

**CORROSION PROTECTION OF ZINC
COATING ON STEELS**

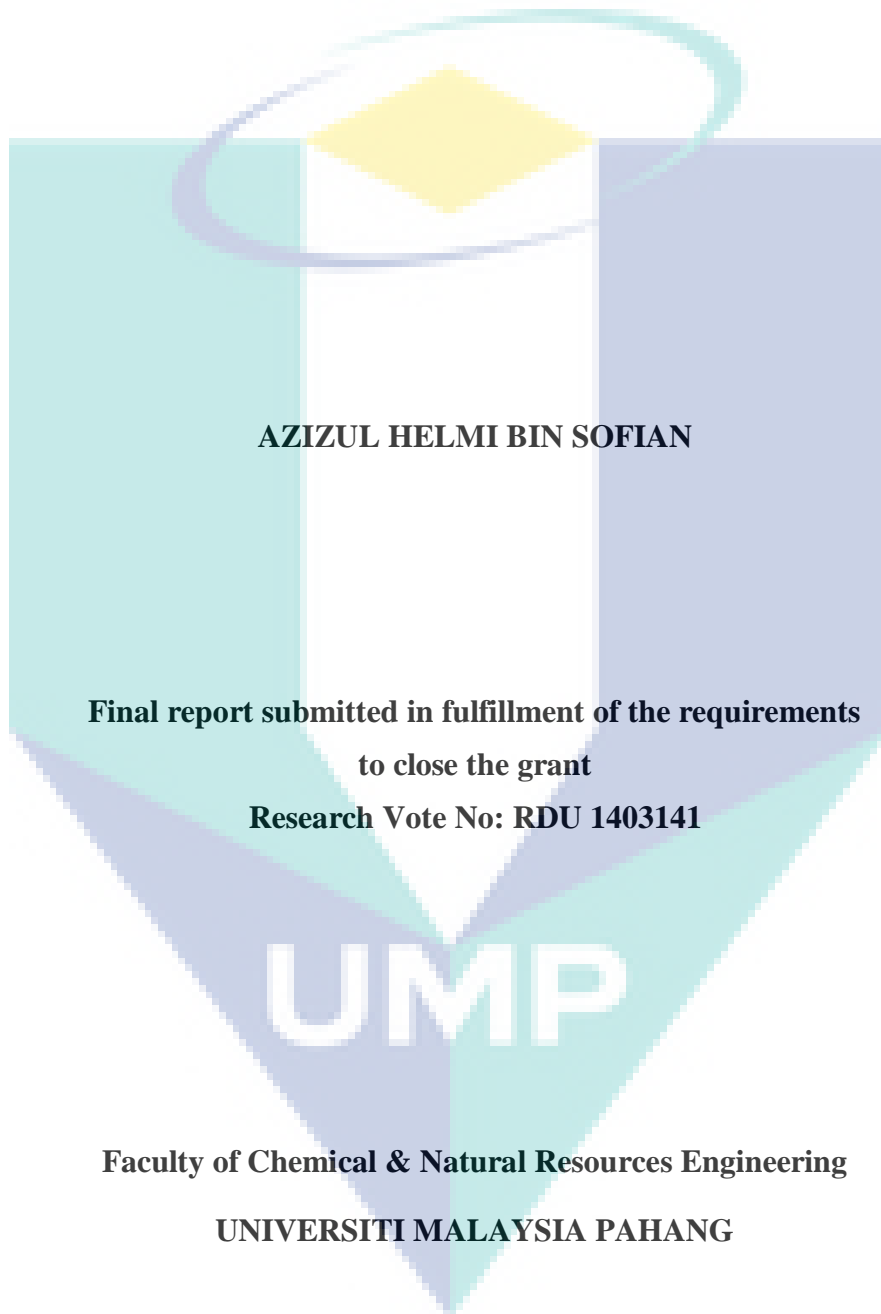


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RESEARCH VOTE NO: RDU 1403141

UNIVERSITI MALAYSIA PAHANG

CORROSION PROTECTION OF ZINC COATING ON STEELS



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**Final report submitted in fulfillment of the requirements
to close the grant**

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UMP

Faculty of Chemical & Natural Resources Engineering

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ABSTRACT

Organic coated steel is widely used in industry because of well-known of its advantage towards corrosion protection. The objective is to understanding the physical appearances and behaviour of organic coating using SEM by investigate the internal reaction and environment nature. Three types of organic coating were used namely epoxy, zinc oxide and zinc primer spray paint to evaluate the effectiveness of coatings towards corrosion protection. There were four methods to identify the effectiveness of organic coating by mechanical testing including adhesion test, immersion test, temperature and thickness with different layer (1, 3, 5 layers). In addition, the kinetic growth can be analysed or interpret by using scanning electron microscope (SEM) for morphology observation. First, the samples mixed with solvent, ratio (1:1) and dry for 24 hours. After that, the sample were placed in hot area by taking the daily temperature and reference of humidity from Malaysia Meteorology Department. Besides that, cross-cut test was used to identify the good quality of adhesion. At the same times, samples were immersed in 3.5 % NaCl solution in duration at least 60 days. The thickness was measured by weighing before and after coating. The result for adhesion test for epoxy and zinc-epoxy shows high strength in chemical bonding. The weight percentage for all sample shows gain in weight but only one layer of zinc primer spray paint loss in weight. The temperature was lower for coated sample than uncoated sample. Lastly, the image of sample for immersion test were taken from SEM analysis. This research shows that zinc and epoxy act as anticorrosive protection barrier. The effectiveness of the coating thickness is recommended for future study.

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ABSTRAK

Keluli bersalut organik digunakan secara meluas dalam bidang industri kerana terkenal dengan kelebihanannya melindungi besi daripada berkarat. Objektif projek ini adalah untuk memahami fizikal luaran dan dalaman salutan organik menggunakan SEM dengan mengkaji tindak balas elektrokimia dan alam sekitar. Tiga sampel organik yang digunakan iaitu epoksi, epoksi oksida dan cat sembur primer untuk menilai tahap keberkesanan lapisan untuk perlindungan daripada pengaratan dan beberapa perbezaan lapisan salutan (1, 3 dan 5 lapisan). Terdapat empat kaedah untuk mengenal pasti keberkesanan salutan organik dengan cara ujian mekanikal termasuk ujian rekatan, ujian rendaman dan suhu dan ketebalan. Di samping itu, pertumbuhan kinetik boleh dianalisis atau ditafsir dengan menggunakan ujian seperti imbasan mikroskop elektron (SEM) untuk kajian morfologi. Pertama, sampel akan bercampur dengan pelarut, nisbah (1:1) selama 24 jam. Selepas itu, sampel diletakkan di kawasan panas dengan mengambil suhu harian dan rujukan kelembapan dari Jabatan Meteorologi Malaysia. Selain itu, ujian silang-potong digunakan untuk mengenal pasti kualiti rekatan yang baik. Pada masa sama, sampel direndam dalam 3.5 % NaCl larutan dalam tempoh sekurang-kurangnya 60 hari. Ketebalan boleh diukur dengan menimbang sebelum dan selepas salutan. Berdasarkan keputusan ujian rekatan menunjukkan bahawa epoksi dan komposit epoksi dengan zink dalam kondisi yang baik dan menunjukkan ikatan yang kuat dalam tindak balas kimia. Peratusan berat untuk semua sampel menunjukkan kenaikan dalam berat tetapi hanya satu lapisan zink primer penghakis berat. Suhu sampel adalah lebih rendah untuk sampel yang bersalut daripada sampel tidak bersalut. Akhir sekali, imej untuk sampel ujian rendaman diambil daripada analisis SEM. Tujuan kajian ini adalah untuk menilai tahap keberkesanan salutan organik terhadap pengaratan. Kajian ini menunjukkan bahawa zink dan epoxy bertindak sebagai penghalang perlindungan anti-karat. Keberkesanan ketebalan lapisan telah dicadangkan untuk kajian seterusnya.

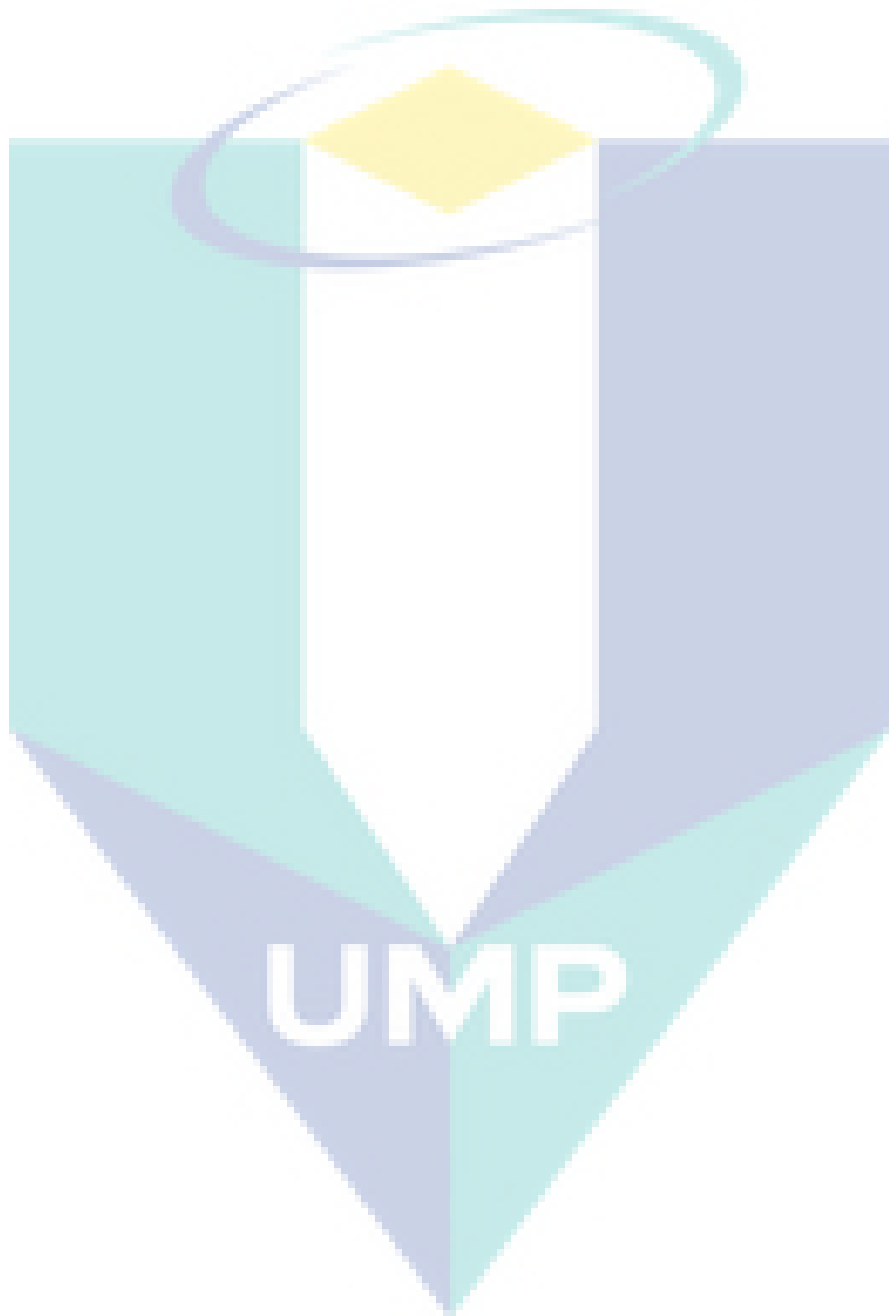


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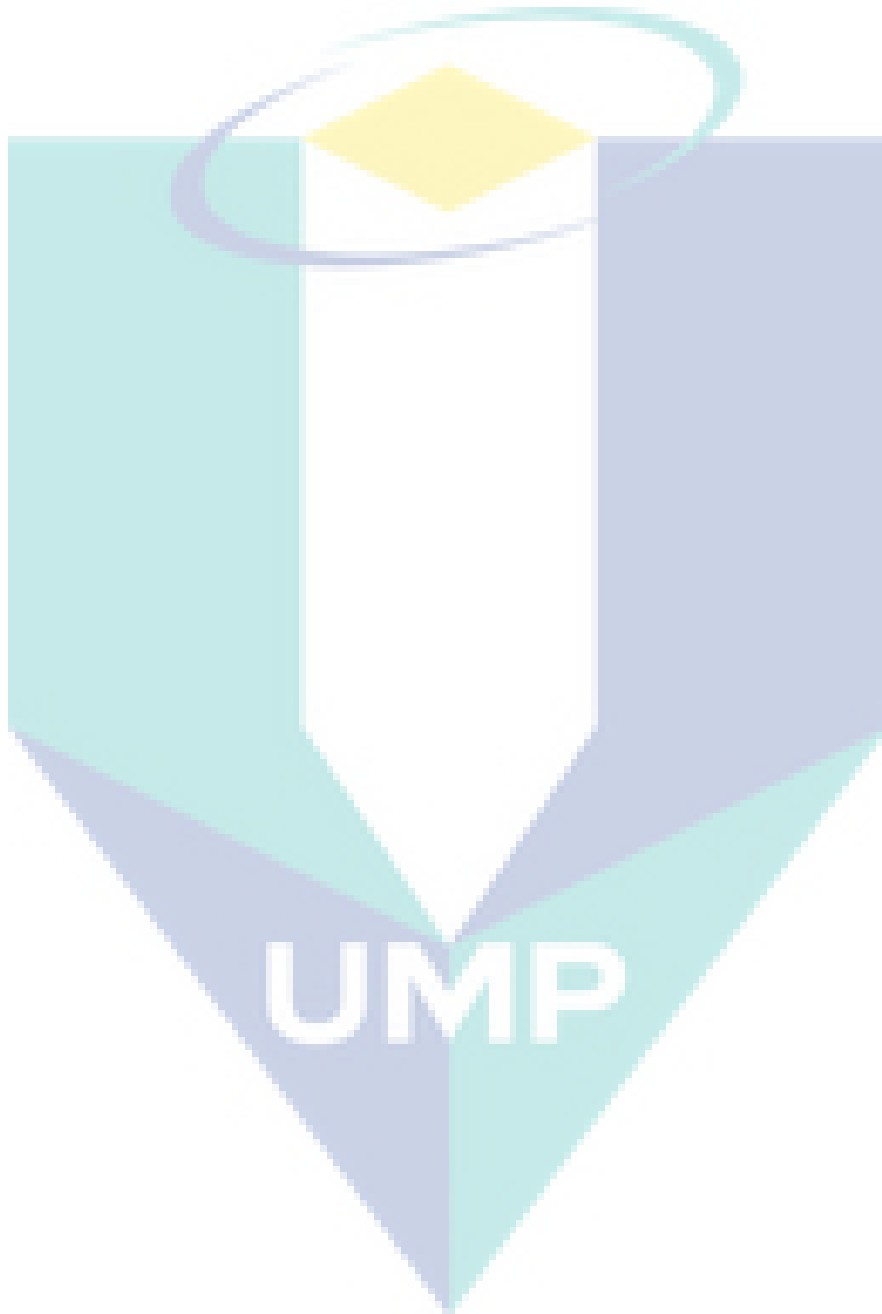
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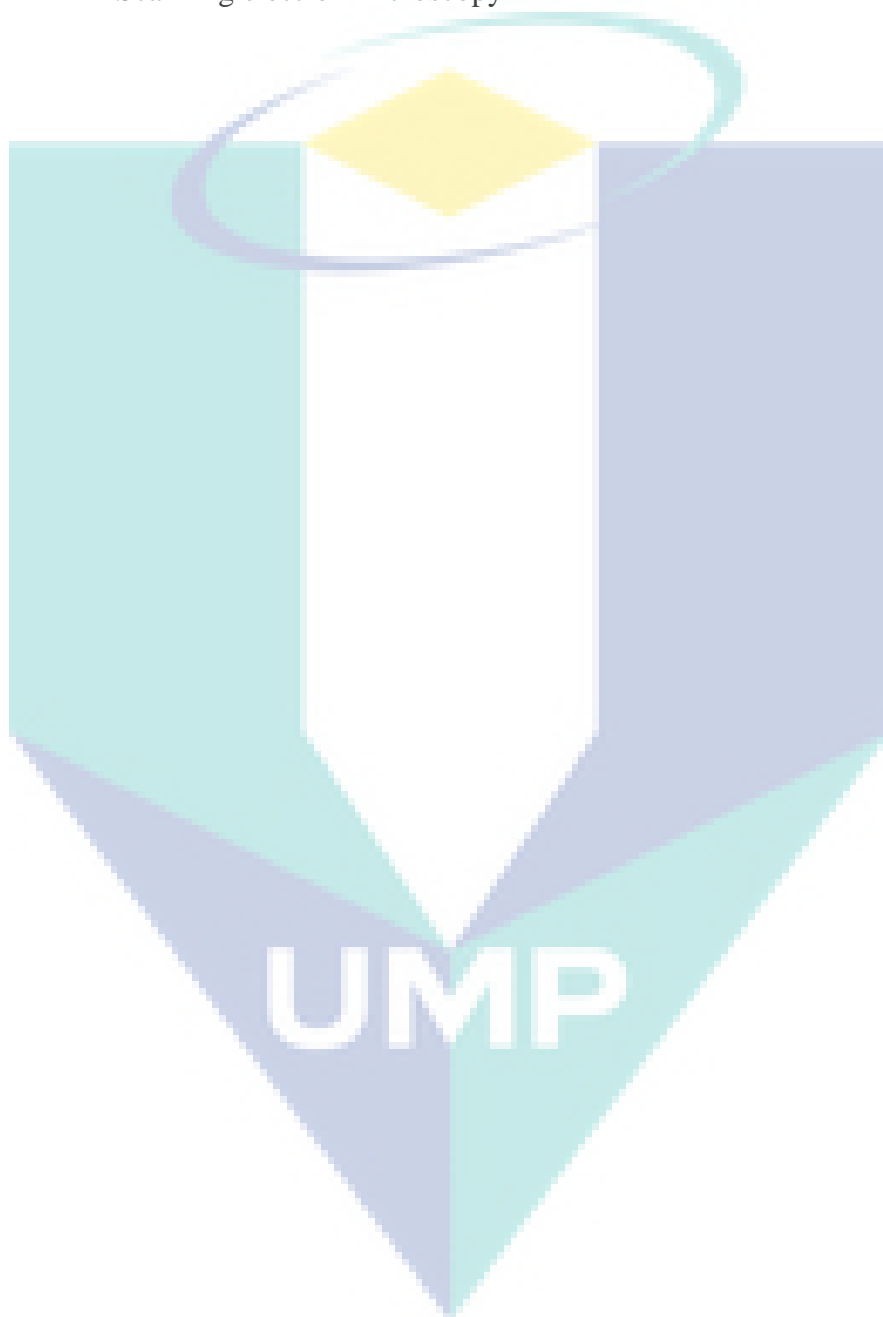
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LIST OF ABBREVIATIONS

EIS Electrochemical impedance spectroscopy

SEM Scanning electron microscopy



CHAPTER 1

INTRODUCTION

1.1 Background

1.1.1 Mechanism of Corrosion

Iron is the main component of steel. The other elements of steel are carbon (C), manganese (Mn), phosphorus (P), sulphur (S), silicon (Si), nickel (Ni) and Chromium (Cr) (The elements of steel, 2013). Steel can corrode if involving oxygen, water and metal able to give up the electrons. Firstly, iron (Fe) lose the electron become a positively charge and bond to negative charge atom become iron hydroxide (Nimmo & Hinds, 2003).



The second steps is iron hydroxide reacts with an excess of oxygen, dissolves in water produce hydrated iron oxide which brown in colour (Nimmo & Hinds, 2003).



1.1.2 What is Corrosion

Corrosion is an environment nature process where the metallic materials changes the physical appearance, back to the original iron ore and loss function of

component (Nimmo & Hinds, 2003). The Gibbs energy is a driving force for all corrosion by lowering the systems (Shaw & Kelly, 2006).

In addition, ISO 8044 defines corrosion is a “Physicochemical interaction, of an electrochemical nature between metal and environment which results in changes of metal properties and may lead to impairment of function of metal, the environment or the technical system of which form apart” (Demadis et al., 2006). So, both chemical and electrochemical reaction with environment can affect the deterioration of metallic materials (ASM, 2000).

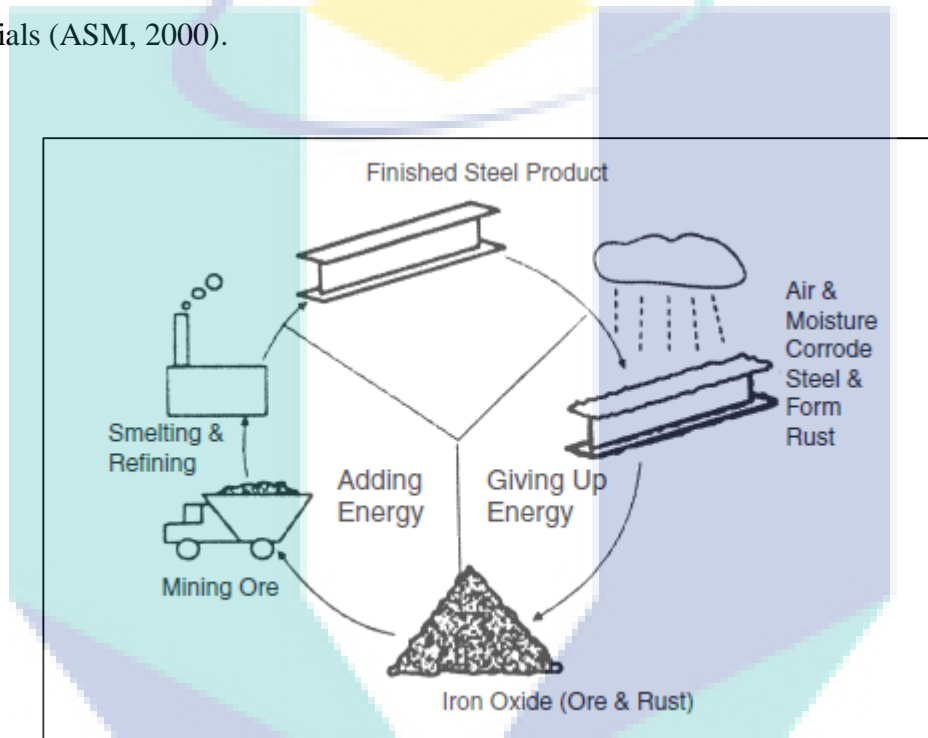


Figure 0.1 Illustration of corrosion cycle of steel (ASM International, 2000)

Based on figure 4 illustrates above, the combination of steel and other chemical element have a natural tendency to return to lowest energy. The production of metal involving adding energy to the system and result the metal has strong driving force will giving up energy return to lowest state. Combination of steel with oxygen and water will form hydrated iron oxide (rust) back to original ore (ASM, 2000).

1.1.3 The Consequences of Corrosion

The corrosion is one of the major harmful effects in industries that need to be controlled before harmful to human and surrounding. The consequences of corrosion are (Tezdogan, 2014):

- Hazards or injuries to people from structural failure or breakdown
- Loss of time to repair the equipment
- Reduced values of good
- Contamination of fluids in vessel and pipe (cloudy air)
- Mechanical damage such as valve, pumps or blockage of pipe

1.1.4 Fire and Explosion of Oil and Gas Company in Washington

There are two terrible cases of fire and explosion happen because of corrosion at Tesoro and Chevron Oil and Refinery Company. On 04 February 2010, Tesoro Company of Oil and Refining at Anacortes, Washington had a fire and explosion accident due to failure process of one parts (E) of heat exchanger. Based on final investigation report by Tesoro Refinery “carbon steel of heat exchanger being weakening because of damage mechanism known as high temperature hydrogen attack (HTHA). HTHA is a damage mechanism that results in fracture, decarburization and cracking and occurs when carbon steel equipment is exposed to high temperature and pressure.”

The temperature is above 500 °F, based on facts “there is problem if the temperature above 400 °F.” In addition, HTHA found in high stress area where no post-weld heat treated. Unfortunately, the explosion caused seven employees were killed. So, in order to prevent this accident choosing the right material selection for low carbon steel, high alloyed steel, using Nelson Curve for safety margin of 30 °C and follow the safety limit.



Figure 0.2: Fire and Explosion at Tesoro Anacortes Refinery (John, 2014)

1.1.5 Fire and Explosion of Oil and Gas Company in California

Next, an accident happened at Richmond, California in Chevron Oil and Refining on 6 August, 2012 at 6.38 p.m. According to the final investigation report, “a carbon steel pipe within the refinery’s crude oil processing unit failed due to thinning caused by a chemical process known as sulfidic corrosion. The corrosion occurred in a pipe component approximately five feet in length that contained lower levels of silicon than other portions of the pipe.”

