A Study of Electrochemical Measurements Under Microemulsion Condition Corrosion

by

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Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Mechanical Engineering
With Honours

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CERTIFICATION OF APPROVAL

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(MECHANICAL)

Approved by,

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(AP Ir Dr Mokhtar B Che Ismail)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
January 2020

CERTIFICATION OF ORIGINALITY

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

NUR ASHILA NADHIRAH BINTI MUZIR

ABSTRACT

Corrosion is one of the main integrity threats in crude oil pipeline transportation. Accurate online corrosion monitoring is critical to the effective implementation of corrosion mitigation method. Current electrochemical methods of linear polarization resistance (LPR) relies on high conductivity of interfacial solution. The challenge in measurement is due to microemulsion condition has low conductivity of interfacial solution which affect the accuracy of the LPR. The study was undertaken to evaluate the effect of microemulsion on the accuracy of linear polarization resistance (LPR) and electrochemical impedance spectroscopy (EIS) electrochemical measurement. The experimental work consists of using mild steel as the working electrode for LPR and EIS test. The solution for both tests is microemulsion solution that includes 30%, 50% and 70% water cutting as the variable in the experiment. Weight loss coupons also will be conducted to examine the corrosion rate in microemulsion condition. The expected outcome of this venture is the reliability that can be compared with both electrochemical approaches when measuring the corrosion rate in low conductivity solution as well as the result from the weight loss coupon method.

ACKNOWLEDGEMENT

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In addition, I would love to thank my parents and families for their endless support for me throughout my study days., I would like to express my gratitude to my colleagues for always supporting and helping each other in facing the journey together. Not only that, I would also like to thank Mr. Jamalulhaq who had provided me guidance and a helping hand in conducting my project experimental work.

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Table of Contents

List of Figures

List of Tables

ABBREVIATIONS AND NOMENCLATURES

LPR	Linear Polarization Resistance
EIS	Electrochemical Impedance Spectroscopy
O/W	Oil-in-water
W/O	Water-in-oil
W/O/W	Water-in-oil-in-water
O/W/O	Oil-in-water-in-oil
EOR	Enhanced Oil Recovery
Rp	Polarization Resistance
i_{corr}	Corrosion Current Density

CHAPTER 1

INTRODUCTION

1.1 Background

The substance that is being transported in pipelines at offshore platform does not contain only crude oil, but also other substances such as water and gas [1]. The other elements are such as hydrogen sulphide gas, carbon dioxide and sand. Water mixing with the oil is a major problem for the industry especially the oil and gas where it leads to corrosion problem along the pipeline.

There are many ways and techniques have been applied to prevent corrosion from happening. Scientists and engineers working together to solve the corrosion problem where, if there are corrosion attack at the pipeline, it can automatically affect the production rate thus, slows the production and lead to profit loss [2].

Corrosion monitoring is one of the techniques that have been applied to overcome the issue. Field Corrosivity Toolbox are one of the corrosion monitoring method being used where it uses linear polarization resistance (LPR) method measuring fluid corrosivity. However, problem arises when the water used for testing contains microemulsion. LPR has failed to provide an accurate result for corrosion monitoring as the water are not conductive enough due to the microemulsion.

1.2 Problem Statement

The presence of microemulsion are affecting the measurement of corrosion rate. Fluid conductivity decreases thus, the rate of corrosion obtained is not accurate. The corrosivity of the fluid contain microemulsions should be measured using corrosion monitoring techniques to predict the corrosion activity that may occur.

1.3 Objective

The objective of this research was to evaluate electrochemical corrosion measured under microemulsion condition as found in crude oil transporting pipelines.

The objectives of this project would be:

1. To run electrochemical impedance spectroscopy (EIS) method measuring the fluid corrosivity.
2. To compare between electrochemical impedance spectroscopy (EIS) and Linear Polarization Resistance (LPR) method for corrosion monitoring.

