A STUDY ON THE PERFORMANCE OF SPLASH ZONE COATING SYSTEM

by

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
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Approved by,

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May 2015

CERTIFICATION OF ORIGINALITY

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

(NUR AMIRA BTE RUBA'AI)

ABSTRACT

Corrosion at offshore risers is one of the main threats in oil and gas industry. Although new advanced technology is being applied to inhibit the corrosion problem in riser, failure due to corrosion is still happening. Cause of the problem is suspected that insufficient of coating thickness applied towards riser's material particularly at splash zone. For that, experimental works is necessary to better understand these parameters and modify specifications for improving fitness of pipeline and reducing corrosion risks. Two methods are applied in these experiments which are **Water Immersion Test (D 870)** and **Pull-off Adhesion Test (D 4541)**. The pull-off adhesion test was conducted in order to determine the strength of adhesion between the coating and the substrate. In addition, water absorption rates (%) in this experiment were recorded and calculated using weight metal loss method. From the analysis of results, the effects of thickness in coating system when exposed to severe environments were known. This study can help to take awareness and action to overcome the corrosion of offshore risers in oil and gas industry in the future.

CHAPTER 1: INTRODUCTION

1.0 Introduction

The offshore environment particularly at splash zone area is highly corrosive which poses additional challenges on the coating system used for protection of marine pipeline risers. Cyclic wetting or drying by oxygenated seawater, mechanical forces from the wave action, temperature changes, UV exposure, as well as marine life growth frequently cause premature failures of the splash zone coatings. When the splash zone coating fails, rapid corrosion will follows. In fact, corrosion rates occur in splash zone generally more frequent then those in the immersed area [2].

Statistical data collected in the last 30 years indicate corrosion as the main failure cause onto risers [1]. Studies published by U.S. Department of the Interior, Minerals Management Service (MMS) indicate that 92% of corrosion failures occurred onto risers in the Gulf of Mexico caused by external corrosion [1, 2]. Further studies, in 2000, Chevron mentioned a high pressure riser failure in one of their platform in West Coast of Africa due to severe corrosion at splash zone caused by coating damage [2].

Riser is a pipe that connects an offshore floating production structure or a drilling rig to a sub-sea system for production purposes such as drilling, production, injection and export, completion and etc [3]. These risers must withstand severe environmental conditions for years. Highly specialized treatment is required to ensure long-lasting service. Among various material technology developments to cope with the corrosion, coating is one of easiest, effective, and economical measure to minimize or control corrosion loss.

Cost of riser failure caused by corrosion has taken the attention around the world as a one of the major challenges. Evidence from the findings in 1978 study “Economic Effect of Metallic Corrosion in the United States” reported that the corrosion of metals costs the U.S economy almost $300 billion per year [2].

Offshore maintenance is very difficult and the cost is exorbitantly high. Hence, from the early construction stage, the selection of quality coating systems and to ensure that they are applied correctly under the right conditions, are huge decisions to make so that life cycle cost savings is maintained. Offshore painting, compared to onshore, is more expensive due to several factors including the logistics of getting worker and materials to the job site, and also limited access to the structure due to offshore weather conditions [5].

In general, durability of paint film depends on these two significant factors; appropriate surface preparation grade and correct coating thickness [4]. Improper surface preparation and incorrect coating thickness can lead to insufficient corrosion protection, rust spotting, erosion, cracking, and extra material costs. The objective of this study is to investigate the performance of anti-corrosive coating system by increasing their thickness. Damaged barrier coating would be significantly reduced when the thickness of the coating is increased because these coatings behave as semi-permeable membranes.

External protective coatings for offshore riser applications may be organic, inorganic, or metallic materials that may be applied by spraying, brushing, dipping, wrapping, or co-extrusion. Several coating systems that are well known in oil and gas market for application of riser protection are Polyester Glass Flake, 3 layers of Polyethylene, Neoprene, High-Density Polyethylene (HDPE), Thermally Sprayed Aluminium (TSA) and etc. These coating systems come with many range size of thickness.

1.1 **Problem Statement**

Corrosion protection of steel by glass flake reinforced polyester coating is widely used in marine division. Even though, glass flake reinforced polyester coating is highly corrosion resistant, its performance is decreased due to insufficient coating thickness and improper surface preparation. If the appropriate thickness is being applied, the physical and protective properties of the coating material can be optimized.

The hypothesis of this study is, the thicker of the coating layers, and if it bears no defects, it will increase the anticorrosive performance of barrier system and is more rust preventive. Although by doing so might increase large capital expenditure, it is surely worth when the risers are highly protected and the service life are extended.

1.2 **Objectives**

The objective of this study is to evaluate the performance of coating system with varies thickness for splash zone application, specifically in terms of:

1) Visual inspection
2) Water absorption rate
3) Adhesion strength of glass flake reinforced polyester coating to the substrate

1.3 Scope of Study

The scope of study for this project as per stated below:

1) This project only focused on external corrosion of risers and the root cause of failure is corrosion defect
2) The samples, API 5L X65 mild steel will be coated with polyester glass flake and immersed into artificial seawater for 28 days
3) This project is planned to be done in the form of experiments. Pull off adhesion test (ASTM D4541) and water immersion test (ASTM D870) are conducted
4) All requirement testing will be done with experimental setup available at Centre of Corrosion Research (CCR) at Block I
5) This project is planned to be completed within 6 months

CHAPTER 2: LITERATURE REVIEW

2.0 Riser System

Overall, riser is defined as a long tube made of metal or plastic that is used to carry water or oil or gas. On the other hand, riser system is a conductor pipes connecting floaters on the surface and wellheads at the seabed. By referred to API 1998, functions of riser are to convey fluids between the wells and the floater for production and injection risers and export fluid from floater to pipeline for export riser [6]. Main function of riser is to transport fluids or gas from seabed to a host platform.

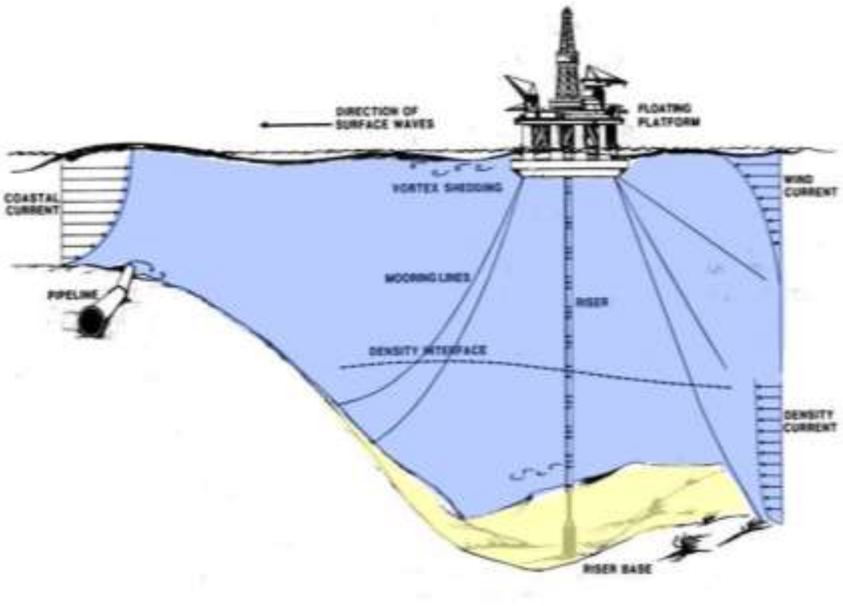


Figure 2.0: Offshore riser system

There are number of riser configurations that have been used in deep water field such as hybrid riser, steel catenary riser, and top tensioned riser. These applications of riser system are vary according to water depth and environmental conditions. Design of riser system for deep water field is obviously more challenging than shallow water. Usually, risers are designed so that they can withstand some corrosion damages, because of economic considerations. These damages must be controlled in order to prevent failures of the risers. Such failure can and has resulted in huge economic and environmental loss.

In the last few years, steel catenary risers have been preferred riser solution for deep water field developments due to its simple engineering concept, cost effective, flexibility in using different host platform and flexibility in geographical and environmental condition.

2.1 Overview of External Corrosion of Riser

Based on offshore riser failure statistics made by U.S Department of the Interior Minerals Management Service (MMS), illustrate that general riser at offshore platform tends to fail caused by corrosion damage is highest compared to other causes [1]. The other causes are natural hazard, structural, erosion and etc. From recent MMS analysis has shown that the riser predominantly fails as a result of corrosion. Figure 2.1.1 indicates that, 92% of riser corrosion failures were due to external corrosion with 8% percentage due to internal corrosion damage.

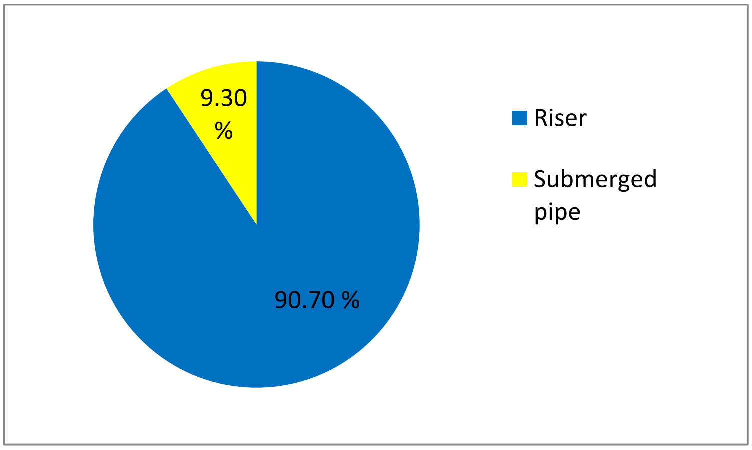


Figure 2.1.1: Location of damage due to external corrosion. (Source: DOI/MMS)

The possible root causes of these elevated riser failure were due to ineffective coatings, ineffective cathodic protection, ineffective design, and etc [7]. Besides, riser also would be degrading because of marine growth. However, in Schumacher report has revealed that biofouling and marine growth in the tidal zone may actually mitigate external corrosion. Integrity of offshore riser also may potentially deteriorate when exposed to aggressive seawater environments such as atmospheric, tidal, splash and submerged zone.

