicates a material that readily allows the movement of electrical charge.

Resistivity of the sample measured according to the standard procedure ASTM F 1896 – 98 (reapproved 2004). Table 4.6, Table 4.7 and Table 4.8; show the resistivity result for each type of filler used in sample.

Table 4.6: Resistivity value for Carbon Electrode (CE) as filler

Sample Name	Resistivity (Ω)	Carbon Electrode (%)
CE0	0	0
CE5	0	5
CE10	0	10
CE15	0	15
CE20	51.12×10^6	20
CE25	44.58×10^6	25
CE30	30.65×10^6	30
CE35	25.34×10^6	35
CE40	24.13×10^6	40

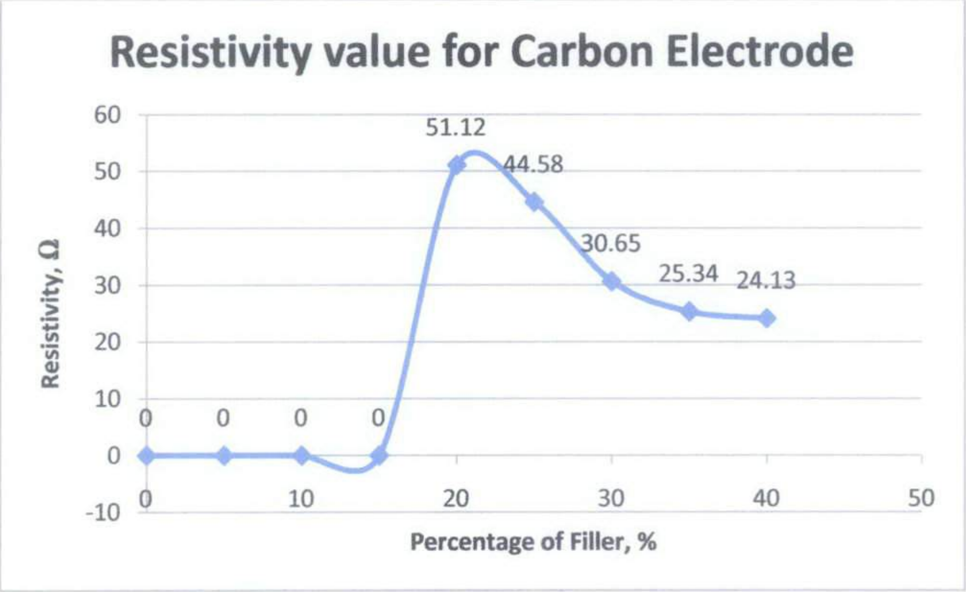


Figure 4.5: Graph of resistivity value for Carbon Electrode (CE) as filler

Table 4.7: Resistivity value for Cathode Waste (CW) as filler

Sample Name	**Resistivity (Ω)**	**Carbon Electrode (%)**
CW0	0	0
CW5	0	5
CW10	0	10
CW15	0	15
CW20	0	20
CW25	60.38×10^6	25
CW30	48.23×10^6	30
CW35	35.71×10^6	35
CW40	33.45×10^6	40

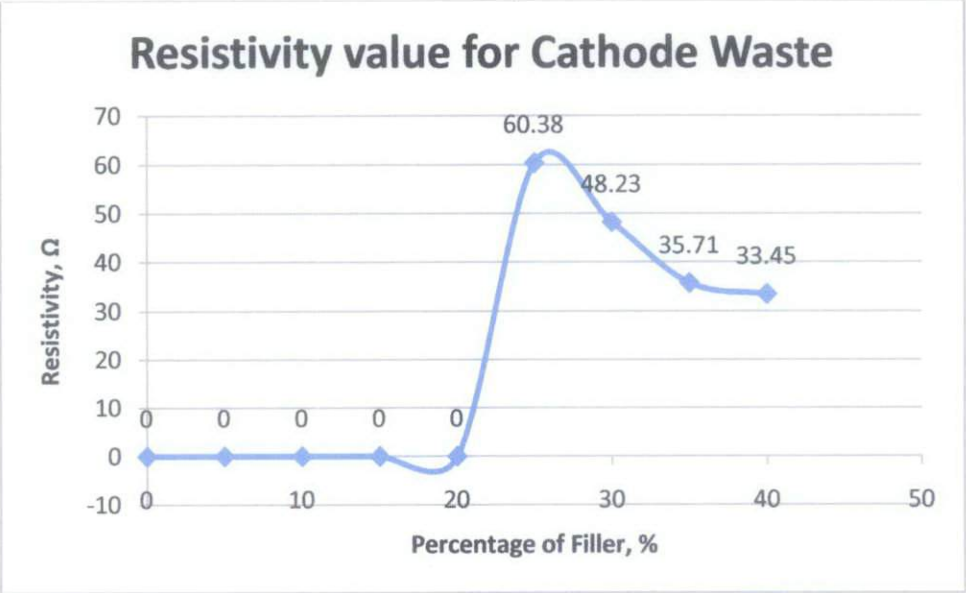


Figure 4.6: Graph of resistivity value for Cathode Waste (CW) as filler

Table 4.8: Resistivity value for Carbon Electrode plus Cathode Waste (MIX) as filler

Sample Name	Resistivity (Ω)	Carbon Electrode (%)
MIX0	0	0
MIX 5	0	5
MIX10	0	10
MIX15	0	15
MIX20	78.65×10^6	20
MIX25	56.23×10^6	25
MIX30	38.22×10^6	30
MIX35	30.67×10^6	35
MIX40	29.17×10^6	40

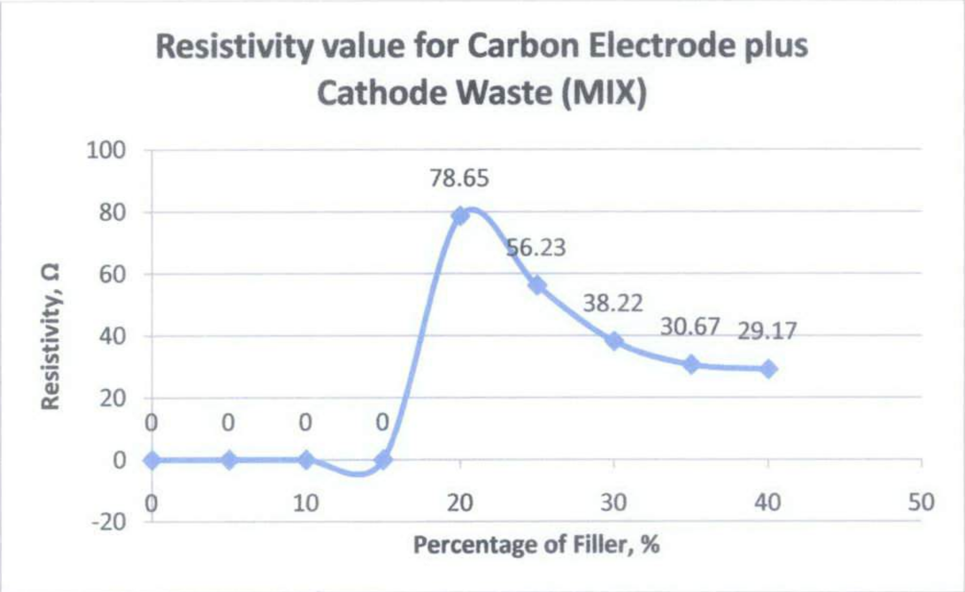


Figure 4.7: Graph of resistivity value for Carbon Electrode plus Cathode Waste (MIX) as filler