Because of high temperature from 450 °F to 1000 °F and no inspection or repairing the carbon steel, causes unexpected explosion. So, to avoid such accident to happen again needed a proper selection of material, high corrosion-resistant material, always monitoring and inspection, and lastly make sure the insulation is installed correctly to prevent the corrosion under insulation (CUI).



Figure 0.3: Fire and Explosion at Chevron's Richmond Refinery Burns
(Lisa, 2015)

1.1.6 Methods to Prevent Corrosion

So, in order to prevent the corrosion there are five methods to solve such as material selection, coatings, inhibitors, cathodic protection and design. Besides that, advantage of corrosion control are longevity (cost expenditures), reduced corrective maintenance (reduce service cost and time), reduce requirement of maintenance, lower risk of failure and fatality and develop new and expanded markets (ASM, 2000).

One of the corrosion method that being used for this project is coatings. Coatings especially in organic coatings is used to evaluate the corrosion protection on carbon steel. A good coating must include proper criteria such as good adhesion towards metal,

property for a long time, ability to resist (electric current occurs), and sufficient atmospheric resistance, withstand freezing temperature and resist solar radiation (Chesnokov et al., 1967).

1.2 Motivation and problem statement

Today, paint coatings are useful for industries, construction, environment and society. This is because in industry, a lot of problem will occur if corrosion is infected. A possibility for plant shutdown or failure because of corrosion will have expensive cost to inspection and repair. Besides that, corrosion is hazardous to society health through breathing, touching and consumed. Through environment, the effect of corrosion will damage building, structures, cracking and fatigue process. Thus, this significant for this study is to evaluate the paint coating towards corrosion protection that can reduce the cost of maintenance and inspection and lower the health problem. The corrosion can be controlled by proper material selection, coating, inhibitors, cathodic protection and design (Patil & Ghanendra, 2013). So this project will evaluate the effectiveness of organic coating to prevent the metallic structure such as carbon steel to become corrode. Choosing the suitable coatings will help to control the rate of corrosion become lower. A lower water uptake resistance corrosion is important in which coatings represent a good quality to be used.

1.3 Objectives

The objective of this study is:

- 1) To understanding the physical appearances and behaviour of organic coating using SEM by investigate the electrochemical reaction and environment nature.

1.4 Scope of this research

A few scopes are identified in order to achieve the objectives. They are:

- 1) Preparation of three sample which are epoxy, zinc oxide and zinc spray paint for single, triple and fifth layers coating.
- 2) Cross-cut tape test will be used to study the quality of adhesion by using cross-cut kits
- 3) The thickness will be measured by weighing balance before and after.
- 4) The samples are placed in hot area, using thermostat to measure the temperature surrounding and refer to Malaysian Meteorology, Kuantan for temperature and humidity.
- 5) In immersion test using 3.5 % NaCl aqueous solution, open to air and held at temperature room (25 °C) for 60 days.
- 6) Studying the internal behaviour of coating, cracking or failure because of corrosion using SEM analysis for morphology observation.

The logo for UIMP (Universiti Malaysia Perlis) is a large, downward-pointing arrow shape. It is composed of four triangular sections meeting at a central point. The top-left and bottom-right sections are light blue, the top-right and bottom-left sections are a darker blue, and the central point is yellow. The letters 'UIMP' are written in white, bold, sans-serif font across the bottom of the arrow.

UIMP

CHAPTER 2

LITERATURE REVIEW

2.1 Overview

In this chapter, previous studies done by other researchers on the paint coating will be reviewed. In industry, paint coating is widely used to protect the metals against corrosion. Furthermore, in this study the paint coating used will be determined by a few methods such as physical test include adhesion test, electric resistance measurement, immersion test, thickness and environment and scanning electron microscopy analysis for morphology observation.

2.2 What is paint

Based on International Standard ISO 4618-1, paint is defined as a pigmented of coating material in liquid or powder form applied to a substrate which form opaque layer of protective and decorative surface. The pigment, binder and thinner should be measure in right proportions to give final finished product is smooth and attractive.

Next, using solvent or binders used to stick as an adhesive for pigment particles. Then, the chemical properties in solid or fluids coating will be modified by additive (Waldie, 1974). So, the flow of process organic coating is the pigment will mixing or binding with solvent as a medium and modify of presenting pigment by right proportion and nature (Mathiazhagan et al., 2011).

2.2.1 Binder

Binders is a drying process for curing which can accelerated by addition of natural or synthetic film forming substances. Natural compound can be classified into

two which are micro and macromolecule. While, synthetic film can be classified into two which are poly-condensation and poly-addition oligomers and chain polymers.

2.2.1.1 Natural Binder

Natural compounds have been used to improve the quality of properties of coating from batch to batch oil and involved in increasing molecular weight of oil by control the oxidation. There are two molecules for natural binder as shown in Table 2.1 below:

Table 0.1: Advantage and Disadvantage of Natural Binder of Micro and Macro molecule of film substances (Karlsson, 2011)

Micro molecule	Macro molecule
<p>Drying oil</p> <ul style="list-style-type: none"> • Glycerol ether (fatty acid) • Unsaturated • Seed and fruits (sunflower) • Advantage : ability to dry in air 	<p>Cellulose derivative</p> <ul style="list-style-type: none"> • Advantage : durability • Disadvantage : slow drying and toxic
<p>Bitumen</p> <ul style="list-style-type: none"> • Oxidation and polymerisation • Advantage : waterproofing • Disadvantage : oxidation (aging) 	<p>Natural rubber</p> <ul style="list-style-type: none"> • Advantages: non-flammable, resistance to acid and alkali, smooth and good adhesion.
<p>Fossil resin</p>	<p>Casein</p>

<ul style="list-style-type: none"> • High hardness • High melting point (> 300 °C) • Low solubility 	<ul style="list-style-type: none"> • Construction and colouring • Advantage : fast drying and durable • Disadvantage : form mold
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2.2.1.2 Synthetic Binder

Synthetic binder have two substances which are poly-condensation and poly-addition oligomers and chain polymers as shown in Table 2.2 below:

Table 0.2: List of Substances for Synthetic Binder (Karlsson, 2011)

Poly-condensation and poly-addition oligomers		Chain polymers
Alkyd- resin	Polyamides	Polyolefin
Polyester	Polyurethane	Polyvinyl chloride
Epoxy resin	Silicon	Polyvinyl acetate
Phenolic resin	Amino acid	Acrylic polymer

2.2.2 Pigment

Pigment can be classified by two types of coating which are organic and inorganic. The function of pigment in paint system are providing colour, opacity and smooth. Organic pigment are usually brighter, stronger, more transparent and more

stable. Besides, inorganic pigments have white and black in colour and cheaper than organic pigment.

2.3 Organic and Inorganic Coatings

Coatings can be divided into two groups, metallic and non-metallic or organic and inorganic. Metallic coatings lead an irreversible disintegration of the material under formation of oxides, hydroxides and salts by induced the chemical reactions between metal surface and environment. The corrosive resistance of the noble metal takes an advantage on active metal when applied the concept of more noble metal coating (ASM, 2000).

Furthermore, coatings are composed of primer and topcoat usually applied as multi-layered systems the overall performances of multi-coat system plays an important role for interactions among different layers (Hegedus, 2004). In addition, specific parts of a coating system have different properties of coatings (Verkholanstev, 2003). The classification of organic coatings is functional coatings (Mathiazhagan et al., 2011).

Organic coating is used as a medium to prevent the metallic structure to lower the corrosion rate. The function is to isolate the metal from corrosive environment (Mak, n.d.). There are three organic coating component are pigment, additive and solvent. Pigment is a substances that gives particular colour when it is present in it or added to it. In 1918, the white pigment replaced the white lead using titanium dioxide compounds (Historical Background, n.d.).

Inorganic coatings function as corrosion protective barrier for ceramic coatings. The corrosion resistance ceramics include glass coatings, chemical-setting silicate cement lining, porcelain enamels and etc. (ASM, 2000). The organic coating was selected in this project research because metals will be used as samples preparation. The difference of organic coatings and inorganic coatings shown in Table 2.3 below.

Table 0.3: Differences of Organic and Inorganic coating (Eddy, 2013)

	Organic	Inorganic
Binder	Epoxy	Silicate
Performances : sea coast marine with blast cleaning	Organic zinc with high build epoxy Life : 13.5 years	Inorganic with high-build epoxy Life : 15 years
Abrasion	Low	High
Solvents	Low	High
Sunlight	Low	High
Top coats	Easier (denser surface)	No over coating

2.3.1 Advantages of organic coating

Organic coatings is widely used in order to protect the metal against corrosion. The key feature of organic coatings is to evaluate the effectiveness of coatings towards corrosion protection. There are four samples used in this experiment are epoxy resin,

epoxy amine, polyurethane and acrylic-based paint. The advantage of organic coatings compared to the other coatings are:

- i. The hydrophobic organic film only water can slightly penetrated.
- ii. Good corrosion resistances and waterproof to water or air.
- iii. Consistent quality of organic coated steel.
- iv. Smooth appearance and variety of metallic colour.
- v. Well-known in domestic appliance market and successfully used for white goods such as refrigerator, washing machines, small kitchen appliances (microwave oven)
- vi. Production of different surface such as smooth, orange peel, grained, textured or embossed in flexibility of the coil coating process.
- vii. Organic coated steel reduces company taxes (waste treatment eliminate) and insurance premium.
- viii. By painting the metallic subtract will enhance the protective ability of steel.
- ix. Powder thermoset coatings have zero volatile organic content (VOC) thus produce eco-friendly coatings.
- x. Organic coated steel can be recycled at the end of life cycle likes the other steel products.
- xi. Human and environment does not involved with danger in various sectors such as construction, domestic appliances and general industry when using the organic coated steel because new transformation have been develop to lower organic- solvent content.

2.3.2 Zinc rich epoxy paint immersed in 3.5 % NaCl

In 2011, zinc rich epoxy paint (ZRP) in 3.5 % NaCl solution have been studied to identify the protective behaviour mechanism for single coat solvent – based using EIS about 100 kHz to 10 mHz. The result obtained the barrier nature of ZRP films reinforced by zinc was still protected the substrate steel even the cathodic protection effect by Zn dust become weak (Hammouda et al., 2011). The increasing barrier

resistance because the zinc corrosion product precipitate inside coating by blocking the pores around the zinc particles originated (Abreu et al., 1996).

The immersion test is used to test the effectiveness of corrosion protection towards metals. Besides, zinc particles provide a cathodic protection of steel substrate at least beginning immersion for solvent-based (ZRP) (Hare, 1998). The barrier effect of the paint was reinforced by formation of zinc rich product in long term protection (Morcillo et al., 1990). A high pigment concentration required above 60 % by volume in solvent-based (ZRP) to ensure the good electrical contacts between zinc pigment and steel substrate (Vilche et al., 2002).

Figure 2.1 below shows the structure formula was under conductive form for ZRP using FTIR spectral characterization (Hammouda et al., 2011).

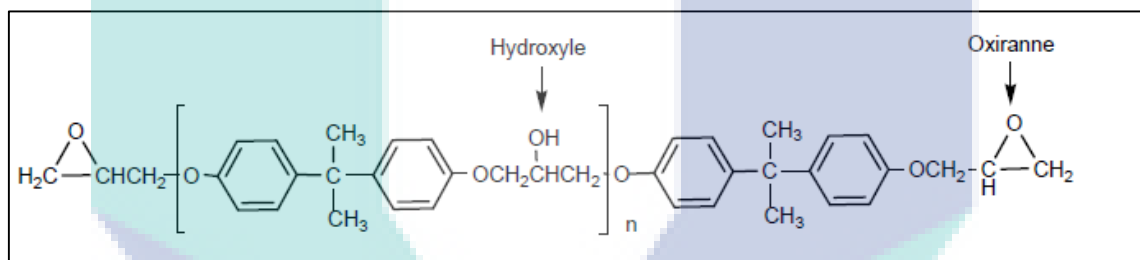
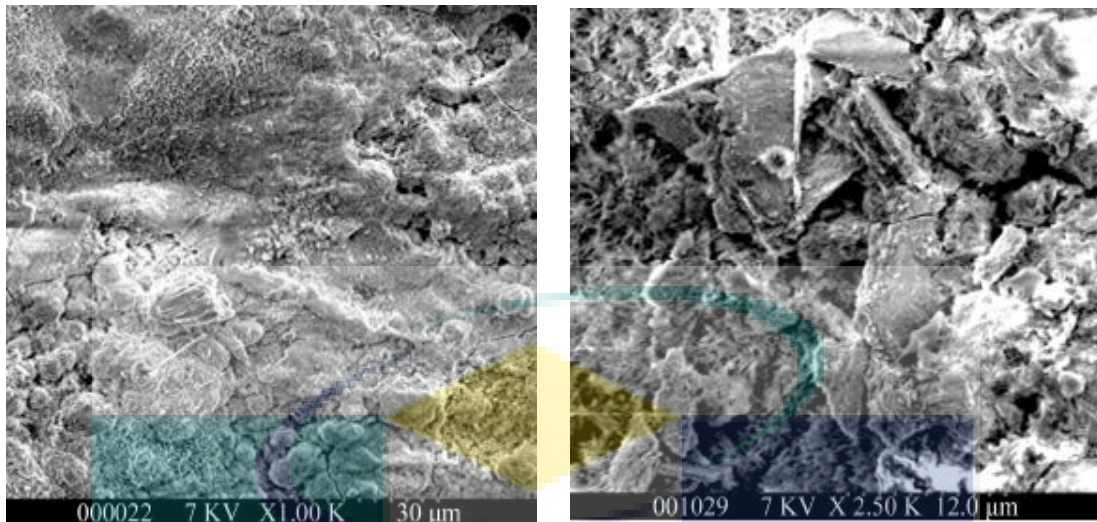


Figure 0.1: Structural formula of zinc rich epoxy paint from FTIR spectra result (Hammouda et al., 2011)

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a

b

Figure 0.2 : SEM image of zinc rich epoxy (Hammouda et al, 2011)

Figure 2.2, a and b shows the SEM image of zinc rich epoxy paint after immersed in 3.5 % NaCl solution with oxidation product. The “white corrosion” appear when involved oxidation of zinc coating with NaCl solution (Kalendova, 2000). The shape of spherical zinc - pigmented coating must be sealed to secure the substrate protection barrier (Hammouda et al., 2011).

Furthermore, figure 2.3 below is white rust occurs on galvanized steel. When the pH is higher with high conductivity water, the zinc surface will attacked by white rust (white Rust, n.d.).



Figure 0.3 : White rust on galvanized steel

2.4 Coating types and systems coating composition

Organic coatings is made up of three components pigment, additive and solvent (Mathiazhagan et al., 2011). There are many coating types such as enamel, lacquers, varnish, paints, and dispersion and emulsion coatings (Khanna, 2008). Furthermore, organic coating composed of single or more layer and classified as primers, intermediate coat and finish coats. In industries, common standard used in paint system is higher film thickness with less coat for environment legislation and improved the durability performances (Paint Coatings, n.d.).

2.4.1 Primers

Primers are coatings that applied before coat for metal substrate that designed to improve the overall adhesion and prepare metal surface to accept the coat. In order to use primers coat, the substrate need to be clean and dry before coat. In addition, industry used primers for long-term protection. Moreover, carbon steel surface must be clean before directly applied primers coat. The purpose is to identify the quality of adhesion. There are two types of primers are pigmented with metallic elements anodic to steel and high adhesion and chemical-resistance properties of the binders (Khanna, 2008).

2.4.1.1 Advantages of primers

The advantages of using primers as first coat before apply main coat are:

- 1) Primers can provide corrosion-resistant barrier when applied on metal subtract.
- 2) Improving the wetting properties of the surfaces for partially cleaned surface, primer is essential to be used for cover the cracking or damage metal subtract.
- 3) Zinc-based primers and epoxy primers are most commonly used to carbon steel that provide the corrosion resistance

2.4.1.2 Primers pigmented with metallic elements anodic and to steel

Zinc-rich primers that shown in figure 2.4 below is used in this types of primers pigmented when anodic metal sacrificially corrode for metal subtract that have damage or exposed. The corrosion will occurs after anodic metal is fully exhausted.



UMP



Figure 0.4: Organic Zinc Rich Primer (Benjamin, 2014)

2.4.1.3 Primers pigmented on high adhesion and chemical-resistance properties of the binder

The effectiveness of high quality of adhesion using two-pack epoxy primers is sufficient to prevent damage of corrosion in coating. The corrosion process will interfere if active inhibitive pigments is used in primers. Figure 2.5 below shows Dulux Duremax @ GPE ZP which contain active inhibitive protection with formulation of zinc phosphate towards steel against corrosion. In addition, the advantage of epoxy resin in Duremax paint are high strength adhesion, excellent cohesion, tightly binding pigment, and protection of passive barrier.

UMP



Figure 0.5: Dulux Duremax @ GPE Zinc Phosphate (Dulux, 2013)

2.4.2 Intermediate and top coats

After primers have finish coated, organic paints coating will be used as intermediate and top coats. Different types of paint coatings will be chosen based on environment, technique and climate condition. There are two classification of intermediate and top coatings are convectional coatings and advance coatings (Khanna, 2008). Table 2.4 below show an examples of both intermediate coatings.

Table 0.4: Examples Convectional and Advance coatings (Khanna, 2008)

Convectional coatings	Advanced coating
Alkyd	Solvent less coatings
Epoxy	Glass flake-modified coatings
Polyurethane	Poly-siloxane coatings

Chlorinated carbon	Poly-urea
--------------------	-----------

2.4.3 Epoxy resin

Epoxy is an organic molecule consists of oxirane groups. Oxirane groups is an oxygen atom bonded with two adjacent carbon atoms in roughly a triangular form. However, oxygen in this component is highly reactive than other organic groups. Next, epoxy also known as glycidol ether because the internal reaction is link to the rest of organic molecule through an ether linkage (Mattson, 2013).

Besides that, epoxy resin is a polymer and containing two or more oxirane groups with organic molecule. In order to make epoxy resin, Bisphenyl A must react with diglycidol ether. Bisphenyl A is made from reaction of phenol and acetone to form di or bis which means two phenol (Mattson, 2013).

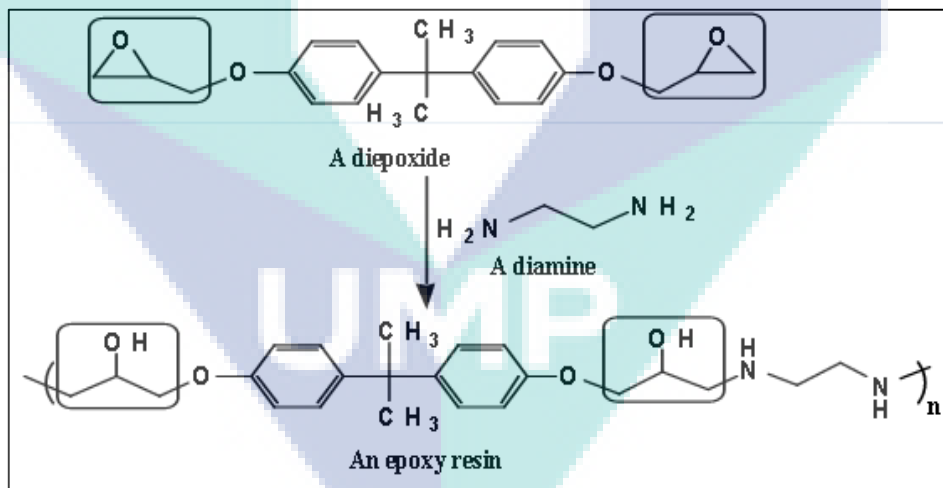
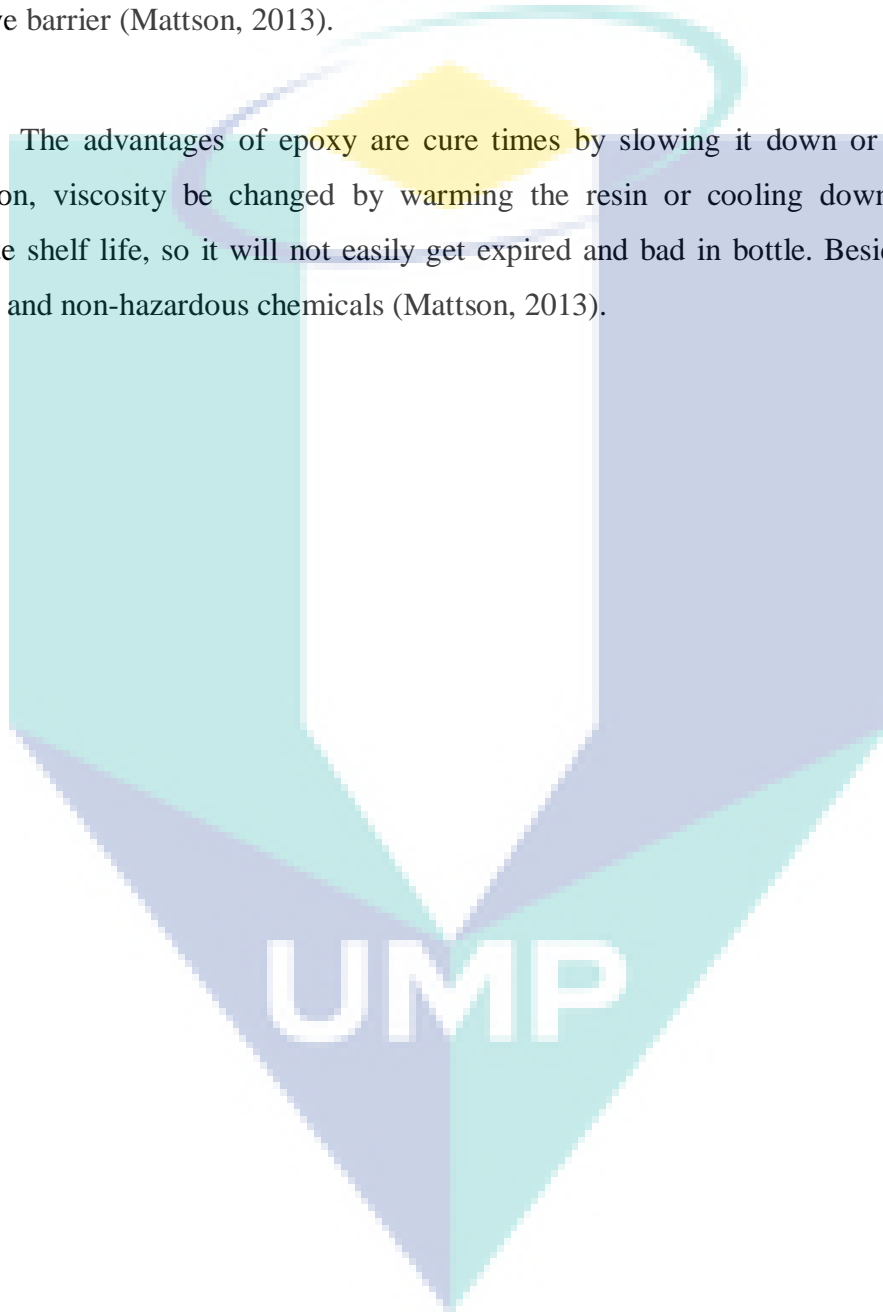


Figure 0.6: Epoxy Resin Formation

Epoxy resin is widely used in industry for protective barrier against corrosion and cure by internal generated heat and thermosetting resin. Resin and hardener are parts of epoxy systems. Furthermore, the cross linkage between polymer and hardener plays an important role to make sure become solid. The advantage of epoxy resin are strength adhesion, excellent cohesion, tightly binding pigment and good protection of passive barrier (Mattson, 2013).

The advantages of epoxy are cure times by slowing it down or speed up. In addition, viscosity be changed by warming the resin or cooling down. Epoxy has infinite shelf life, so it will not easily get expired and bad in bottle. Besides, it has no odour and non-hazardous chemicals (Mattson, 2013).



CHAPTER 3

MATERIALS & METHODOLOGY

3.1 Introduction

The chemical used in this project is to evaluate the effectiveness of paint coatings based on physical appearances and internal behaviour were epoxy, zinc oxide, spray paint, alcohols, and sodium chloride by compared paint coating with market products. Epoxy, zinc rich spray paint and acetone were not available in lab. The only available in lab UMP were ethanol, detergent, sodium chloride. There were three methods to identify the effectiveness of organic coating by mechanical testing including adhesion test, immersion - test, temperature and thickness. Lastly, the sample coated carbon steel were analysed or interpret by using scanning electron microscope (SEM) for morphology observation.

3.2 Materials

The chemical used were epoxy resin and hardener, zinc oxide powder, zinc galvanizing primer spray paint , spray paint (zinc primer), ethanol, acetone, detergent, and sodium chloride (figure a). Epoxy and hardener (slow setting epoxy adhesive), spray paint and acetone were obtained from ACE HARDWARE MID VALLEY MEGAMALL, Kuala Lumpur. Zinc oxide reagent plus powder (99.9%), sigma brand were obtained from VNK SUPPLY & SERVICES, TMN SIERRA PERDANA MASAI JOHOR. Meanwhile, chemical that obtained from lab were epoxy (fast cure kit), detergent, ethanol (99.8 % purity) and sodium chloride (99 % purity). Besides, sand paper used (#180, #220, #1000 and #1500) was obtained from (ACE HARDWARE MID VALLEY MEGAMALL, Kuala Lumpur.