1.4 Scope of Study

The project focused on the offshore pipeline that transport the crude oil. In the pipeline, the oil is transmitted along with other substances mainly water that can lead to corrosion problems. The microemulsion inside the water is an issue that affecting the measurement of corrosion monitoring. Three corrosion monitoring methods that has been performed were linear polarization resistance (LPR), electrochemical impedance spectroscopy (EIS) and weight loss coupon test.

CHAPTER 2

LITERATURE REVIEW

2.1 Corrosion

Corrosion is degradation of material when it reacts with the environment. There are two principles of corrosion that is thermodynamics principle and electrochemical principles [3]. As for thermodynamic principles, it is the spontaneous chemical reaction direction. It is used to determine whether the corrosion is possible theoretically or otherwise. Meanwhile, principles of electrochemical are mainly used to determine the corrosion behaviour of the materials.

Corrosion is a natural phenomenon which is impossible to be prevented fully. However, it can be controlled, and one can measure the possibility of when, where and why corrosion can occur at respective area. The important elements for electrochemical corrosion to occurs consists of an anode, a cathode, metallic and electrolyte [4]. An anode is where oxidation occurs that is, the metal tends to corrode while the cathode is reduction reaction takes place. Metallic is the electrically conductive path where there is connection between the anode and cathode. The electrolyte is defined as the environment which is the ionic conductive path that contains the electroactive species.

In pipelines transporting the crude oils, it also contains other foreign substance. The active mechanisms that lead to corrosion and the rate depends on the chemical composition of the fluid [5]. The composition of water in oil and gas pipelines are main criteria of fluid’s corrosivity.

Corrosion monitoring is a method that measures and observe the change of materials. The change of materials can be monitored by either the alteration of physical, chemical, mechanical properties, the weight loss or the loss of integrity of components.

2.2 Corrosion Monitoring

Most of the corrosion monitoring probes focuses on providing the level of damage has been sustained [6] . The invention of corrosion monitoring should not only to sustain the damage but also to prevent corrosion from happening. Conventional corrosion instrumentation is known to be incapable of detecting the localized corrosion such as cracking, pitting and crevice attack. It also failed in providing the real-time indication of the damage progress [6]. To overcome this issue, on-line corrosion monitoring is introduced to measure the corrosion rate where the conventional corrosion instrumentation incapable of. Electrochemical impedance spectroscopy (EIS), electrochemical noise (EN) and harmonic analysis (HA) is the example of the on-line corrosion monitoring.

For the industries, plants or machines are being inspected and monitored from time to time. The term inspection and monitoring may confuse some people who does not know the meaning of it. Inspection is a condition periodic evaluation done by the expert technician to achieve small number of very quality measurements [6]. Meanwhile, monitoring is said to be a low-cost techniques to obtain a large volume of moderate- quality measurements. For example, inspection is being done once or twice a year while monitoring is done to predict the conditions of the plants or machines.

Corrosion monitoring should be implemented to have the continuous data and information that can help to predict the rate of attack. By this said, the operator or workers can take immediate action if the data taken is significantly different from usual. Most importantly, it helps to ensure the operability and overall integrity of the respective subject. Table 2.1 showed the corrosion monitoring advantages.

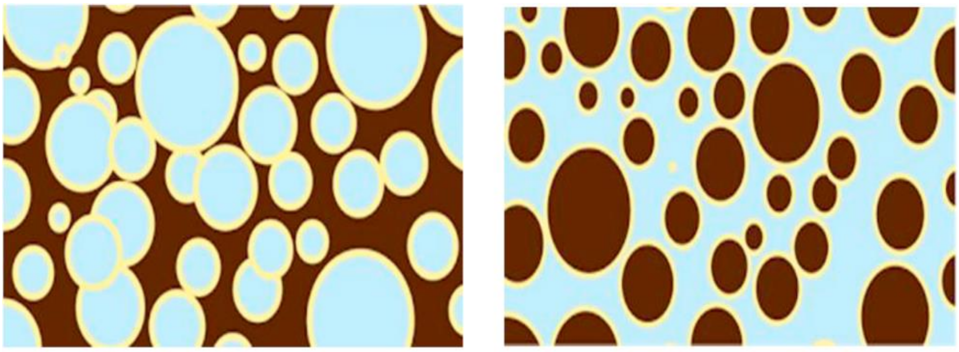
TABLE 2.1 Advantages of Corrosion Monitoring