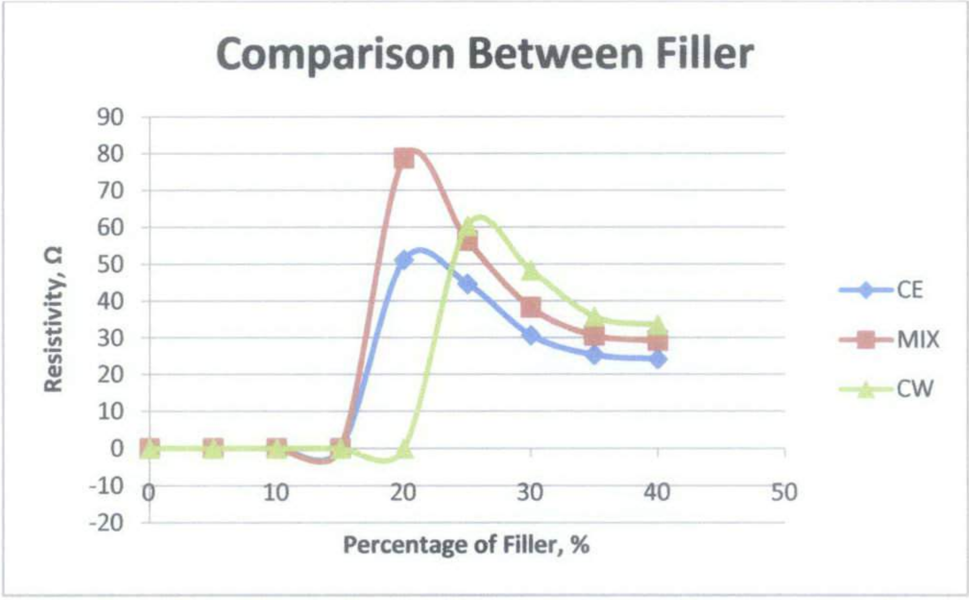


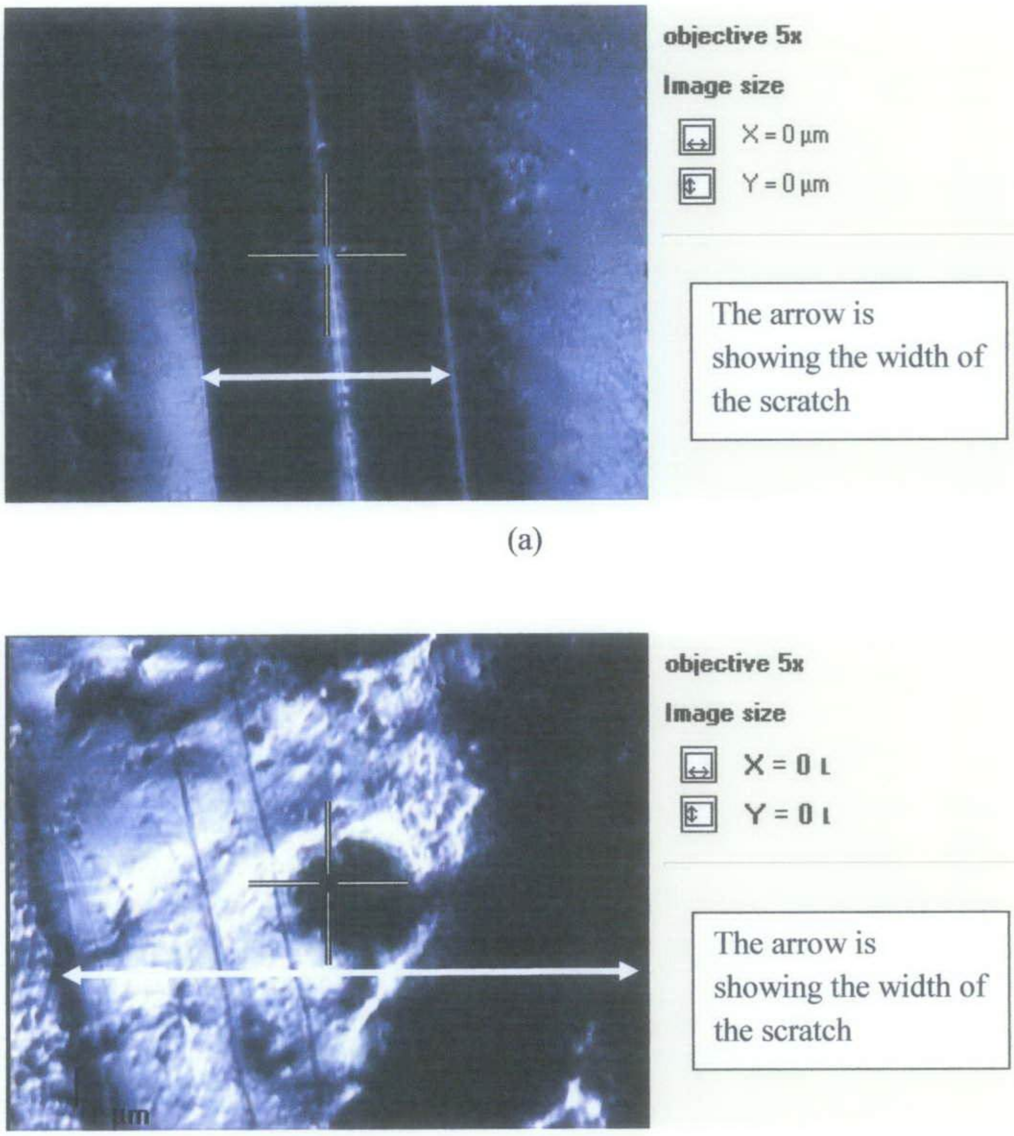
Figure 4.8: Comparison graph of resistivity value for all filler

All the filler has the lowest resistivity value at 40% of the epoxy mixture. The lowest value of resistivity however is epoxy mixture with 40% of CE filler. This is because the carbon inside CE is more reactive than the manganese inside the CW. The combination filler between CE and CW at mixing ratio 1:1 gave the resistivity value range between resistivity value of CE and CW. Addition of CE into CW improve the resistivity but not enough to surpass the CE resistivity value.