3.2.1 Materials

- I. Epoxy Resin
- II. Epoxy Hardener
- III. Zinc oxide
- IV. Spray paint
- V. Ethanol
- VI. Detergent
- VII. Acetone
- VIII. Grit sand paper (#180,#220,#1000, #1500)
- IX. Carbon steel
- X. Sodium chloride (NaCl powder)
- XI. Tapes
- XII. Sealer bag (8" x 5" x 0.05 mm)
- XIII. Aluminium foil
- XIV. Gloves

3.2.2 Apparatus

- I. Brush
- II. Spatula
- III. Beaker (#50ml,#100ml, #25 ml, #1000ml)
- IV. Knife
- V. Cellotape
- VI. Close container
- VII. Jug (500 ml)
- VIII. Box
- IX. Thermostat
- X. Scanning electron microscope (SEM image)



Figure 3.1: Epoxy and Hardener, Slow setting epoxy adhesive



Figure 3.2 : Fast Cure Epoxy Kit



Figure 3.3: Zinc Oxide Reagent Plus powder



Figure 3.4: Spray Paint

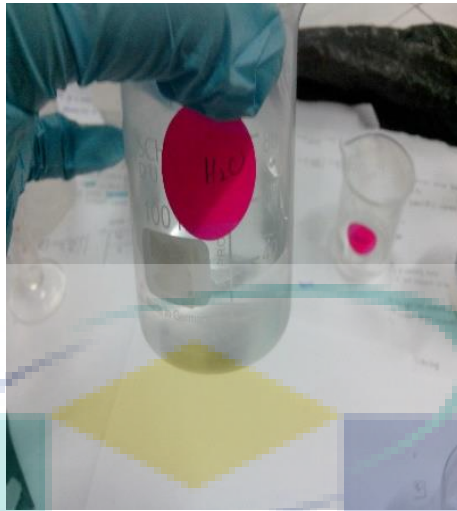


Figure 3.5: Water (H_2O)



Figure 3.6 : Ethanol

UMP



Figure 3.7: Plastic cup

UMP

3.3 Overall Methodology

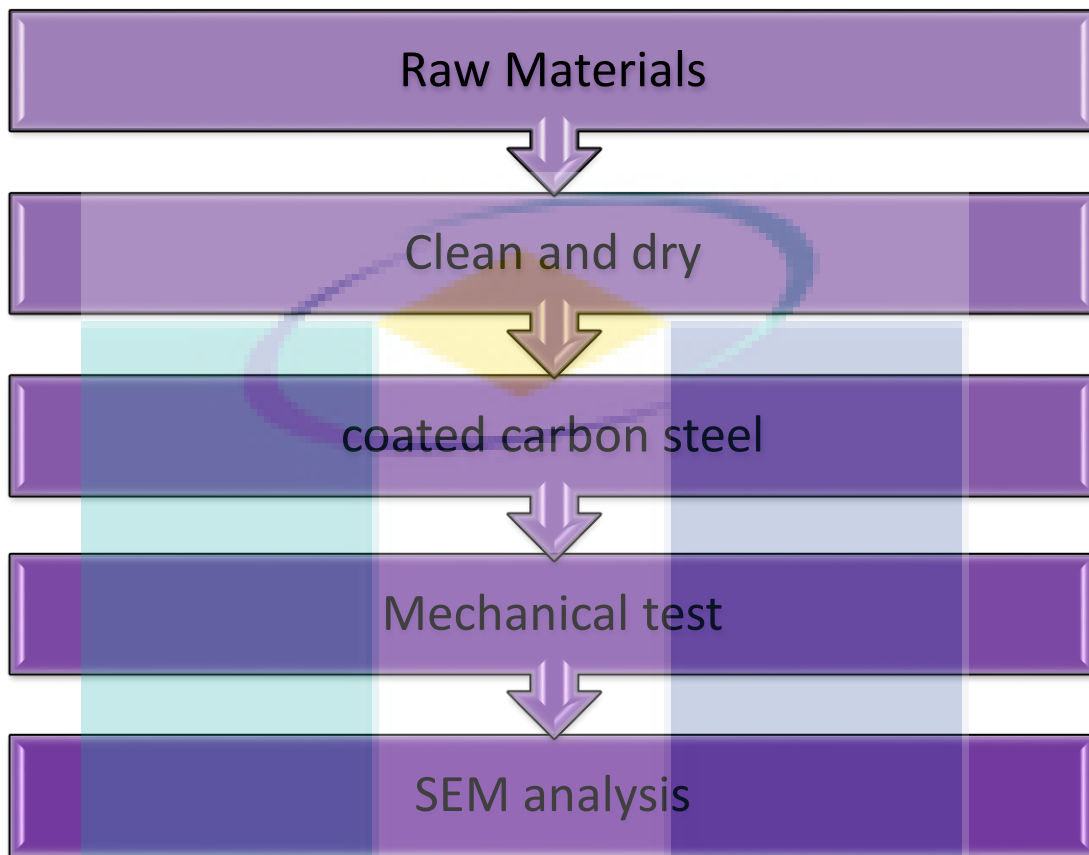


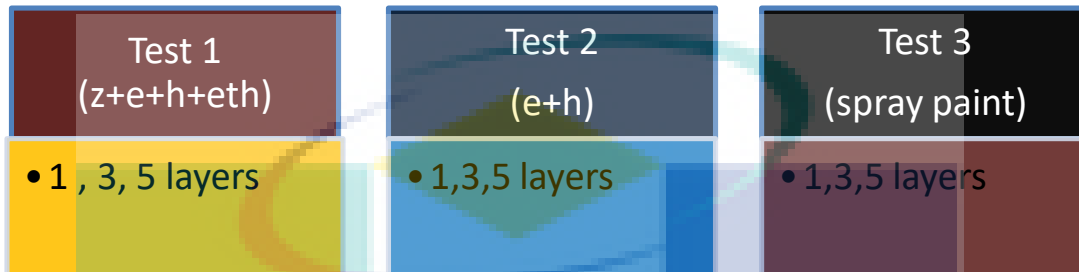
Figure 3.8: Overall flow chart of samples carbon steel coated for organic coatings

3.4 Experimental Methodology

Sample

- Same concentration but different layer
- For immersion test , adhesion test and temperature
- Material – epoxy & hardener (slow setting epoxy adhesive) , epoxy (fast cure epoxy kit), zinc powder , spray paint

- Total sample : 45 (9 samples for immersion , 27 samples for adhesion , 9 sample for temperature)
- *z = zinc powder , * e= epoxy resin , * h = hardener , * eth = ethanol



3.4.1 Processing Raw Materials

Firstly, the sample of carbon steel were prepared with sizes (20 x 20 x 0.2 mm). After that, specimen were place at room temperature (25 °C) cleaned by sand paper with grid (#180, #220, #1000, #1500) on flat surface. Next, preparation of three samples epoxy, zinc oxide and zinc rich spray paint were coated on sample with same concentration using brush but different layer of (single, triple and fifth layers). The samples were leaved about 24 hours before undergo mechanical test.

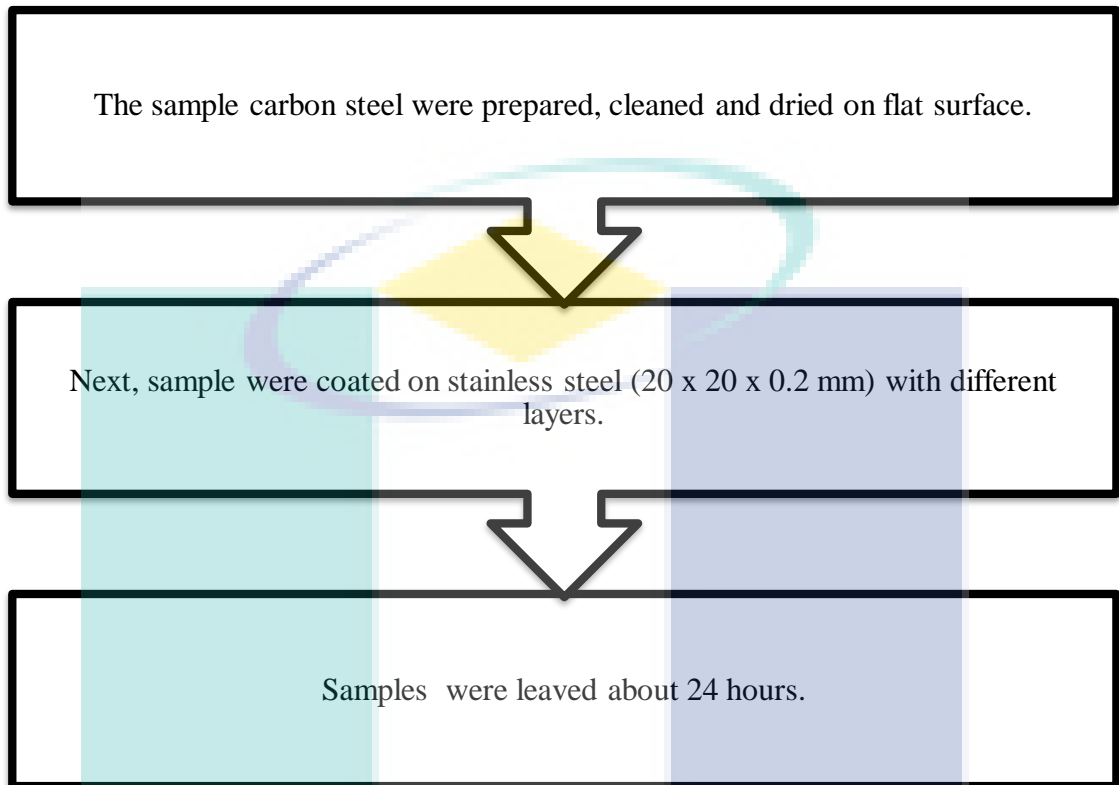


Figure 3.9: Flow chart for processing raw material

3.4.2 Sample preparation

The steel samples were cut and cleaned under the following conditions:

Sizes : 20 x 20 x 0.2 mm

Medium : Distilled water and 5 % ethanol

Temperature : Room temperature (25 °C)

Equipment : Shearing machine, ultrasonic cleanser, oven

Firstly, the sample of carbon steel were cut into many pieces with sizes (20 x 20 x 0.2 mm) using shearing machine equipment. Next, sample were place at room temperature (25 °C) cleaned by sand paper with grid (#180, #220, #1000, #1500) on flat surface with continuously water flowing. At the same times, the sample were dipped into 5 % of ethanol to make rust surface easily removed. Each sample takes three hours to fully clean. After that, the sample were put into ultrasonic cleanser for two minutes (30 °C) and baked into oven with temperature 60 °C in one hour.

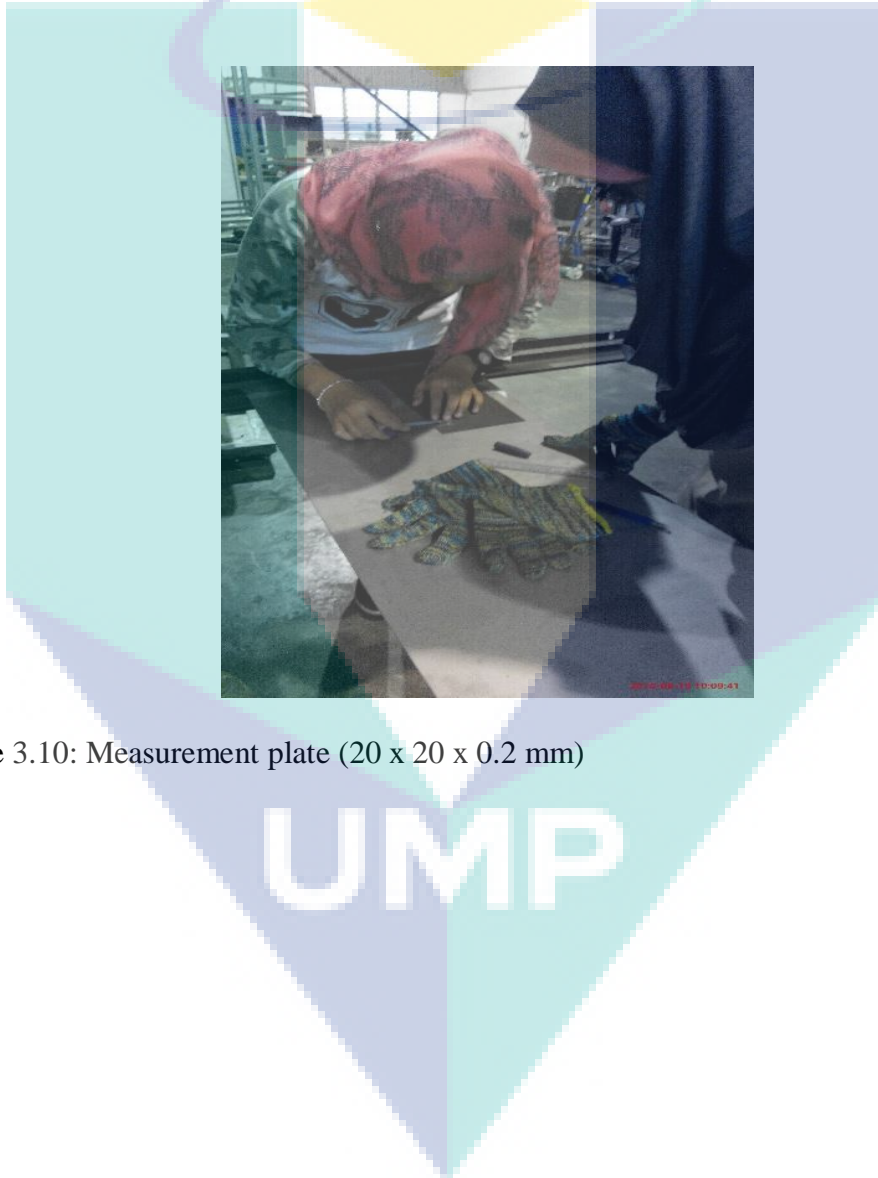


Figure 3.10: Measurement plate (20 x 20 x 0.2 mm)



Figure 3.11: Training session using Shearing Machine by Mr. Abd Razak Bin Abd Hamid



Figure 3.12: Cut plate with carefully handle

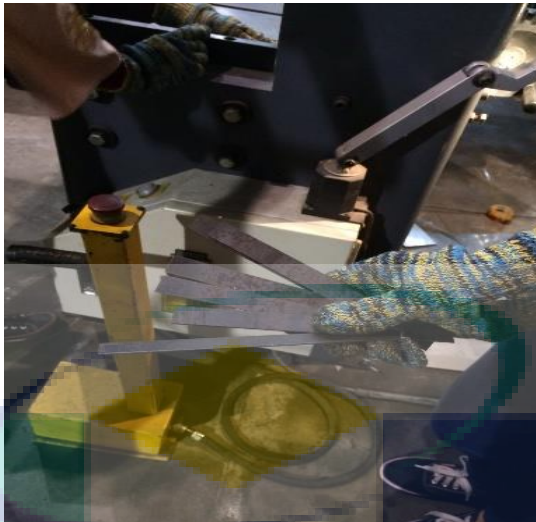


Figure 3.13: Cut plate into many pieces



Figure 3.14: Sample for Experiment

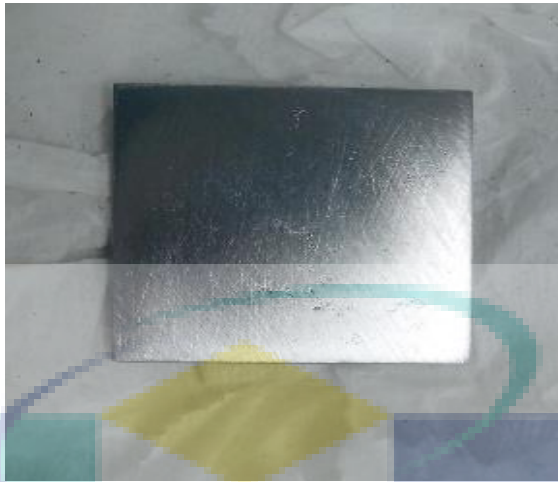


Figure 3.15: Sample after cleaned with sand paper (#180, #220, #1000, #1500)



Figure 3.16: Ultrasonic Cleaner (2 min, $T= 30\text{ }^{\circ}\text{C}$)



Figure 3.17: Dried into Oven 60 °C, 1hr



Figure 3.18: 9 sample fully cleaned and ready for next phase

3.4.3 Preparation of Coated Sample

Firstly, there were three tests to coat samples were carried out before choose the best experimental method for mechanical test. Test A and B were not successful but Test C was successful as shown in Table 3.1.

Table 3.1: Three test were carried out for Test 1 coated samples using Zinc oxide, Epoxy and Ethanol

Test	Test A	Test B	Test C
Sample	1 layer	1 layer	1 layer
Medium	2 % ethanol	1 % ethanol	2 % ethanol
Temperature	25 °C	25 °C	25 °C
Duration	3 days	3 days	2 hours
Successful	Not	Not	Yes

Test A – Not Successful

Chemical : Resin + Hardener + Zinc Powder + ethanol

Epoxy : Fast cure epoxy kit

Techniques : Mixed both resin and hardener with ratio (5:1) and stirred with spatula about 10 minute. Next, added zinc powder (10 g) little by little, so that the solution was not saturated. Mixed well all the solution for 10 minutes. Lastly, added some ethanol about 2 % to make sure the solution was smooth.



Figure 3.19: Testing A



Figure 3.20: Testing A (image from bottom beaker)

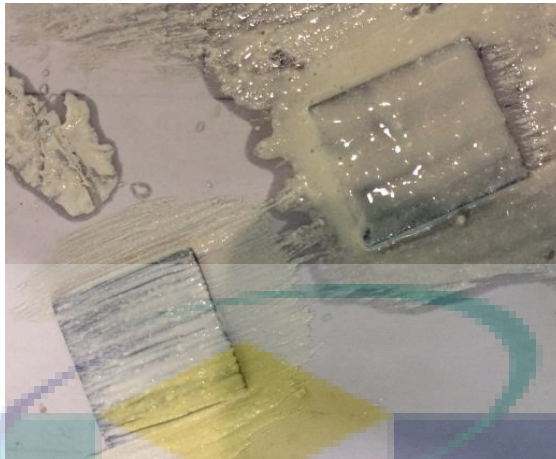


Figure 3.21: Samples was not successful coated

Table 3.2: Physical Morphology from Test A

Physical morphology	1	3	5
Appearance			√
Odour		√	
Colour			√
Temperature			√

*1— not good 3— medium 5— very good

Result : Test A was not successful carried out when used fast cure epoxy kit because the sample was not cure and dried enough even though takes 3 days. Therefore, this fast cure epoxy cannot be used in this project and too much zinc powder caused the solution become saturated.

Test B – Not Successful

Chemical : Resin + Hardener + Zinc Powder + ethanol

Epoxy : Fast cure epoxy kit

Techniques : Mixed both resin and hardener with ratio (5:1) and stirred with spatula about 10 minute. Next, added zinc powder (5g) little by little, so that the solution was not saturated. Mixed well all the solution for 10 minutes. Lastly, added some ethanol about 1% to make sure the solution was smooth.

The logo for UWP (Universiti Wawasan Putrajaya) is a large, downward-pointing arrow shape. It is composed of several overlapping geometric shapes in shades of teal, light blue, and yellow. The letters 'UWP' are written in a bold, white, sans-serif font across the bottom of the arrow.

UWP

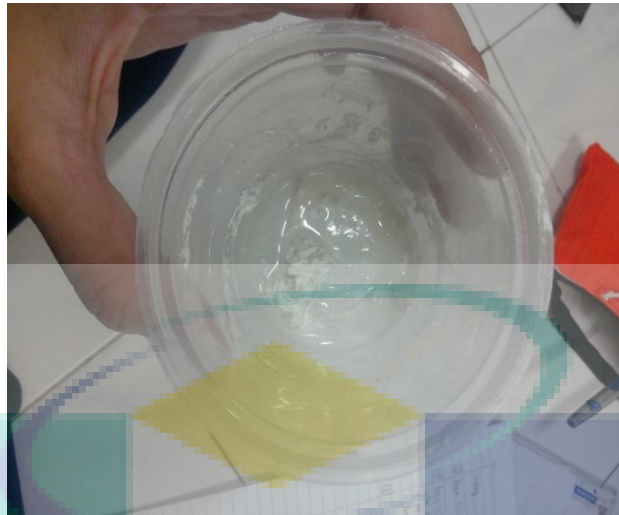


Figure 3.22: Testing B

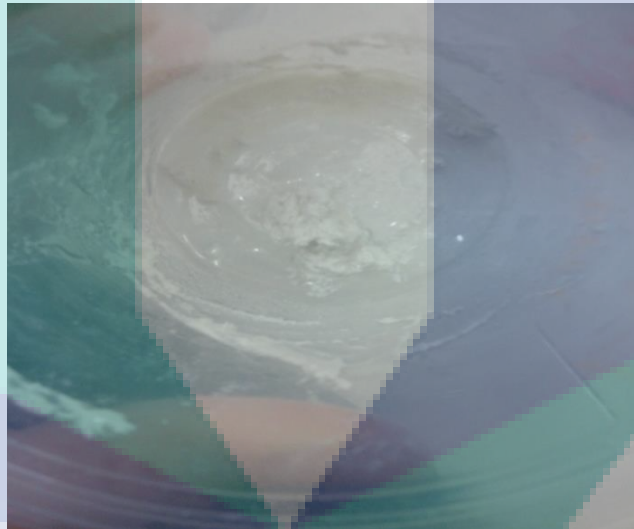


Figure 3.23: Testing B (closure look inside plastic cup)

Table 3.3: Physical Morphology for Test B

Physical morphology	1	3	5

Appearance		√	
Odour	√		
Colour		√	
Temperature (hot)			√

*1— not good 3— medium 5— very good

Result : Test B was unsuccessful carried out when used fast cure epoxy kit because the sample is not dried and cure enough even though takes 3 days. Therefore, this fast cure epoxy cannot be used in this project and solution become saturated.

Test C – Successful

Chemical : Resin + Hardener + Zinc Powder + ethanol

Epoxy : Slow setting epoxy adhesive

Techniques : Mixed both resin and hardener with ratio (1:1) and stirred with spatula about 10 minute. Next, added zinc powder (5g) little by little, so that the solution was not saturated. Mixed well all the solution for 10 minutes. Lastly, added some ethanol about 2% to make sure the solution was smooth.

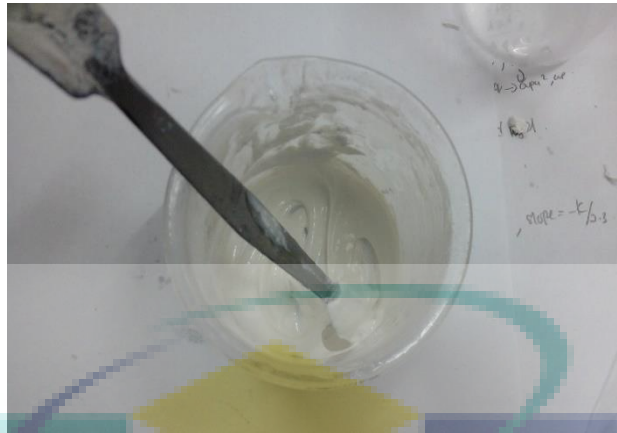


Figure 3.24: Testing C



Figure 3.25: Testing C (closure look image)

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Figure 3.26: Testing C were successful and coated for (1, 3, 5 layers)

Table 3.4: Physical Morphology for Test C

Physical morphology	1	3	5
Appearance			√
Odour		√	
Colour			√
Temperature		√	

*1— not good 3— medium 5— very good

Result :Test C was carried out were successful when used slow setting epoxy adhesive because the sample was cure and dried enough fast in 2 hours. Therefore, this slow setting epoxy adhesive can be used in this project and the solution became smooth.

Next, after choose the best method to coat the samples for Test 1 proceed to make same method for Test 2 which were epoxy coated only.

Epoxy coated sample

Chemical : Resin + Hardener

Epoxy : Slow setting epoxy adhesive

Techniques : Mixed both resin and hardener with ratio (1:1) and stir with spatula about 10 minute. In addition, the solution must stirred in slow motion to prevent formation of bubble.