No.	**Advantages of corrosion monitoring**
1	To identify when maintenance action should be taken
2	To establish the inspection intervals
3	To determine if there are active corrosion occurs
4	To ensure the effectiveness of cathodic protection applied

2.3 Emulsion in Crude Oil

Emulsions is defined as metastable systems formed when there are amphiphilic polymers, surfactant molecules or solid particles presents [1]. In oil production, emulsions occur in pipelines, well bores and at surface facilities. The emulsions increases when production time of the oil wells increases [1].

There are few types of emulsions that is water-in-oil emulsion (W/O), oil-in-water (O/W) emulsion and also multiple emulsion [2]. W/O is where there are water globules scattered throughout the continuous oil phase while O/W is otherwise. O/W is oil globules dispersing throughout the water continuous phase. Figure 2.1 shows the W/O and O/W emulsion. Multiple emulsion is where there are water-in-oil-in-water (W/O/W) and oil-in-water-in-oil (O/W/O) emulsion. The most common emulsion that occurs in oil and industries are the W/O emulsions which also known as “chocolate mousse” [2].



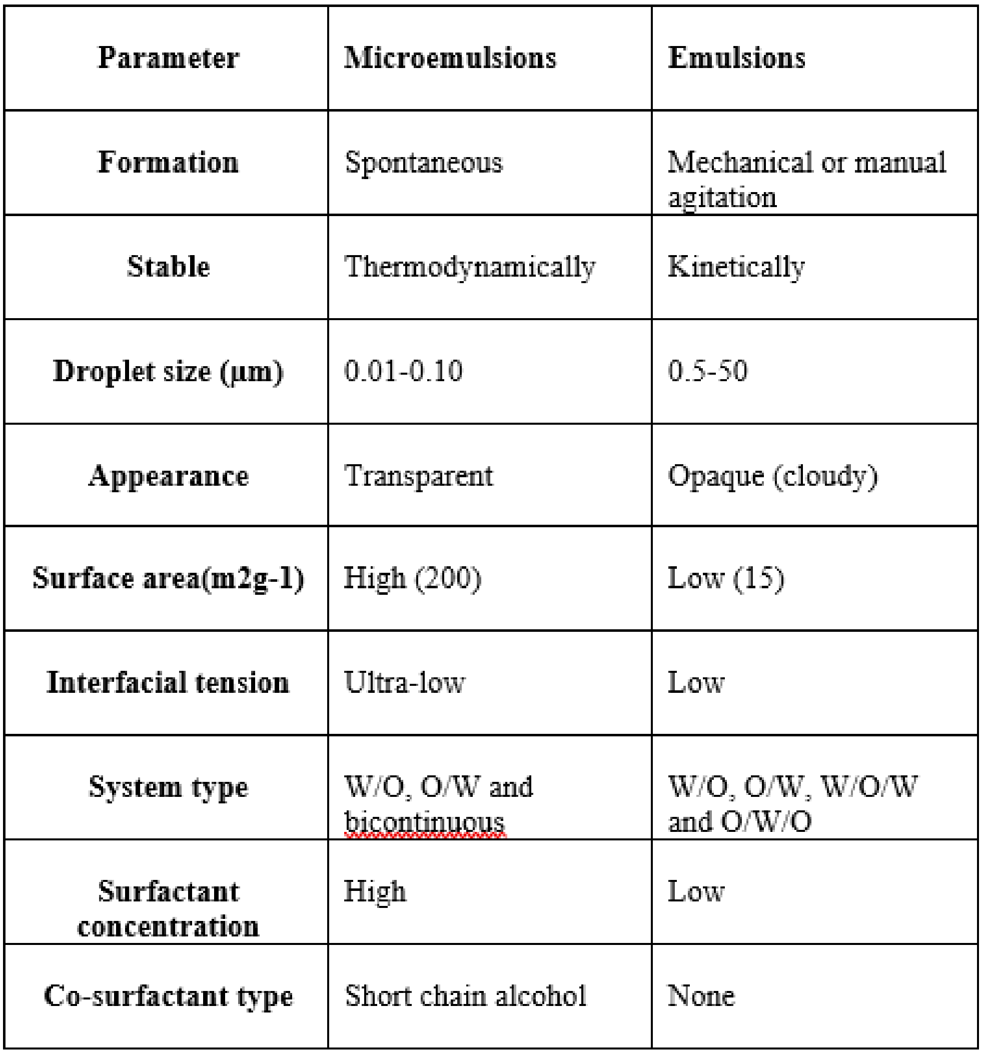
(a) Water-in-Oil Emulsion (b) Oil-in-Water Emulsion (Nalco)