From the Table 4.6 to Table 4.8, there are some value of resistivity is zero. Zero value means that the sample has very high resistivity that outside the range of the multimeter can detect. Note that zero value does not mean that the sample is high in term of conductivity. The lowest resistivity is at 24.13 x 10^6 Ω (CE – 40%) followed by 29.17 x 10^6 Ω (MIX – 40%) and the highest is 33.45 x 10^6 Ω (CW – 40%)

4.6 Scratch Resistance Test

In order to test the adhesion properties of the blended epoxy resin to the aluminum plate substrate, scratch resistance test was carried out. Test was done using CTM Instrument – Scratch Machine to ensure the quality of the coating properties. The samples undergone the test was prepared using 1:1 mixing ratio with addition of filler varies from 0% to 40% with increment of 5% of the coating weight.



The arrow is showing the width of the scratch

(a)

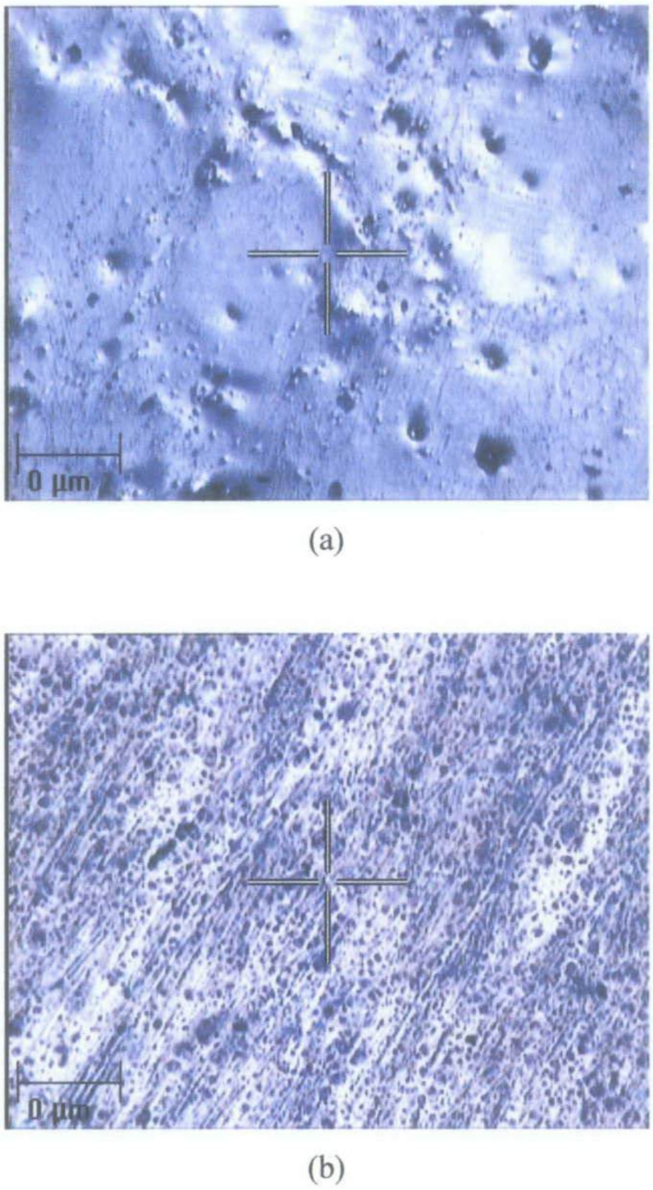
The arrow is showing the width of the scratch

(b)

Figure 4.9: Scratch test result with (a) 5% filler and (b) 40% filler

The result from the scratch test concluded that addition of filler to the epoxy mixture reduce the mechanical properties of the samples. In Figure 4.9 showed (a) with 5% filler has a small width of scratch compare to the sample with 40% filler. Increment of filler in the coating will reduce the adhesion of coating on to the aluminum plate.

4.7 Microstructure Study



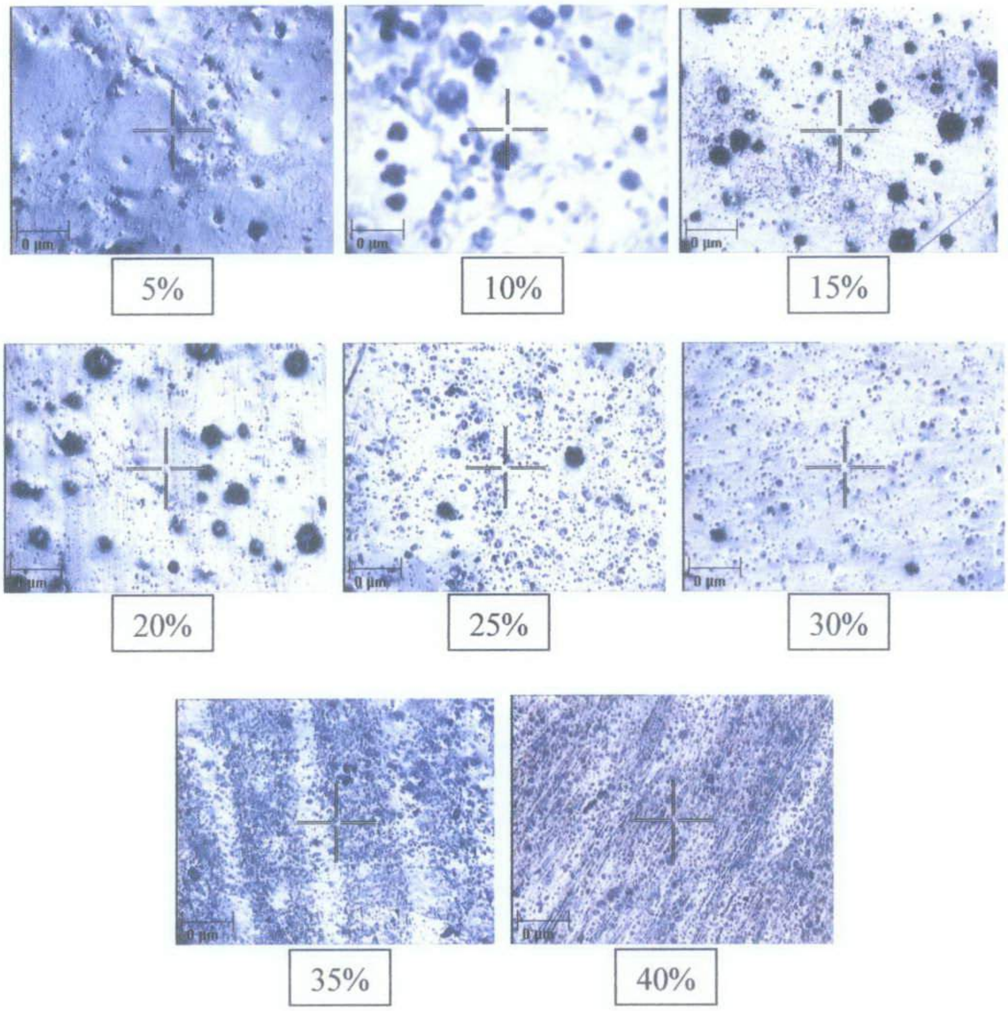
(a)

