CHAPTER 1

INTRODUCTION

1.1 Background Study

Polyurea is a type of elastomer due to the reaction product of an isocyanate component and a synthetic resin blend component through step-growth polymerization. The technology of polyurea coatings and their hybrids offer the industry a variety of performance characteristics. Among the characteristics that are claimed by the industry are that polyurea is fast curing whereby it may take only 45s to cure compared to epoxy, the current normal coating used in industry.

Polyurea also complies with volatile organic compound (VOC) regulations. In other words, it is environmentally compliant. It is also high build where it provides multiple coat performance with a single application. Polyurea is believed to exhibit a lot of other excellent properties that will be discussed later in this report.

Currently the study of coating application using polyurea has not being explored much. There are some loose ends in the application of polyurea as surface coating. These setbacks however is said to be encountered for further development of the research as well as an alternative to the industry. Extensive experimental studies will be conducted such as Water Absorption Test, Pull-Off Adhesion Test and Scanning Electron Microscope (SEM) to study the characteristic and performance of coating.

1.2 Problem Statement

Polyurea is used as corrosion protection. As we all know, in oil and gas industry the usage of carbon steel is widely used. The carbon steel used needs to be protected from range of corrosion types. The carbon steel needs to be protected from range of corrosion types such as due to carbon dioxide, sulphur reduced bacteria and microbiologically induced corrosion. This is where the role of surface coating comes in, as a form of corrosion protection to various component used in engineering services. The current usage of surface coating is using Epoxy, whereby the curing time may take a long time, up until 24 hours.

However, when using polyurea, curing may take a lot less of time compared to what is instilled in the normal coating used now. Using polyurea will save a lot of time as well as energy of applying it as coating. This shows that polyurea has the capability of being an alternative approach for the industry in surface coating application. Thus, it is concluded that it is advantageous to the industry if the research is being done closely and more detail.

The project is said to be relevant as corrosion protection is essential in many kinds of industry. For instance in oil and gas industry, the usage of pipeline is very important as these pipelines will determine the flow of oil and gas and thus the financial state of a country. This shows how serious corrosion problems can be, as it involves large amount of money. Thus, a precise and accurate research on how to encounter corrosion is viable and having an alternative approach such as this is relevant and exceptional.

1.3 Objective and Scope of Study

The objective of this research is to study on the effects of water absorption at different coating thickness and environmental condition. Also, the objective is to compare polyurea with the current widely used coating epoxy on water absorption, pull-off adhesion test and analysis using Scanning Electron Microscope.

1.4 Relevance of the project

This is one year research project which includes the literature review, analytical analysis as well as experimental work. It is planned to identify the effects of water on the adhesion properties of polyurea by varying temperature and coating thickness in order to identify its resistance in water service environment that may cause corrosion.

CHAPTER 2

LITERATURE REVIEW AND THEORY

2.1 Introduction to corrosion

Definition of corrosion: 1) “Corrosion is the deterioration of a material, (usually steel), because of a reaction with its environment”. [1] 2) “The destruction of steel by an electrochemical process that is recognised by the formation of rust or pits”. [1]

“The formation of corrosion is known as an electrochemical process. This process requires 4 conditions. It is important to understand that all four of these must be present for corrosion to occur: Anode, Cathode, Metallic Pathway and Electrolyte and Oxygen.”[1]

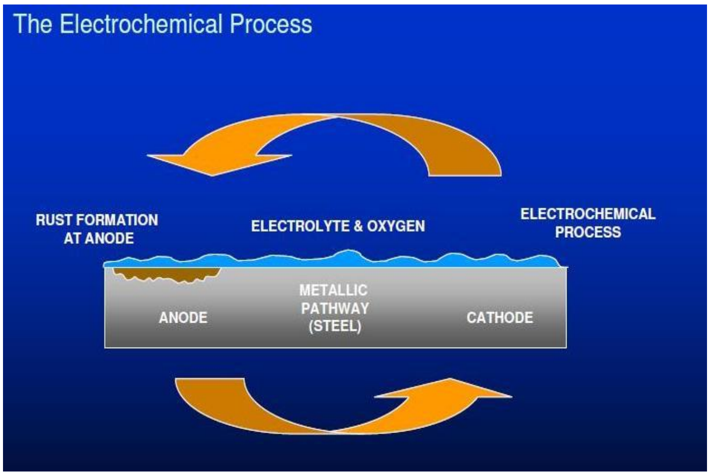


Figure 1: The Electrochemical Process

“The electrochemical process can be described as a flow of electricity. During this process the iron reacts with the electrolyte and oxygen to form iron oxides at the Anode area. Iron oxide is the chemical name for corrosion, which is a much more stable form of iron. Both the anode and the cathode must be in contact with the same electrolyte for corrosion to occur.” [1]

2.1.1 Classification of Environments

The ISO 12944 environment categories are shown below (Note: Increasing Metal loss C1 to C5)

Corrosive Category	Description
C1	Very low corrosive environment
C2	Low corrosive environment
C3	Medium corrosive environment
C4	High corrosive environment
C5-I (I = Industrial)	Very high industrial corrosive environment
C5-M (M = Marine)	Very high marine // offshore corrosive environment
Im1	Fresh water immersion
Im2	Seawater immersion
Im3	Buried in soil (underground)

Table 1: Classification of Environments. [1]

Corrosion is the destructive attack of a metal by chemical or electrical reaction with its environment.” Rusting applies to the corrosion of iron or iron-base alloys with formation of corrosion products consisting largely of hydrous ferric acid oxides. Nonferrous metals therefore corrode but do not rust. Coatings play an important role in corrosion control, used either alone or as part of an overall strategy of corrosion control. While theoretically, coatings may be employed in a variety of ways to modify, hinder, or thwart the corrosion reaction, three or four devices find practical application (see Table 2). [2]

Barrier coatings	Resistance inhibition, oxygen deprivation
Inhibitive pigments	Modification of interfacial environment (establishment of passive films)
Zinc-rich coatings	Preventions of current discharge from metal to environment by affixation of anode film

Table 2: Basic Techniques Employed in Corrosion Control by Coatings. [2]

These devices are utilized in three types of coatings employed in corrosion control. There are barrier coatings, functioning via resistance inhibition and/or oxygen deprivation, inhibitive coatings, functioning by modifying the interfacial chemical environment against the metal surface, and zinc-rich coatings, which prevent current discharge from the steel to the environment by employing a more anodically active metal (almost exclusively zinc) as a pigment at loadings high enough to ensure electrical contiguity with the substrate and low electrical resistance across the film.[2]

The form of corrosion is generally well known from the book of corrosion engineering one. Usually the different type of corrosion represent corrosion phenomena is categorized according to their appearance. Dillon considered Fontana's basic forms of corrosion and divided them into three groups based on their ease of identification. The three categorized used are:

- Group 1 : Readily can be identified by ordinary visual examination
- Group 2 : Required supplementary means of examination
- Group 3: Only can be verified using microscopic method (electron, optical and etc.)

For the industries, there are three common types of corrosion protection:

a. Coating system
b. Cathodic protection
c. Sacrificial protection

The corrosion has 3 significant effects in the study. The first one is the economic effect, due to material losses resulting from corrosion. The second one is to improve the safety of the equipment especially for critical equipment such as vessels and rotors. The third one is conservation, applied primarily to metal resource, where the resource is limited.

Failure of various kinds and the need for expensive replacements may occur even though the amount of metal destroyed is quite small.

2.1.2 Ways to overcome corrosion

1. Passive Barrier Protection

Passive barrier protection works by coating the steel with a protective coating system that forms a tight barrier to oxygen, water and salts (ions). The lower the permeability of the coating system to water, the better the protection. Two-pack epoxy coatings and chlorinated rubbers applied at sufficiently high film builds offer the most successful corrosion protection through passive barrier protection. [3]

2. Active Protection

Active corrosion protection occurs when a primer containing a reactive chemical compound is applied directly to the steel. The reactive compound disrupts the normal formation of anodes on the surface of the steel in some way. For example, inorganic zinc inhibitive pigments, such as zinc phosphate, offer active anti-corrosive protection to the steel substrate. [3]

3. Sacrificial Protection (Cathodic Protection or Galvanic Protection)

The most widely used metal for the protection of steel is zinc. Zinc metal in direct contact with the steel substrate offers protection through the preferential oxidation of zinc metal. Zinc is a great choice in protecting steel, as not only does it corrode in preference to the steel, the rate of corrosion is generally lower. This rate, however, is accelerated in the presence of ions such as chlorides in coastal locations. [3]

2.2 Introduction to coating

Construction metals are selected because of their mechanical properties and machineability at a low price, while at the same time they should be corrosion resistant. Very seldom can these properties be met in one and the same material. This is where coatings come into play. By applying an appropriate coating, the base metal with the good mechanical properties can be utilized while the appropriate coating provides corrosion protection. [4]

Definition of coating: “A clear or pigmented film-forming material that protects the surface to which it is applied from the effects of the environment”. [2] When uncoated carbon steel structures come into contact with their environment, the usual result is the formation of corrosion. Coatings are designed to help prevent and seriously slow down the corrosion process. [5]

The basic parameters that influence the performance properties of the paint films are film thickness, film density, internal stresses and pigment volume concentrations. In order to evaluate the performance properties quantitatively, an accurate determination of these parameters is essential. Since both the protective and the appearance properties of paint films are dependent to a great extent on the film thickness, its accurate estimation is essential.