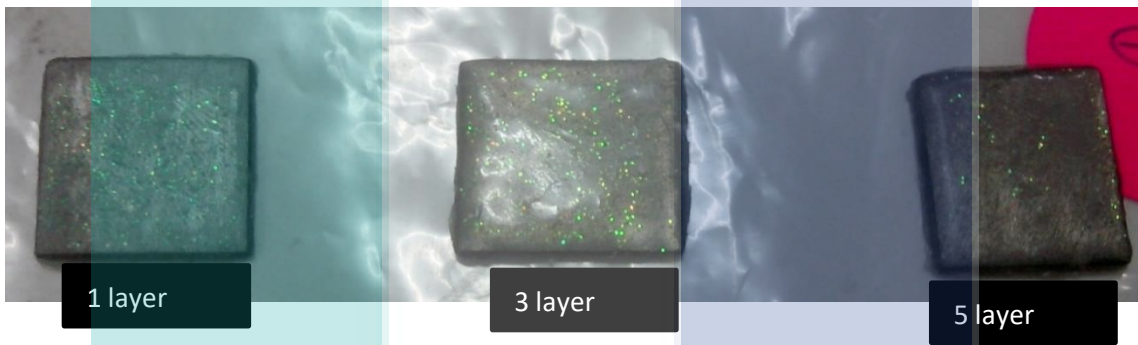


Figure 3.27: Epoxy coated sample for (1, 3, 5 layers)

Table 3.5: Physical Morphology for Epoxy coated sample

Physical morphology	1	3	5
Appearance			√
Odor			√

Colour			√
Temperature		√	

*1— not good 3— medium 5— very good

Result : Test D was carried out successful when used slow setting epoxy adhesive because the sample was cure fast and dried enough even in 2 hours. The solution also was not form bubble. Therefore, this slow setting epoxy adhesive can be used in this project and the solution became smooth.

Lastly, using zinc primer spray paint for Test 3 (one, triple and fifth layers) to compare with this project.

Zinc primer spray paint

Chemical : Zinc primer spray paint

Techniques : Swept horizontally and vertically past the sample. Make sure to use a large plastic or cloth that provide plenty backdrop to spray. Last, set up high place for consistency and it is easy to spray than floor.

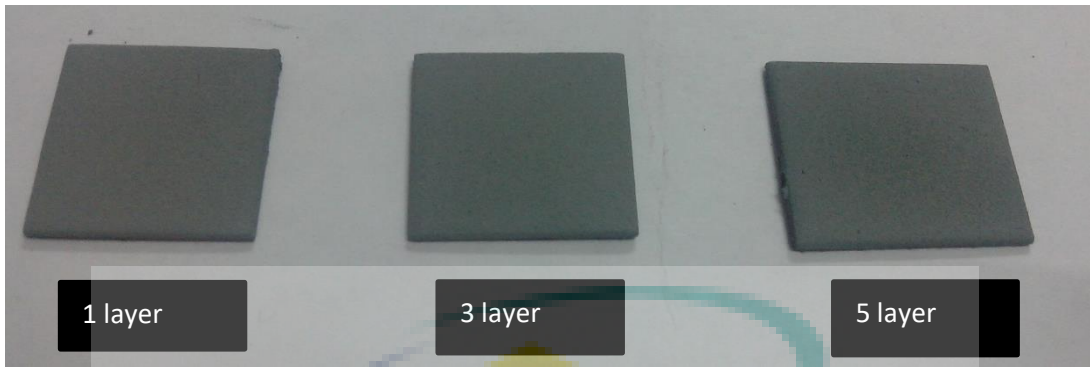


Figure 3.28: Zinc rich spray paint for (1, 3, 5 layers)

Table 3.6: Physical Morphology for Zinc rich spray paint

Physical morphology	1	3	5
Appearance			√
Odor	√		
Colour			√
Temperature		√	

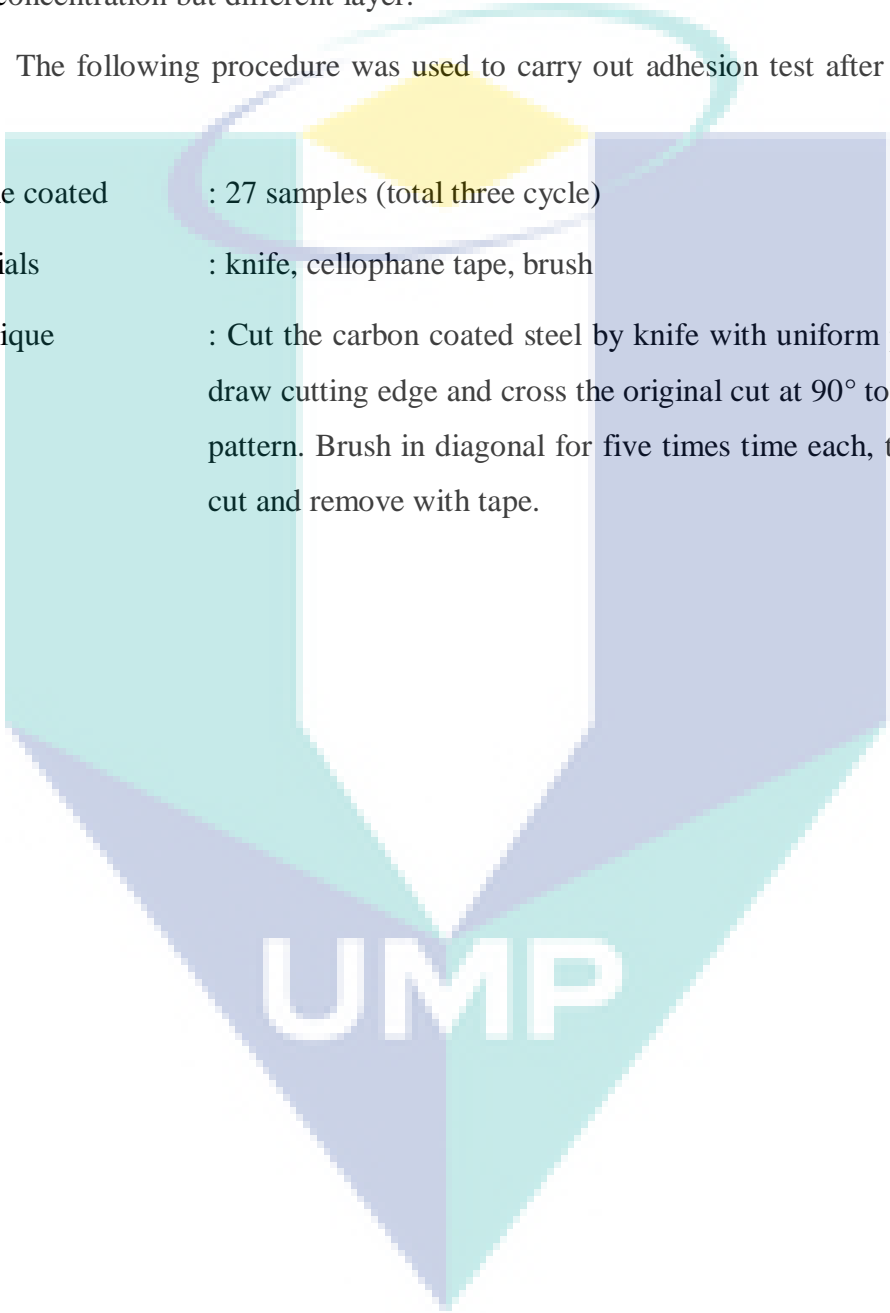
*1— not good 3— medium 5— very good

Result : Using spray paint fast dry is suitable in this research project because the sample was dried enough even in 15 minute. Therefore, this spray paint can be used in this project for comparison purpose.

3.4.4 Adhesion test – ASTM D 3359

The aim was to determine the quality of adhesion on carbon steel coated. Cross-cut tape test was an adhesion test which follow standard ASTM D 3359. Furthermore, there were three test required to repeat for consistency. Each test need 9 samples with same concentration but different layer.

The following procedure was used to carry out adhesion test after leave for 24 hours:



Sample coated	: 27 samples (total three cycle)
Materials	: knife, cellophane tape, brush
Technique	: Cut the carbon coated steel by knife with uniform pressure and draw cutting edge and cross the original cut at 90° to make lattice pattern. Brush in diagonal for five times time each, tape over the cut and remove with tape.

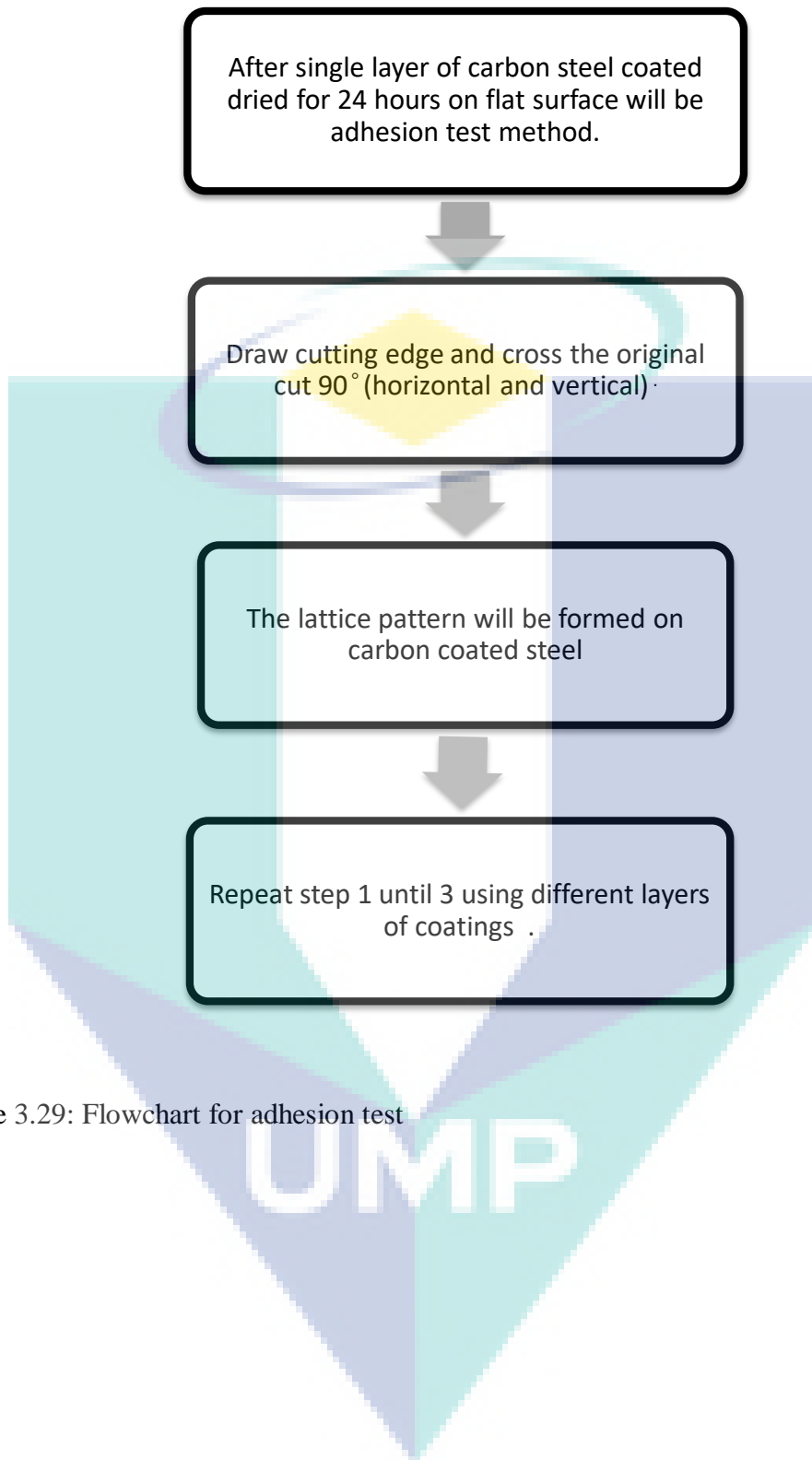
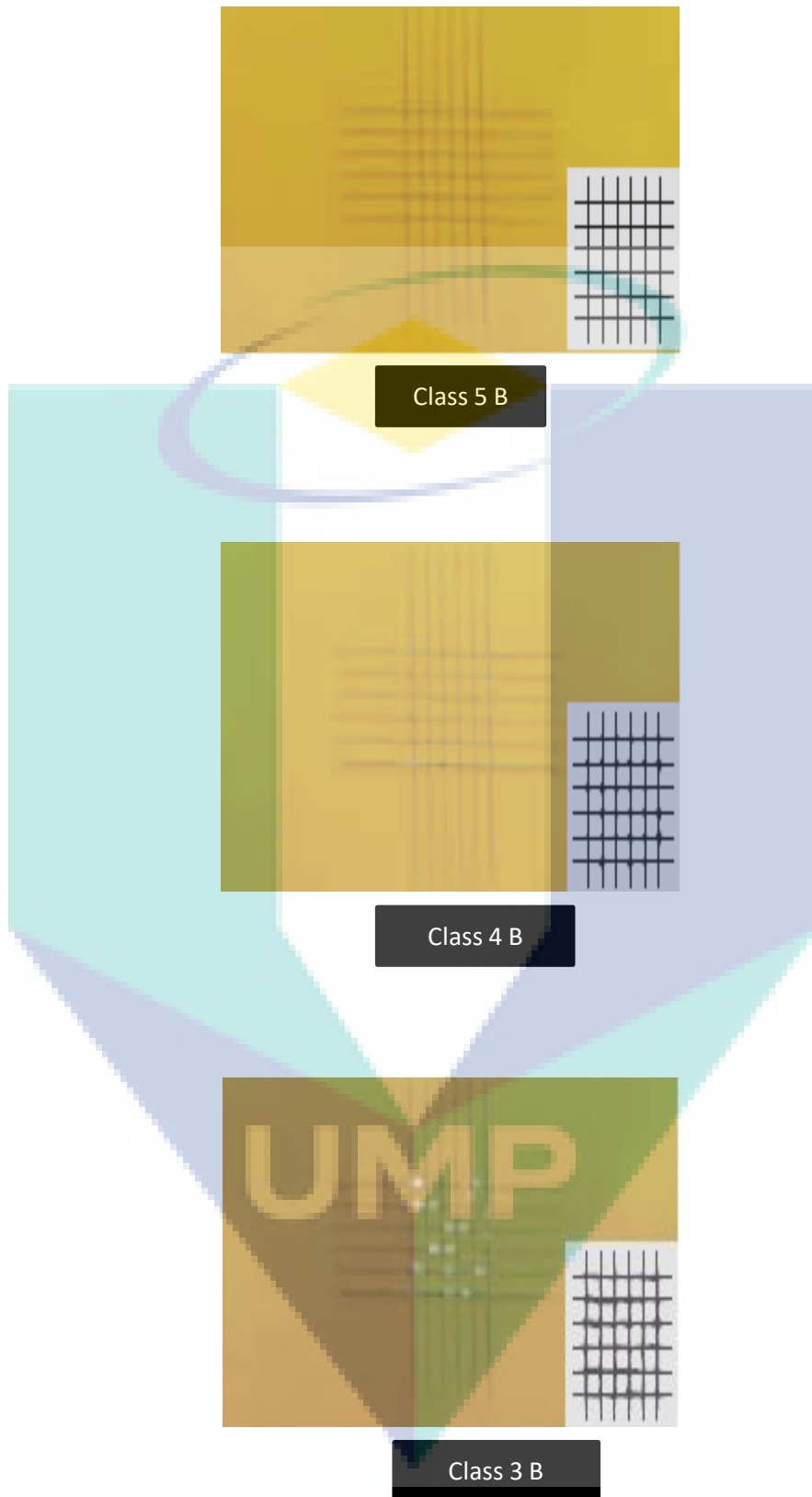
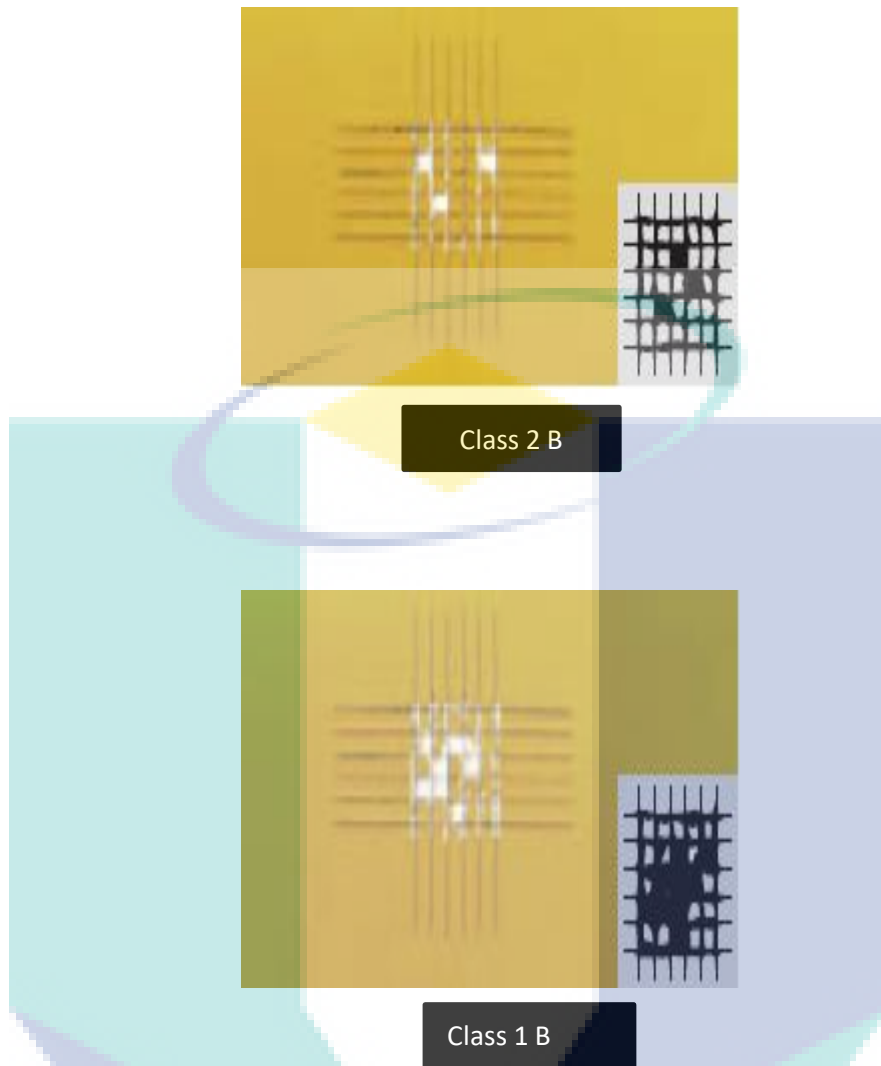


Figure 3.29: Flowchart for adhesion test

STANDARD ASTM D 3599





- **ASTM CLASS 5B** - “edge cut are completely smoothly, none of square lattice is detached.”
- **ASTM CLASS 4B** – “small flakes of the coating at intersection of cuts. Not significantly greater than 5 % is affected.”
- **ASTM CLASS 3B** – “Coating has flakes along edge and intersection. Significantly greater than 5 % but not significantly greater than 15 %.”
- **ASTM CLASS 2B** – “Coating has flakes along edges of cuts, fully in different part of squares. Significantly 15 % but not greater than 35 %.”
- **ASTM CLASS 1B** – “Coating has flaked along edges of cuts, some squares have detached fully and significant.”

A) Epoxy + hardener (1, 3, 5 layers) – cross cut test (lattice pattern)

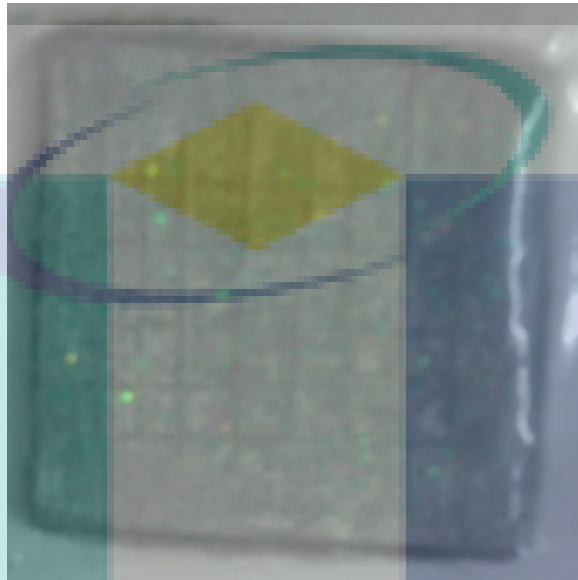


Figure 3.30: Epoxy adhesion test for 1 layer



Figure 3.31: Epoxy adhesion test for 3 layers

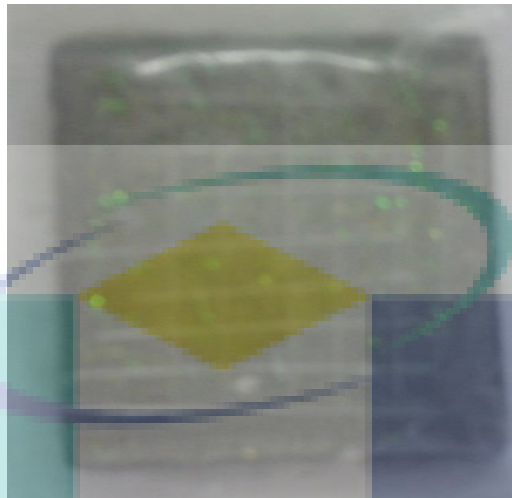


Figure 3.32: Epoxy adhesion test for 5 layers

B) Zinc powder + Epoxy + Hardener + Ethanol



Figure 3.33: Zinc with epoxy adhesion test for 1 layer



Figure 3.34: Zinc with epoxy adhesion test for 3 layers



Figure 3.35: Zinc with epoxy adhesion test for 5 layers

B) Zinc spray paint



Figure 3.36: zinc primer spray paint adhesion test for 1 layer



Figure 3.37: zinc primer spray paint adhesion test for 3 layers

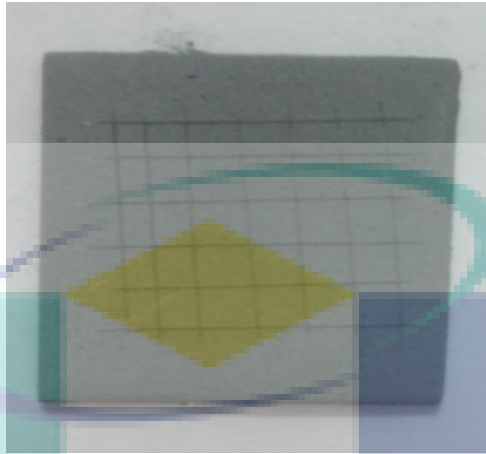


Figure 3.38: zinc primer spray paint adhesion test for 5 layers

3.4.5 Immersion test

After sample coated steel carbon dried for 24 hours, method to test the effectiveness of paint coating was immersion test. The aim was to study the effectiveness of water-based anti-corrosive paint system for protection of carbon steel and weight loss. The procedure were to immersed samples into 3.5 % solution into beaker in open air, held at temperature room for 60 days. The observation was recorded during two months and examine using SEM images (S.J Choi et al, 2014).