FIGURE 2.1 Microemulsion In (A) Water-In-Oil And (B) Oil-In-Water [1].

Emulsions in pipelines, are the unwanted substances as it can lead to numerous problems such as reduces quality of crude oil, increases the operating cost and causes corrosion to occurs which will contaminate the pipeline and the crude oil itself [2]. In addition, Santos and others (2017) states that emulsion will increase the oils' viscosity which will affect the production rate to decrease [7].

Crude oils contain few components that can lead to formation of emulsions that is also known as emulsification. The components that is important for emulsion stability is the resin and asphaltenes of the crude oil [1]. Other than that, chemical enhanced oil recovery (EOR) techniques also known to process stable water-in-oil emulsions and oil-in-water emulsions. The alkali, surfactants and polymers used in EOR technique are the factors for the stable emulsions produced.

TABLE 2.2 Characteristics Of Emulsions And Microemulsions [8].



Parameter	Microemulsions	Emulsions
Formation	Spontaneous	Mechanical or manual agitation
Stable	Thermodynamically	Kinetically
Droplet size (µm)	0.01-0.10	0.5-50
Appearance	Transparent	Opaque (cloudy)
Surface area(m2g-1)	High (200)	Low (15)
Interfacial tension	Ultra-low	Low
System type	W/O, O/W and bicontinuous	W/O, O/W, W/O/W and O/W/O
Surfactant concentration	High	Low
Co-surfactant type	Short chain alcohol	None

Referring to Table 2.2 above, there are listed few characteristics that differentiate emulsions and microemulsions.

2.4 Microemulsion

Microemulsion are emulsions of when droplet size of dispersed phase are not more than 0.1 µm. Microemulsion were characterized as dispersion of two non-miscible liquids which usually are water and oil and stabilized by a surfactant [8]. In addition, Thulio, Carlos and others stated that microemulsion are thermodynamically stable systems of two non-miscible liquids with surfactants and/or co-surfactants being adsorbed in the interface between two phases [9]. Microemulsion are thermodynamically stable compared to emulsion because the interfacial tension of microemulsion are lower which leads to the interfacial energy to be very small. Hence, free energy of the droplet formation will be lower than zero ($\Delta G > 0$).

Microemulsion is transparent and it occurs in both as oil-in-water or water-in-oil [1]. Referring to Figure 2.2, it shows the type of microemulsion consist of oil-in-water, water-in-oil and bicontinuous microemulsion. Bicontinuous microemulsion is where the oil and aqueous phase microdomain are randomly linked to approximately equal volume [10].