All types of coating materials are composed three basic components. [5] These are:-

- Solvent
- Binder
- Pigment

Apart from that, the majority of coatings are applied on external surfaces to protect the metal from natural atmospheric corrosion and atmospheric pollution. [4]

This shows how crucial the selection of coating is in industry whereby a lot of factors need to be considered before making a choice of selection. Surface coatings is a substance applied to other materials to change the surface properties, such as color, gloss, resistance to wear or chemical attack, or permeability, without changing the bulk properties.

This includes such materials as paints, varnishes, enamels, oils, greases, waxes, concrete, lacquers, powder coatings, metal coatings, and fire-retardant formulations.

2.2.1 Mild Steel

Steel is derived from iron. Iron ore requires great thermal energy (around 1,500°C) to reduce to its metallic form of iron. The iron is then alloyed with carbon and metals such as nickel or tungsten to produce steel. [3]

Steels are described as mild, medium- or high-carbon steels, according to the percentage of carbon they contain. Mild steel is an iron alloy that contains less than 0.25% carbon. [3]

Mild steel is very reactive and will readily revert back to iron oxide (rust) in the presence of water, oxygen and ions. The readiness of steel to oxidize on exterior exposure means that it must be adequately protected from the elements in order to meet and exceed its design life. [3]

Steel is an excellent choice of building material due to its high flexural and compressive strength. It allows the design of much taller multistorey buildings and structures with wider spans because of its high strength to mass ratio. It is also lighter to transport, quick to erect and is versatile in design. [3]

2.2.2 Surface Preparation

The surface has to have the contaminants removed before applying it to the substrate. The coating may not meet the required quality standards i.e. appearance, adhesion, or corrosion resistance if contaminants are left on the surface. There are four principal ways of preparing surfaces which are:

i. Solvent degreasing. [6]
ii. Abrasion including emery paper, sand, shot, or grit blasting. [6]
iii. Chemical etching and anodizing. [6]
iv. The use of surface primers. [6]

Below are surface preparation used in this project.

1. Mechanical Cleaning – Blasting
    - Blasting with sand or grit will remove scale, rust, or previous coatings from substrate and create a surface profile that provides good coating adhesion. [7]
    - Blasting operations can be manual batch systems or automated in-line systems. Blasting systems can use high pressure nozzles or a centrifugal wheel to deliver the blast media. [7]
    - Nozzle blast systems require compressed air for delivery of the media, while a wheel system uses centrifugal force. [7]
    - Even though the compressed air is an added cost, it may be necessary to direct nozzles into hard to reach areas of a part.
2. Chemical Cleaning – Hand Wipe
    - The most inexpensive method of cleaning from an equipment perspective is manual cleaning of parts. [7]

- This would normally involve hand-wiping parts with a solvent or aqueous (water based) solution. [7]
- Manual cleaning can be somewhat effective in terms of soil removal but is very costly in terms of labor and very slow in terms of production time. [7]
- Hand wiping with a clean cloth does not require any capital investment, but it is not very effective at soil removal; it has a high labor content, is inconsistent and may create health and safety problems. [7]

2.3 Polymeric coating

2.3.1 Polyurea

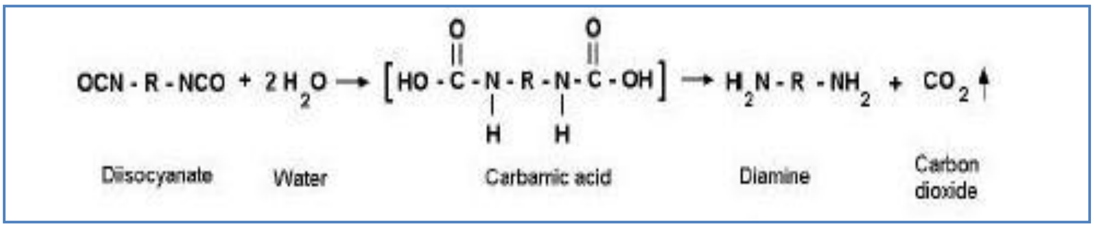
For this project, the polymer that will be used as surface coating is polyurea. The first actual reference to polyurea came in 1948 when some researchers were evaluating thermal properties / melting points of various polymer systems. They were comparing polyesters, linear polyethylene, polyurethanes, polyamides and polyureas; and, found that the polyureas had far superior thermal properties and an extremely high melting point. [8]

Polyurea were first developed in the 1970s, but the development of any practical application was impeded by their extremely short set times of 1 to 3 sec. In the early 1980s sophisticated, plural-component heated equipment was able to mix quickly and dispense the polyureas into a usable form. Throughout the 1980s and 1990s, improvements in polyurea chemistry led to products with set times that ranged from 3 sec to 25 min. [3]

Polyurea is a type elastomer that is derived from the reaction product of an isocyanate component and a synthetic resin blend component through step-growth polymerization. It is also truly a remarkable coatings, linings and joint sealant technology. It is being used successfully for so many different applications today.

The isocyanate can be monomer based, a prepolymer, a polymer or a blend. For the prepolymer, amine- and/or hydroxyl-terminated resins can be used. On the other hand, the resin blend should only contain amine-terminated resins and/or chain extenders and not any hydroxyl reactive polymer components. [9]

Polyureas, as the name suggests, are closely related to polyurethanes. Polyureas, on the other hand, require no catalyst, so they are able to cure at any temperature and in the presence of moisture. The fast curing ability of polyurea is inherent in their chemistry, which gives them several unique advantages. Because of the speed of the reaction times, polyurea systems can be sprayed on damp or wet surfaces with no detrimental reactions on performance. The polyurea elastomers can be sprayed directly on water or ice and at temperatures lower than -40F with minimal effects on tack-free times. [3]



$$\mathrm{OCN{-}R{-}NCO} + 2\,\mathrm{H_2O} \rightarrow \left[\mathrm{HO{-}\overset{\overset{\displaystyle O}{\|}}{C}{-}\underset{\underset{\displaystyle H}{|}}{N}{-}R{-}\underset{\underset{\displaystyle H}{|}}{N}{-}\overset{\overset{\displaystyle O}{\|}}{C}{-}OH}\right] \rightarrow \mathrm{H_2N{-}R{-}NH_2} + \mathrm{CO_2}\uparrow$$

Figure 2: Polyurea Chemistry. [3]

Among the claims of the properties of polyurea are:

1. Moisture Resistance – The reaction of component A and component B (polyamines) or a polyurea is so fast that the moisture reaction can’t occur. Therefore, the polyurea systems are not sensitive to moisture and humidity and do not normally produce carbon dioxide bubbles. (The A and B components react together faster than the A component can react with moisture and water. This feature is an advantage in high humidity climates or regions. [10]
2. Spraying over wet surfaces – Another claim in the industry is that the polyureas can be sprayed on moist or wet substrates without any foaming, moisture reaction or detrimental effect. Part of this claim is true. However, there could be detrimental results in regards to adhesion. It should be fairly obvious that if there is a substantial amount of water or moisture the adhesion will be compromised. [10]

3. Fast Cure Properties – Polyurea systems are being promoted as fast cure, which is true, and they can gel as fast as 3-10 seconds. The polyurethane can be catalyzed as fast as 5 seconds but when necessary can be slowed down to a few minutes or more. Both systems can be considered fast gel/cure systems and can be used to increase fabrication or production efficiency in all applications. However, it is important to note that sometimes a polyurea can be too fast and may not produce the results required for a specific application. [10]
4. Fast Cure Surface Finish – The polyurethane reaction profile can be adjusted to achieve an aesthetically smooth gloss finish. Most polyurea are so fast that the surface has a rough orange peel texture to it. [10]

Polyurea coating used for this project is according to PTS Standards. The surface preparation is done according to Sa 2.5. Coating thickness per application is 500microns. For normal application is 1000microns. For harsh environment i.e. sub sea, sub surface, acid protection, heat, etc. thickness used is normally 2000microns.