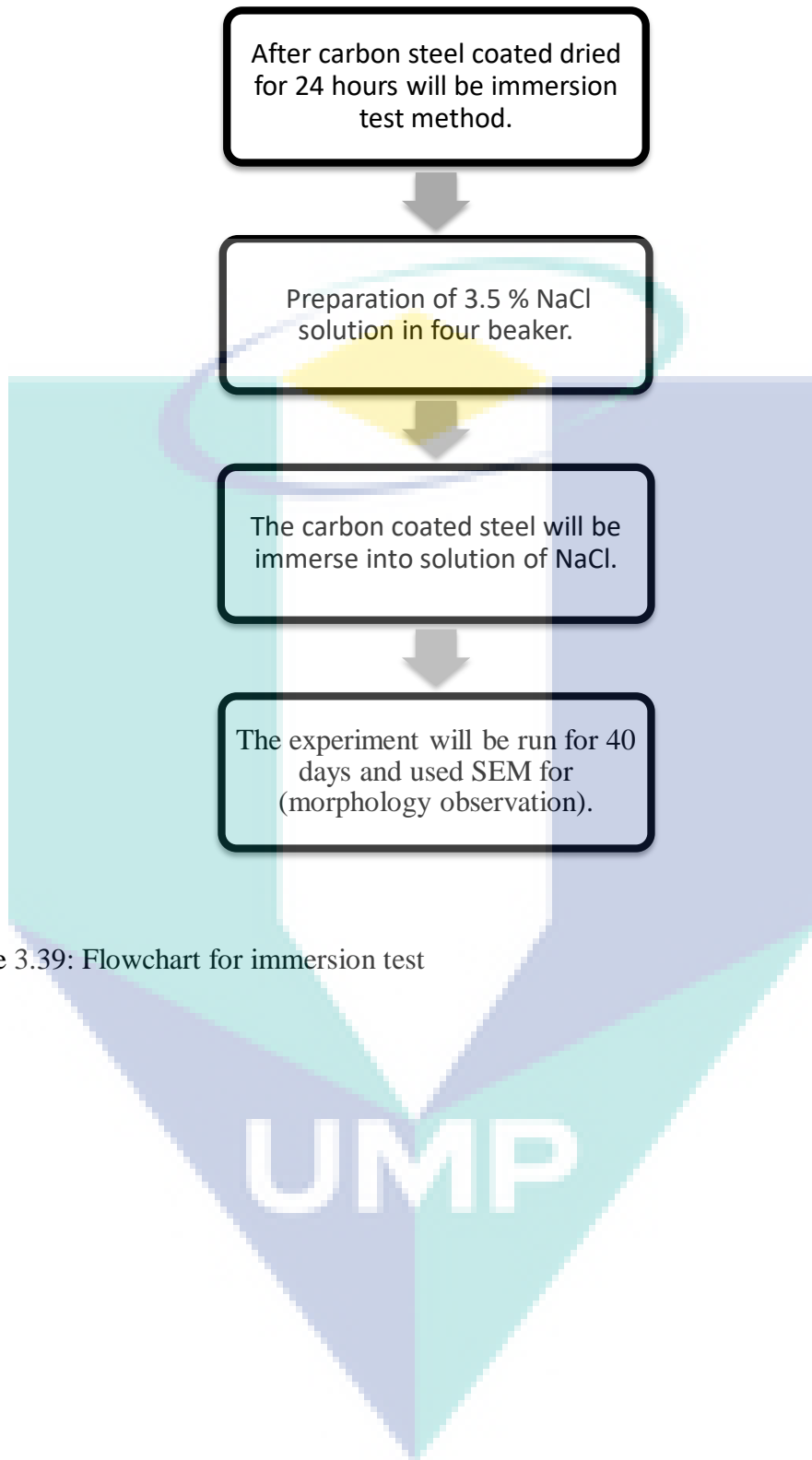


Figure 3.39: Flowchart for immersion test

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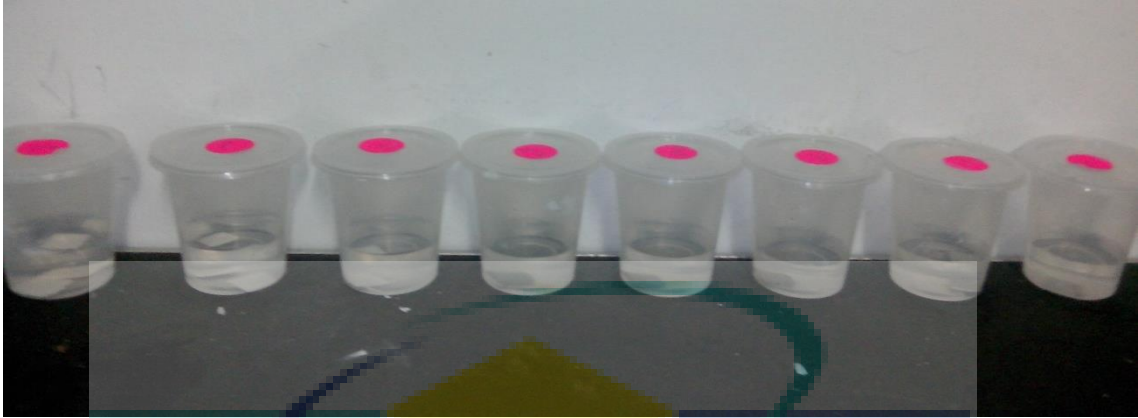


Figure 3.40: Immersion test for 9 sample (1, 3, 5 layers)

3.4.6 Temperature and environment

After sample coated steel dried for 24 hours, temperature and environment are method will be test on paint coatings. The aim is to determine the corrosion rate based on weight loss method influenced of chloride ion, pH, and temperature on corrosion resistance. Four sample will be coat with different layer (one, double and triple layer) are placed on hot area. The experiment is repeated and coated with double layer and triple layer about 2 months. The magnetic thickness and thermostat gauge is used to measure thickness and temperature. The observation and data is recorded. The sample are placed in hot area by taking the daily temperature and humidity from Malaysian Meteorology Department.

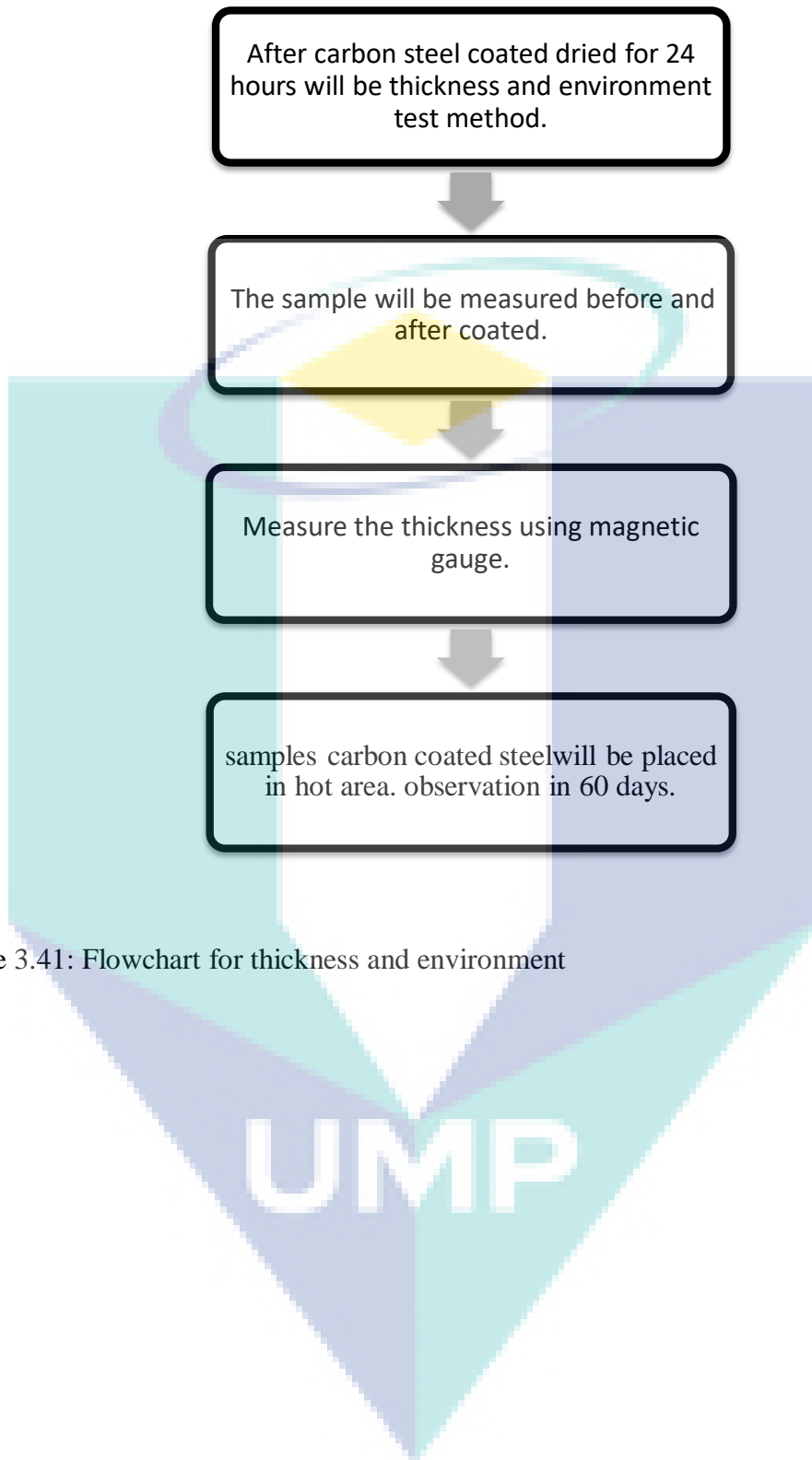


Figure 3.41: Flowchart for thickness and environment

CHAPTER 4

RESULTS & DISCUSSION

4.1 Introduction

This chapter present a comprehensive study to evaluate the efficiency organic coating on coated carbon steel and the effect of using epoxy, zinc oxide and zinc rich spray paint. The expected result discussed was about the physical test include adhesion test, immersion test, temperature, thickness and internal behaviour using SEM analysis for morphology observation.

4.2 Explanation for Unsuccessful Coating Method

There were three method used for coated sample for adhesion test, immersion test, temperature and thickness. The three test were Test A, Test B and Test C. Test A and B method were unsuccessful, while Test C was successful.

The possible causes and solution for epoxy mixture and zinc oxide powder have not cured even after recommendation time passed (West System, 2015):

Causes

- Off ratio – ratio too much or little hardener can affect the cure time
- Low temperature – epoxy mixtures cure slower at low temperature
- Insufficient mixing
- Incorrect product

Solution

- Off ratio – remove epoxy and do not apply any additional non-curing, check the ratio for resin to hardener (5:1 or 3:1).

- Low temperature – applied heat to maintain the chemical reaction and can faster the cure. If use faster hardener, it is designed to cure at low temperature.
- Insufficient mixing – mix resin and hardener thoroughly to avoid hardener rich the areas.
- Incorrect products – resin will not cure properly if mixing with other hardener brand

TEST A

The chemical used in Test A were zinc oxide powder, fast cure epoxy and ethanol. When mixed zinc oxide with epoxy, the solution became saturated as shown in figure 3.25. The ratio for epoxy was 5:1 and 10 g for zinc oxide powder. Therefore, Test A was not successful carried out when used fast cure epoxy kit because the sample was not cure and dried enough even though takes 3 days. Furthermore, even though the ratio for mixed both resin and hardener correct, there were some problem when curing process. This fast cure epoxy cannot be used in this project and too much zinc powder caused the solution become saturated.

In order to interpret this problem, based on four possible caused and solutions if epoxy mixture and zinc oxide powder not cured even after the recommendation time as discussed above, back to the properties and component in fast cure epoxy. Firstly, fast cure epoxy kit obtained from BUEHLER CASTABLE (COLD) MOUNTING COMPOUND (Epo –Kwick Fast Cure Epoxy).

The properties of fast cure kit epoxy were obtained from Bluehler Company:

- Air cure in 45 minutes using SAMP-KUPS
- Good flow characteristics
- Good edge protection and hard mounts
- Transparent
- 185 °F (85 °C) cure temperature

So, coating sample for method Test A cannot be used and unsuccessful, because of when coating the sample operated at room temperature (25 °C), while the temperature

to cure the sample using Bluehler product for fast cure epoxy kit was 85 °C. Therefore, the problem were solved by changing the epoxy product that can operate at room temperature which was epoxy and hardener (slow setting epoxy adhesive). The fast cure epoxy cannot be use when the temperature was lower than even after recommendation time for 3 hours.

TEST B

The chemical used in Test A were zinc oxide powder, fast cure epoxy and ethanol. When mixed zinc oxide with epoxy, the solution became saturated as shown in figure 3.27. The ratio for epoxy was 5:1 and 5 g for zinc oxide powder. Therefore, Test B was not successful carried out when used fast cure epoxy kit because the sample was not cure and dried enough even though takes 3 days. There were some problem during curing process.

In order to interpret this problem, based on four possible caused and solutions if epoxy mixture and zinc oxide powder not cured even after the recommendation time as discussed above, back to the properties and component in fast cure epoxy kit. The solution after mixed with zinc oxide powder became saturated and hot. Furthermore, the epoxy was not smooth and harden faster.

So, coating sample for method Test B cannot be used and unsuccessful, because of off-ratio, insufficient mix and clear coating become cloudy before mixed with zinc oxide. Therefore, the problem was solved by remove the epoxy coating. Measure the epoxy and hardener with ratio 5:1 correctly and apply heat to speed the cure. Method for Test A and Test B were likely same but the difference was mixture ratio and mass of zinc oxide. Therefore, fast cure epoxy cannot be used when the temperature was lower than cure even after recommendation time for 3 hours and must mixing the epoxy and zinc with correct ratio based on their own properties and uses.

4.3 Adhesion Test

The aim were to determine the quality of adhesion coated on carbon steel. Adhesion test was performed on sample coated epoxy, zinc with epoxy and zinc spray paint. The adhesion result were shown in Table 4.1 below by comparing with standard ASTM D 3599.

Table 4.1: Percentage perfection of coating for adhesion test

5	0% (Perfect adhesion)
4	<15%
3	5-15%
2	15-35%
1	35-65%
0	>65%

*** Same concentration but different layer**

Table 4.2: Sample for different layers

sample	1	3	5	1	3	5	1	3	5
layer									
	Z + E + H + eth	Z +E + H + eth	Z + E + H + eth	E	E	E	Spr ay	Sp ray	Spra y

- Z = zinc , E = ethanol , H = hardener , eth = ethanol
- **Red colour – Z + E + H +Ethanol**
- **Blue colour – E + H**
- **Purple colour – Spray paint only**

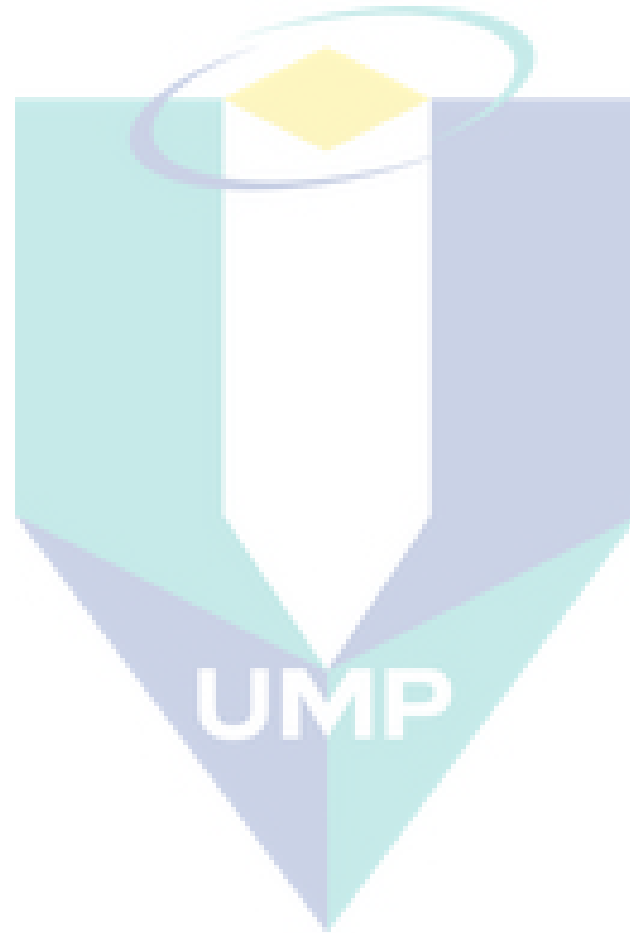
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Table 4.3: Result adhesion test

1				2 (repeat)				3 (repeat)			
Res ult	ASTM	%	Quality	Resul t	ASTM	%	quality	R esult	ASTM	%	quality
	D 3359	(Perfect adhesion)			D 3599	(Perfect Adhesio n)			D 3599	(Perfect Adhesi on)	
1	1B	> 65	Bad	1	4B	<5	moder ate	1	5B	0	good

3	5B	0	Good	3	5B	0	good	3	5B	0	good
5	5B	0	Good	5	5B	0	good	5	5B	0	good
1	5B	0	Good	1	5B	0	good	1	5B	0	good
3	5B	0	Good	3	5B	0	good	3	5B	0	good
5	5B	0	Good	5	5B	0	good	5	5B	0	good
1	2B	35	Bad	1	2B	35	bad	1	2B	35	bad
3	2B	15	bad	3	2B	20	bad	3	2B	20	bad
5	5B	0	Good	5	5B	0	good	5	5B	0	good

* Good – none detached, * Moderate – small flakes, * Bad – flakes and detached



Adhesion test must followed standard ASTM D 3599 test method to evaluate the effectiveness of quality adhesion test on subtract by applying and removing pressure - sensitive tape over cross cut test. The important of adhesion test were to determine the strength of bond. Furthermore, the sample were repeated three times to obtained consistency of adhesion test as shown in Table 4.2.

Next, total sample for adhesion test were 27 samples. Each sample have same concentration but different layers. The sample were divided into three different coating mixture (epoxy, zinc-epoxy and spray). First cycle for adhesion test, the zinc- epoxy for first layers was not in good condition which coating has flaked along edges of cuts, some squares have detached fully and significant. The perfect adhesion percentage was zero, so for first layers for zinc-epoxy shows bad quality more than 65 % and not bond very well. So, the causes for this problem because of insufficient mixing or off ratio.

However, the others two zinc-epoxy were well-bonded for three and fifth layers. Next, epoxy were well-bonded and 0 % (perfect adhesion) with edge cut were completely smoothly, none of square lattice was detached. Besides that, zinc galvanizing primer spray paint for one and triple layers shows bad result for adhesion test because the coating has flaked along edges of cuts, fully in different part of squares. Significantly 15 % but not greater than 35 % small flakes of the coating at intersection of cuts. This problem can be solved by right technique such as swept horizontally and vertically in order to have smooth surface .While, the others three and fifth layers shows the best result which 0 % perfection means the edge cut were completely smoothly, none of square lattice was detached.

Table 4.4: The quality of Adhesion Test for one cycles

Sample coated Layer	Quality of Adhesion Test		
	1 layer	3 layers	5 layers
Epoxy	Good	Good	Good

Zinc-epoxy	Bad	Good	Good
Zinc spray paint	Bad	Bad	Good

Second cycle for adhesion test shows an improvement strength of zinc-epoxy first layer. The epoxy first layer only has small flakes of the coating at intersection of cuts and not significantly greater than 5 % was affected rather than first cycle which were not well-bonded. The others three and fifth layers shows a good adhesion test same as epoxy layers from one to fifth layers. All of those layers were well mixed and well-bonded, harden, smooth and good appearances. Besides, standard ASTM D 3599 for 0 % perfection describe that the edge cut are completely smoothly, none of square lattice was detached. For zinc primer spray paint shows same bad quality of adhesion for first triple layers. Therefore, for second cycle of adhesion test were describe below:

Table 4.5: The quality of Adhesion Test for second cycles

Sample coated Layer	Quality of Adhesion Test		
	1 layer	3 layers	5 layers
Epoxy	Good	Good	Good
Zinc-epoxy	Moderate	Good	Good
Zinc spray paint	Bad	Bad	Good

Lastly, for last cycle for adhesion test shows that zinc-epoxy first layer has good quality which the edge cut are completely smoothly, none of square lattice were detached. The others three and fifth layers of zinc - epoxy shows a good adhesion test same as epoxy layers. However, zinc primer spray paint shows same quality with second cycle which in bad quality of adhesion. All of those layers were well mixed and well-bonded, harden, smooth and good appearances. Therefore, for third cycle of adhesion test were describe below:

Table 4.6: The quality of Adhesion Test for third cycles

Sample coated Layer	Quality of Adhesion Test		
	1 layer	3 layers	5 layers
Epoxy	Good	Good	Good
Zinc-epoxy	Good	Good	Good
Zinc spray paint	Bad	Bad	Good

As a conclusion, from one until third cycle adhesion test shows that epoxy, zinc – epoxy and zinc galvanizing spray paint were in good quality just first and triple layer of zinc mixed with oxide were in bad conditions. Therefore, this project research of organic coating are shown improvement from commercialized products.

4.4 Immersion test

Immersion test was a part of corrosion testing where have two criterion involved which were immersion test without mass loss and immersion test with mass loss. Both criterion has been accepted by standard ASTM G31 (mass loss) and ASTM B 895-99 (without mass loss). The weight data was obtained for 2 months shown in Table 4.3.

4.4.1 Immersion test without Mass Loss

Immersion test without mass loss includes testing in neutral salt solution. ASTM B 895-99 for salt solution immersion test. The procedure was simple, inexpensive and flexible. If the immersion test less than 25 % of surface covered by stain or rust, the corrosion resistance have a little effect on mechanical properties in which weight loss were unreliable for some of the rust. Therefore, cause the weight gain because of penetration of salt into pores instead of weight loss (Corrosion Test and Standard, 2000).

ASTM G1 were used to evaluate the measuring weight loss or gain for metal specimen. The initial weight of the test samples is measured and recorded. So, the data in Table 4.2 can be calculated for weight change either in loss or gain by this formula.

Weight change by percent:

$$\% \text{ weight change} = [\text{Final weight} - \text{initial weight} / \text{initial weight}] \times 100$$

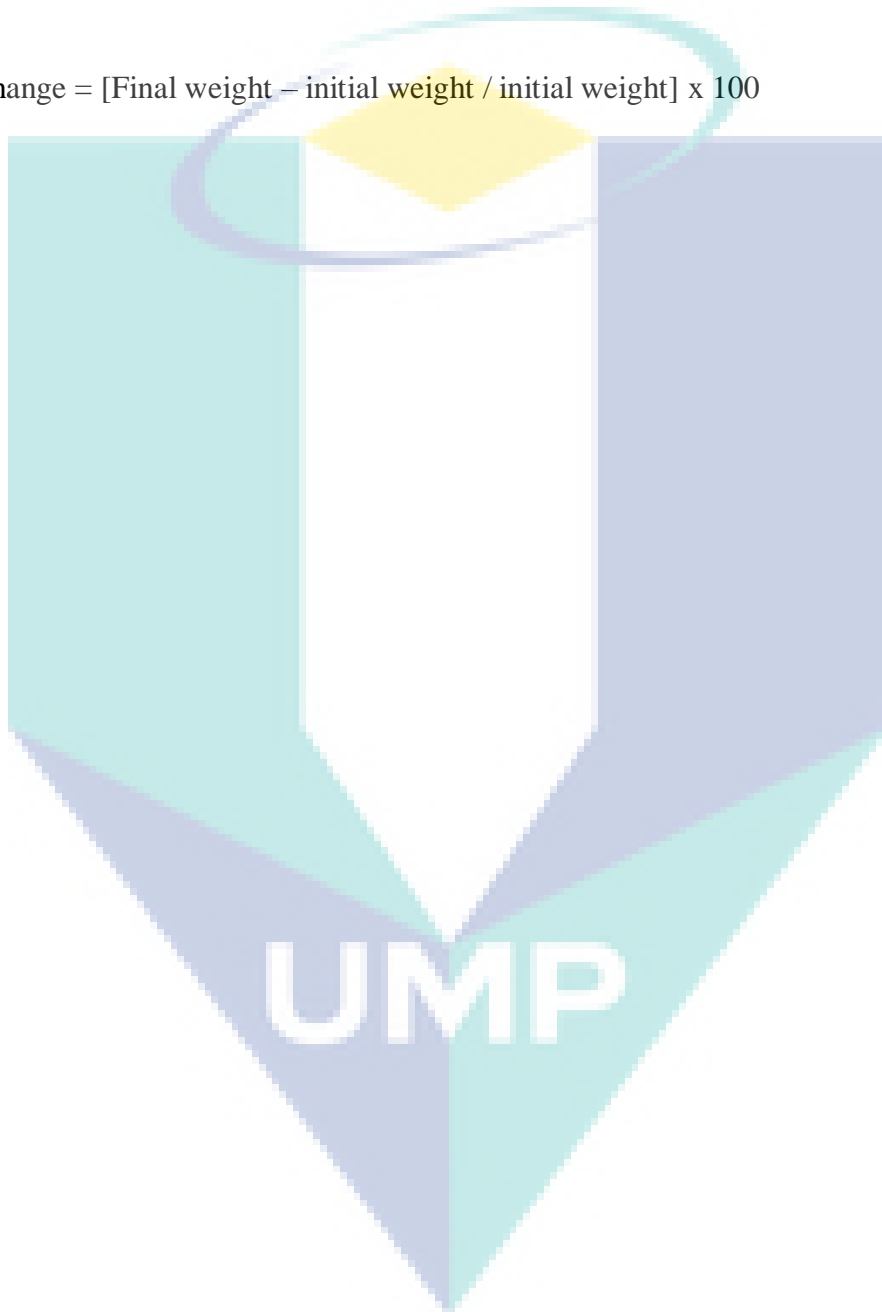


Table 4.7: Result weight for Immersion Test

sample / layer	1	3	5	1	3	5	1	3	5
Initial (without coated)	3.2958 g	2.4465 g	2.9964 g	2.0766 g	2.3672 g	2.0981 g	2.6001 g	2.4357 g	2.1648 g
1 week	3.3404 g	2.4944 g	3.5862 g	2.1176 g	2.4010 g	2.1591 g	2.6069 g	2.4657 g	2.2048 g

22/10/2015									
2 week	3.3400 g	2.5029 g	3.7606 g	2.4985 g	2.4974 g	2.7950 g	2.5966 g	2.4707 g	2.4697 g
29/10/2015									
3 week	3.3437 g	2.5011 g	3.7901 g	2.4993 g	2.4986 g	2.7990 g	2.5950 g	2.4699 g	2.4320 g
5/11/2015									
5 week	3.3501 g	2.4998 g	3.8332 g	2.5024 g	2.5060 g	2.8015 g	2.5940 g	2.4664 g	2.4001 g
19/11/2015									
6 week	3.3556 g	2.5628 g	3.8239 g	2.5023 g	2.5050 g	2.7989 g	2.5936 g	2.4562 g	2.3987 g

26/11/2015									
7 week	3.3576 g	2.5637 g	3.8224 g	2.5122 g	2.5105 g	2.8016 g	2.5832 g	2.4553 g	2.3995 g
3/12/2015									
8 week	3.3577 g	2.5655 g	3.8243 g	2.5232 g	2.5110 g	2.8016 g	2.5432 g	2.4532 g	2.3996 g
10/12/2015									

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Based on result of immersion test from Table 4.7 above shows that weight data consists of weight gain and loss. Firstly, the initial data (red colour) for epoxy composite with zinc coating for first layer was 3.298 g, triple layers (2.4465 g) and fifth layers (2.9964 g). After two months immersed in 3.5 % of NaCl solution, the weight increased for first layer was 3.3577 g, triple layers was 2.5655 g and 3.8243 g. This means that, the weight was gained between periods of time. These was because penetration of salt into pores instead of weight loss based on ASTM B 895-99 (Corrosion Test and Standard, 2000). Corrosion of a block of metal does not changed the weight of metal in the system and only increase in weight due to addition of oxygen molecules. In addition, these was because density of oxygen (1.429 g/l) is lighter than steel which was (7.86 g/l) and resulting molecule was larger.