(b)

Figure 4.10: Surface picture of sample taken using optical camera at 5X magnification. (a) 5% filler (b) 40% filler

The black dot in the both figures both show how the filler disperse in the sample. In Figure 4.10 (a) there are only few black dot compared with Figure 4.10 (b). This means that with the increase of the filler in the epoxy mixture also increased the dispersion of the filler in the sample. When the filler disperse evenly in the sample, resistivity value also will be decreased.

4.7.1 Carbon Electrode (CE) Microstructure



5%

10%

15%

20%

25%

30%

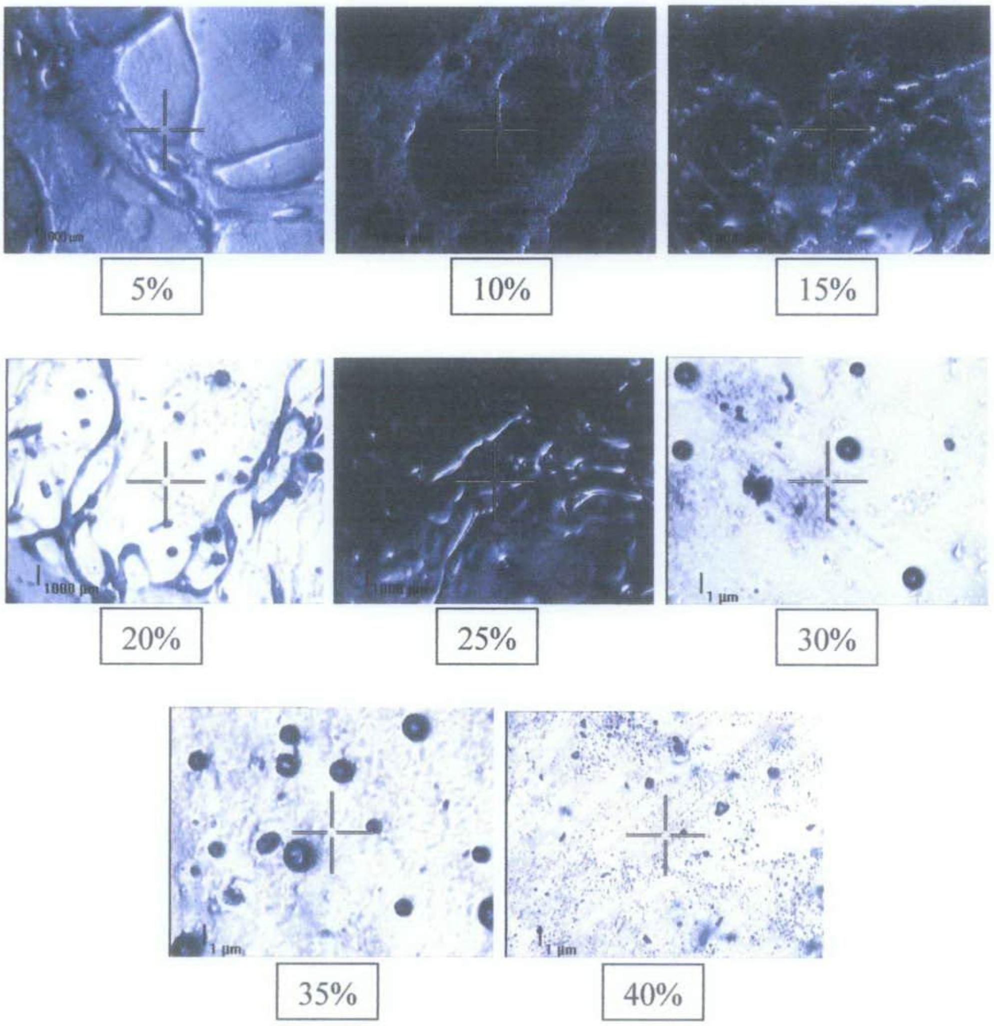
35%

40%

Figure 4.11: Carbon Electrode (CE) microstructure with variation of filler from 5% to 40% at 5X magnification

At 5% sample the dispersion is the worst compared to the 40% sample. The sample's dispersion starting to scattered evenly at 25% sample. The figure above showed that with increased of filler percentage the dispersion of the conductive paint became more evenly and well scattered in the sample.