2.3.2 Epoxy

Epoxy or polyepoxide is a thermosetting epoxide polymer that cures (polymerizes and crosslinks) when mixed with a catalyzing agent or "hardener". Most common epoxy resins are produced from a reaction between epichlorohydrin and bisphenol-A. [11]

The applications for epoxy based materials are extensive and include coatings, adhesives and composite materials such as those using carbon fiber and fiberglass reinforcements. The chemistry of epoxies and the range of commercially available variations allows cure polymers to be produced with a very broad range of properties. [11]

In general, epoxies are known for their excellent adhesion, chemical and heat resistance, good to excellent mechanical properties and very good electrical insulating properties, but almost any property can be modified (for example silver-filled epoxies with good electrical conductivity are available, although epoxies are typically electrically insulating).[11]

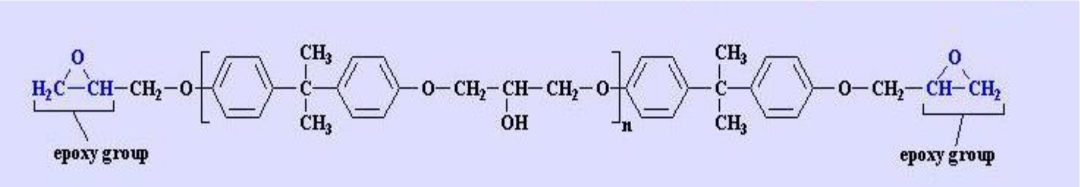


Figure 3: Structure of Epoxy. [11]

Epoxy adhesives can be developed that meet almost any application. They are exceptional adhesives for wood, metal, glass, stone, and some plastics. They can be made flexible or rigid, transparent or opaque/colored, fast setting or extremely slow. Epoxy adhesives are almost unmatched in heat and chemical resistance among common adhesives. In general, epoxy adhesives cured with heat will be more heat- and chemical-resistant than when cured at room temperature. [11]

In the unhardened state the chemical structure, from which the epoxy adhesive get its name, is characterised by the ring-like shape of the epoxide group. The ring structure is broken by an active molecule – typically an amine – and the two monomers link. The constant repetition of this process hardens the adhesive by forming a polymeric mass. This mechanism requires exact quantities of resin and hardener, hence the need for precise mix ratios and the thorough mixing of resin and hardener. [6]

2.4 Water absorption on polymeric materials

Definition of water absorption: 1) “The amount of water absorbed by a composite material when immersed in water for a stipulated period of time.”[10] 2) “The ratio of the weight of water absorbed by a material, to the weight of the dry materials. All organic polymeric materials will absorb moisture to some extent resulting in swelling, dissolving, leaching, plasticizing and/or hydrolyzing, events which can result in discoloration, embrittlement, loss of mechanical and electrical properties, lower resistance to heat and weathering and stress cracking.”[12]

Water absorption is used to determine the amount of water absorbed under specified conditions. Among the factors affecting water absorption are type of plastic (polymer), additives used, temperature and length of exposure.

According to ASTM D570 – 98 (2005) Standard Test Method for Water Absorption of Plastics, the test method covers the determination of the relative rate of absorption of water by plastics when immersed. The test method also intended to apply to the testing of all types of plastics, including cast, hot-molded, and cold-molded resinous products, and both homogeneous and laminated plastics in rod and tube form and in sheets 0.13mm [0.005in.] or greater in thickness.[13]

The procedure starts with the specimens being dried in an oven for a specified time and temperature and then being cooled prior to weighing. The weight data is then being taken followed by the specimens being emerged in water at agreed upon conditions i.e. 23°C for 24 hours. The specimens are then being removed, patted dry with lint free cloth and weighed. [13]

For testing, the specimens are dried and cooled. Three testing procedures are commonly employed. Only data from the same testing procedures are readily comparable. [15]

Procedure	Test Description
Water Absorption @ 24 hrs	Test specimens are immersed in distilled water at a specified temperature for 24 hours. Testing is most commonly done at 23°C (73.4°F)
Water Absorption @ Saturation	Test specimens are immersed in distilled water at a specified temperature until the water absorption essentially ceases.
Water Absorption @ Equilibrium	Test specimens are exposed to a humid environment at a specified temperature for 24 hours. Testing is most commonly performed at 50% relative humidity (RH) and 23°C (73.4°F).

Table 3: Examples of Testing Procedures. [15]

Water absorption is expressed as increase in weight percent.

$$\text{Percent Water Absorption} = [(\text{Wet weight} - \text{Dry weight})/\text{Dry weight})] \times 100\%$$

The percent increase in weight of a material after exposure to water under specified conditions. Water absorption can influence mechanical and electrical properties. [15]

2.5 Fick's Second Law

The rate of moisture diffusion is adequately described by Fick's second law of diffusion or Fick's Law with constant diffusion coefficient. [17]

$$\frac{\partial c}{\partial t} = D \frac{\partial^2 c}{\partial x^2} \qquad \text{Equation 2.5.1}$$

Fickian describe the non-steady state diffusion of a substance in Cartesian coordinate, x, y, z.

$$\frac{\partial c}{\partial t} = D\left[\frac{\partial^2 c}{\partial x^2} + \frac{\partial^2 c}{\partial y^2} + \frac{\partial^2 c}{\partial z^2} \right] \qquad \text{Equation 2.5.2}$$

Where c is the concentration in dimensions of [(amount of substance) length^{-3}].[mol m^{-3}], t is time [s], D is the diffusion coefficient in dimensions of [length^2 time^{-1}], [m^{2}s^{-1}], X is the position [length], [m]

The analytical solution of Equation (2.5.1) for the concentration profiles of a diffusing substance in an isotropicplane sheet of finite thickness can be described as a function of time, t and distance, x:

$$\frac{c_{x,t}}{c_\infty} = 1 - \frac{4}{\pi} \sum_{m=0}^{\infty} \frac{1}{2m+1} \exp\left[\frac{D\,(2m+1)^2}{h^2} \pi^2 t \right] \sin\left[\frac{(2m+1)\pi x}{h} \right] \qquad \text{Equation 2.5.3}$$

where c_∞ is the saturation concentration of the absorbed substance. Upon solving Equation (2.5.1) with the following boundary conditions $c = 0$ when $t = 0$, $0 \leq x \leq h$, $c = c_\infty$ when $t = h$, $\partial c/\partial x = 0$, $t > 0$, the relative moisture uptake is expressed as:

$$\frac{M_t}{M_\infty} = 1 - \sum_{n=0}^{\infty} \frac{8}{(2n+1)^2\pi^2} \times \exp\left[- \frac{D(2n+1)^2\pi^2}{h^2} t \right] \qquad \text{Equation 2.5.4}$$

Where M_t is the mass gain at reduced time and M_∞ is the maximum mass gain at the equilibrium state. At the initial stage of diffusion, Equation (2.5.4) can be approximated by:

$$\frac{M_t}{M_\infty} = 4 \left[\frac{Dt}{\pi h^2} \right]^{1/2} \qquad \text{Equation 2.5.5}$$

Therefore, the diffusivity D can be calculated from initial slope of moisture gain, M_t/M_∞ versus time ($t_{1/2}$).

$$D = \frac{\pi}{16} \left[\frac{\mathrm{Mt}/M_\infty}{\sqrt{t/h}} \right] \qquad \text{Equation 2.5.6}$$

A relationship has been developed to predict the moisture content that has a form similar to Equation (2.5.5)

$$\frac{M_t}{M_\infty} = 1 - \exp\left[-7.3\left(\frac{Dt}{h^2}\right)^{0.75} \right] \qquad \text{Equation 2.5.7}$$

The rate of diffusion depends on the structure, melting temperature, bonding, size of diffusing atoms and density. Diffusion is faster in open crystal structures, low melting temperature materials, materials with secondary bonding, small diffusing atoms and low density materials.

2.6 Pull-off Adhesion Test

The equipments used in this testing are dolly, adhesive, substrate and adhesion tester. The thickness of the coating will be varied in this test. Firstly, the fixture (dolly/stub) is affixed by an adhesive coating. The load will then be applied to the surface increasingly till the dolly is pulled off.