Next, for epoxy composite data (blue colour) from Table 4.3 shows that weight were gained between one layers, triple layers and fifth layers. The initial weight for epoxy resin coated with steel for one layer was 2.0766 g, triple layers was 2.3672 g and for fifth layers was 2.0981 g. Besides, the weight after two months for first layers increased to 2.5232 g, triple layers was 2.5110 g and fifth layer was 2.7980 g. These was likely same with epoxy composite with zinc coating that increased weight gain. Therefore, both epoxy composite with zinc and epoxy resin were same in criterion of immersion without mass loss.

Lastly, for zinc galvanizing primer spray paint was obtained to study the comparison of commercialized product with this research. The initial weight for first layers was 2.6001 g decreased to 2.5432 g, triple layers was 2.4357 g increased to 2.4532 g and fifth layers was 2.1648 g increased to 2.3996 g. There were two criterion involved in this zinc spray reaction which immersion test with mass loss and without mass loss. For first layer, involved with immersion test with mass loss, while differ for triple and fifth layers which involved with immersion without mass loss. So, the immersion with mass loss because metallic oxide easily discard off parent object. If the metal was disturbed it was easy to be removed and loss weight (Engineering Forum, 2015).

Table 4.8: Differences in weight for Immersion

sample layer	1	3	5	1	3	5	1	3	5
1 week 22/10/2015	0.0446 g	0.0479 g	0.5898 g	0.0410 g	0.0338 g	0.0610 g	0.0068 g	0.0300 g	0.0400 g
2 week 29/10/2015	0.0442 g	0.0564 g	0.7642 g	0.4219 g	0.1302 g	0.6969 g	0.0035 g	0.0350 g	0.3049 g
3 week 5/11/2015	0.0479 g	0.0546 g	0.7937 g	0.4227 g	0.1314 g	0.7009 g	0.0051 g	0.0324 g	0.2672 g

5 week 19/11/2015	0.0543 g	0.0533 g	0.8368 g	0.4258 g	0.1388 g	0.7034 g	0.0061 g	0.0305 g	0.2353 g
6 week 26/11/2015	0.0598 g	0.1163 g	0.8275 g	0.4257 g	0.1378 g	0.7008 g	0.0065 g	0.0205 g	0.2339 g
7 week 3/12/2015	0.0618 g	0.1172 g	0.8260 g	0.4356 g	0.1433 g	0.7035 g	0.0169 g	0.0196 g	0.2347 g
8 week 10/12/2015	0.0619 g	0.1190 g	0.8279 g	0.4466 g	0.1438 g	0.7035 g	0.0569 g	0.0175 g	0.2348 g
Average weight	0.0535 g	0.0807 g	0.7808 g	0.3742 g	0.1227 g	0.6100 g	0.0145 g	0.0265 g	0.2215 g

Layer									
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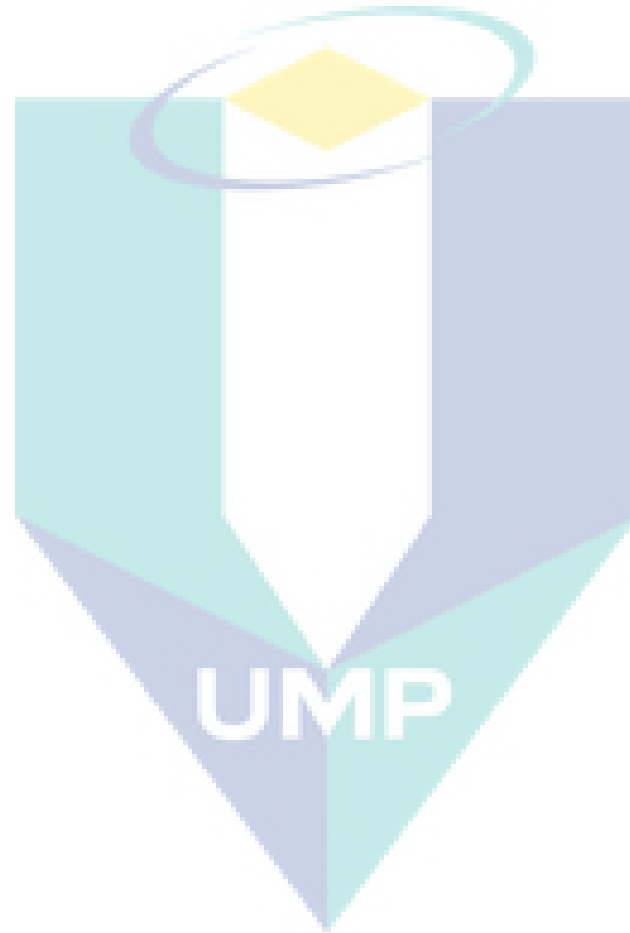


Table 4.8 shows the differences in weight of immersion test for epoxy composite with zinc, epoxy resin and zinc galvanizing spray coating. The differences of each sample were calculated using formula weight difference which initial weight minus with final weight after been immersed in 3.5 % salt solution. The average for each coating divided with seven weight data to get average value of each sample.

Firstly, the differences of epoxy composite with zinc for one layer was 0.0046 g and after two months was 0.0619 g. The average value for first layer was 0.0535 g. From this result, shows that the weight difference increased. Next, for triple layers was 0.0479 g and final was 0.1190 g with average value of 0.0807 g. Besides, the fifth layers was 0.5898 g to 0.8279 g. Each of layers in epoxy composite with zinc shows increased data at weight differences. This calculation was to be done to easily calculated weight percentage that shown in Table 4.9 below.

Next , epoxy resin for the one layer have average weight for first layer 0.3472 g, triple 0.1227 g and fifth layers was 0.6100 g. The initial weight difference in first week for the first layer was 0.0410 g and final weight was 0.4466 g. In addition, the initial weight for triple layers increased from 0.0338 g to 0.1438 g. Besides, final weight of fifth layer was higher than initial which 0.7035 g.

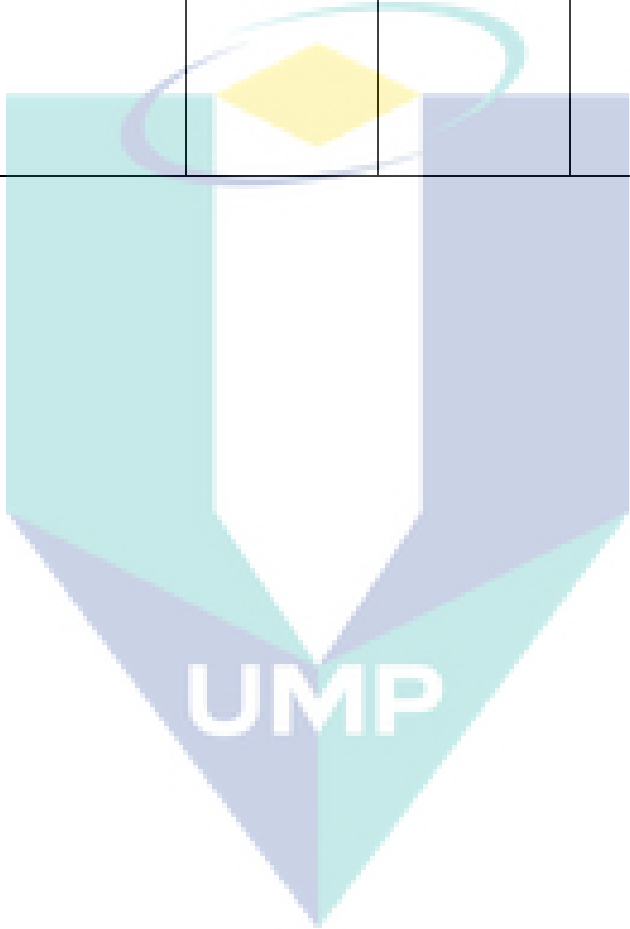
Lastly, zinc primer spray paint average value were 0.0145 g for one layer, 0.0265 g for triple layer and 0.2215 g for fifth layer. For one layer, the weight differences decreased, however, for triple and fifth layer shows increased in weight loss. These was because of the immersion of salt solution for one layer was loss in mass but triple and fifth layers without mass loss.

Table 4.9: Weight percentage for immersion test

sample / layer	1	3	5	1	3	5	1	3	5
1 week 22/10/2015	1.35 %	1.95 %	19.68 %	1.97 %	1.43 %	2.91 %	0.26 %	1.23 %	1.85 %
2 week 29/10/2015	1.34 %	2.25 %	25.50 %	20.3 %	5.45 %	33.2 %	0.15 %	1.44 %	14.08 %

3 week	1.45 %	2.23 %	26.50 %	20.4 %	5.55 %	33.0 %	0.20 %	1.40 %	12.34 %
5/11/2015									
5 week	1.65 %	2.19 %	27.90 %	20.5 %	5.59 %	33.5 %	0.23 %	1.26 %	10.86 %
19/11/2015									
6 week	1.78 %	4.75 %	27.60 %	20.5 %	5.82 %	33.4 %	0.25 %	0.84 %	10.80 %
26/11/2015									
7 week	1.88 %	4.79 %	27.60 %	21.0 %	6.05 %	33.5 %	0.65 %	0.80 %	10.84 %
3/12/2015									

8 week	1.88 %	4.86 %	27.60 %	21.5 %	6.07 %	33.5 %	2.19 %	1.09 %	10.84 %
10/12/2015									



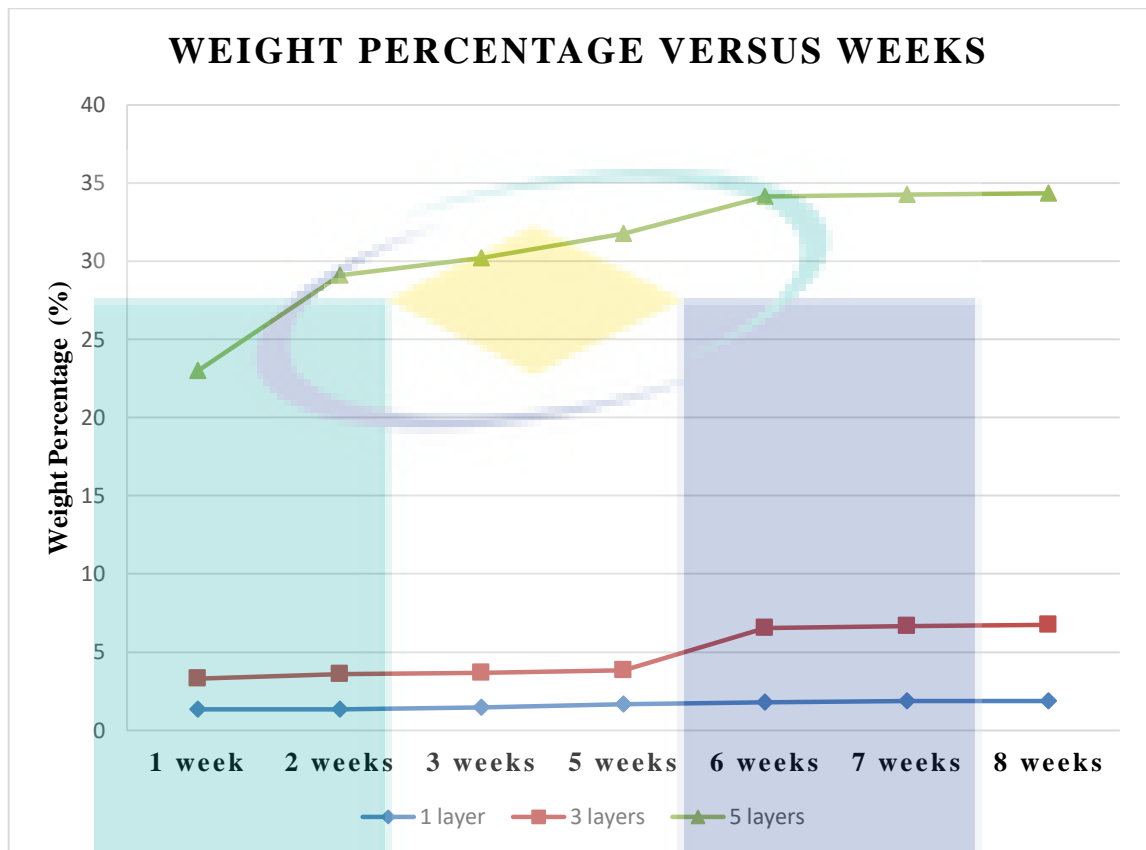


Figure 4.1: Weight percentage versus weeks for epoxy composite with zinc oxide

From Table 4.9 above shows the weight percentage of each layer of corrosion test and the data were interpret in figure 4.1 above. Firstly, the weight percentage was calculated based on ASTM G 31. The calculation can be both gain in weight and loss weight. From figure 4.1 above, shows that fifth layers have high weight percentage than triple and one layer. This is because of thickness of coating and absorption of oxygen into metal causes the weight increased. These result was reasonable because during immersion test in 3.5 % salt solution, some of sample were corrode at the bottom but the upper surface kept maintained. Therefore, the corrosion testing based on ASTM B 895-99 plus-minus 25 % shows the corrosion resistance have a little effect on mechanical properties in which weight loss were unreliable for some of the rust.

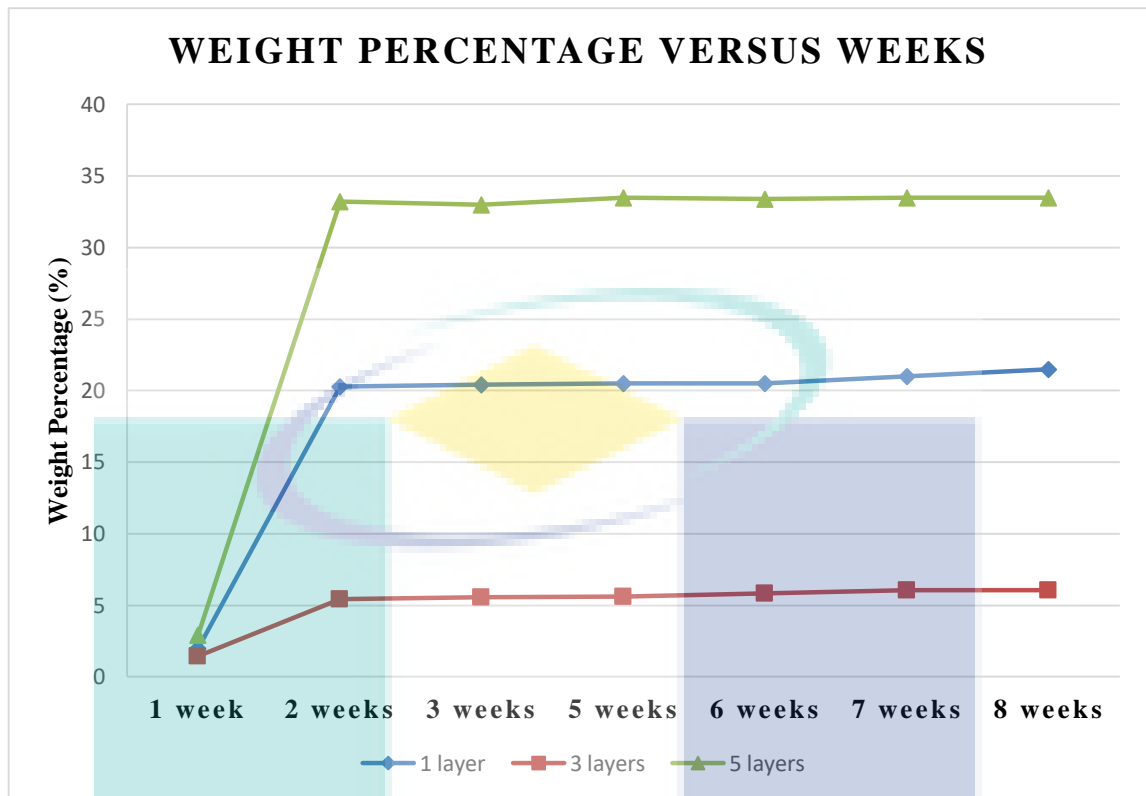


Figure 4.2: Weight percentage versus weeks for epoxy resin

From Table 4.9 above shows the weight percentage of each layer epoxy resin for corrosion test and the data were interpret in figure 4.2 above. The initial weight percentage of epoxy for one layer was 1.97 % and final was 21.50 %. Triple layer of epoxy was 1.23 % for first week and 1.09 % for final week. Next, fifth layers was 33.5 % for final week. The calculation can be both gain in weight and loss weight. From figure 4.2 above, shows that fifth layers have high weight percentage than one layer. The triple layers has lower weight percentage than one layer because the initial weight of triple layers was lower than one layer. The epoxy resin and epoxy composite with zinc shows both same result which highest weight percentage was fifth layers of immersion test. Therefore, the weight percentage kept increased because the molecule was not discard easily and absorption of oxygen into metal causes the weight increased. These result was reasonable because during immersion test in 3.5 % salt solution, some of sample were corrode at the bottom but the upper surface kept maintained. Therefore,

the corrosion testing based on ASTM B 895-99 plus-minus 25 % shows the corrosion resistance have a little effect on mechanical properties in which weight loss were unreliable for some of the rust.

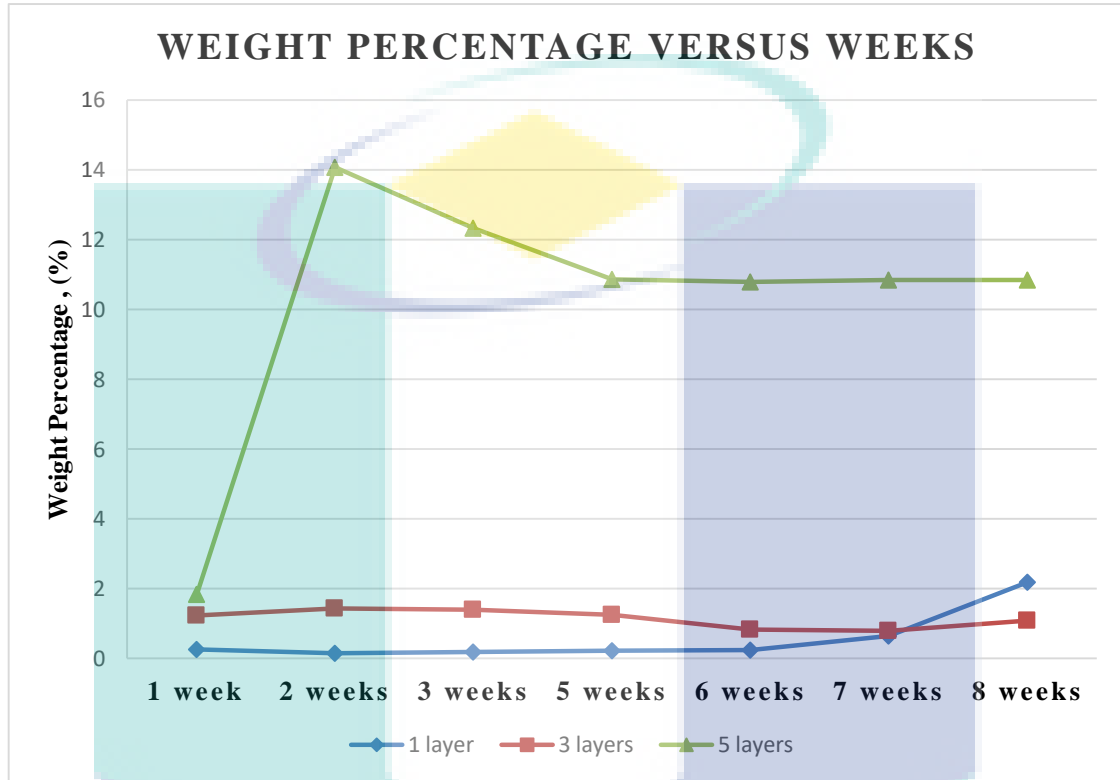
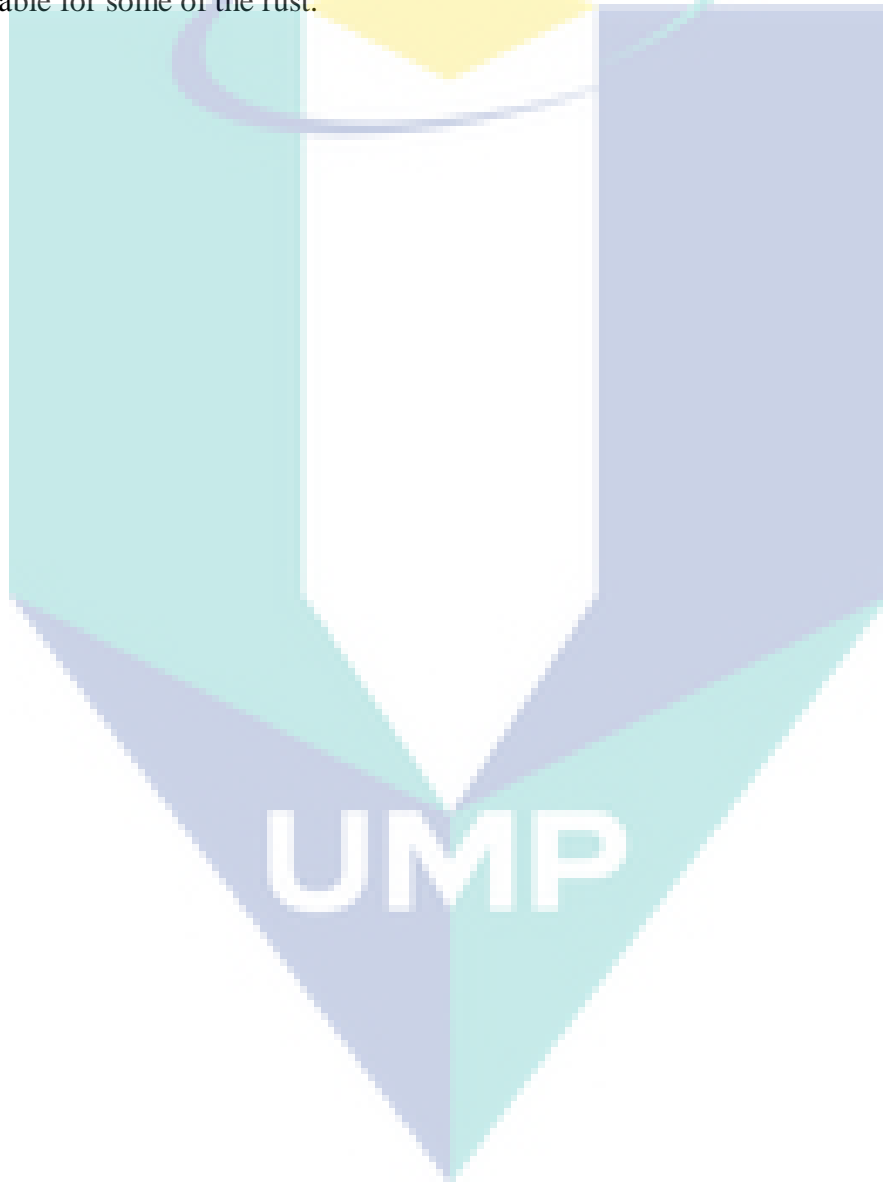


Figure 4.3: Weight percentage versus weeks of zinc galvanizing primer spray paint

From Table 4.9 above shows the weight percentage of each layer zinc rich spray paint for corrosion test and the data were interpret in figure 4.3 above. The initial weight percentage of epoxy for one layer was 0.26 % and final was 2.19 %. Triple layer weight percentage was 1.43 % for first week and 6.07 % for final week. Next, fifth layers was 10.84 % for final week. The calculation can be both gain in weight and loss weight. From figure 4.3 above, shows that fifth layers have high weight percentage than one layer. The triple layers has lower weight percentage than one layer because the initial weight of triple layers was lower than one layer. The epoxy resin and epoxy composite

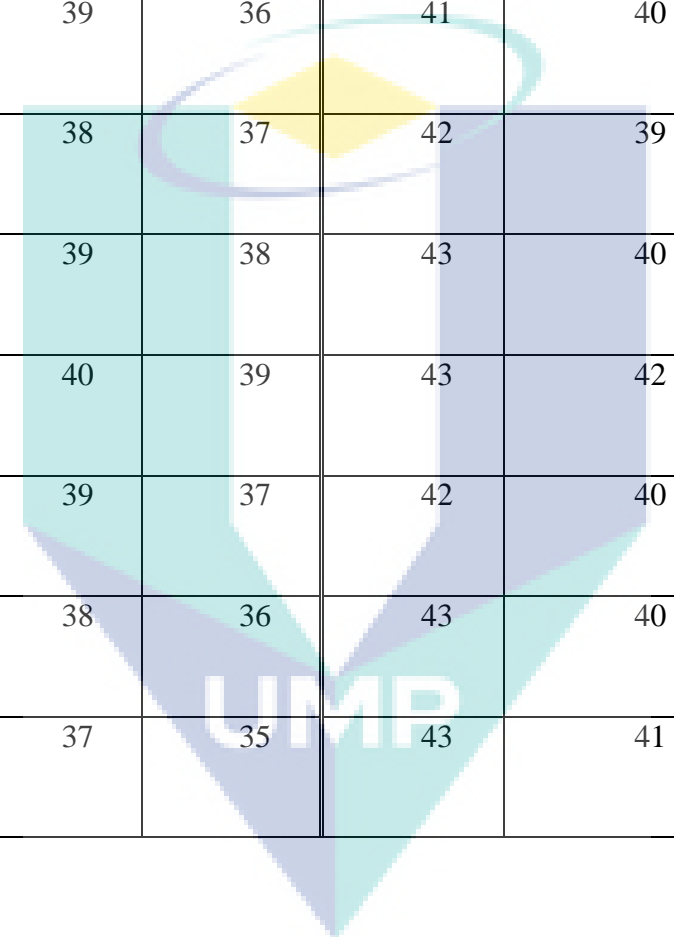
with zinc shows both same result which highest weight percentage was fifth layers of immersion test. Therefore, the weight percentage kept increased because the molecule was not discard easily and absorption of oxygen into metal causes the weight increased. These result was reasonable because during immersion test in 3.5 % salt solution, some of sample were corrode at the bottom but the upper surface kept maintained. Therefore, the corrosion testing based on ASTM B 895-99 plus-minus 25 % shows the corrosion resistance have a little effect on mechanical properties in which weight loss were unreliable for some of the rust.