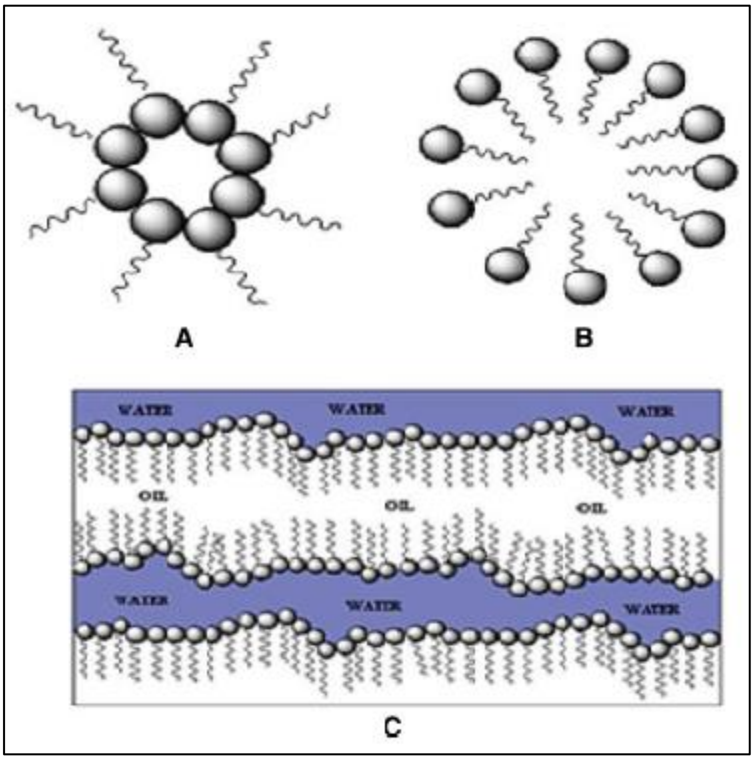


FIGURE 2.2 Water-in-Oil (A), Oil-in-Water(B) and Bicontinuous Microemulsion (C) [11].

In addition, surfactant is known to help emulsification process by reduces the interfacial tension and stabilization by having the double layer forces and solvation forces among the particles [10]. Barbosa, and his collaborators prepared the microemulsion by mixing babassu oil, ethanol solution and isobuthanol in aqueous solution and the experiment is done by titration procedure [8]. Co-surfactant is added to increase the flexibility of amphiphilic monolayer.

2.5 Linear Polarization Resistance

Linear polarization resistance (LPR) is one of the corrosion monitoring techniques which gives the corrosion rate to be measured directly in real time. The measurement includes the polarizing the metal and measures the resulting current which was introduced by Stern and Geary [12]. Small voltage perturbation is applied where the current will be linearly related with the voltage and thus, give out a constant called polarization resistance (R_p).

$$\mathrm{Rp} = \frac{\Delta \mathrm{E}}{\Delta \mathrm{i}} \tag{1}$$

Rp – polarization resistance (Ohm)

ΔE – potential difference (V)

Δi – change in current (A)

Corrosion rate can be measured by converting the polarization resistance using Stern-Geary equation.

$$i_{corr} = \frac{\mathrm{B}}{R_p} \tag{2}$$

Where, i_{corr} – corrosion current density, $A.cm^{-2}$

B – Stern-Geary constant

Equation (3) shows calculation of corrosion rate if given R_p and i_{corr}.