The best repeatability can be obtained when the pull-off acts perpendicular to the surface tested. The force required to pull the dolly or withstand it will yield the tensile strength. Failure will occur along the weakest plane and will be exposed by the fractured surface shown after the test.

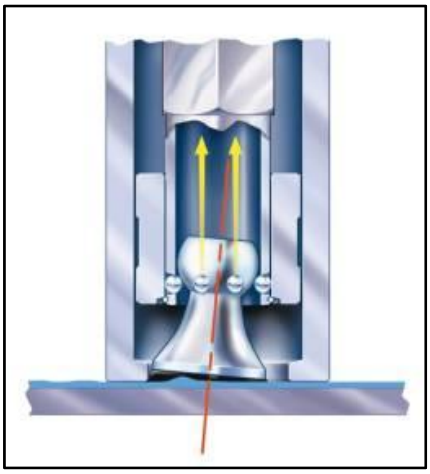


Figure 4: Cross sectional view of actuator in Pull-off Adhesion Testing

2.7 Scanning Electron Microscope

The scanning electron microscope (SEM) is a microscope that uses electrons rather than light to form an image. There are many advantages to using the SEM as an adjunct to the optical (light) microscope. The SEM has a large depth of field, which allows a large amount of the sample to be in focus at one time.

The SEM also produces images of high resolution, which means that closely spaced features can be examined at a high magnification. Preparation of the samples is relatively easy since most SEMs only require the sample to be electrically conductive.[16]

For conventional imaging in the SEM, specimens must be electrically conductive, at least at the surface, and electrically grounded to prevent the accumulation of electrostatic charge at the surface.

Conductive materials in current use for specimen coating include gold, gold/palladium alloy, platinum, osmium, iridium, tungsten, chromium and graphite. Coating prevents the accumulation of static electric charge on the specimen during electron irradiation. [18]

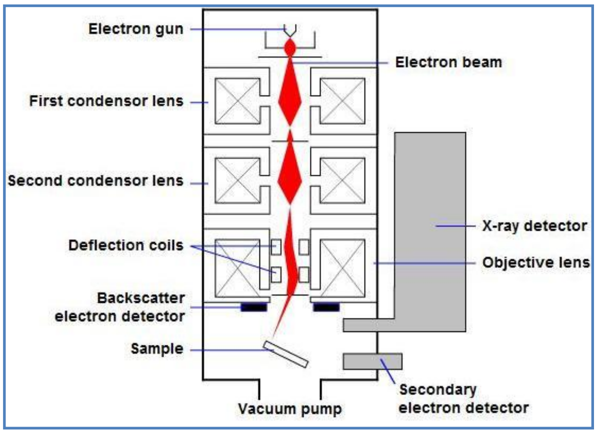


Figure 5: Schematic Diagram of SEM. [18]

CHAPTER 3

METHODOLOGY

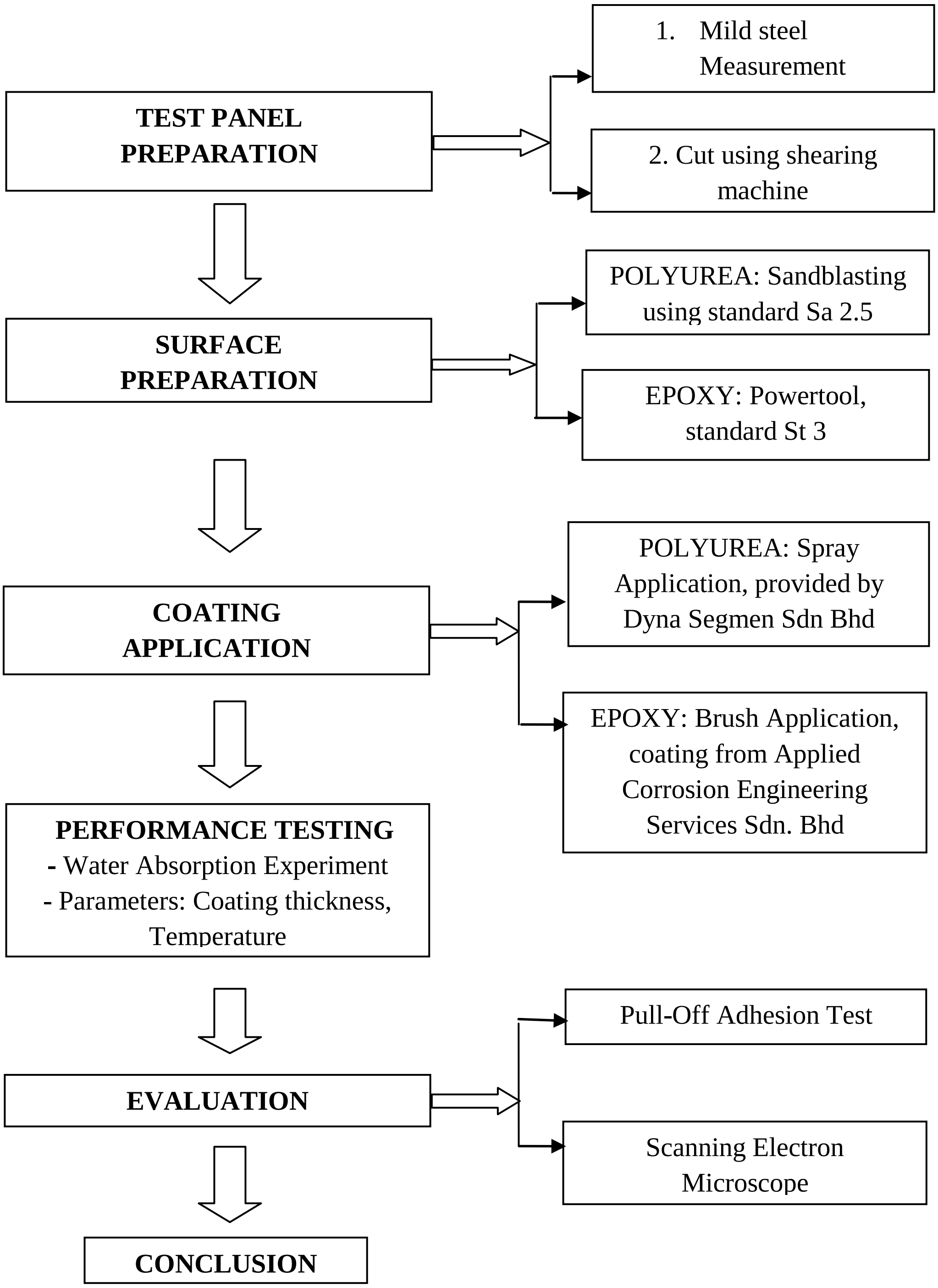


Figure 6: Methodology Flow Process

3.1 Test Panels preparation

3.1.1 Steel Preparation

1. Carbon steel (1m X 1m) is being measured into specified requirement.
2. Next, it is been cut into small specimens (mainly 10cm X 10cm).

3.1.2 Surface Preparation

A. Polyurea
   1. Polyurea coated steels are prepared by sand blasting of standard Sa 2.5 using sand blasting equipment.

B. Epoxy
   1. The test panels' surface was cleaned using water soluble degreaser. Then, it is followed by fresh water and allowed to dry to remove any obvious dirt, salt, chemical or grease contamination.
   2. Manual cleaning onto the test panels surface by using powertool (St 3) was applied until scales were removed.
   3. The new surface is cleaned using fresh water and allowed to dry.
   4. The new surface profiles of the manually cleaned surface were measured to determine the average roughness. The lower the surface average roughness will give a lesser effective of the surface profile to the paint bonding. Note that the low quality of surface profile will affect the bonding between paint layer and the surface metal which will also affect the testing involved in this project i.e. adhesion test.

3.2 Coating application

1. Coating being applied with thickness of 1000microns, 2000microns and 3000microns.
2. Application is according to PTS Standards.

3.2.1 Polyurea coating

a. Polyurea coating is being applied by using Spray application.
b. Coated steel is provided by Dyna Segmen Sdn Bhd

3.2.2 Epoxy coating

1. Epoxy coating is being applied by using Brush application.
2. Coating is been provided by Applied Corrosion Engineering Services Sdn

3.3 Performance and Corrosion Testing.

3.3.1 Water Absorption

1. Each sample thickness is measured and recorded.
2. Sample is then divided into respective thickness (i.e. 1000microns, 2000microns and 3000microns) and condition (23°C and 50°C).
3. The initial weight of the sample is measured and recorded.
4. The samples are then immersed in water at room temperature and 50°C.
5. The samples are taken out from water for every 24 hours for weighing until constant value is obtained. The weights are recorded.
6. Steps (3) to (5) is used with other samples too.