2.2 How Corrosion Occurs

To select the method of corrosion control, it is very important to understand how corrosion occurs. The corrosion of steel proceeds in the presence of oxygen and water, and is restrained by removing one of them. The corrosion of steel in water, as illustrated in Figures below, can generally attributed to corrosion in the air, because on a steel surface in a humid atmosphere, there is a thin water layer formed by the adsorption of water molecules in the air. Each point on a steel surface has its own tendency to dissolve in water. This is called electrolytic solution pressure.

This tendency depends on the condition of the surface and water contacting each point. At the point of high dissolution, metallic iron dissolves in the form of ferrous ions. Since the water must remain electrically neutral, hydrogen ions are deposited as a thin layer of hydrogen along the low dissolution surface of the steel. As a result, an electric circuit is formed between the high and low points of dissolution. These points and the circuits are called anodes, cathodes, and corrosion cells, respectively.

The existence of a layer of hydrogen on the cathodes obstructs the further progress of the reaction, but free oxygen in the water removes the obstruction as it combines with hydrogen. The ferrous ions dissolved in the water combine with hydroxyl ions and finally precipitate on the surface of the steel as rust after being oxidized to form ferric ions. The general methods of controlling corrosion are as follows:

1) Minimize the difference in electrolytic solution pressure between anodes and cathodes by making the steel surface uniform
2) Use a coating to restrain the supply of water and oxygen to the steel surface
3) Restrain the corrosion reaction by the adsorption of inhibitors in the anodes of cathodes. The inhibitors are intentionally added to the water or supplied by the dissolution of anticorrosive pigments in the coating material

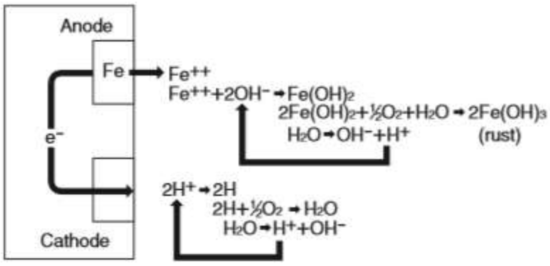


Fig. 1 Corrosive Action

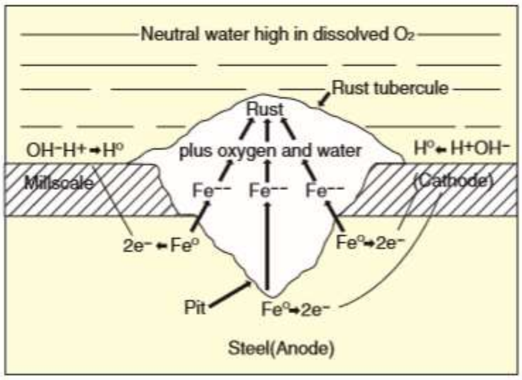


Fig. 2 Corrosion at Break in Mill Scale
(Steel Structures Painting Manual,
Steel Structures Painting Council)

2.3 Environments of Seawater

All offshore risers exposed to several different environments according to the position of its parts relative to seawater level. They are known as atmospheric, splash, tidal, and submerged zone that can be described as follows:

Atmospheric zone: the zone above the splash zone

Splash zone: the zone of the riser that is alternately in and out of the water because of the influence of the tides, winds, and waves. The zones range is:

1) Upper limit is defined as 65% of the maximum wave height above the High Astronomical Tide (HAT)
2) Lower limit is defined as 35% of the maximum wave height above the Low Astronomical Tide (LAT)

Tidal zone: the zone of the riser section lying between mean low tide and mean high tide

Submerged zone: the region below the tidal zone including seawater, sea bottom and buried or mud zone

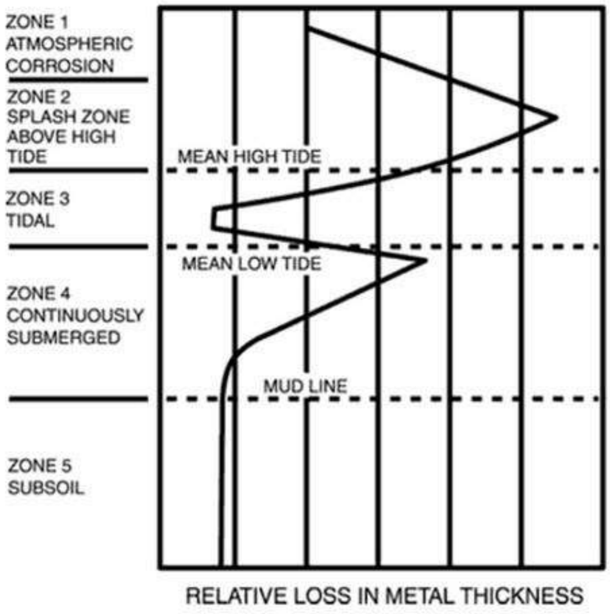


Figure2.3.1: Curve represent the most severe corrosion occurs in the splash zone for steel pilings. (Source: http://m.international-pc.com/Pages/news.html)

Steel structure situated above the seawater is atmospheric zone. In this zone the corrosion rate of unprotected steel is typically in range of 80 – 200µm per year. High corrosion rates are caused by extended periods of wetness and high concentrations of chlorides that accelerate corrosion. Another factor that needs to be considered in the atmospheric zone is UV exposure; light from the sun may affect on some types of corrosion protection.

An area alternately between above and below the water line due to the action waves is called the splash zone, and here the corrosion stresses are even higher. The corrosion rates of 200 - 500µm per year have been measured in this area. Compared to the atmospheric zone, risers in splash zone area usually receive the worst abuse during their service life. Additional stresses such as mechanical forces from the wave action and cyclic wetting or drying by oxygenated seawater are causing corrosion rate is higher.

Continuously, immersed zone show corrosion rates of unprotected steel in the range of 100 - 200µm per year. In this area the corrosion rate is expected to be very low in comparison with other zones because the seawater is stagnant and has the lowest temperature and oxygen concentration.

2.4 Protection through Coating System

The most common method use for the protection of materials in offshore environments is the use of various types of coatings and for the immersion zone, coatings combined with cathodic protection. If the composite materials are used, corrosion is no longer an issue. However, water molecules can still diffuse into the network of composites to affect the mechanical properties. When moisture diffuses into the composites, it can degrade the fiber-matrix interfacial bonding, lower the glass transition temperature, swell, plasticize, hydrolyze, and sometimes micro crack the matrix.

For proper performance of a coating system and ensure durable protection under such extreme conditions, it is critical to respect the fundamental parameters in coating selection. These parameters include the:

a) Type and condition of substrate
b) Environment and possible additional stresses
c) Surface preparation
d) Quality of coatings
e) Selection of coating systems
f) Application
g) Quality control

2.4.1 Substrate

By referring to the PETRONAS standard (PTS 31.40.00.20), the most frequently used riser materials are metallic. Non-metallic materials such as glass reinforced plastic (GRP) and flexible pipe may be cost effective for specific applications especially when the fluid is corrosive [8]. Materials are able to resist corrosive conditions are austenitic stainless steel, duplex stainless steel, high nickel alloys and high grade carbon steel above X60 [8, 9].

But, not all types of austenitic stainless steel are resistant to corrosive environment for example SS304. It is due to absence of molybdenum component in its chemical composition. It is differing to SS316; it contains (2% - 3%) of molybdenum. Molybdenum is added to improve the corrosion resistance to chlorides such as seawater. Without molybdenum, the material will easy to degrade when exposed to saltwater. Even though SS316 is better quality than SS304, it is not guaranteed that the degradation of material will not occur. Therefore, a special coating must be applied on the material to minimize the degradation problem. Disadvantage of SS316 is it can only operate at temperature of below 60˚C of seawater, or it will expose to stress corrosion cracking (SCC) [9].

Meanwhile, duplex stainless steel have roughly twice the strength compared to austenitic stainless steel and improved resistance to localized corrosion, particularly pitting, crevice corrosion and stress corrosion cracking [10]. They are characterized by high chromium (19% – 32%) and molybdenum (up to 5%) and lower nickel content. Unfortunately, in recent years, utilization of duplex stainless steel for riser materials is decreased. It is due to higher alloy content makes them more expensive compared to carbon steel. It is generally uneconomic for normal applications.

Carbon steel with an improved resistance to seawater corrosion is normally used for offshore risers in splash zone. For example, the high strength C-Mn steel ASTM A690 is used for sheet pilling because it has two to three times better resistance to splash zone corrosion than the commonly used ASTM A36 steel [11]. Other materials can be used also in splash zone are CuNi, Monel 400, and titanium. Using of the high alloy materials may be only option in the case of high temperature or critical service risers.

2.4.2 Exposure environment

Seawater is a complex fluid that affects nearly all common structural materials to some extent. There are two competing processes that operate simultaneously in seawater environments which are the chloride ion (Cl^-) activity and dissolved oxygen. Below are listed three important factors that contributing to seawater corrosion:

a) Chloride Concentration

Seawater is known as excellent electrolyte. The presence of a large amount of dissolved salts, sodium chloride (NaCl) that are ionized make it an excellent conductor and influence the corrosion rates and corrosion resistance of metals in seawater. It is normally more corrosive than fresh water because of the higher conductivity and the penetrating power of the chloride ion through surface films on a metal [19].