4.5 Temperature

Table 4.10: Temperature of epoxy composite with zinc with and without coating surface

Temperature for Zinc- epoxy at upper surface with coating (°C)				Temperature Zinc-epoxy at bottom surface without coating (°C)		
Time (s)	1	3	5	1	3	5
5	27	26	26	27	28	27
10	29	28	27	30	32	29
15	38	36	34	40	39	41
20	39	37	35	41	40	39



25	38	36	35	40	39	37
30	41	39	36	41	40	38
35	40	38	37	42	39	36
40	42	39	38	43	40	37
45	42	40	39	43	42	40
50	41	39	37	42	40	39
55	42	38	36	43	40	38
60	42	37	35	43	41	42

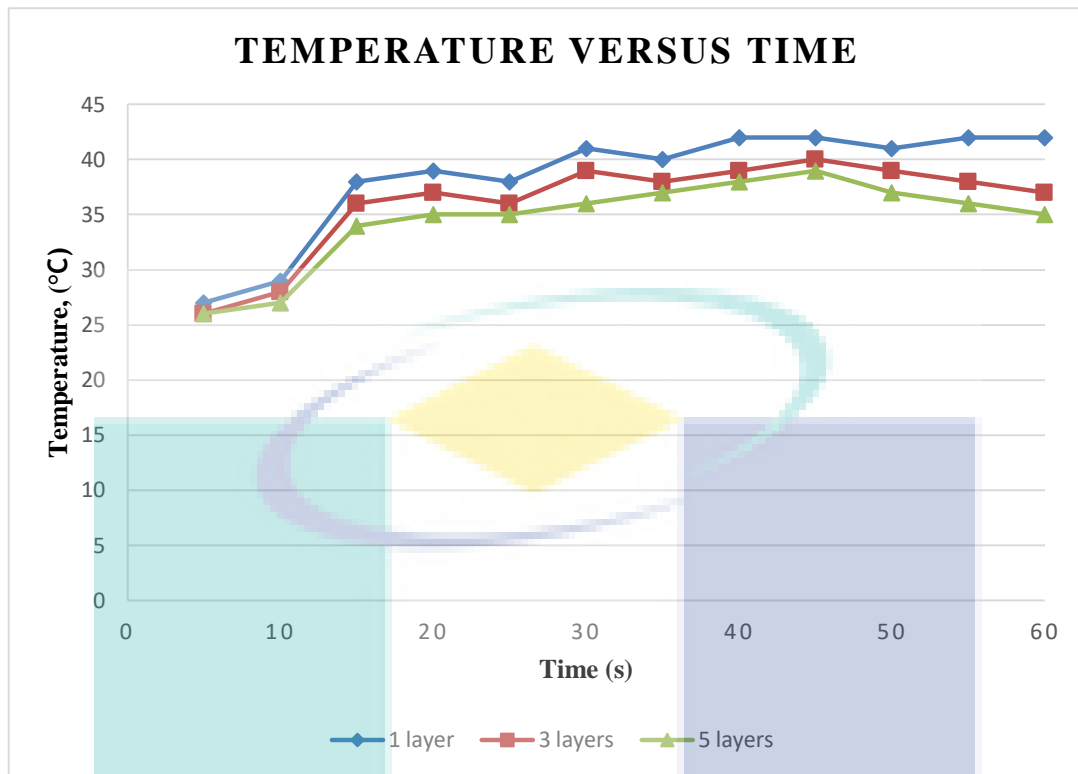


Figure 4.4: Temperature of zinc-epoxy at upper surface with coating

Based on table 4.10 above shows the result data for temperature of epoxy composite with zinc at upper surface of coating and bottom without coating. The time taken to take the data about 1 hours for both side upper and bottom surface of coated sample. Firstly, temperature for first layer was 27 °C and final temperature was 42°C. Between the temperature data, each 5 minutes the temperature increased for 1°C. Next, for triple layers the temperature for initial was 26 °C same as fifth layer and final was 37 °C and 35 °C. Besides that, figure 4.4 shows the temperature for fifth layers of zinc-epoxy at upper surface was lower than triple and one layer. These was because the voids that was produced by a diffusion of zinc from the outer free zinc layer into inner alloy shrink and not form a gap and known as Kirkendall Effect with no peeling. The protection of zinc coating depend on thickness. Besides, the temperature below than 480 °F (250°C) can protect the steel from corrosion (Bernardo, 2013).

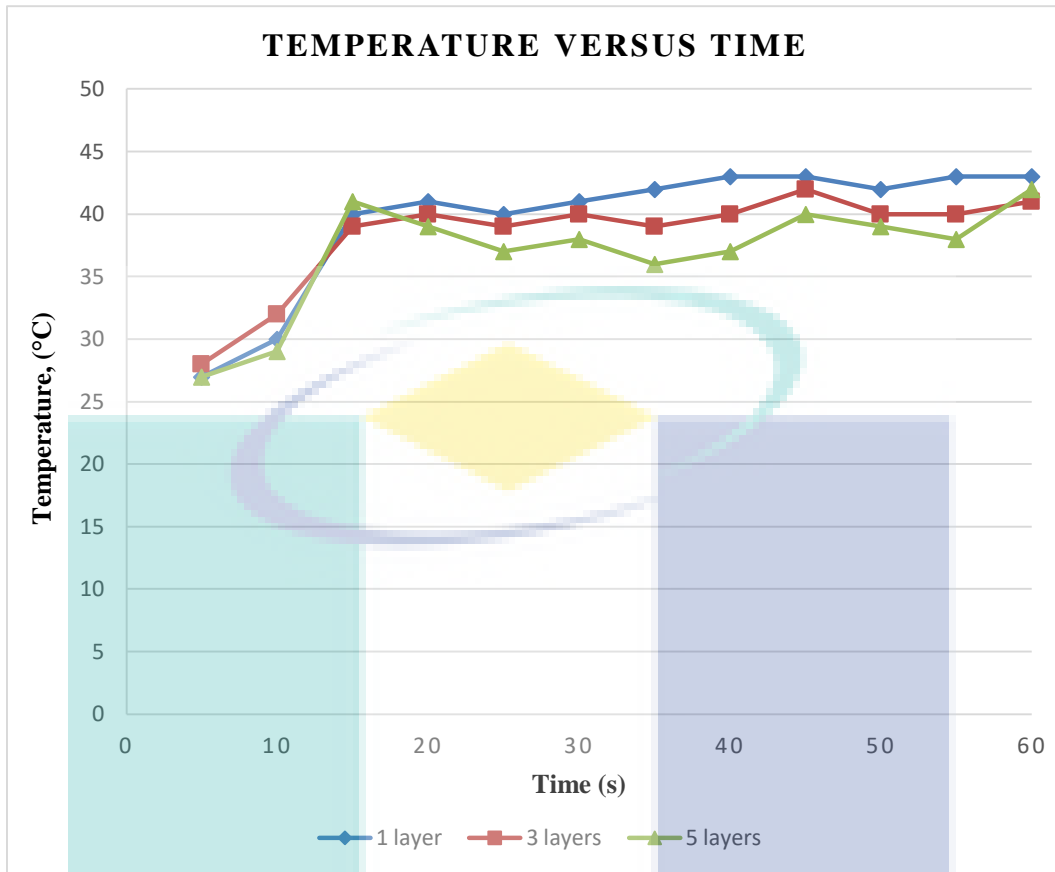


Figure 4.5: Temperature of zinc-epoxy at bottom surface without coating

Based on table 4.10 above shows the result data for temperature of epoxy composite with zinc at upper surface of coating and bottom without coating. The time taken to take the data about 1 hours for both side upper and bottom surface of coated sample. Firstly, temperature were taken from bottom surface without coating, for first layer was 27 °C same both with fifth layer and final temperature was 43°C and 42 °C means higher than upper surface. Between the temperature data, each 5 minutes the temperature increased for $\pm 2^{\circ}\text{C}$. Next, for triple layers the temperature for initial was 28 °C and final was 41 °C. Besides that, figure 4.5 shows the temperature for fifth layers of zinc- epoxy at upper surface was lower than triple and one layer. The temperature for zinc-epoxy at bottom surface was higher than upper surface because heat penetrated easily into surface without coating (Bernando, 2013).

Table 4.11: Temperature of epoxy with and without coating surface

Temperature for epoxy at upper surface with coating (°C)				Temperature epoxy at bottom surface without coating (°C)		
Time (s)	1	3	5	1	3	5
5	27	26	26	27	28	29
10	29	28	27	29	30	32
15	31	29	28	32	31	30

20	33	31	29	35	33	32
25	32	33	31	37	35	33
30	33	32	31	36	37	40
35	35	33	30	39	40	41
40	38	37	34	41	42	40
45	41	39	37	42	41	43
50	43	42	40	45	43	42
55	42	41	39	44	42	40
60	43	40	40	45	43	42

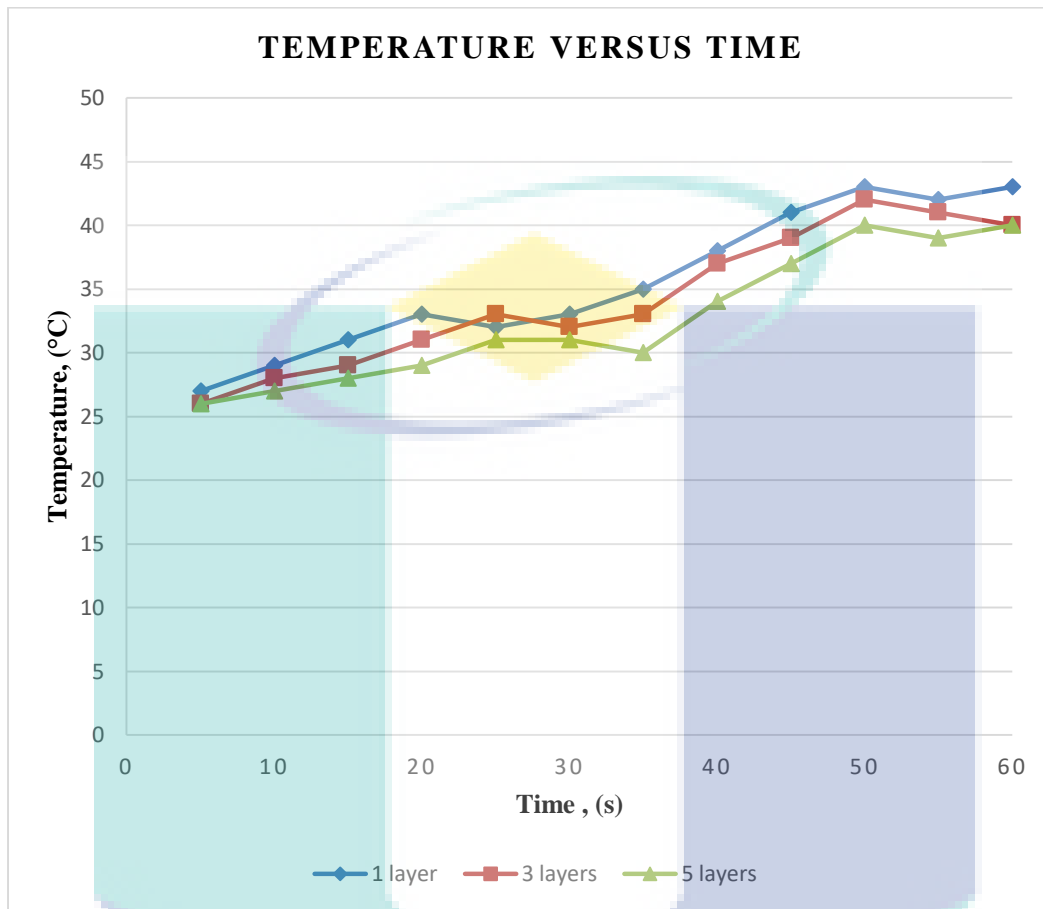


Figure 4.6: Temperature of epoxy at upper surface with coating

Based on table 4.11 above shows the result data for temperature of epoxy at upper surface of coating and bottom without coating. The time taken to take the data about 1 hours for both side upper and bottom surface of coated sample. Firstly, temperature for first layer was 27 °C and final temperature was 42°C. Between the temperature data, each 5 minutes the temperature increased for $\pm 3^{\circ}\text{C}$. Next, for triple layers the temperature for initial was 26 °C same as fifth layer and final was same 40 °C. Besides that, figure 4.6 shows the temperature for fifth layers of epoxy at upper surface was lower than triple and one layer. These because the epoxy were formulated

at lower temperature, as the thickness increased, the temperature of coating can lower the chemical reaction process in high environment conditions. The temperature below 50 °F (10°C) will stop the reaction and evaporation of solvent in coating also inhibited (Williams, 2008).

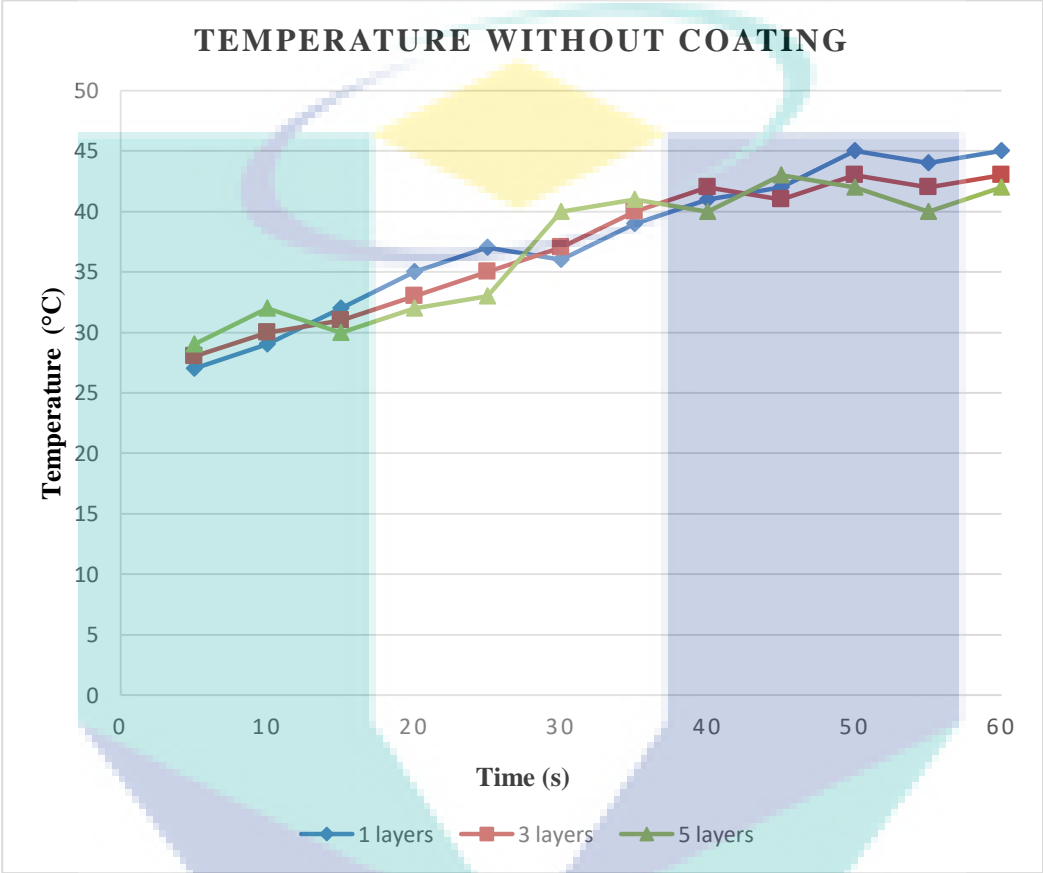


Figure 4.7: Temperature of epoxy at bottom surface without coating

Based on table 4.11 above shows the result data for temperature of epoxy at upper surface of coating and bottom without coating. The time taken to take the data about 1 hours for both side upper and bottom surface of coated sample. Firstly, temperature were taken from bottom surface without coating, for first layer was 27 °C and final temperature was 45°C means higher than upper surface. Between the temperature data, each 5 minutes the temperature increased for ± 2°C. Next, for triple

layers the temperature for initial was 28 °C and final was 43 °C. While, final temperature for fifth layers was 42 °C. Besides that, figure 4.7 shows the temperature for fifth layers of epoxy at fifth layer was lower than triple and one layer. The temperature for steel at bottom surface was higher than upper surface because heat penetrated easily into surface without coating (Bernando, 2013).

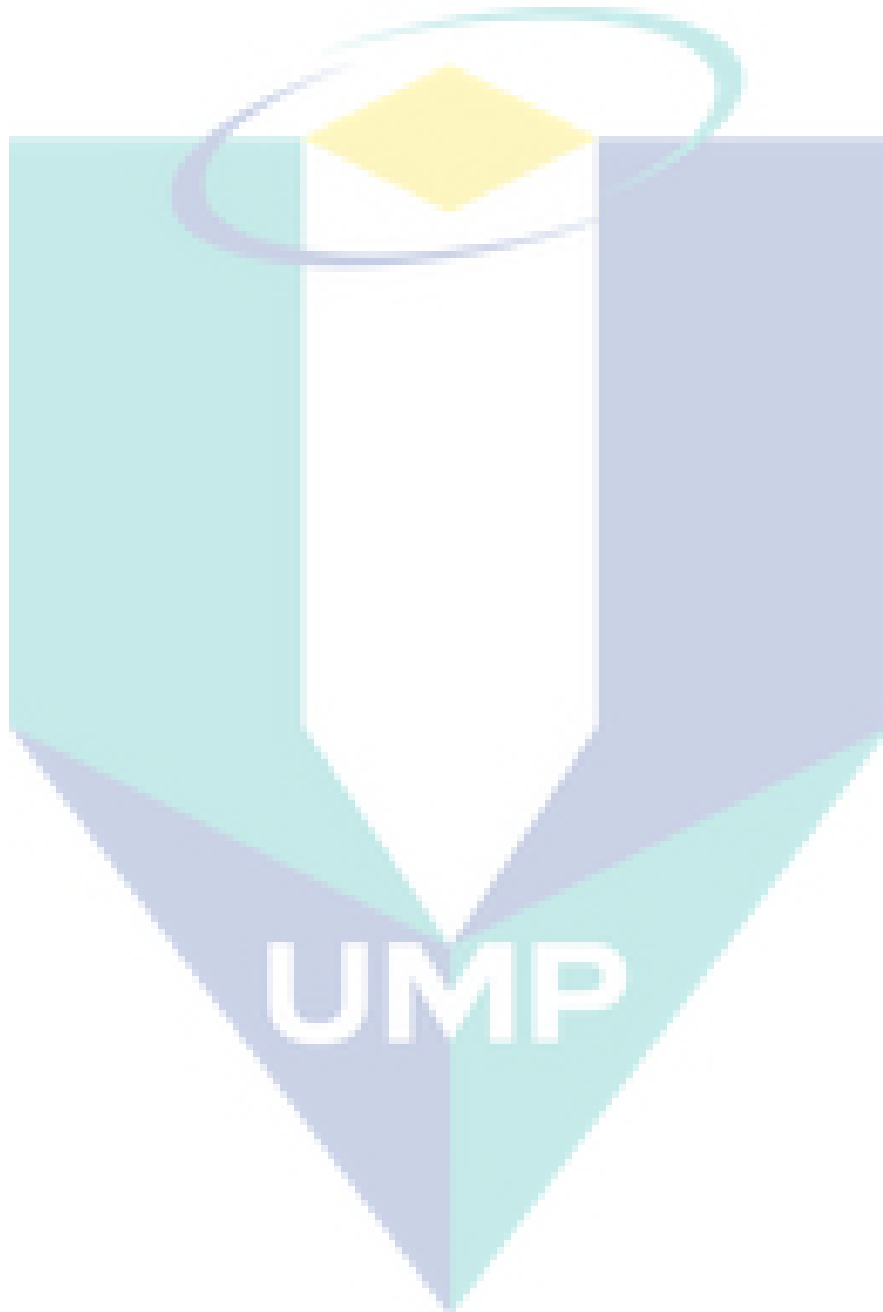
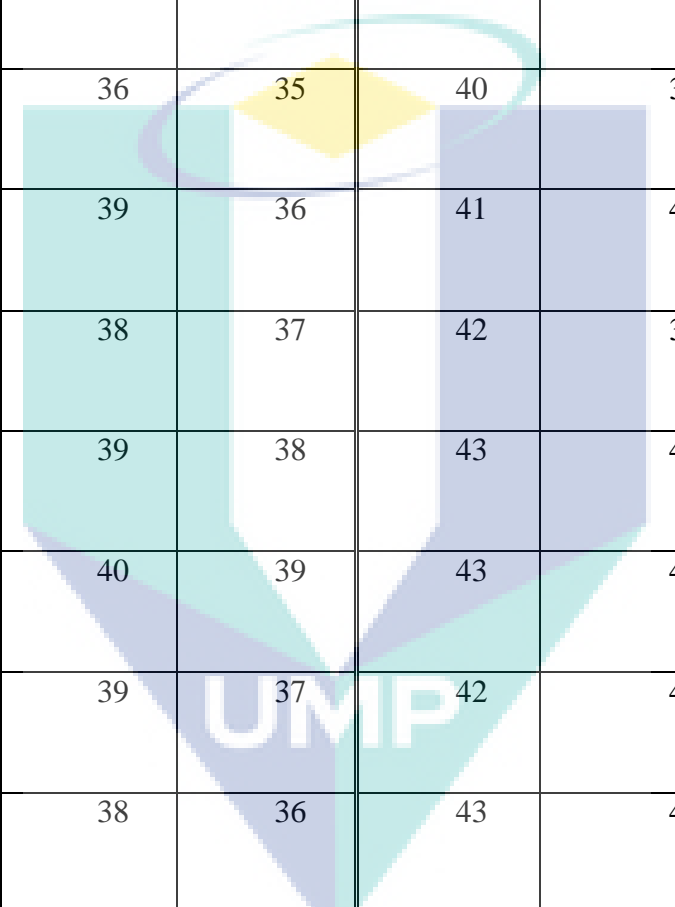