3.3.2 Pull-off Adhesion Test

1. The surface of the dolly and the coating are prepared where the dolly is to be applied by roughening with an abrasive paper.
2. These areas are then de-greased by using a suitable solvent to clean both surfaces.
3. A small quantity of adhesive is mixed and an even film is applied onto the prepared surface of the dolly.
4. The dolly is placed onto the prepared test surface and pressure is applied to squeeze out excess adhesive which should then be removed.
5. The adhesive is allowed to cure for the recommended time.
6. Around the base of the dolly is cut very carefully using the dolly cutter. This is only necessary when lateral bonding in the coating is greater than adhesion, for example, elastomeric coatings.

7. The base support ring is placed over the dolly ensuring that it lies flat on the surface.
8. The handwheel or nut is slackened on the Adhesion Tester. The dragging indicator is set to zero (0) on the scale and the claw is engaged carefully with the dolly.
9. The Adhesion Tester is held steadily with one hand to prevent rotation and the handwheel or nut is tightened slowly and evenly to apply an increasing force to the dolly and hence to the coating.
10. Continue until the coating fails and the dolly is removed from the surface, or until the specified test force is indicated. The pull-off force is read from the position of the dragging indicator after the test is complete.

3.4 Scanning Electron Microscope

1. Sample heated in oven at 100°C to remove water, solvent, or other materials that could vaporize while in the vacuum.
2. The duration of heating is approximately 10 minutes.
3. The samples are mounted firmly in the small chamber.
4. The sample is ready to be placed in SEM.
5. The fractured surfaces were examined in the SEM and the changes of the coating were observed and studied.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Preparation of samples

The preparations of samples include the steel preparation, surface preparation and coating application. For polyurea, the coating application was done by company, Dyna Segmen Sdn. Bhd. Also, epoxy coating was applied to the steel, whereby it serves as a comparison with polyurea coated specimens.

Figure 7: Polyurea coated specimens

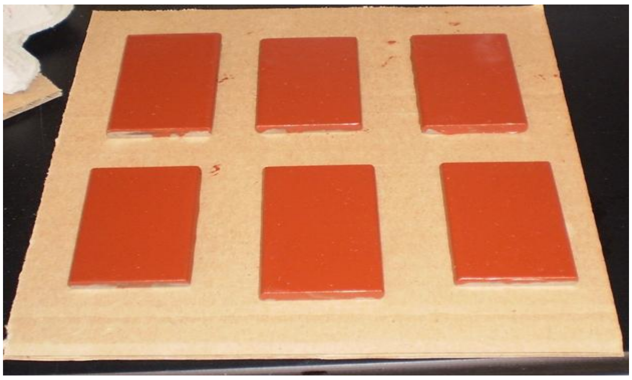


Figure 8: Epoxy coated specimens

4.2 Water absorption of Polyurea

The samples were submerged in the water basin (at 23°C) and water bath (50°C) for 15 days. The Percentage of Water Uptake versus Days graphs were plotted.

From the graph shown, sample with coating thickness of 1mm/1000microns absorbed the lowest percentage of water. This is followed by 2mm and 3mm. The trend of the graph also shows that polyurea is prone to water absorption whereby it absorbs a lot of water in a short time.

Based on literature research, polyurea has a high absorption on water. Water absorption may limit the protection of substrate against corrosion. Thus, in order to decrease water absorption inside a polymeric material, an extensive research to produce an isocyanate pre-polymer with enhanced hydrophobic character is done. This is to enhance the water proofing capabilities of surface coating polyurea.

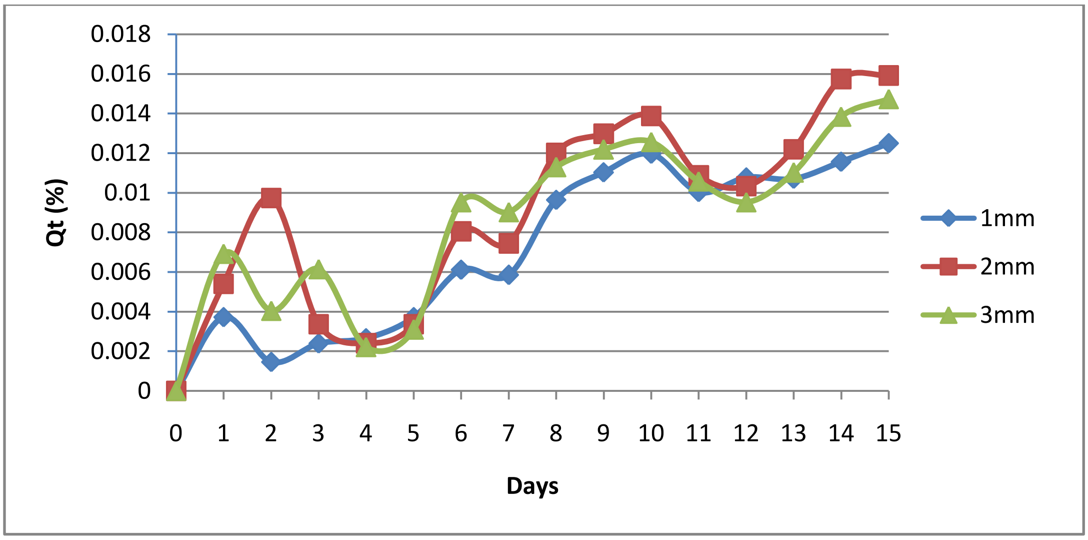


Figure 9: Percentage of Water Uptake, Qt(%) versus Days at 23°C

Below graph shows the percentage of water uptake at 50°C. The trend line shows a similar pattern. However, graph below shows a steeper slope compared to water uptake at 23°C. This means that the absorption happens at a much higher rate than at 23°C. Highest absorption also happens at a higher temperature as can be seen in the graph.

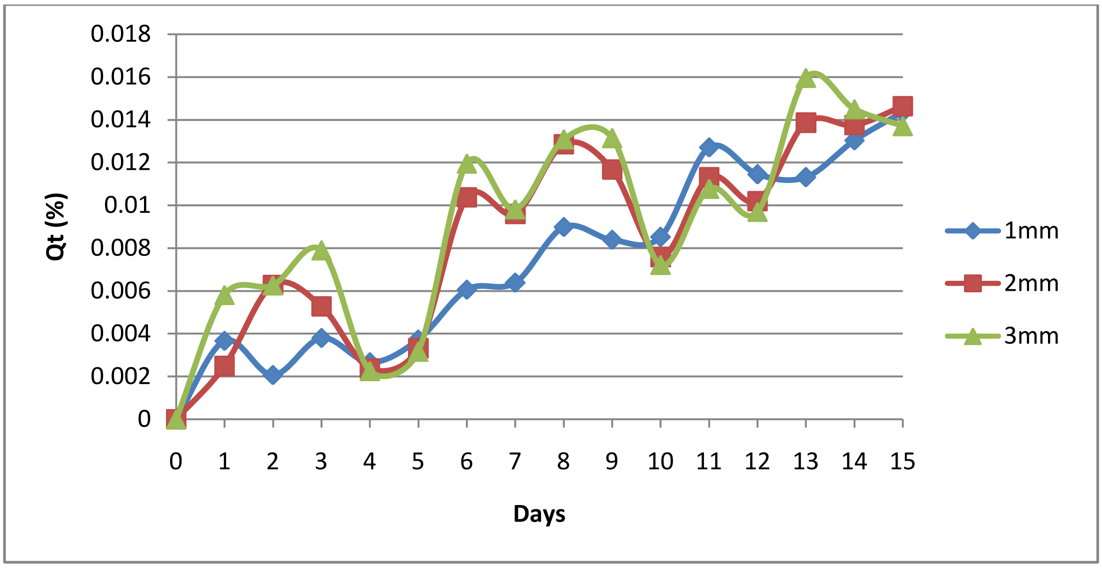


Figure 10: Percentage of Water Uptake, Qt(%) versus Days at 50°C

The next graph shows the difference of trendline between two specimens of 1mm coating thickness submerged in a different condition. However, the trendline difference is small due to short time period of experiment. In order to have a better accuracy, a longer time of experiment should be done.