4.7.2 Cathode Waste (CW) Microstructure



5% 10% 15%

20% 25% 30%

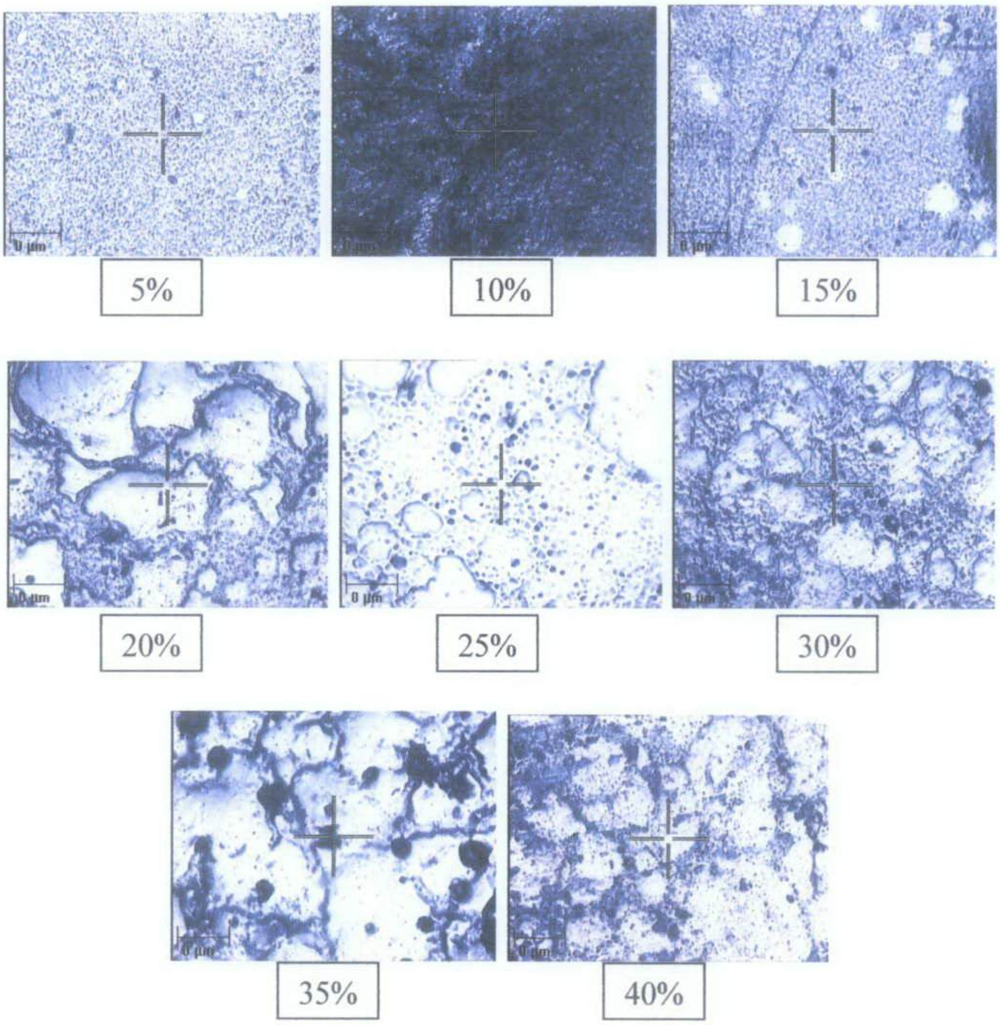
35% 40%

Figure 4.12: Cathode Waste (CW) microstructure with variation of filler from 5% to 40% at 5X magnification

At 5% sample the dispersion is the worst compared to the 40% sample. The sample's dispersion starting to scattered evenly at 30% sample. The figure above showed that with increased of filler percentage the dispersion of the conductive paint became more evenly and well scattered in the sample.

Compared to CE sample the CW samples have worst dispersion of filler. The samples starts to scatter evenly only at 30% sample compared to CE at 25% sample. This result explained why the CW samples have the highest resistivity value. Because of the dispersion is low, there was not enough contact areas between the filler particle to conduct the electric through the paint.

4.7.3 Carbon Electrode mix with Cathode Waste (MIX) Microstructure



5%

10%

15%

20%

25%

30%

35%

40%

Figure 4.13: Carbon Electrode mix with Cathode Waste (MIX) microstructure with variation of filler from 5% to 40% at 5X magnification

At 5% sample the dispersion is the worst compared to the 40% sample. The sample's dispersion starting to scattered evenly at 20% sample. The figure above showed that with increased of filler percentage the dispersion of the conductive paint became more evenly and well scattered in the sample. Compared to the CW samples dispersion results, MIX samples have better dispersion of filler, but if compared to the CE samples, the dispersion of filler in CE samples is better. Thus, the result explains why the resistivity value of MIX samples was in between of CE samples and CW samples.

CHAPTER 5
CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The conclusion was done based on the result and discussion in previous chapter. In the resistivity test the lowest value of resistivity is $24.13 \times 10^6\ \Omega$ at 40% of carbon electrode (CE) filler. This value is the best value derived from the experiment to conduct electricity compared to the others. Based on the resistivity result, the best material for filler is CE and the worst is cathode waste. In conclusion, the higher percentages of CE filler in the sample generate lower the resistivity value.

In the preliminary examination, some experiments were done to determine optimum mixing ratio of epoxy to hardener, particle distribution, particle size and curing time. The optimum mixing ratio is 1:1 between epoxy and hardener. This is because the mixture has good viscosity, fast curing time and good adhesion to the applied substrate.

In particle distribution analysis of the filler, the most of fillers were distributed at size less than 150 microns. Based on this result, the optimum size is less than 150 microns in term of quantity available. Other result that support particle size of less than 150 microns is the best is sizes more than 150 microns such as 300 microns and 600 microns have low dispersion rate. The filler particle does not disperse and mix well with the epoxy mixture. Besides that, the epoxy which is act as binder does not has enough strength to bind larger particle of filler causing the paint almost impossible to apply on to the aluminum plate.

In term of mechanical properties such as adhesion of coating, the conclusion was done based on the width of scratch after the scratch test. 5% sample has the smallest scratch width compared to the 40% sample. So, the mechanical properties in term of adhesion of the sample to the materials decrease with the increasing of the percentage of filler. In microstructure study, result shows that CE sample was the best in term of filler dispersion in the paint, while CW sample has the worst filler dispersion in the paint. In MIX sample combination of CE and CW filler has improve the dispersion slightly but still not surpass the CE sample's dispersion.

5.2 Future Work

From the studies that have been done in this project, there are still some improvements that can be done to generate more accurate and more details. One of the improvements is to use finer particle size of filler. Finer particle size will give better particle dispersion and more contact area between the particles and reduce the resistivity value.

The thickness of the sample should be more consistent. It is hard to get consistent value of thickness because there is no device or tools that can control the sample thickness during epoxy mixing and sample preparation on this study. Hegmen gauge can be used for this purpose in future work.

Proper or advance technique can be used in the future to get better value of the resistivity. Four point probe machine can be used to generate accurate data since the machine have greater sensitivity.

Beside scratch test to determine the mechanical properties, impact test and hardness test also can be done. The microstructure and composition of the sample can be studied using Scanning Electron Microscopic (SEM) while the filler composition can be studied using XRD machine.

The used of other filler materials that have higher reactivity and lower resistivity values also can improve this study.