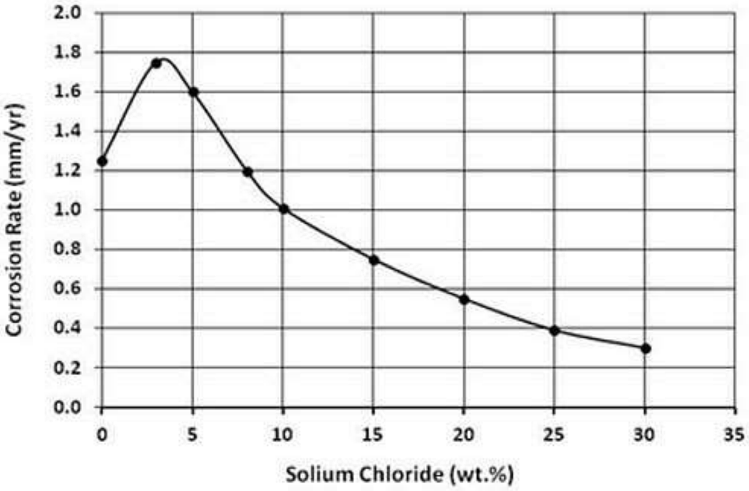


Figure 2.4.1: Relationship between corrosion rate (mm/yr) and sodium chloride (wt%).
Source: http://www.corrosion-doctors.org/Corrosion-by-Water/Types-of-water.html

From the graph above, it shows the combination of chloride concentration and dissolved oxygen concentration results in the maximum corrosion rate. The highest oxygen concentration can be achieved at 3.5 weight percent sodium chloride. One of previous research work has shown that oxygen solubility can affect the rate of corrosion at solution of sodium chloride concentration. The more diluted the solution would be easier for oxygen to diffuse or reach to cathodic areas, causing more corrosion in the metal surface.

b) Oxygen and Temperature

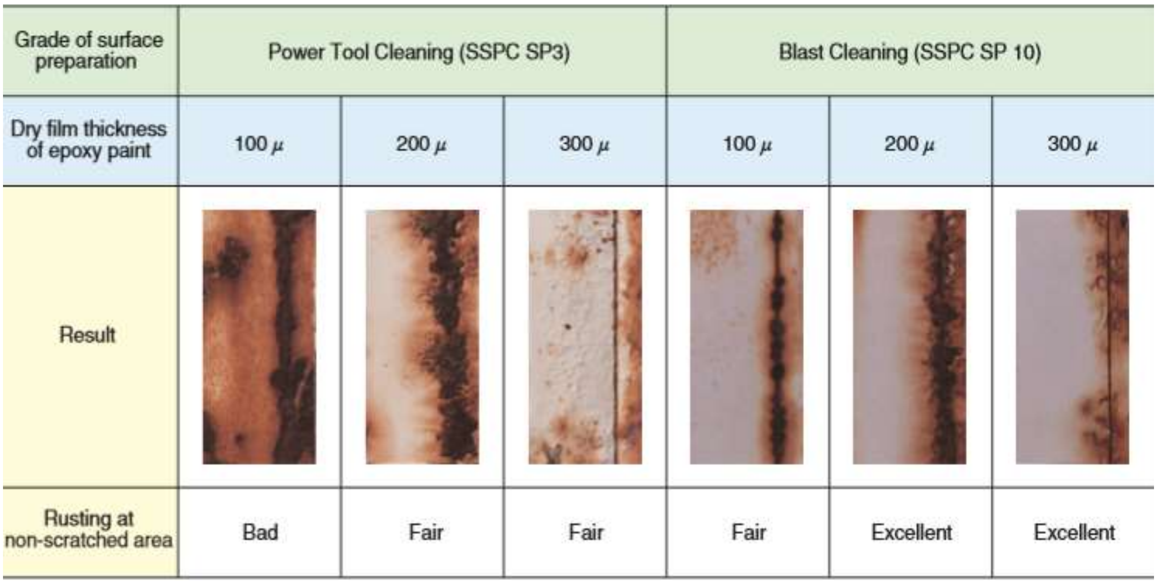
In general, there are several factors that can influence the oxygen concentration. Agitation of seawater due to waves can increase the oxygen concentration in the seawater. Temperature is the other factor and it produces two opposite effects. At first view, temperature can have an impact on the solubility of dissolved oxygen and the diffusion rate of dissolved oxygen. The diffusion rate of oxygen in seawater increases as the temperature of the water rises. In response, the corrosion rate increases as a result of the increasing limit current density of the oxygen reduction cathodic reaction. On the other hand, at high temperatures, the solubility of oxygen in seawater decreases. This effect can reduce the corrosion rate. Despite the above mentioned effects, the solubility of oxygen in saline water is not affected by temperature as much as the diffusion coefficient is affected.

Temperature also affects the corrosion rate by changing these two parameters which are oxygen solubility and diffusivity. The solubility of oxygen in water decreases significantly with the increase in temperature and slightly with concentration of dissolved salts (Shrier, 2004). Also by increasing temperature can increase the solution electrical conductivity thus enhanced the corrosion rate of material.

2.4.3 Surface preparation

Surface preparation is a very important factor affecting the durability of paint film. It is degree of cleaning to remove rust, mill scale, oil or grease and the roughness as well as preparation; rounding and grinding of sharp edges, welding seams, and other imperfections of steel work that are critical in this phase. Paint adheres better to a clean and smooth surface; consequently it will enhance steel to be more resistant towards corrosive environment.

Figure 2.4.3 shows the result of coastal weathering test which was done with different dry film thicknesses of catalyzed epoxy paint on surfaces treated by power-tool cleaning (SSPC-SP3) and blast cleaning (SSPC-SP10) [15]. From observation, high grade surface preparation give results in long-term durability of paint film. Poor surface preparation does not provide long-term durability. Therefore, the coating shall be planned so that the surface preparation should be of the highest grade possible.



Grade of surface preparation	Power Tool Cleaning (SSPC SP3)			Blast Cleaning (SSPC SP 10)		
Dry film thickness of epoxy paint	100 μ	200 μ	300 μ	100 μ	200 μ	300 μ
Result						
Rusting at non-scratched area	Bad	Fair	Fair	Fair	Excellent	Excellent

Figure 2.4.3: Coastal Weathering Test Results with Catalyzed Epoxy Paint

2.4.4 <u>Selection of Coatings systems</u>

The coating systems should be selected with due consideration to the environment as well as the special stresses. For the atmospheric zone, typically use a zinc-rich primer followed by epoxy intermediate coats and a UV durable topcoat such as epoxy siloxane, has minimum 320 µm dry film thickness (DFT) in no less than 3 coats [16]. The splash zone will often be protected by epoxy or polyester coatings, in a thickness that takes into account the special stresses and normally more than 600 µm DFT in total. For optimum protection against impact damages in such areas, zinc-rich primers are normally avoided while fiber reinforced coatings are recommended as a means of increasing the impact resistance [17]. Finally, immersed areas will be coated with epoxy barrier coatings in a film thickness of no less than 450 µm DFT in minimum 2 coats. It is important that the epoxy coating system is compatible with the cathodic protection system used.

For this study only focused on riser at splash zone. Thus, by followed PETRONAS standard for protective coatings and linings, most appropriate coatings system used to protect riser from corrosion damage is polyester glass flake [12]. Apart from polyester glass flake, neoprene coating system also suitable for riser splash zone. The benefits of this coating system are providing abrasion resistant waterproofing protection, excellent resistance to salts and good resistance to alkalis and etc [13].

2.4.5 Application

It is important that the correct film thickness is applied within the normal tolerances of a quality paint application. Not too thin as this will result in premature corrosion either because of pinholes in the film or just because of insufficient thickness, but not too thick either as this also can result in adverse effects such as solvent retention, reduced adhesion, cracking etc [4]. Furthermore, more coats will generally give better protection than one coat in the same total film thickness. Stripe coating on welding seams, edges, corners and areas that are difficult to reach by the airless spray is mandatory for a later high durability of the coating system.

2.4.6 Quality Control

The final parameter and the most important ones is quality control throughout the process. Special focus needs to be put on the surface preparation and coating application processes, where several check points need to be confirmed to ensure the proper final result.

2.5 Coating Standards

The most important standards that apply within the field of offshore coatings are the following:

a) PTS 38.48.00.31- September 2012

b) NORSOK M501-revision 5

c) ISO 12944-1998/2007

d) ISO 20340-200

2.6 Polyester Glass Flake Coating

The purposes of external pipe coatings are as corrosion protection for metal surface and acts as barrier to inhibit the contact between chemical compounds or corrosive materials. Polyester resin based linings are known for their solvent and chemical resistance. The word “lining” in the coating industry is commonly defined as a material used to protect the surface of a tank, vessel, or similar structure from highly corrosive exposures. However, in recent years, glass flake reinforced polyester coatings have been used in the offshore industry for 30 years and well-known as high performance coating system. This glass-flake reinforced polyester coating is suitable for immersion and underground services such as splash zone, decks, and jetty piles. It has proven highly effective in extremely corrosive environments. Moreover, polyester glass flake specially formulated to give optimum ease of application and short cure cycles, offers a remarkably low permeation rate, combined with excellent resistance to abrasion and cathodic disbondment [24].

Until today, most of the research was done focused on internal and external corrosion of carbon steel in the corrosive environments. Even from the past study, corrosion rate of steel has been predicted roughly for different environments. Meanwhile for this study, the intention is to focus on surface coating technologies for corrosion prevention particularly for the sections of the riser which located at the air or water interface where accelerated corrosion and severe pitting corrosion may occur. Specific coatings and thickness should be applied on these risers because they have to be mechanically resistant for tidal and splash zone offshore conditions.

CHAPTER 3: METHODOLOGY

3.1 Overall Project’s Methodology

For this project, sample of carbon mild steel will be used in all experiments. Several conditions will be monitor and set in order to achieve the objective which is to evaluate the performance of coating system at varies thickness and with the presence of corrosive environments. Below is shown the flow of project to conduct the experiment.