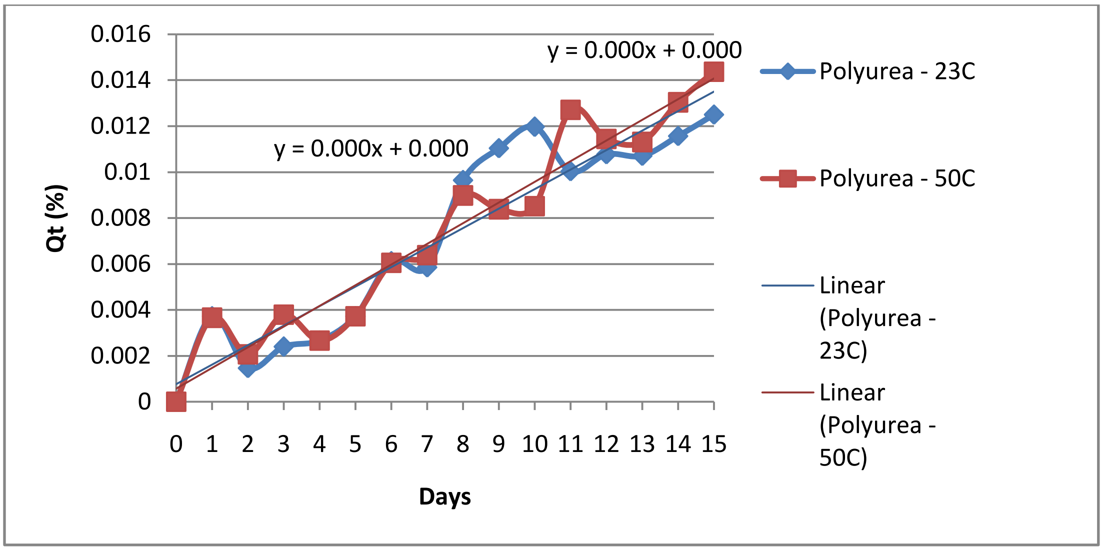


Figure 11: Trendline comparison of Qt(%) versus Days for 1mm coating

Comparison with Epoxy Coating

Based on the graph below, it shows that the absorption rate of polyurea is higher than epoxy. This can be seen by the percentage water uptake of polyurea where it is more than epoxy. The slope of graph for polyurea is steeper, which indicates that the rate of absorption is higher. This shows that polyurea is more prone towards loss of adhesion compared to epoxy. High water absorption to some extent will cause other failures such as loss of mechanical properties, lower resistance to heat and weathering and corrosion to the specimens.

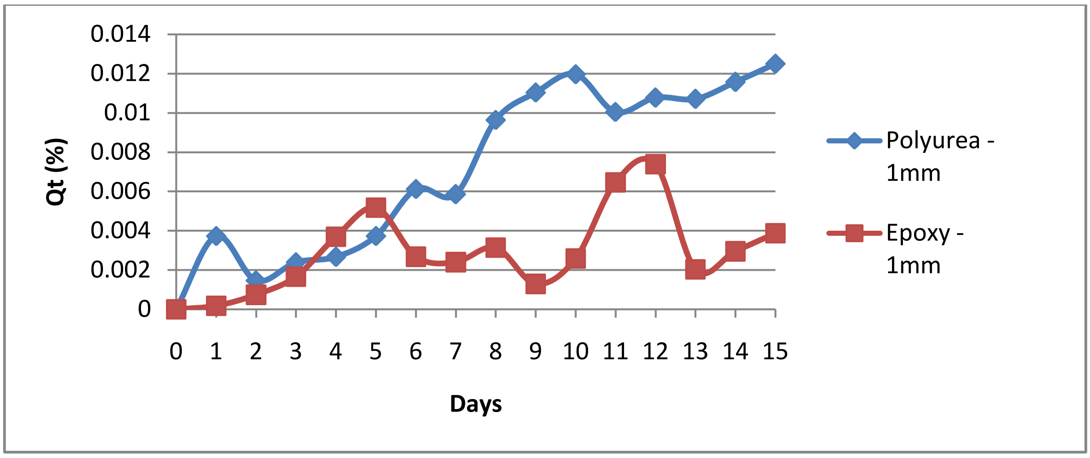
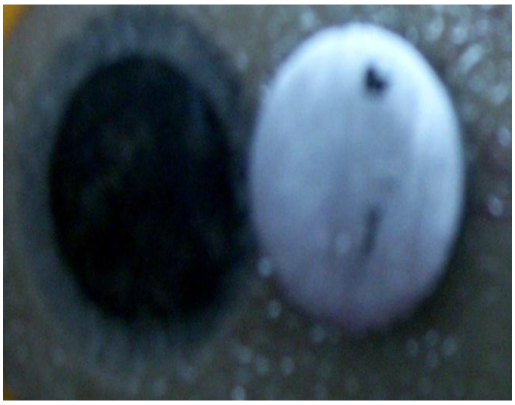
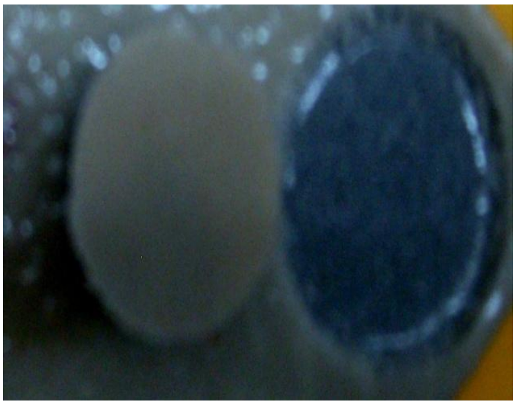


Figure 12: Polyurea and Epoxy - Percentage of Water Uptake, Qt(%) versus Days at 23°C

4.3 Pull Off Adhesion Test

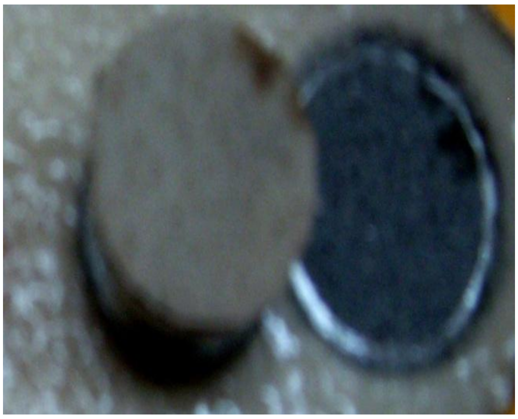
4.3.1 Polyurea – Sample A1

a. Thickness: 1mm coating
b. Immersed at 23°C

 Section 1 - No coating present on the dolly - 100% Glue Failure	 Section 2 - 3Mpa / N/mm^2

4.3.2 Polyurea – Sample A2

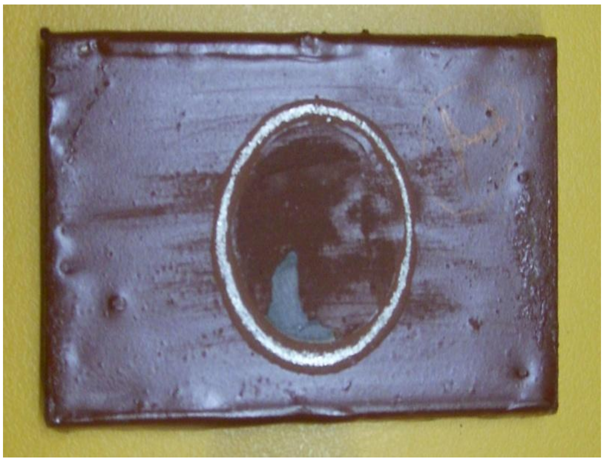
a. Thickness: 1mm coating
b. Immersed at 50°C

 Section 1 - 2Mpa / N/mm^2	 Section 2 - No coating present on the dolly - 100% Adhesive Failure