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APPENDICES

APPENDIX A: GANNT CHART

First phase of the project / 1st semester – January 2008

No.	Details	Weeks																
		1	2	3	4	5	6	7		8	9	10	11	12	13	14	15	16
1	Project topic selection	■	■						SEMESTER BREAK									
2	Meeting with supervisor		■	■	■	■	■	■		■	■	■	■	■	■	■	■	■
3	Preliminary research work			■	■	■												
4	Preliminary report preparation			■	■	■												
5	Submission of preliminary report (15th Feb 2008)				■	■												
6	Seminar 1 preparation						■	■		■								
7	Progress report preparation						■	■		■								
8	Submission of progress report									■								
9	Research the suitable material for the experiment							■		■	■	■	■	■	■	■	■	
10	Research for the experiment that need to be carry out							■		■	■	■	■	■	■	■	■	
11	Get familiar with the interface study & analysis											■	■	■	■	■	■	
12	Seminar 1										🟦	🟦	🟦	■				
13	Interim report preparation													■	■	■	■	
14	Submission for interim report																🟦	■
15	Oral presentation preparation																■	■
16	Oral presentation																🟦	■

🟦 Scheduled task

■ Completed task

Second phase of the project / 2nd semester – July 2008

No.	Details	Weeks																				
		1	2	3	4	5	6	7	8	9	10		11	12	13	14	15	16	17	18	19	20
1	Project work continue	⬛	⬛	⬛	⬛	⬛	⬛	⬛	⬛	⬛	⬛	SEMESTER BREAK	⬛	⬛	⬛	⬛	⬛					
2	Meeting with supervisor	🟦	⬛	⬛	⬛	🟦	🟦	🟦	⬛	⬛	⬛		🟦	⬛	🟦	⬛	⬛	🟦	🟦	⬛	🟦	⬛
4	Progress report 1 preparation			⬛	⬛																	
5	Submission of progress report 1				⬛																	
7	Progress report 2 preparation							⬛	⬛													
8	Submission of progress report 2								⬛													
9	Seminar (compulsory)								⬛	🟦	🟦											
10	Poster Exhibition												🟦	⬛								
11	Submission of dissertation (soft bound)													🟦	🟦	⬛	⬛					
12	Oral presentation																⬛	⬛				
13	Submission of project dissertation (Hard Bound)																		⬛	⬛	⬛	⬛

🟦 Scheduled task

⬛ Completed task

APPENDIX B

Material Safety Data Sheet: Epoxy Resin DER 331

SECTION I - Material Identity

Item Name	
Part Number/Trade Name	D.E.R.(R) 331 EPOXY RESIN
National Stock Number	8030000861506
CAGE Code	71984
Part Number Indicator	A
MSDS Number	8217
HAZ Code	B

SECTION II - Manufacturer's Information

Manufacturer Name	DOW CHEMICAL U.S.A
Emergency Phone	517--636-4400

MSDS Preparer's Information

Date MSDS Prepared/Revised	NONE
Active Indicator	N

Alternate Vendors

SECTION III - Physical/Chemical Characteristics

Specification Number	NR
Boiling Point	NR
Container Pressure Code	4
Temperature Code	8
Product State Code	U

SECTION IV - Fire and Explosion Hazard Data

Flash Point Method	UNK

SECTION V - Reactivity Data

Stability	YES
Stability Conditions to Avoid	EXCESS HEATING OVER LONG PERIODS OF TIME DEGRADES THE RESIN
Materials to Avoid	BASE,ACID,AMINES AND OXIDIZING MATERIALS
Hazardous Decomposition Products	EPOXY RESINS:PHENOLICS,CARBON MONOXIDE
LD50 - LD50 Mixture	ORAL:RATS:>5000MG/KG

SECTION VI - Health Hazard Data

Route of Entry: Skin	YES
Route of Entry: Inhalation	NO
Health Hazards - Acute and Chronic	EXCEPT FOR SKIN SENSITIZATION,REPEATED EXPOSURES TO LOW MOLECULAR WEIGHT DIGLYCIDYL ETHER OF BISPHENOL&ARE NOT ANTICIPATED TO CAUSE ANY SIGNIFICANT ADVERSE EFFECTS
Carcinogenity: NTP	NR
Carcinogenity: IARC	NR
Carcinogenity: OSHA	NR
Symptoms of Overexposure	EYE:TEMP.EYE IRRIT.CORNEAL INJURY UNLIKELY;SKIN:REPEATED EXPOSURE MAY CAUSE IRRIT.POSS.ALLERGIC REACTION;INGEST:SINGLE DOSE ORALTOXICITY IS LOW;INHALATION:VAPORS ARE UNLIKELY DUE TO PHYSICAL PROPERTIES

Medical Cond. Aggrevated by Exposure	NR

SECTION VII - Precautions for Safe Handling and Use

SECTION VIII - Control Measures

Respiratory Protection	NO RESPIRATORY PROTECTION SHOULD BE NEEDED
Ventilation	GOOD GENERAL VENTILATION SHOULD BE SUFFICIENT FOR MOST CONDITIONS
Protective Gloves	IMPERVIOUS
Eye Protection	SAFETY GLASSES & CHEMICAL GOGGLES
Supplemental Health/Safety Data	1ST AID:EYE:IRRIGATE IMMED'Y WITH WATER FOR AT LEAST 5 MINUTES; SKIN:WASH OFF IN FLOWING WATER OR SHOWER;INHAL:REMOVE TO FRESH AIR IF EFFECTS OCCUR.CONSULT A PHYSICIAN; NOTE TO PHYSICIAN:NO SPECIFIC ANTIDOTE.SUPPORTIVE CARE.TREATMENT BASED ON JUDGEMENT
Disposal Code	0

SECTION IX - Label Data

Protect Eye	YES
Protect Skin	YES
Protect Respiratory	YES
Chronic Indicator	UNKNOWN
Contact Code	SLIGHT
Fire Code	UNKNOWN
Health Code	UNKNOWN
React Code	UNKNOWN

SECTION X - Transportation Data

Container Quantity	1
Unit of Measure	PT

SECTION XI - Site Specific/Reporting Information

SECTION XII - Ingredients/Identity Information

Ingredient #	01
Ingredient Name	REACTION PRODUCTS OF EPICHLOROHYDRIN AND BISPHENOL A
CAS Number	25085998
NIOSH Number	NR
Proprietary	NO
Percent	100
ACGIH TLV	NR

APPENDIX C

MATERIAL SAFETY DATA SHEET FOR POLYAMIDE

SECTION 1 - MATERIAL IDENTIFICATION

PRODUCT NAMES

EP84-0216 Activator

EP9027 Activator

EP9098 Activator

REVISION DATE JANUARY 1999

****

EMERGENCY OVERVIEW

****

HMIS/NFPA HEALTH 2 FLAMMABILITY 1 REACTIVITY 0

PHYSICAL FORM Mobile liquid

COLOR Amber

ODOR Irritating

HAZARDS Moderate eye irritant. Moderate skin irritant. Respiratory Irritant. May cause skin sensitization.