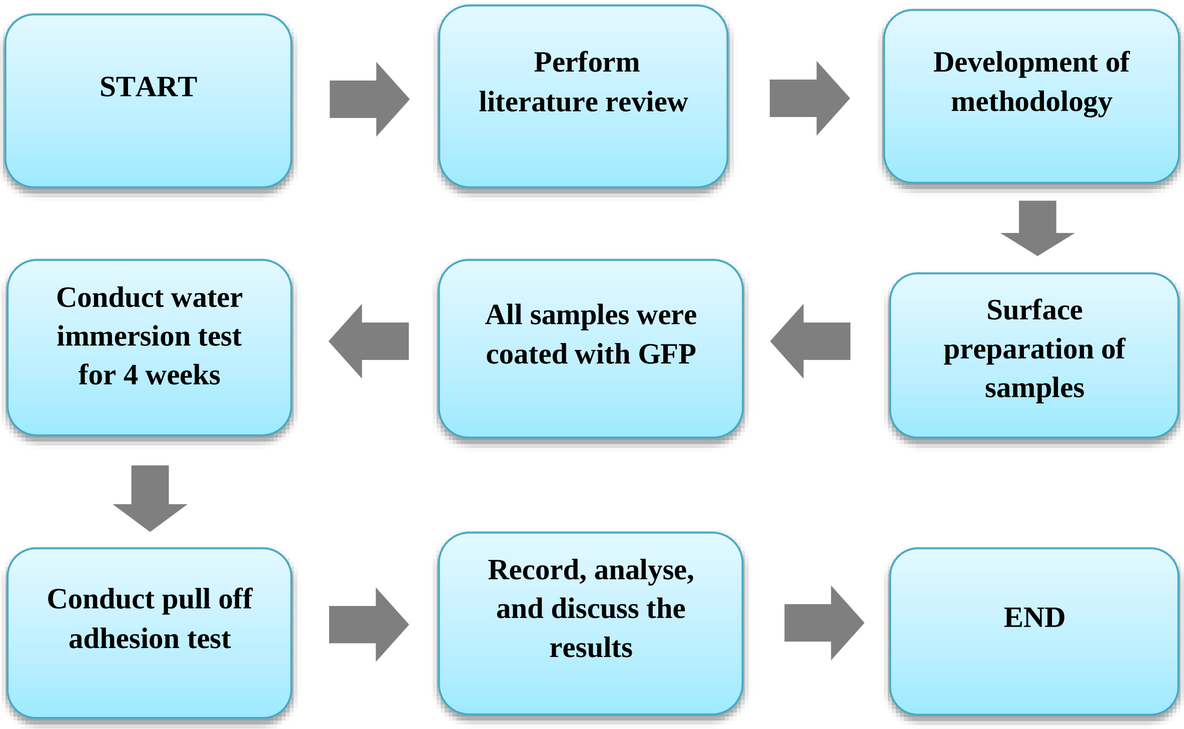


Figure 3.1.1: Flow chart of the project’s methodology

3.2 Sample Preparation

The sample of mild steel will be fabricated into a desired shape before being used for experiment purposed. Figure 3.2 shows the steps in preparing the sample:

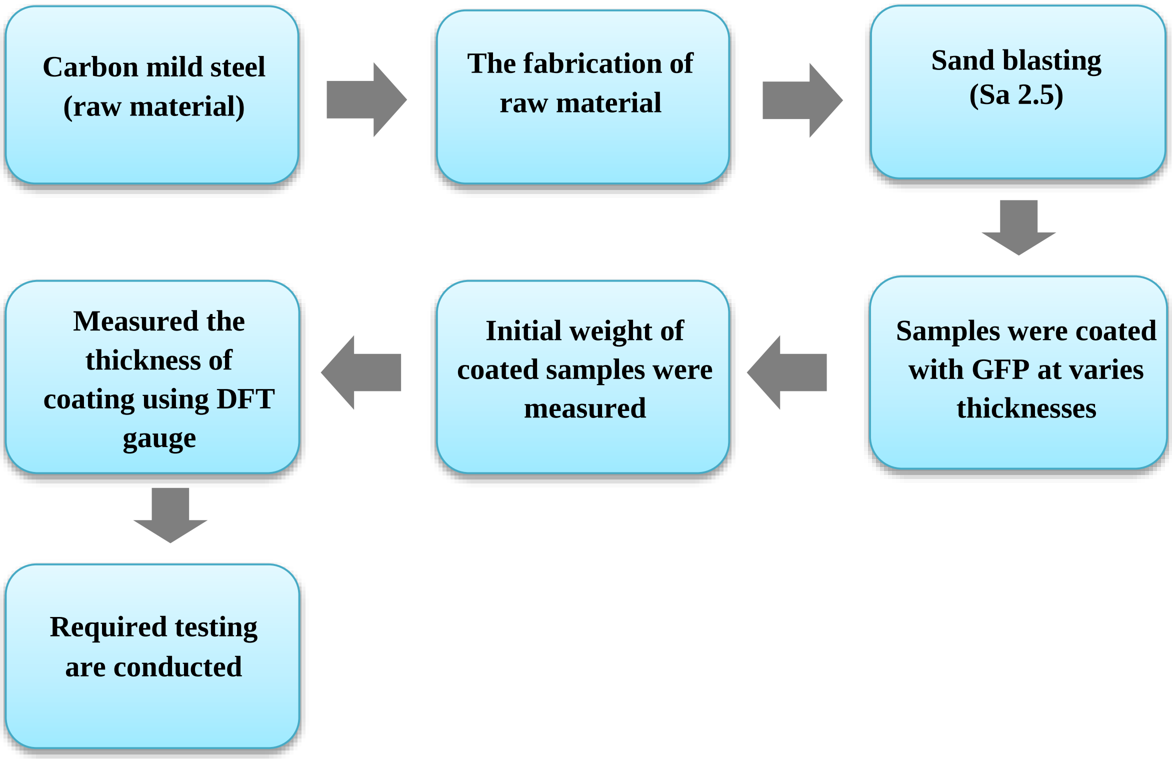


Figure 3.2.1: Procedures of sample preparation

Step 1: Sample Surface Preparation

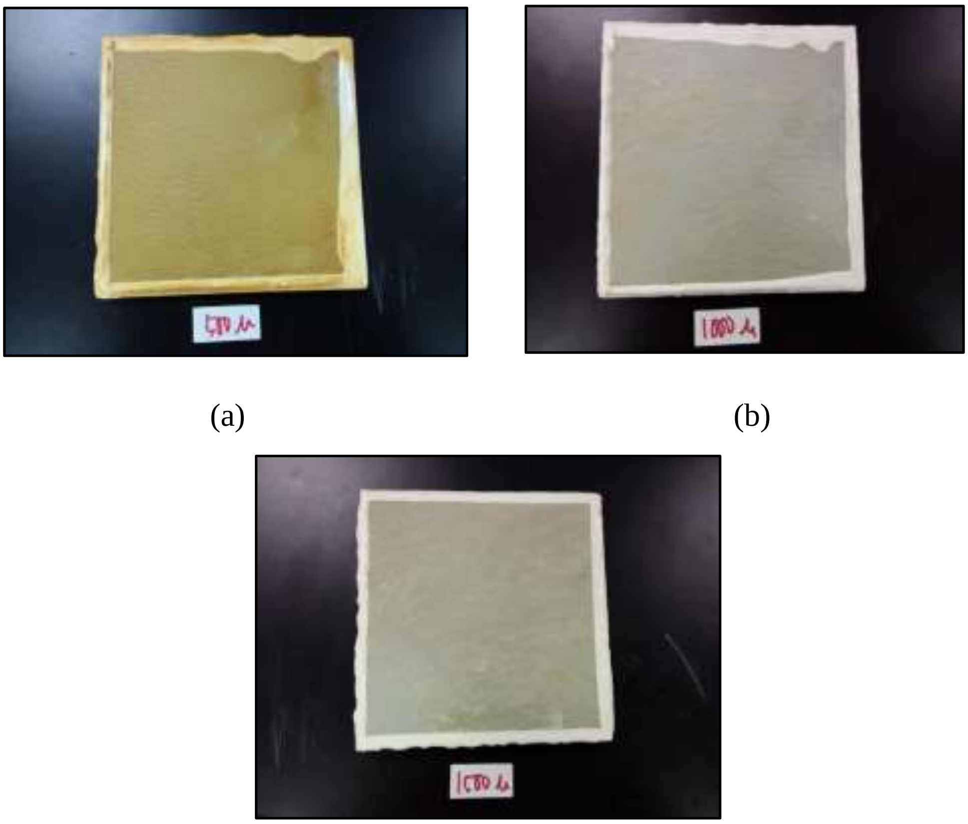
To secure lasting adhesion of coating system to substrate, all surfaces shall be completely dry and free from all surface contamination. The oil, grease, unwanted corrosion products, and other contaminants were cleaned using blast cleaning before the substrate to be coated. For surface preparation of carbon steel, it is recommended to apply the Near White Blast system (Sa 2½).

Step 2: Coating Application

Blast cleaned samples shall be coated within 4 hours. If delayed beyond this period, the sample will shows any visible rust strain and it must be blast cleaned again. Airless spray is used in this experiment to coat all samples. The position of spray gun should not be closer than 300mm or more than 600mm from the surface to be coated. Table 3.1below is the details of coating application:

Table 3.2.1: Coating parameters

No	Description	
1	Type of coating system	Polyester Glass Flake
2	Coating thickness	500μ, 1000μ, and 1500μ
3	System ID	One coat
4	Curing temperature and time	23ºC, 2 hours



(a) (b)

(c)

Figure 3.3: The samples with different coating thickness

Step 3: Measuring the Coating Thickness

The coating thickness was measured using Dry Film Thickness (DFT) Gauge. Seven readings were recorded at different spot.

3.3 Tools and Equipment

These are tools and equipment required to carry out the experiments. Table 3.3.1 shows the summary of equipment and their function.