4.3.3 Epoxy

a. Thickness: 1mm coating

b. Immersed at 23°C



- Partial Cohesive Failure
- 5 MPa / N/mm^2

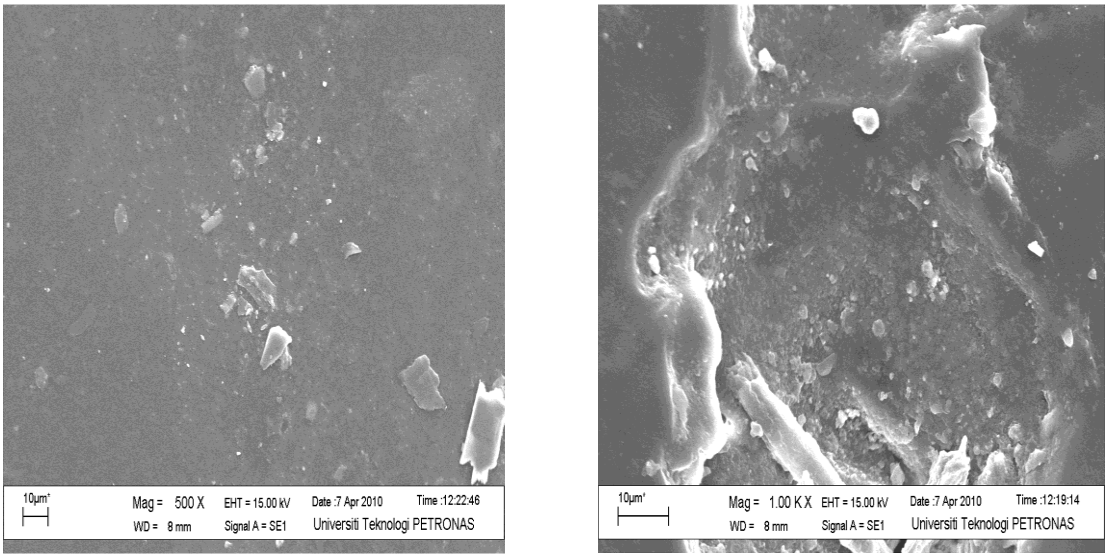
3. Epoxy
    a. Thickness: 1mm coating
    b. Immersed at 50°C

- Partial Cohesive Failure
- 30Mpa / N/mm^2

4.4 Scanning Electron Microscope

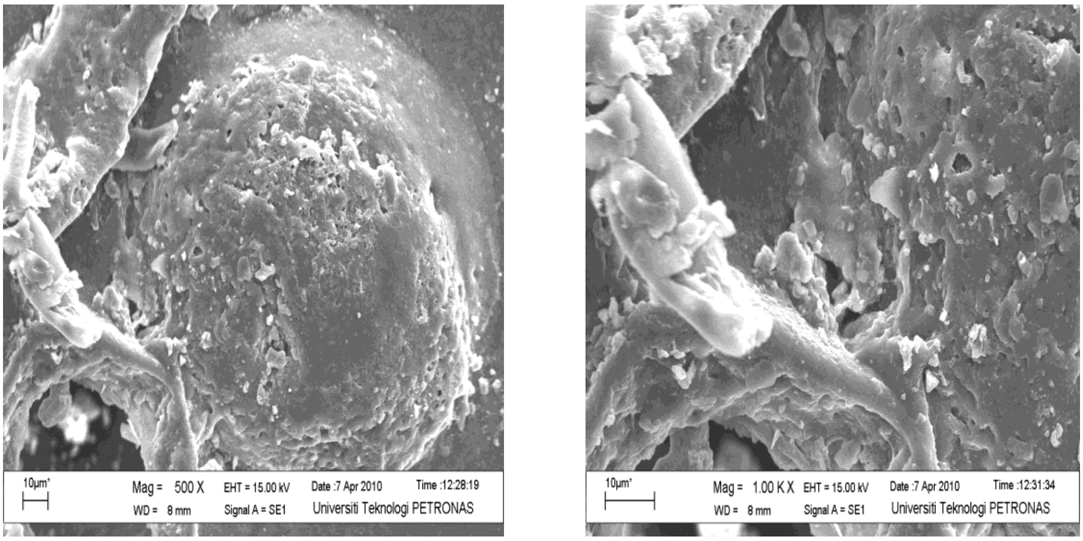
4.4.1 Polyurea coating

a. 1mm at 23°C



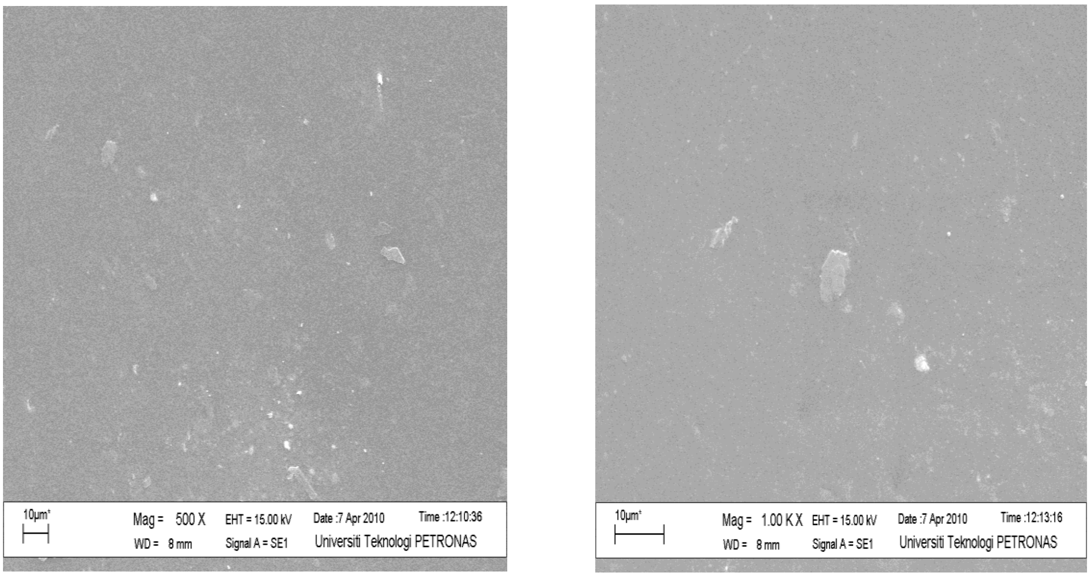
Thickness of 1mm will result in a mild absorption on the coating. Due to the insufficient thickness, it may encourage corrosion to happen to the steel provided it is not fully protected by the coating. Normal environmental condition will also give a mild absorption, whereby the effects can only be seen slightly. The thickness and environmental condition plays an important role in order to compare coating performance.

b. 1mm at 50°C



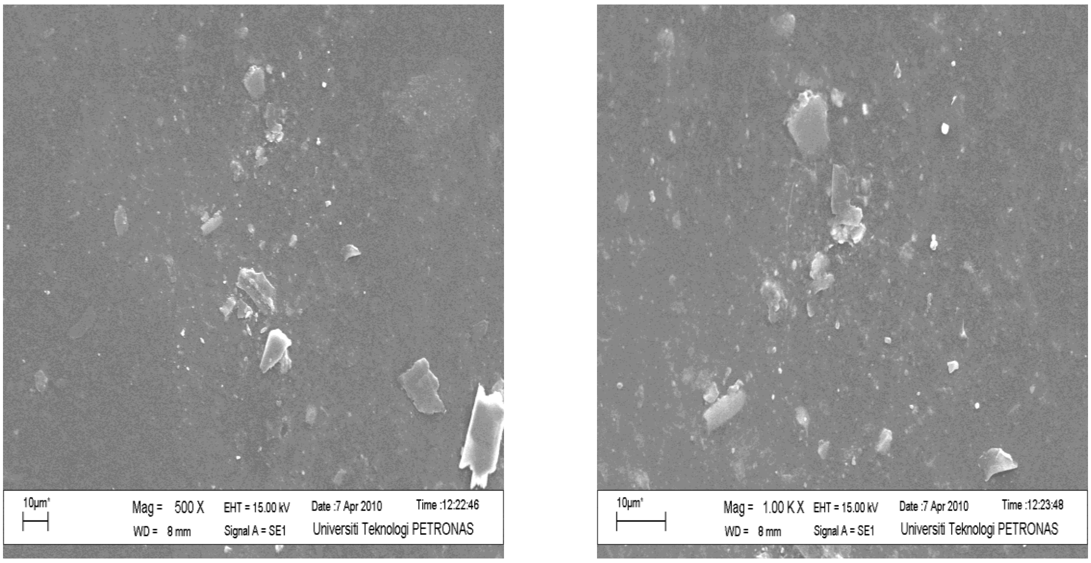
For coating thickness of 1mm at higher temperature, it can be seen that the coating layer is affected due to harsher condition prior to this sample. The surface structure shows defects of coating. The variations of colours i.e. light and dark colour in the scan shows the undistributed thickness of coating throughout the sample.

c. 2mm at 23°C



For coating thickness of 2mm at 23°C, the surface is clear and can only be seen a mild defect. This shows that the thickness of 2mm is the optimum thickness at room temperature to be used as it can withstand the condition for quite a long time.

d. 2mm at 50°C



With coating thickness of 2mm at 50°C, the surface also shows a mild effect. At a harsher environment, the coating shows a slight effect on the coating layer compared to at normal condition. For higher temperature condition, it is recommended to have a higher coating thickness, if not a better controlled application of coating onto the steel.