EXTINGUISHING MEDIA Ignition will give rise to a Class B fire. In case of large fire use: alcohol foam, water spray. In case of small fire use: carbon dioxide (CO2), dry chemical, dry sand or limestone.

****

C.A.S. CHEMICAL NAME	Mixture
SYNONYMS	None
CHEMICAL FAMILY	Polyamide
EMPIRICAL FORMULA	Mixture
INTENDED USE	Curing Agent, Epoxy
REVISION NOTES	Updated health hazard information. SIGNS AND SYMPTOMS OF EXPOSURE (Acute effects) SIGNS AND SYMPTOMS OF EXPOSURE (Possible Longer Term Effects) Updated handling and storage information.

SECTION 2 - INGREDIENTS

Num	%	CAS Number and Chemical Name
1.	>90.00	68082-29-1 POLYMER OF C-18 UNSAT'D FATTY ACID DIMERS W/ TETA & TOFA
2.	<10.00	112-24-3 TRIETHYLENETETRAMINE (TETA)

OSHA (ACGIH) EXPOSURE LIMITS

TWA		STEL		CEILING	
ppm	mg/m3	ppm	mg/m3	ppm	mg/m3

1. OSHA N/E N/E N/E N/E N/E N/E

ACGIH N/E N/E N/E N/E N/E N/E

---

2. OSHA N/E N/E N/E N/E N/E N/E

ACGIH N/E N/E N/E N/E N/E N/E

N/E = Not Established.

SECTION 3 - HEALTH HAZARDS

ROUTES OF EXPOSURE

Eye Contact

Skin Contact

Ingestion

Inhalation

Skin Absorption

EXPOSURE STANDARDS

No standards established for the product. Maintain air contaminant concentrations in the workplace at the lowest feasible levels.

HEALTH HAZARDS

Moderate eye irritant.

Moderate skin irritant.

Respiratory Irritant.

May cause skin sensitization.

TARGET ORGANS

Eye
Skin
Respiratory system

SIGNS AND SYMPTOMS OF EXPOSURE (Acute effects)

Product vapor in low concentrations can cause lacrimation, conjunctivitis and corneal edema when absorbed into the tissue of the eye from the atmosphere. Corneal edema may give rise to a perception of "blue haze" or "fog" around lights. The effect is transient and has no known residual effect. Inhalation of mists may cause irritation in the respiratory tract. Inhalation of vapors may cause irritation in the respiratory tract. Contact with the skin or eyes causes moderate eye and skin irritation, redness and discomfort which is transient. Coughing and chest pain may result. Product is absorbed through the skin and may cause nausea, headache and general discomfort.

SIGNS AND SYMPTOMS OF EXPOSURE (Possible Longer Term Effects)

Repeated and/or prolonged exposure may cause allergic reaction/sensitization. Repeated and/or prolonged exposures may result in adverse respiratory effects (such as cough, tightness of chest or shortness of breath), adverse eye effects (such as conjunctivitis or corneal damage), and adverse skin effects (such as rash, irritation or corrosion). Effects from inhalation of vapors may be delayed. Repeated and/or prolonged exposure to low concentrations of vapor may cause: sore throat, eye irritation which are transient.

MEDICAL CONDITIONS GENERALLY AGGRAVATED BY EXPOSURE

Asthma

Chronic respiratory disease (e.g. Bronchitis, Emphysema)

Eye disease

Skin disorders and Allergies

CARCINOGENS UNDER OSHA, ACGIH, NTP, IARC, OTHER

This product contains no carcinogens in concentrations of 0.1 percent or greater.

SECTION 4 - FIRST AID

EYE CONTACT

Hold eyelids apart and immediately flush eyes with plenty of water for at least 15 minutes. Seek medical advice.

SKIN CONTACT

Remove product and immediately flush affected area with water for at least 15 minutes. Remove contaminated clothing and shoes. Seek medical advice.

INHALATION

Move patient to fresh air. If breathing has stopped or is labored give assisted respiration (e.g. mouth-to-mouth). Supplemental oxygen may be indicated. Prevent aspiration of vomit. Turn victim's head to the side. Seek medical advice.

INGESTION

If swallowed, call a physician immediately. Remove stomach contents by gastric suction or induce vomiting only as directed by medical personnel. Never give anything by mouth to an unconscious person.

SECTION 5 - FIRE AND EXPLOSION DATA

FLASH POINT (closed cup)	200.00 C (392.00 F)
UPPER EXPLOSION LIMIT (UEL)	No Data
LOWER EXPLOSION LIMIT (LEL)	No Data
AUTOIGNITION TEMPERATURE	No Data

FIRE HAZARD CLASSIFICATION (OSHA/NFPA)

Class IIIB

EXTINGUISHING MEDIA

Ignition will give rise to a Class B fire. In case of large fire use: water spray, alcohol foam. In case of small fire use: carbon dioxide ($CO2$), dry chemical, dries sand or limestone.

SPECIAL FIRE FIGHTING PROCEDURES

Firefighters should wear butyl rubber boots, gloves, and body suit and a self-contained breathing apparatus.

UNUSUAL FIRE AND EXPLOSION HAZARDS

May generate toxic or irritating combustion products. Sudden reaction and fire may result if product is mixed with an oxidizing agent. May generate carbon monoxide gas. May generate toxic nitrogen oxide gases. May generate ammonia gas.

Personnel in vicinity and downwind should be evacuated.

SECTION 6 - ACCIDENTAL RELEASE MEASURES

CONTAINMENT TECHNIQUES (Removal of ignition sources, dicing etc)

Shut off or remove all ignition sources. Stop the leak, if possible. Ventilate the space involved. Reduce vapor spreading with a water spray. Construct a dike to prevent spreading (includes molten liquids until they freeze).

CLEAN-UP PROCEDURES

If recovery is not feasible, admix with dry soil, sand or non- reactive absorbent and place in an appropriate chemical waste container. Transfer to containers by suction, preparatory for later disposal. Place in metal containers for recovery or disposal. Flush area with water spray. Clean-up personnel must be equipped with self contained breathing apparatus and butyl rubber protective clothing. For large spills, recover spilled material with a vacuum truck.

OTHER EMERGENCY ADVICE

Open enclosed spaces to outside atmosphere. Wear protective

clothing, boots, gloves, and eye protection.

SECTION 7 - HANDLING AND STORAGE

STORAGE

Keep away from: acids, oxidizers. Keep in cool, dry, ventilated storage and in closed containers. Do not store in reactive metal containers.

HANDLING

Avoid contact with skin or eyes. When handling, does not eat, drink, or smoke.