Table 3.3.1: List of equipment and its function

No	Equipment	Function
1	Dry Film Thickness (DFT) Gauge 	To measure the thickness of the coating system
2	Water Bath 	As an apparatus to immerse the samples at operating temperature of 50ºC
3	Adhesion Hydraulic Tester & Dollies 	To measure the force required to pull a specified diameter of coating away from its substrate

3.4 Attainable Test Matrix

Table 3.4.1: Test matrix of the experiment

Sample & Coating thickness	Sample A (500μ)	Sample B (1000μ)	Sample C (1500μ)
Type of coating system	Polyester Glass Flake Coating		
Temperature used	50 ºC		
Salt concentration used	5.0 wt% NaCl + 1000 ml distilled water		
Type of testing will conducted	1) Water Immersion Test (D 870) 2) Pull Off Adhesion Test (D 4541)		
Test duration	30 days		

3.4 Experimental Procedures

Experiment A: Water Immersion Test (ASTM D 870)

Objectives:

1) To obtain the water absorption rate (%) of coated sample
2) To simulate the real condition of seawater environment into coated sample

Procedures:

First precaution should be taken before conducting this testing is all samples must be left for a week to ensure the coating system is fully dried. Initial weight of coated samples has to measure using a weighing machine in the beginning of this test. Next, the samples will immerse in synthetic seawater solution at 50 ± 2ºC for 28 days. The test solution was made by adding 5.0 wt% of sodium chloride, NaCl into 1wt% of distilled water. The samples have to fully immerse in synthetic seawater solution within 28 days. During this period of 28 days, the samples are taken out of the solution on day 3, day 10, day 14, and day 28 to weight the samples in order to obtain water absorption. Water absorption can be calculated using this formula from ASTM D570:

$$\frac{\text{Wet weight} - \text{Dry weight}}{\text{Dry weight}} \times 100\%$$

In the meantime, visual inspection also can be conducted to observe any changes occur on the samples such as discoloured of surface, blistering, and etc. After this testing was done, the samples will undergo a pull off adhesion test. The pull off adhesion test is performed using ASTM D4541 within 8 hours of removing the samples from synthetic seawater solution.

(a) *(b)*

Figure 3.3: (a) Test solution preparation (b) Fully immersed sample in synthetic seawater solution

Experiment B: Pull-off Adhesion Test (ASTM D4541)

Objectives:

1) To measure the force required to pull a specified diameter of coating away from its substrate
2) To evaluate the adhesiveness of polyester glass flake coating system

Procedures:

The major components of a hydraulic adhesion tester are the dolly, a pressure gauge, and a pressure source. Size of dollies were used is 20 mm diameter (outside) and 3 mm diameter (inside). The pull-off test is performed by securing a loading fixture perpendicular to the surface of coating with araldite glue. After the araldite glue is cured at 80ºC for 24 hours, scoring is done as per recommended by the standard. Typically, the glue and the coating are scored through the metal substrate around the circumstances of the dolly. This defines the area of the coating and glue being stressed. Next, the force applied to the loading fixture is gradually increased and monitored until either a plug of material is detached or whether the surface remains intact at a prescribed force. When a plug of material is detached, the exposed surface represents the plane of limiting strength within the system.

The precautions that must be consider in this test are the adhesion of the glue between dolly and coating must be strong, so that the failure is between coating and substrate and it must be rigid to ensure deformations occurs only in the coating. Besides, the pulling direction must be perpendicular to the plane of the coating and substrate so the stress is perfectly across the area of the dolly-coating joint.

(a) (b) (c)

Figure 3.4: (a) The dollies attached on the sample. (b) The scoring was performed. (c) The force is applied on the dolly.

3.5 **Project's Schedule**

Table 3.5.1: Project's schedule for FYP 1 & FYP 2

No	Project activities	FYP 1							FYP 2						
	Weeks	1-2	3-4	5-6	7-8	9-10	11-12	13-14	1-2	3-4	5-6	7-8	9-10	11-12	13-14
1	Select and briefing on FYP project title with supervisor & co-supervisor	🟧													
2	Identify and define the problem statements, objectives, & scope of study	🟧	🟧												
3	Perform literature review by reading research paper or case study regarding corrosion at seawater environment		🟧	🟧	🟧										
4	Study on methodology & setup test matrix for the experiment					🟧									
5	Contact and sent samples of study to Jotun company for coating					🟧	🟧	🟧							
6	Prepare and understand the procedures involved to conduct the experiments					🟧	🟧	🟧							
7	Starts performed experiments; water immersion test & pull-off adhesion test								🟥	🟥	🟥	🟥	🟥		
8	Observe any changes occur on the samples												🟥	🟥	
9	Record, analyze, and discuss gathered results												🟥	🟥	
10	Writing a technical paper report & dissertation report											🟥	🟥	🟥	🟥
11	Viva presentation														🟥
12	Submit all reports to supervisor, co-supervisor and coordinator of FYP														🟥

3.6 Gantt Chart and Key Milestone

The tables 3.6.1 and 3.6.2 have shown Gantt Chart and Key Milestone of the project for FYP 1 and FYP 2:

Table 3.6.1: FYP 1 project's Gantt Chart and Key Milestone

No	Project activities	Weeks													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Selection of project tittle	🟨													
2	Literature review / research work		🟨	🟨	🟨	🟨	🟨								
3	Submission of Extended Proposal							🟨 ◆							
4	Proposal Defense Presentation								🟨	🟨 ◆					
5	Project work continues										🟨	🟨	🟨		
6	Submission of Interim Draft Report													🟨 ◆	
7	Submission of Interim Report														🟨 ◆

Table 3.6.2: FYP 2 project's Gantt Chart and Key Milestone

No	Project activities	Weeks													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Sample preparation	🟥	🟥	🟥											
2	Experimental setup				🟥	🟥	🟥	🟥	🟥	🟥	🟥				
3	Submission of Progress Report							🟥	🟥 ◆						
4	Collect data and analyze result									🟥	🟥	🟥	🟥		
5	Pre-SEDEX										🟥	🟥 ◆			
6	SEDEX Exhibition												🟥 ◆		
7	Submission of Draft Final Report												🟥 ◆	🟥	
8	Submission of Dissertation													🟥	🟥 ◆
9	Submission of Technical Report													🟥	🟥 ◆
10	Viva presentation														🟥 ◆

CHAPTER 4: RESULTS & DISCUSSIONS

4.1 The Baseline Results of Substrate Weight

Figure 4.1: Coated specimen was weighted by weighing machine.

Table 4.1: Initial substrate's weight before experiment was conducted

Type of Experiment	Experiment A			Experiment B		
Substrate Thickness	**< 500 µ**	**1000 µ**	**1500 µ**	**< 500 µ**	**1000 µ**	**1500 µ**
1st reading	1399.6 g	1351.6 g	1461.4 g	1317.5 g	1346.2 g	1465.8 g
2nd reading	1399.8 g	1351.7 g	1461.5 g	1317.5 g	1346.3 g	1466.0 g
3rd reading	1399.7 g	1351.7 g	1461.4 g	1317.5 g	1346.3 g	1466.2 g
Total	4199.1 g	4055.0 g	4384.3 g	3952.5 g	4038.8 g	4398.0 g
Average	**1399.7 g**	**1351.7 g**	**1461.4 g**	**1317.5 g**	**1346.3 g**	**1466.0 g**

4.2 The Baseline Results of Dry Film Thickness

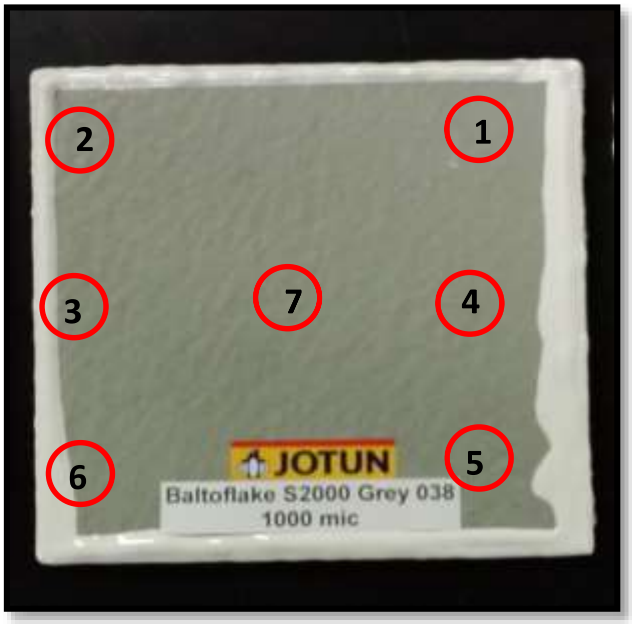


Figure 4.2: Coated specimen with polyester glass flake, 1000 μ. The circle red has shown the specific areas where all thickness is measured.

Table 4.2: Substrate's thicknesses were measured using Dry Film Thickness (DFT) Gauge

Type of Experiment	Group A			Group B		
	A1	**B1**	**C1**	**A2**	**B2**	**C2**
1st reading	289.56 μ	947.42 μ	1457.96 μ	416.56 μ	1097.28μ	1394.46 μ
2nd reading	922.02 μ	1043.94μ	1137.92 μ	326.44 μ	982.98 μ	1524.00 μ
3rd reading	477.52 μ	1016.00μ	1127.76 μ	403.58 μ	828.04 μ	1457.96 μ
4th reading	414.02 μ	1094.74μ	1485.90 μ	589.28 μ	1176.02μ	1478.28 μ
5th reading	302.26 μ	904.24 μ	1536.70 μ	452.12 μ	1099.82μ	1531.62 μ
6th reading	393.70 μ	922.02 μ	1239.52 μ	343.28 μ	957.58 μ	1440.18 μ
7th reading	360.68 μ	1054.10μ	1391.92 μ	394.36 μ	1104.90μ	1501.14 μ
Total	3159.76μ	6982.46μ	9377.68 μ	2925.62μ	7246.62μ	10327.64μ
Average	**451.40 μ**	**997.50 μ**	**1339.67 μ**	**417.94 μ**	**1035.23 μ**	**1475.40 μ**

4.3 Visual Inspection

After being immersed in synthetic seawater solution for 28 days, the coated samples undergo visual inspection. Any changes occur on the samples are observed and recorded. The results on the inspection are shown below:

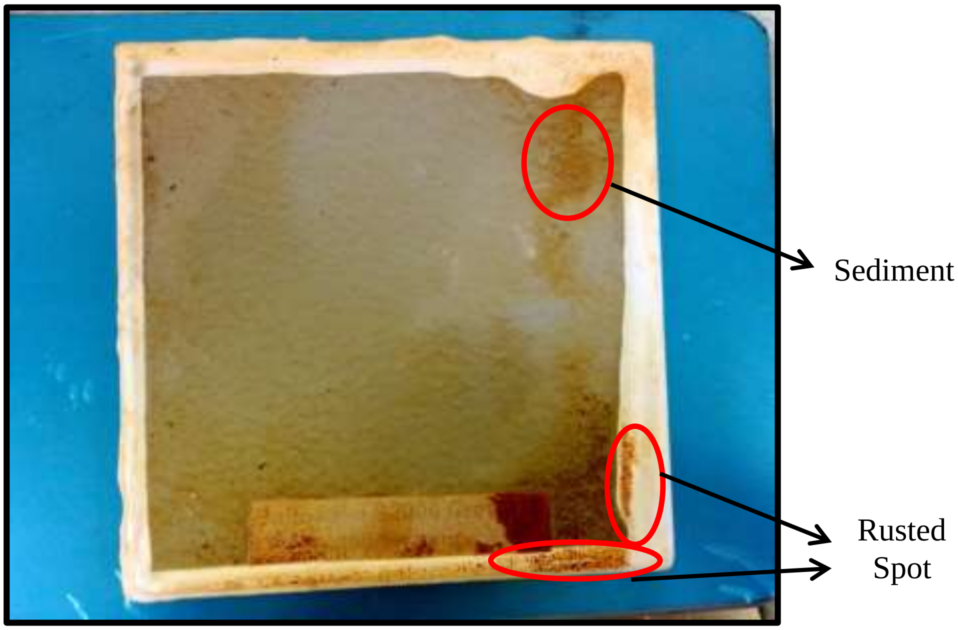


Figure 4.3.1: Coated sample with 500µ of coating thickness

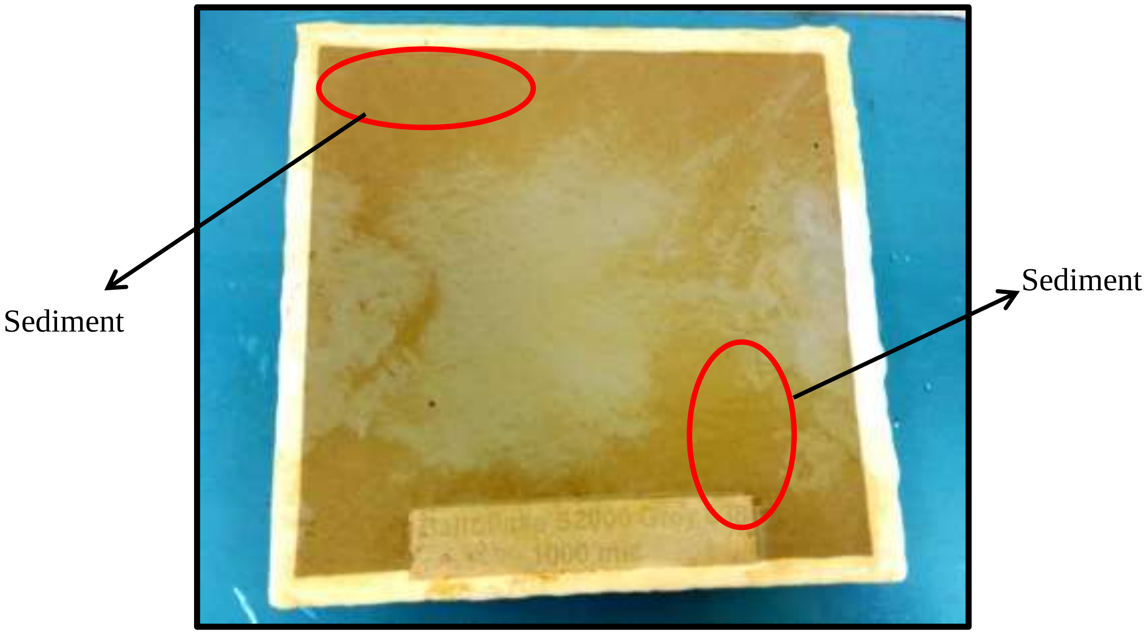


Figure 4.3.2: Coated sample with 1000µ of coating thickness

Figure 4.3.3: Coated sample with 1500µ of coating thickness

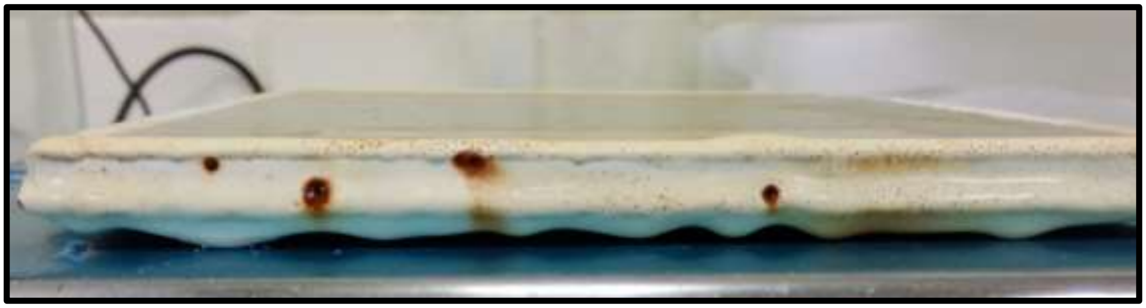


Figure 4.3.4: Rusted spot was clearly seen at the Epoxy area

All figures above have shown the effects on the surface samples after being immersed in artificial seawater solution for 28 days. In performing the visual inspection, the region that has to be focused on the sample is the grey part which is the coating that is tested and examined. The white colour at the edge of the sample is the epoxy as the seal to the coating system. The epoxy gives no effect to the study of this project. It is just the standard practice implemented by Jotun in coating the sample.

By looking at Figure 4.3.2, it is clearly seen that no rusting spots or blistering appeared on the surface area of samples. These observations are also similar for 500µ and 1500µ coating thickness. For 1500µ coating thickness, it is obviously seen that the surface area of sample was covered by the dark brown colour. However, this colour is not showing the sample has rusted, but it is the sediment from the synthetic seawater solution. Even though rusting or corrosion not occurs at the coating area, it happens at epoxy area. Figure 4.3.4 shows the rusted spots appeared at the edge of sample. From these results, it has shown that glass flake polyester coating more highly corrosion resistant compared to epoxy coating.

4.4 Water Absorption Rate

By using a formula in ASTM D570, the equation to calculate water absorption rate is:

$$\boxed{\frac{\text{Wet Weight} - \text{Dry Weight}}{\text{Dry Weight}} \text{ X } 100\%}$$

1) **< 500μ, initial weight: 1399.7 g**

Table 4.3: Experimental result of water absorption rate for 500μ

Group A	After immersion in artificial seawater at 50 ± 2°C			
	3 days	**10 days**	**14 days**	**28 days**
1st reading	1402.4 g	1401.2 g	1400.1 g	1399.8 g
2nd reading	1402.6 g	1401.5 g	1400.3 g	1399.8 g
3rd reading	1402.7 g	1401.3 g	1400.1 g	1399.7 g
Total	4207.7 g	4204.0 g	4200.5 g	4199.3 g
Average	1402.6 g	1401.3 g	1400.2 g	1399.7 g
% of water absorption rate	**0.20**	**0.11**	**0.03**	**0.00**

2) **1000µ, initial weight: 1351.7 g**

Table 4.4: Experimental result of water absorption rate for 1000µ

Group A	After immersion in artificial seawater at 50 ± 2ºC			
	3 days	**10 days**	**14 days**	**28 days**
1st reading	1355.2 g	1354.5 g	1353.2 g	1351.8 g
2nd reading	1354.1 g	1352.9 g	1353.4 g	1351.9 g
3rd reading	1353.9 g	1352.8 g	1353.2 g	1351.8 g
Total	4063.2 g	4060.2 g	4059.8 g	4055.5 g
Average	1354.4 g	1353.4 g	1353.3 g	1351.8 g
% of water absorption rate	**0.19**	**0.13**	**0.11**	**0.01**

3) **1500µ, initial weight: 1461.4 g**

Table 4.5: Experimental result of water absorption rate for 1500µ

Group A	After immersion in artificial seawater at 50 ± 2ºC			
	3 days	**10 days**	**14 days**	**28 days**
1st reading	1464.3 g	1463.2 g	1462.4 g	1461.5 g
2nd reading	1464.2 g	1463.1 g	1462.6 g	1461.6 g
3rd reading	1463.7 g	1463.2 g	1462.3 g	1461.7 g
Total	4392.2 g	4389.5 g	4387.3 g	4384.8 g
Average	1464.1 g	1463.2 g	1462.4 g	1461.6 g
% of water absorption rate	**0.18**	**0.12**	**0.07**	**0.01**

4) **< 500µ, initial weight: 1317.5 g**

Table 4.6: Experimental result of water absorption rate for 500µ

Group B	After immersion in artificial seawater at 50 ± 2ºC			
	3 days	**10 days**	**14 days**	**28 days**
1st reading	1320.3 g	1319.5 g	1318.3 g	1317.3 g
2nd reading	1320.3 g	1319.2 g	1318.2 g	1317.2 g
3rd reading	1320.0 g	1319.5 g	1318.3 g	1317.2 g
Total	3960.6 g	3958.2 g	3954.8 g	3951.7 g
Average	1320.2 g	1319.4 g	1318.3 g	1317.2 g
% of water absorption rate	**0.20**	**0.14**	**0.05**	**0.02**