4.5 Discussion

Based on the water absorption graphs, high absorption rate and amount of water absorbed in polymeric material can be seen at larger thickness of coating and at high temperature (50°C). A comparison on water absorption between polyurea and epoxy shows that rate of absorption in polyurea is higher than epoxy. This indicates that polyurea is more prone towards loss of adhesion compared to epoxy.

Loss of adhesion may cause a lot of problems particularly in industry. As mentioned earlier, coating is used to overcome corrosion problems, namely pipelines regardless of the location (land, under ground, or seabed). Coating used for the pipelines is a type of polymer which exists in chain and consists of components such as C, H and O. The problem may rise due to water absorption because this indicates that the polymer may have unreacted species inside it. Some parts of the polymer may not react and form a bond which is referred to the unreacted species. The diffusion of such components i.e. Carbon may bring harm to the environment. In terms of marine life, it may endanger the marine species as such release may increase the acidity of ocean and thus, reduces the pH of ocean. Meanwhile, high release of these components may also increase the toxicity and harm the human's health.

The result of pull-off adhesion test shows that higher bond strength in epoxy compared to polyurea. This may due to failed coated edges of polyurea specimens, which speed up the corrosion at the edges of specimen. However, in terms of the surface of polyurea specimen, it is not corroded compared to epoxy coating. This indicates that polyurea has high bond strength provided that a better coating application is done.

The SEM analysis verifies the previous testing results whereby a suitable application of polyurea is 2mm of coating when considering high temperature environment.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

1) High water absorption can be seen in polyurea (slope 0.0008x) compared to epoxy (slope 0.0002x). Thus, Polyurea is more prone towards loss of adhesion compared to epoxy.

2) High absorption in polyurea also indicates a possibility of high release of unreacted components inside polyurea, which may harm the environment.

3) Spray versus Brush Application – Brush application still gives good results when being compared to Spray application.

4) Sa 2.5 versus St 3 – St 3 surface preparation also gives good results when being compared to Sa 2.5.

4) However, the bond strength of polyurea is also high (referring to pull-off adhesion test) which indicates that a good future prospect of the coating as to become one of the options to be used in industry.

5.2 RECOMMENDATION

It is recommended that an extended analysis of coated steels by utilizing extraction to verify the content of unreacted species inside the polymer is done. Apart from that, a more careful procedure in coating is important in order to get a more accurate data for analysis. In addition, time allocation of water absorption should be longer in order to have a better accuracy of results.

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APPENDICES

APPENDIX I

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
A1	151.02	150.68	150.82	150.68	150.82	151.38	151.34	151.91	152.12	152.26	152.12	152.26	152.07	152.2	152.34
B1	167.38	168.1	167.04	168.1	167.04	167.82	167.72	168.48	168.64	168.79	168.64	168.79	168.51	169.1	169.13
C1	181.85	181.33	181.71	181.33	181.71	182.32	182.23	182.64	182.8	182.87	182.8	182.87	182.59	183.1	183.26
A2	150.89	150.65	150.91	150.65	150.91	151.25	151.3	151.69	151.6	151.69	151.6	151.69	152.04	152.3	152.5
B2	169.2	169.84	169.67	169.84	169.67	170.53	170.4	170.95	170.75	170.95	170.75	170.95	171.12	171.1	171.25
C2	178.31	178.39	178.68	178.39	178.68	179.4	179.02	179.6	179.61	179.6	179.61	179.6	180.11	179.85	179.71
D1	108.34	108.4	108.5	108.4	108.5	108.61	108.58	108.66	108.46	108.6	108.46	108.6	108.54	108.64	108.74
D2	107.91	107.9	107.95	107.9	107.95	108.25	108.05	107.93	108.06	108.9	108.06	108.9	108.11	108.14	108

Table 4: Weight Sample after submerge for 15 Days

Note:

A = 1mm

B = 2mm

C = 3mm

1 = At 23°C

2 = At 50°C

APPENDIX II

Figure 13: Measuring steel

Figure 14: Shearing Machine

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Figure 15: Surface Preparation of steel

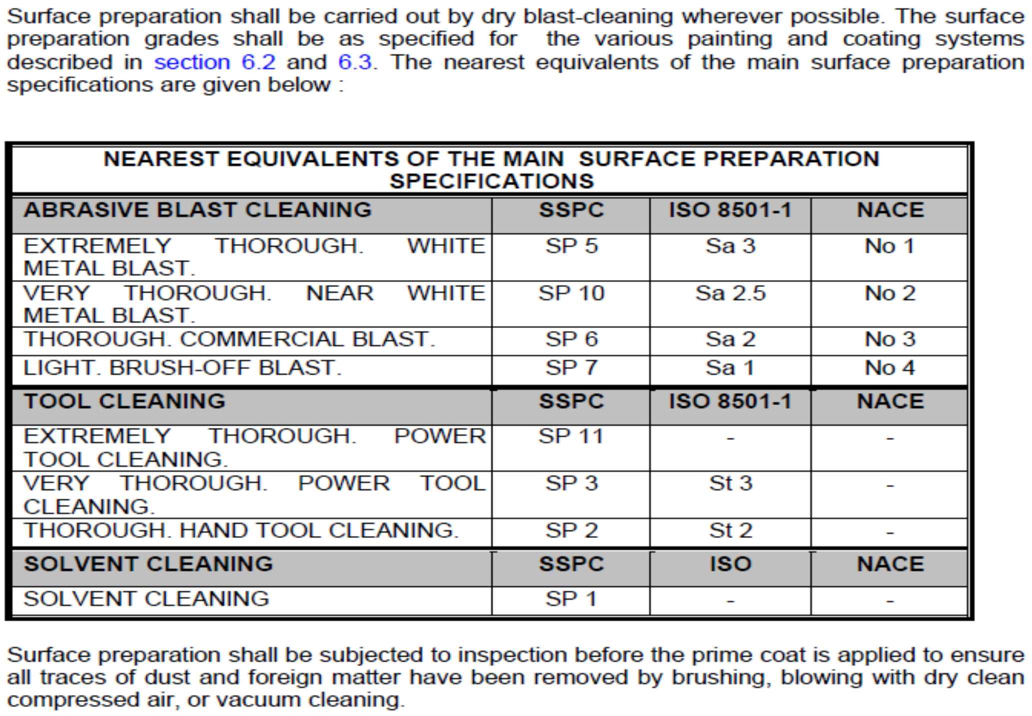
Figure 16: Polyurea Coating Equipment

Figure 17: Grinding polyurea coated steel

Figure 18: Thickness Gage

Figure 19: Water Bath

Figure 20: Pull-off Adhesion Tester



Surface preparation shall be carried out by dry blast-cleaning wherever possible. The surface preparation grades shall be as specified for the various painting and coating systems described in section 6.2 and 6.3. The nearest equivalents of the main surface preparation specifications are given below :

NEAREST EQUIVALENTS OF THE MAIN SURFACE PREPARATION SPECIFICATIONS			
ABRASIVE BLAST CLEANING	**SSPC**	**ISO 8501-1**	**NACE**
EXTREMELY THOROUGH. WHITE METAL BLAST.	SP 5	Sa 3	No 1
VERY THOROUGH. NEAR WHITE METAL BLAST.	SP 10	Sa 2.5	No 2
THOROUGH. COMMERCIAL BLAST.	SP 6	Sa 2	No 3
LIGHT. BRUSH-OFF BLAST.	SP 7	Sa 1	No 4
TOOL CLEANING	**SSPC**	**ISO 8501-1**	**NACE**
EXTREMELY THOROUGH. POWER TOOL CLEANING.	SP 11	-	-
VERY THOROUGH. POWER TOOL CLEANING.	SP 3	St 3	-
THOROUGH. HAND TOOL CLEANING.	SP 2	St 2	-
SOLVENT CLEANING	**SSPC**	**ISO**	**NACE**
SOLVENT CLEANING	SP 1	-	-

Surface preparation shall be subjected to inspection before the prime coat is applied to ensure all traces of dust and foreign matter have been removed by brushing, blowing with dry clean compressed air, or vacuum cleaning.

Figure 21: Surface Preparation