OTHER PRECAUTIONS

Emergency showers and eye wash stations should be readily accessible. Adhere to work practice rules established by government regulations (e.g. OSHA). Do not use sodium nitrite or other nitrosating agents in formulations containing this product. Cancer-causing nitrosamines could be formed.

SECTION 8 - PERSONAL PROTECTION / EXPOSURE CONTROLS

EYE PROTECTION

Splash-proof eye goggles. In emergency situations, use eye goggles with a full face shield.

HAND PROTECTION

Impermeable gloves. Polyvinyl chloride gloves.

RESPIRATORY PROTECTION

Not required under normal conditions in a well-ventilated workplace. An organic vapor respirator National Institute for

Occupational Safety and Health (NIOSH) approved for organic vapors is recommended under emergency conditions.

PROTECTIVE CLOTHING

Long sleeved clothing.

ENGINEERING CONTROLS

No specific controls needed.

WORK AND HYGIENIC PRACTICES

Provide readily accessible eye wash stations and safety showers.

Wash at the end of each work shift and before eating, smoking or using the toilet.

SECTION 9 - TYPICAL PHYSICAL AND CHEMICAL PROPERTIES

PHYSICAL FORM	Mobile liquid
COLOR	Amber
ODOR	Irritating
pH	No Data
VAPOR PRESSURE (mm Hg at 21C (70F))	<10.34
VAPOR DENSITY (Air = 1)	No Data
BOILING POINT	>176.67 C (>350.01 F)
MELTING POINT	No Data
SOLUBILITY IN WATER	Slight (0.1 - 1%)
SPECIFIC GRAVITY (Water = 1)	0.96
MOLECULAR WEIGHT	Mixture

SECTION 10 - STABILITY AND REACTIVITY

CHEMICAL STABILITY

Stable

CONDITIONS TO AVOID (if unstable)

Not applicable

INCOMPATIBILITY (Materials to Avoid)

Mineral acids (i.e. sulfuric, phosphoric, etc.). Organic acids (i.e. acetic acid, citric acid etc.). Oxidizing Agents (i.e.perchlorates, nitrates etc.). Sodium or Calcium Hypochlorite.

CAUTION! N-Nitrosamines, many of which are known to be potent carcinogens, may be formed when the product comes in contact with nitrous acid, nitrites or atmospheres with high nitrous oxide concentrations. Product slowly corrodes copper, aluminum, zinc and galvanized surfaces. Reaction with peroxides may result in violent decomposition of peroxide possibly creating an explosion. Nitrites, nitrosating agents. A reaction accompanied by large heat release occurs when the product is mixed with acids. Heat generated may be sufficient to cause vigorous boiling creating a hazard due to splashing or splattering of hot material.

HAZARDOUS DECOMPOSITION PRODUCTS (from burning, heating, or reaction with other materials).

Carbon Monoxide in a fire. Carbon Dioxide in a fire. Ammonia when heated. Nitrogen Oxides in a fire. Irritating and toxic fumes at elevated temperatures. Nitric acid in a fire. nitrosamines.

Nitrogen oxide can react with water vapors to form corrosive nitric acid (TLV=2 ppm).

HAZARDOUS POLYMERIZATION

Will not occur

CONDITIONS TO AVOID (if polymerization may occur)

Not applicable

SECTION 11 - TOXICOLOGICAL PROPERTIES

ACUTE ORAL TOXICITY (LD50, RAT)

>2000.00 mg/kg (No deaths)

ACUTE DERMAL TOXICITY (LD50, RABBIT)

>2000.00 mg/kg (No deaths)

ACUTE INHALATION TOXICITY (LC50, RAT)

No Data

OTHER ACUTE EFFECTS

No Data

IRRITATION EFFECTS DATA

Moderate irritant to the eyes of a rabbit. Moderate irritant to the skin of a rabbit. Projecting respiratory irritation due to moderate skin irritation.

CHRONIC/SUBCHRONIC DATA

No delayed, subchronic or chronic test data are known.

SECTION 12 - ECOLOGICAL INFORMATION

No Data

SECTION 13 - DISPOSAL CONSIDERATIONS

WASTE DISPOSAL

Comply with all Federal, State and Local Regulations.

SECTION 14 - TRANSPORT INFORMATION

DOT NON-BULK SHIPPING NAME	RESIN COMPOUND - Not DOT Regulated
DOT BULK SHIPPING NAME	Refer to Bill of Lading.
IMO SHIPPING DATA	Refer to Bill of Lading.
ICAO/IATA SHIPPING DATA	RESIN COMPOUND - Not IATA Regulated

SECTION 15 - REGULATORY INFORMATION

US FEDERAL REGULATIONS

TOXIC SUBSTANCES CONTROL ACT (TSCA)-

All components are included in the EPA Toxic Substances Control Act (TSCA) Chemical Substance Inventory.

OSHA Hazard Communication Standard (29CFR1910.1200) hazard class(es)

Irritant. Sensitizer.

EPA SARA Title III Section 312 (40CFR370) hazard class

Immediate Health Hazard. Delayed Health Hazard.

EPA SARA Title III Section 313 (40CFR372) toxic chemicals above "de minimis" level is

None

STATE REGULATIONS

PROPOSITION 65 SUBSTANCES (component(s) known to the State of California to cause cancer and/or reproductive toxicity and subject to warning and discharge requirements under the "Safe Drinking Water and Toxic Enforcement Act of 1986")

None

NEW JERSEY TRADE SECRET REGISTRY NUMBER(S)

None

SECTION 16 - INTERNATIONAL REGULATIONS

CANADA

DSL

Included on Inventory.

WHMIS HAZARD CLASSIFICATION

Class D Division 2B,

WHMIS TRADE SECRET REGISTRY NUMBER(S)

None

WHMIS HAZARDOUS INGREDIENTS

TRIETHYLENETETRAMINE (TETA)

WHMIS SYMBOLS

Stylized T,

EUROPEAN ECONOMIC COMMUNITY (EEC)

EINECS/ELINCS MASTER INVENTORY

Included on Inventory.

EEC SYMBOL

IRRITANT (Xi)

EEC RISK (R) PHRASES

May cause sensitization by skin contact (R43). Irritating to eyes and skin (R36/38).

EEC SAFETY PHRASES

In case of contact with eyes, rinse immediately with plenty of water and seek medical advice (S26). Wear suitable protective clothing and gloves (S36/37).

AUSTRALIA

AICS

Included on Inventory.

JAPAN MITI

Included on Inventory.

PHILIPPINES PICCS

Included on Inventory.

KOREA ECL

Included on Inventory.

CHINA SEPA

Included on Inventory.

END OF DOCUMENT