5) **1000µ, initial weight: 1346.3 g**

Table 4.7: Experimental result of water absorption rate for 1000µ

Group B	After immersion in artificial seawater at 50 ± 2ºC			
	3 days	**10 days**	**14 days**	**28 days**
1st reading	1349.7 g	1349.3 g	1347.9 g	1346.6 g
2nd reading	1348.7 g	1348.3 g	1347.6 g	1346.7 g
3rd reading	1348. 4 g	1348.0 g	1347.8 g	1346.6 g
Total	4046.8 g	4045.6 g	4043.3 g	4039.9 g
Average	1348.9 g	1348.5 g	1347.8 g	1346.6 g
% water absorption rate	**0.19**	**0.16**	**0.10**	**0.02**

6) **1500µ, initial weight: 1466.0 g**

Table 4.8: Experimental result of water absorption rate for 1500µ

Group B	After immersion in artificial seawater at 50 ± 2ºC			
	3 days	**10 days**	**14 days**	**28 days**
1st reading	1469.9 g	1468.7 g	1467.7 g	1466.7 g
2nd reading	1469.1 g	1468.7 g	1467.8 g	1466.7 g
3rd reading	1469.8 g	1468.9 g	1467.6 g	1466.6 g
Total	4408.8 g	4406.3 g	4403.1 g	4400.0 g
Average	1469.6 g	1468.7 g	1467.7 g	1466.7 g
% water absorption rate	**0.19**	**0.13**	**0.07**	**0.02**

4.4.1 Effects of Water Absorption Rate for Glass Flake Polyester Coating

Graphs below were plotted to illustrate the result and to see the relationship between the parameter of the experiment and Glass Flake Polyester (GFP) coating performance at various thickness.

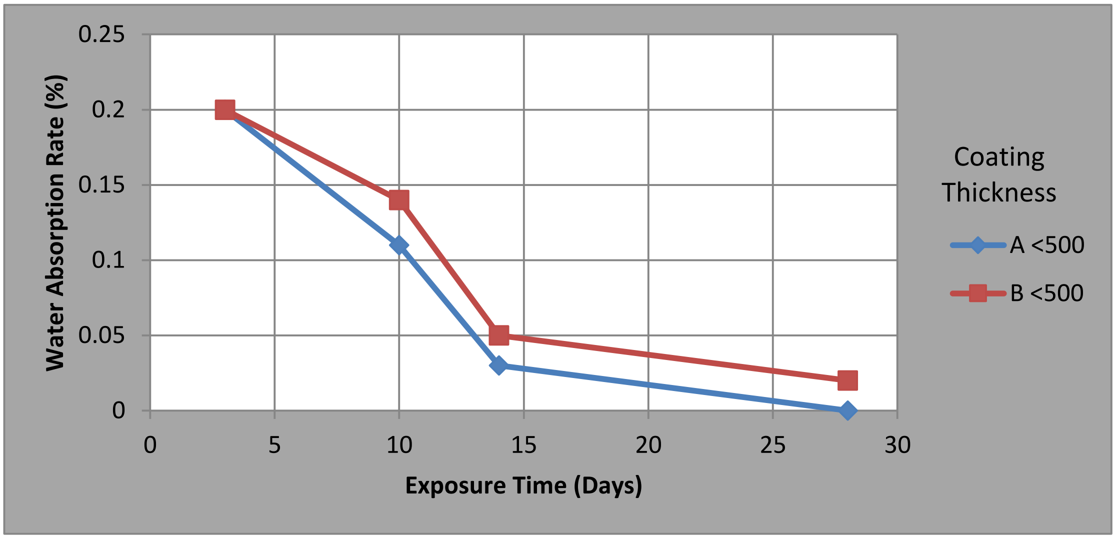


Figure 4.3.1.1 shows the graph of the percentage of water absorption rate for 500µ

The graph above shows the effects of water absorption rate on glass flake polyester coating at different exposure time. On day 3, the graph indicates that the water absorption rate of glass flake polyester coating for group A and group B are the same which equal to 0.20 percent. Group B actually is the repetition of group A for the same testing. It was performed in order to validate the results. After 10 days of immersion period in synthetic seawater solution, water absorption rate starts to reduce to 0.11 percent for group A and 0.14 percent for group B. The water absorption rate slightly decreased on day 14 where the percentage is 0.03 and 0.05 for group A and B respectively. The percentage maintain decreasing day by day until there is no more water being absorbed into the coating system on the day 28. Base on the result, it shows that this coating has high permeability and provides good corrosion protection to the alloy. It is necessary to have high permeation resistance in order to avoid any failure by blistering or adhesion loss of a polymer coating system.

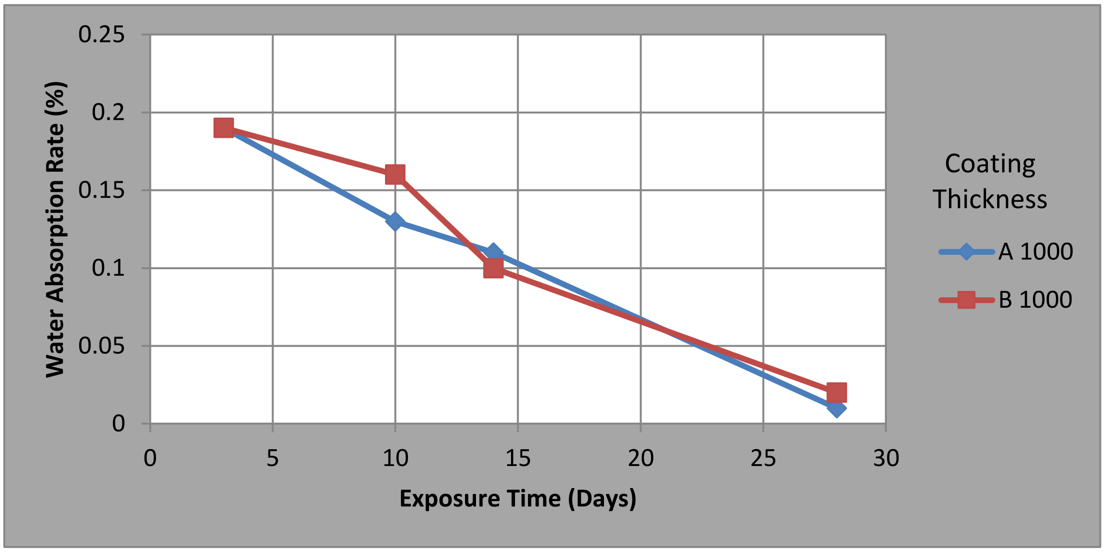


Figure 4.3.1.2 shows the graph of the percentage of water absorption rate for 1000µ

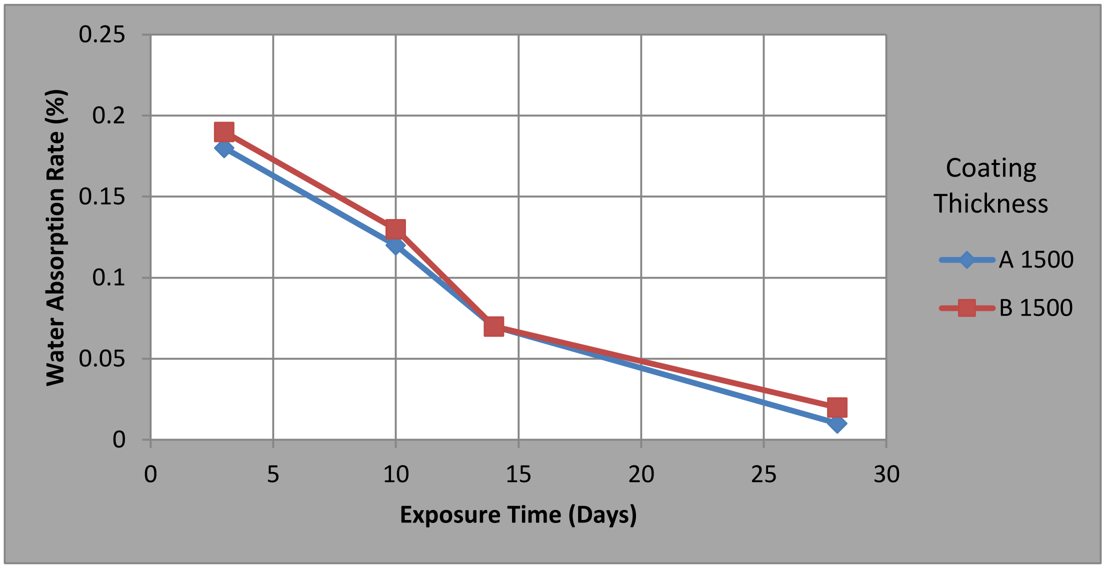


Figure 4.3.1.3 shows the graph of the percentage of water absorption rate for 1500µ

The graphs above show the relationship between the exposure times of sample has immersed in artificial seawater solution with the amount of water absorption rate being absorbed into GFP coating. The results presented in the graph shows that only small amount of water had entered the interface of the glass flake polyester coating specimens. By looking at Figure 4.3.1.3 below, it is clearly seen that as the exposure times getting longer, the amount of water absorption rate decreased until approximately to 0.00 percent. This finding is consistent with the finding of William [9] where he mentioned the effective way to reduce permeability by adding aligned glass flake fillers into the polymer coating. The flake fillers make the path of water molecules or other contaminants through the resin much longer. Besides, it also can increase ability of coating to withstand the higher temperature immersion service.

From these results also can indicate the bond strength between glass flake reinforcement and polyester resin are stronger. It is due to water molecules could not displace these bonds and thus no or little water entered the interface. On the other hand, a result collected from Nippon Paint Group [23] on the performance of glass flake shows water absorption rate of a coating can decrease sharply with increasing content of glass flake.

As the conclusion, the results show that there is not much different in water absorption rate of different thickness i.e. 500µ, 1000µ and 1500µ. However, in real application, it is recommended to use thicker coating system due to environmental condition such as mechanical stresses from wave action, cyclic wetting or drying oxygenated seawater and etc.