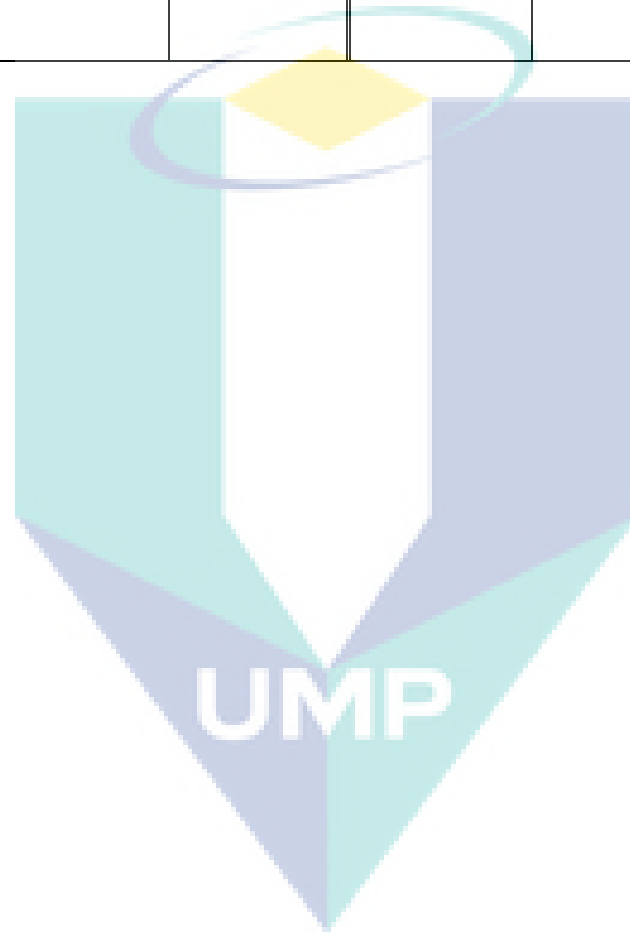
Table 4.12: Temperature of zinc primer spray paint with and without coating surface

Temperature for zinc primer spray paint at upper surface with coating (°C)				Temperature zinc primer spray paint at bottom surface without coating (°C)		
Time (s)	1 layer	3 layers	5 layers	1 layers	3 layers	5 layers
5	27	26	26	27	28	27
10	29	28	27	30	32	29
15	38	36	34	40	39	41



20	39	37	35	41	40	39
25	38	36	35	40	39	37
30	41	39	36	41	40	38
35	40	38	37	42	39	36
40	42	39	38	43	40	37
45	42	40	39	43	42	40
50	41	39	37	42	40	39
55	42	38	36	43	40	38

60	41	38	39	43	41	42
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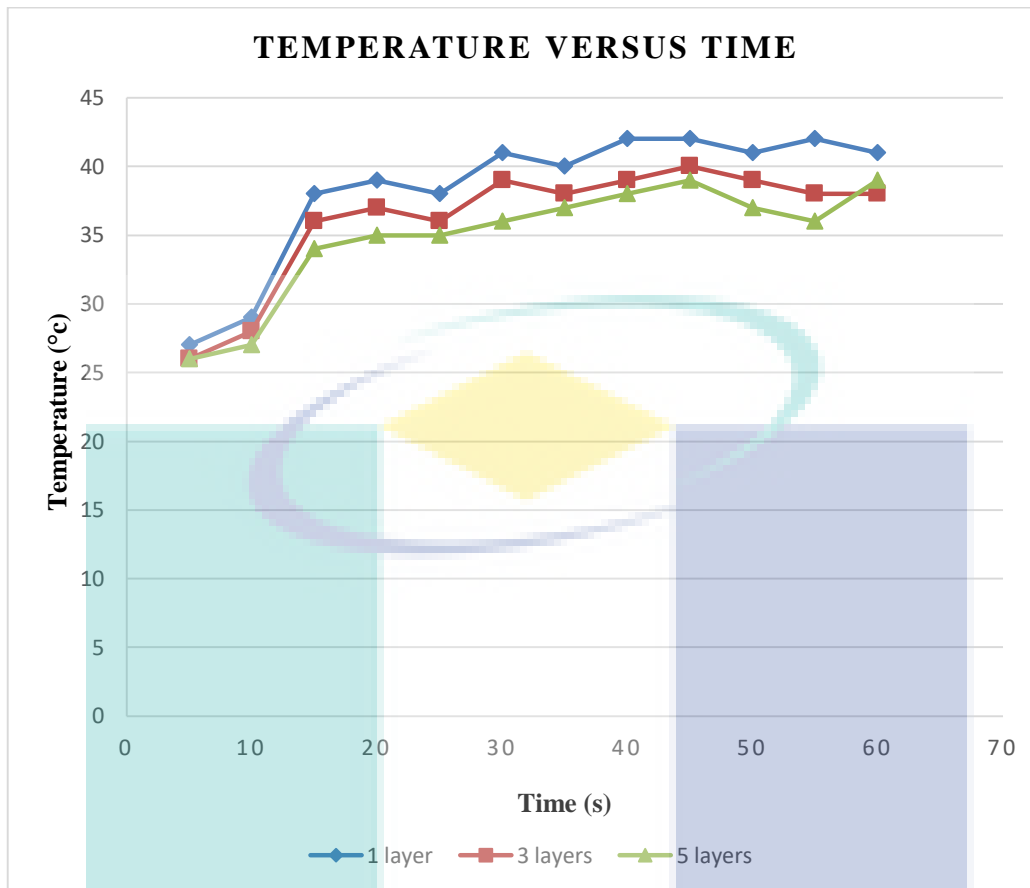


Figure 4.8: Temperature of zinc primer spray paint at upper surface with coating

Based on table 4.12 above shows the result data for temperature of epoxy at upper surface of coating and bottom without coating. The time taken to take the data about 1 hours for both side upper and bottom surface of coated sample. Firstly, temperature for first layer was 27 °C and final temperature was 41°C. Between the temperature data, each 5 minutes the temperature increased for $\pm 1^\circ\text{C}$. Next, for triple layers the temperature for initial was 26 °C same as fifth layer and final was 38 °C and 39 °C. Besides that, figure 4.8 shows the temperature for fifth layers of zinc spray paint at upper surface was lower than triple and one layer. These because the corrosion rate of zinc was low ranges about 0.13 $\mu\text{m}/\text{yr}$ in dry rural atmosphere and 0.013 mm/yr in moist industrial atmosphere. Zinc coating have high degree of resistance to atmospheric corrosion (Corrosion of Zinc, 2015).

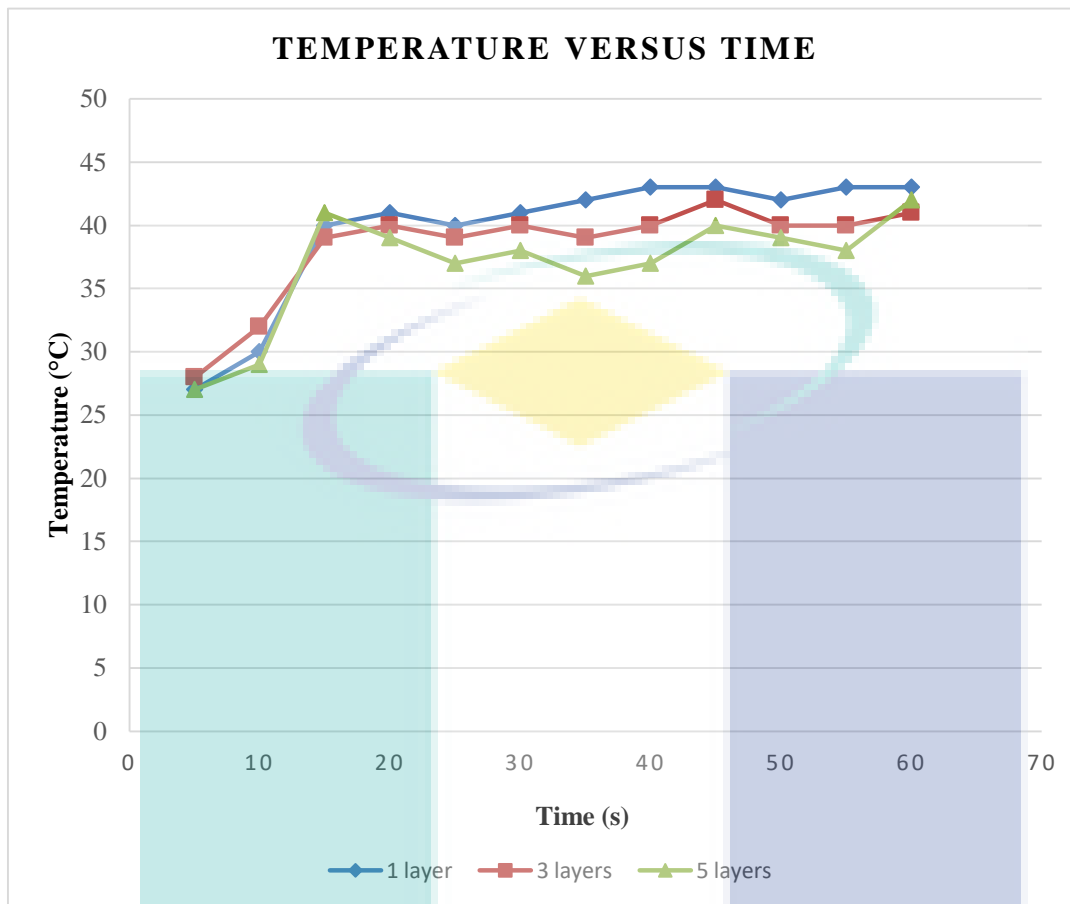
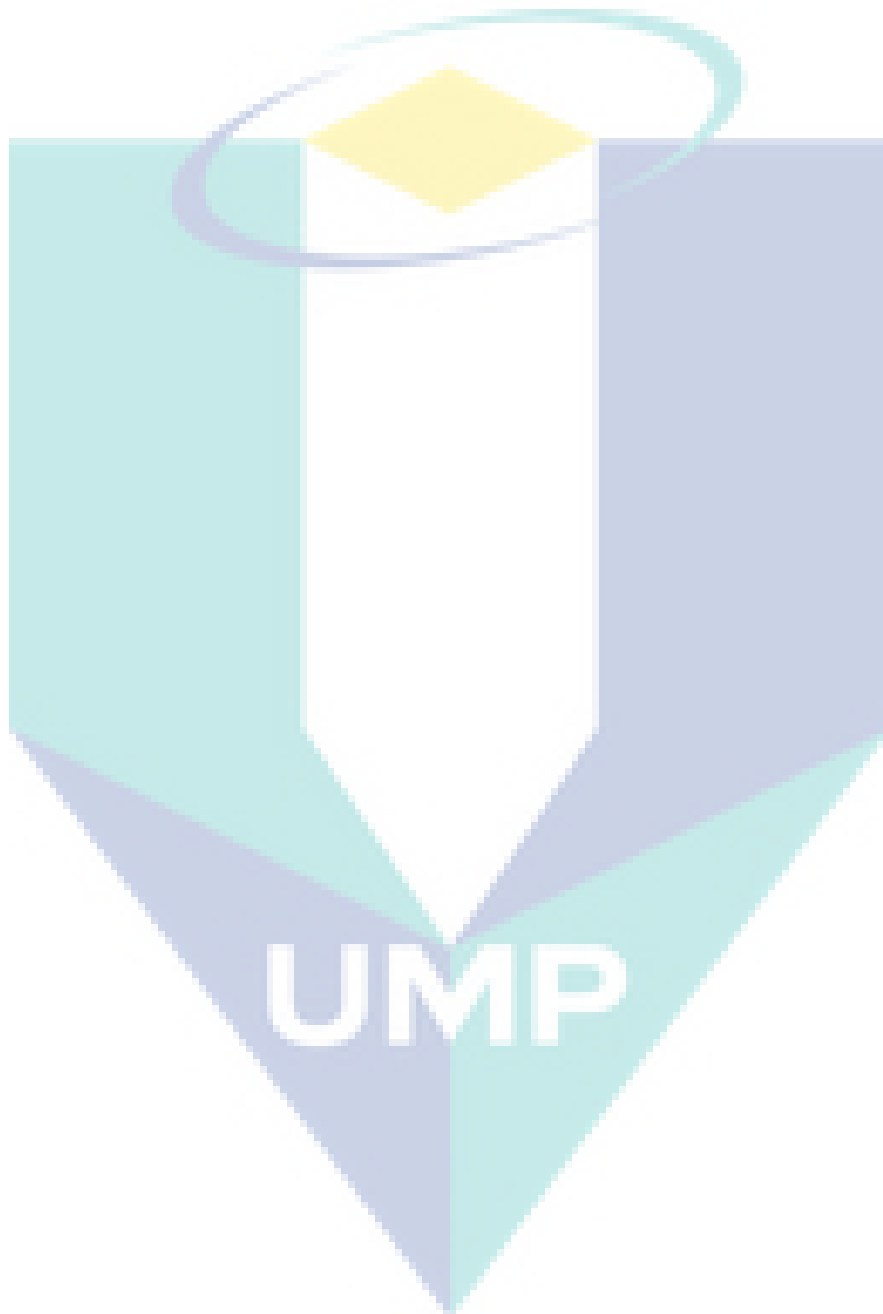


Figure 4.9: Temperature of zinc primer spray paint at bottom surface without coating

Based on table 4.12 above shows the result data for temperature of epoxy at upper surface of coating and bottom without coating. The time taken to take the data about 1 hours for both side upper and bottom surface of coated sample. Firstly, temperature were taken from bottom surface without coating, for first layer was 27 °C same for fifth layer and final temperature was 43 °C and 42 °C means higher than upper surface. Between the temperature data, each 5 minutes the temperature increased for $\pm 3^{\circ}\text{C}$. Next, for triple layers the temperature for initial was 28 °C and final was 31 °C. Besides that, figure 4.9 shows the temperature for fifth layers of epoxy at fifth layer was lower than triple and one layer. The temperature for steel at bottom surface was higher

than upper surface because heat penetrated easily into surface without coating (Bernando, 2013). The metal generally conduct heat better than solid, the electron were not stuck together but free flow among the atoms. So, thermal conductivity of metal was proportional to electrical conductivity (Department of Physics, 2012).



4.6 SEM analysis

The coated carbon steel structure was examined by using SEM analysis. When using different layers coat, the structure has changed and this will affect the behaviour and effectiveness of paint coating. Zinc is in the form of spherical (Kalendova, 2003). Zinc metal powder often added to coating material as a “sacrificial anode”. In addition, zinc metal reacts with oxygen will form zinc oxide. So, zinc oxide act as highly anti-corrosive properties.

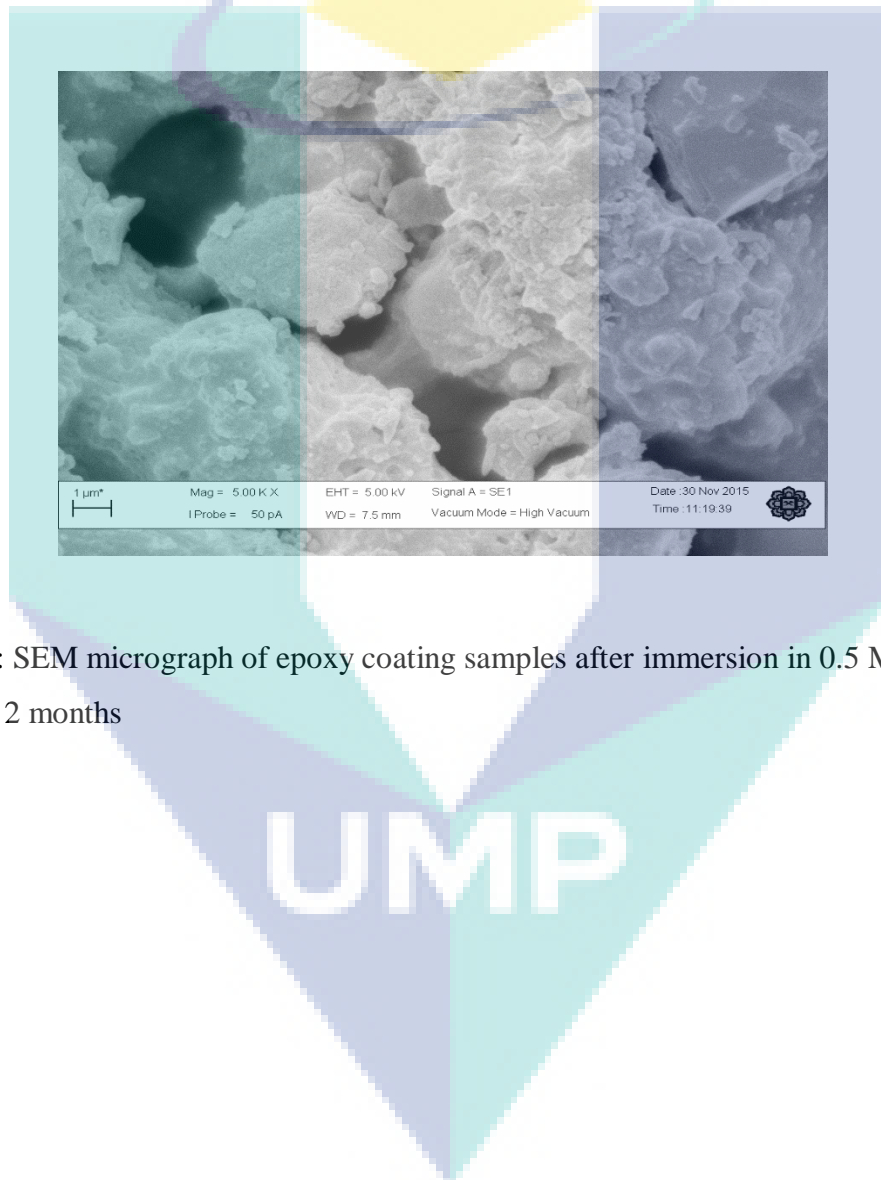


Figure 4.10: SEM micrograph of epoxy coating samples after immersion in 0.5 M NaCl solution for 2 months

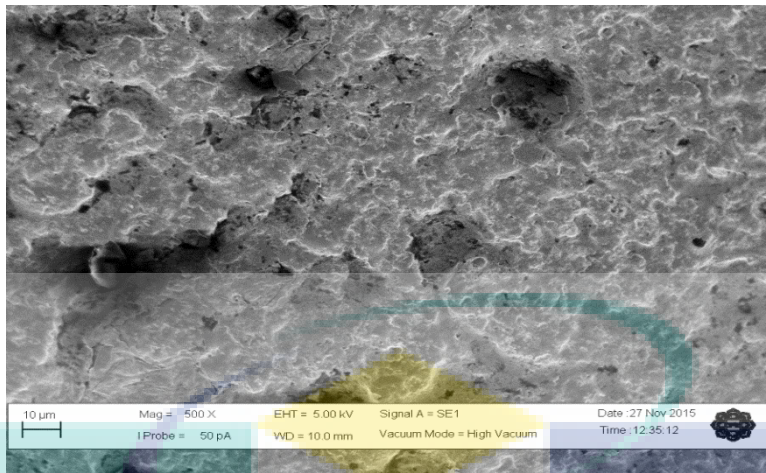


Figure 4.11: SEM micrograph of zinc-epoxy coating samples after immersion in 0.5 M NaCl solution for 2 months

UMP

CHAPTER 5

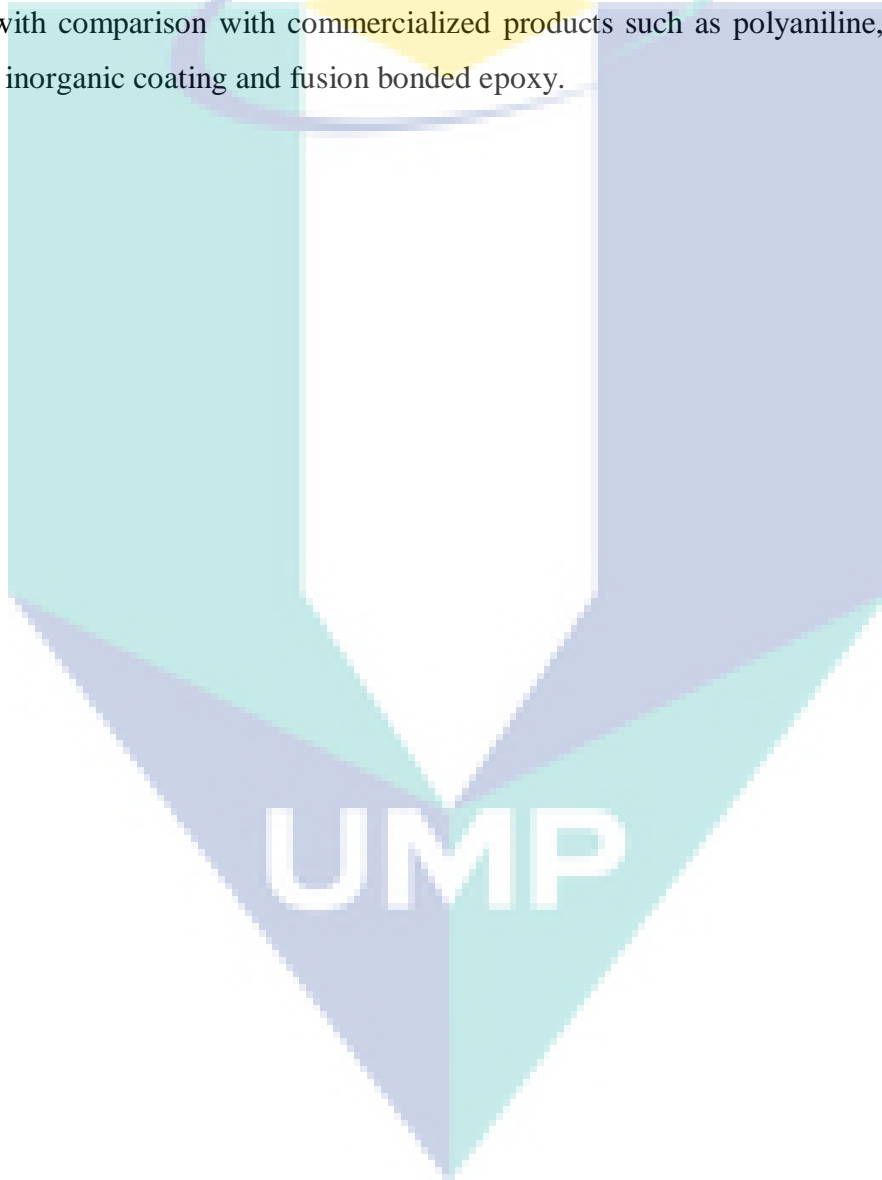
CONCLUSION & FUTURE WORKS

5.1 Conclusion

Based on research work, I achieved my objective successfully by understanding the physical appearances and behaviour of organic coating using SEM by investigate the electrochemical reaction and environment nature. There are four mechanical test involved in my research which are immersion test, adhesion test, thickness and temperature. Furthermore, the scope for this research are fulfil the requirement of objective by preparing of three sample are epoxy, epoxy composite with zinc and zinc primer spray paint for first, triple and fifth layers coating. Next, cross-cut tape test used to study the quality of adhesion by using cross-cut kits. The result shows that epoxy, epoxy composite with zinc and zinc primer spray paint for first, triple and fifth layer are good quality for adhesion test. Furthermore, the thickness are measured by weighing balance before and after. The result of weight percentage shows that epoxy and epoxy composite with zinc are gain in weight while zinc primer spray paint loss in weight. However, for triple and fifth layers of spray paint shows gain in weight. The samples are placed in hot area, using thermostat to measure the temperature surrounding and refer to Malaysian Meteorology Department. Epoxy, zinc-epoxy and zinc spray paint that coated to subtract have lower temperature than without coating. The more thicken the coating, the lower the effect of temperature surrounding. Lastly, the behaviour of coating, cracking or failure because of corrosion using SEM analysis for morphology observation and shows that zinc and epoxy acts as anticorrosive protection barrier. Therefore, this research shown improvement of commercialized products as zinc primer spray paint was used to compare with the sample.

5.2 Future works

The application work is limited by using small sample plate only for laboratory research without dealing with real parts of pipeline in plant. For more advanced, recommend to use a large plate or large pipe instead small plate immersed in sea about 2 months and see the effect of corrosion. Furthermore, in this research number of sample use are 3 organic coating which are epoxy, epoxy composite with zinc and zinc primer spray paint. Therefore, for the future work need to increase until 4 or 5 with and without composite with comparison with commercialized products such as polyaniline, hybrid organic and inorganic coating and fusion bonded epoxy.



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The logo for UMP (Universitas Muhammadiyah Purwokerto) is a large, stylized letter 'U' shape. The top part of the 'U' is a horizontal bar with a yellow center and light blue sides. The two vertical sides of the 'U' are light blue. The bottom part of the 'U' is a downward-pointing triangle, split into two colors: light blue on the left and light purple on the right. The letters 'UMP' are written in white, bold, sans-serif font across the bottom of the 'U' shape.

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