4.5 Adhesive Bond Strength Of Polyester Glass Flake To Metal Surface

The experiment was conducted using nine pieces of 20 mm dollies and Elcometer 108 hydraulic adhesion tester. Three dollies are adhered on each sample using araldite (epoxy) glue. The size of each sample is (120 x 120 x 3) mm. The selected test area must be a flat surface and large enough to accommodate the specified number of replicate tests as per recommended by D4541 standard. Also, the distance between each dollies must be separated at least can accommodate the detaching apparatus. Figure 4.5.1 below shows the dollies adhered to the coated sample at the acceptable spacing.

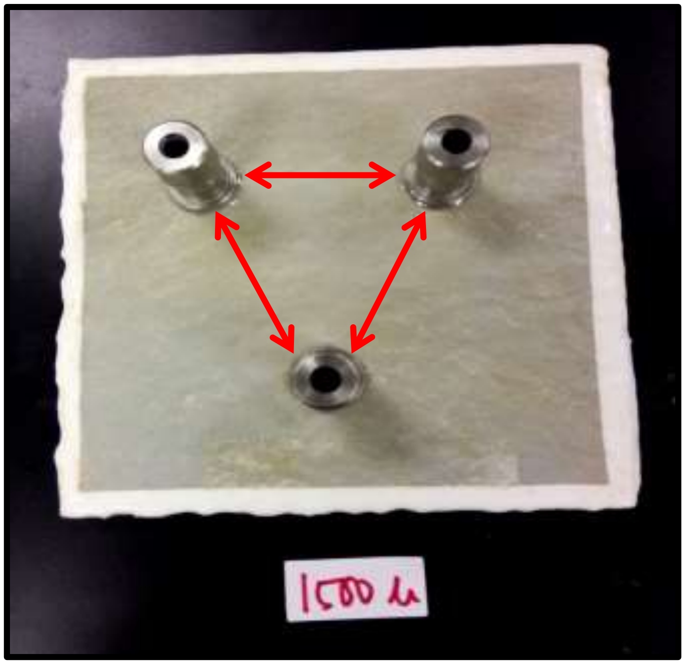


Figure 4.5.1: Three dollies are adhered in each sample using araldite glue with the spacing

Before the adhesion tester is applied to the dolly and ready to be pulled off, we have to do the scoring and remove the plug that contain in the dolly. Scoring is done to clean excess glue from the circumference of the dolly and to scribe the coating, in order to define the area over which the dolly loads the coating during the test. These dollies and pull-off were done specifically for this part of the study, so the laboratory conditions and techniques used were consistent for the three sample sets here. The results of adhesion strength for 500µ, 1000µ, 1500µ coating thickness are given in Table 4.5.

Table 4.5: Result of adhesion strength for 500µ, 1000µ, and 1500µ coating thickness

Coating Thickness	Adhesion strength of GFP to substrate (MPa)			
	1st reading	2nd reading	3rd reading	Average
<500µ	15.7	17.8	12.2	15.2
1000µ	11.8	6.2	5.3	7.8
1500µ	5.6	9.4	7.2	7.4

Based on results in Table 4.5, it indicates that force required to pull a glass flake polyester coating away from its substrate for 500µ coating thickness is 15.2 Mpa (2205 psi). Meanwhile, for 1000µ and 1500µ coating thickness, the force required are 7.8 Mpa (1131 psi) and 7.4 Mpa (1073 psi) respectively. It shows coating at 500µ is well adhered to the substrate as more force is needed to pull out its layer. As the coating layer getting thicker, the less force is required which indicate the adhesiveness of the coating reduced. The reason for the reduced adhesiveness could be because of more voids is present in thicker layer. The voids might be form during coating process.

However, the adhesion strength measured is not very accurate. It is based on the observation to the surface of the sample after the pull off test is done. All the figures below show the effects of araldite glue that is not strong enough to fully pull out polyester glass flake from the substrate.

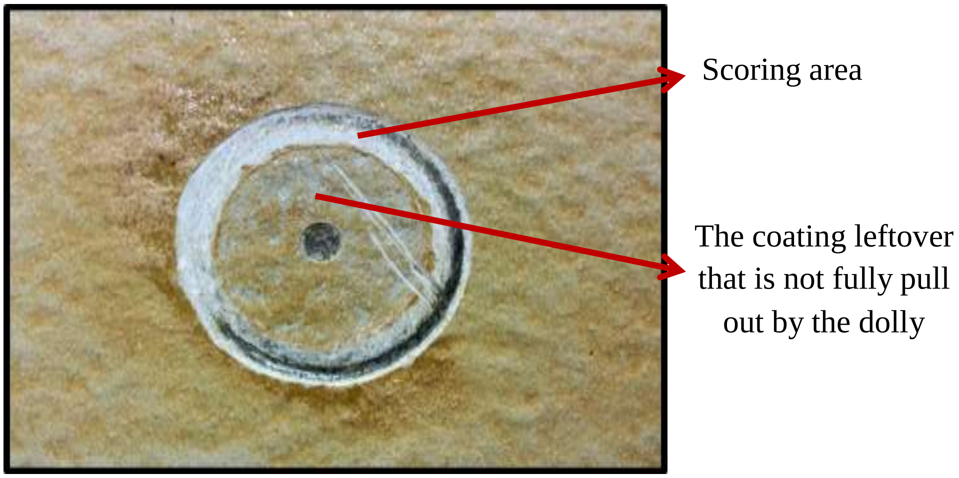


Figure 4.5.2: Surface of 500µ after the pull off adhesion test is done

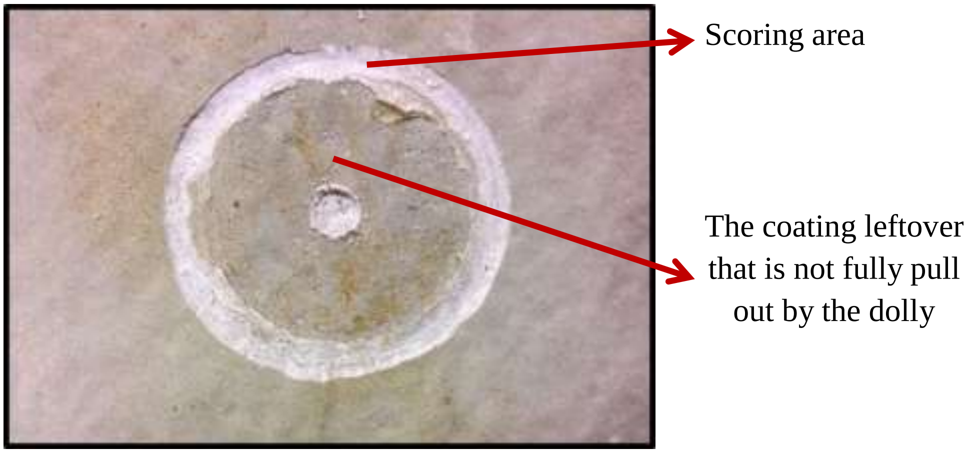


Figure 4.5.3: Surface of 1000µ after the pull off adhesion test is done

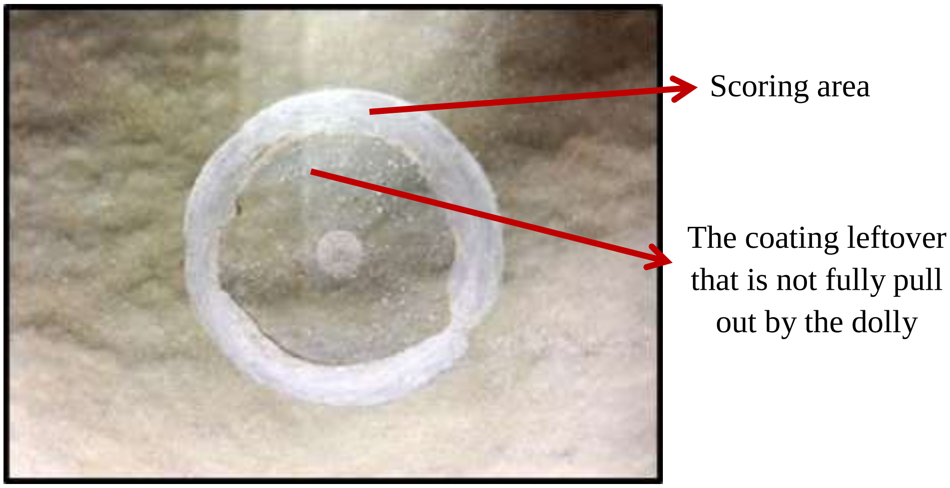


Figure 4.5.4: Surface of 1500µ after the pull off adhesion test is done

The coating is not fully pulled out from the substrate. There is coating leftover remain on the sample due to the strength of the araldite that is not sufficient to pull out the polyester glass flake from the substrate. Even though in many case studies, epoxy and cyanoacrylate are commonly used as adhesive glue, it is not working for this study because the type of coating system is different and stronger compare to the coating system used in other case studies. The example of coating systems that commonly tested in other case studies is coal tar epoxy, polyurethane, and etc. The polyester glass flake could be fully pulled out by the dolly if there is type of glue that can provide higher strength of adhesiveness compare to the araldite.

CHAPTER 5: CONCLUSIONS & RECOMMENDATIONS

5.1 Conclusions

Based on results collected from the testing, glass flake reinforced polyester coatings have proven that these coatings are excellent in chemical resistance, offer low penetration rate, and provide prevention of cracks and pilling to occur. Furthermore, these coatings also have a proven track record in many environments, including seawater; the expectation of its service life could be 20 or 30 years longer in aggressive service applications.

5.2 Recommendations

- The experiment duration should be extended to more days/months/year
- Repetition of the test due to essential for validate the results
- For further study, it is recommended to conduct experiment at real condition of seawater which expected to have more corrosion rates
- In order to conduct pull-off adhesion test for glass flake reinforced coatings, use type of glue that can provide higher strength of adhesiveness compare to the araldite

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