$$CR = \frac{0.00327 \times i_{corr} \times {}^{M}/_{n}}{\rho} \tag{3}$$

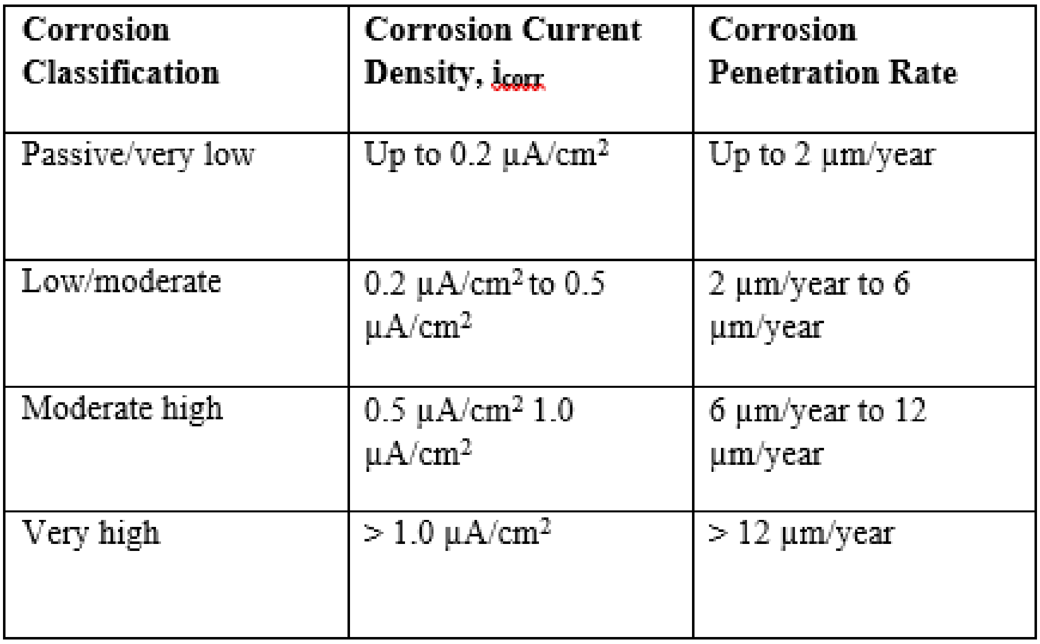
Where, CR – corrosion rate, mm/yr

M/n – Equivalent weight, gm

P – density of metal

Table 2.3 states the typical corrosion rates values by LPR test.

TABLE 2.3 Values of Corrosion Rate from LPR Measurements (Sadowski, 2010).



Corrosion Classification	**Corrosion Current Density, i_{corr}**	**Corrosion Penetration Rate**
Passive/very low	Up to 0.2 µA/cm^2	Up to 2 µm/year
Low/moderate	0.2 µA/cm^2 to 0.5 µA/cm^2	2 µm/year to 6 µm/year
Moderate high	0.5 µA/cm^2 1.0 µA/cm^2	6 µm/year to 12 µm/year
Very high	> 1.0 µA/cm^2	> 12 µm/year

2.6 Electrochemical Impedance Spectroscopy

Electrochemical Impedance Spectroscopy (EIS) is method to study the response of system to small application of amplitude AC signal in different frequencies [13]. AC potential applied to an electrochemical cell and current will be measured. EIS uses small excitation signal as expected response is pseudo-linear. Impedance is defined as ability of a circuit resisting electrical current flow. Impedance is more general circuit parameter compares to other parameter such as the resistance and current. Resistance, R is the ratio of voltage, V and current, I.

$$V = I \times R \tag{4}$$

An imposition of current or frequency dependent voltage will form a non-sero frequency which Ohm's law is:

$$V = I \times Z \tag{5}$$

Where Z is the proportionality factor where the total of all elements that can against the current or also known as the impedance. Impedance consists of real and imaginary components.

In electrochemical experiments, working electrode will be perturbed with sinusoidal potential signal V(t) with numbers of discrete frequency f as shown in equation 5:

$$V(t) = V_A \sin(\omega t) \tag{6}$$

Where the radial frequency, ω is equal to $2\pi f$. Sinusoidal current signal I(t) will be the response which:

$$I(t) = I_A \sin(\omega t + \varphi) \tag{7}$$

The sinusoidal current signal I(t) have same frequency as the potential signal with certain phase angle shift, φ. Impedance can be calculated by:

$$Z = \frac{V(t)}{I(t)} = Z_{real} + jZ_{img} \tag{8}$$

The complex unit, j is equal to $\sqrt{-1}$. The absolute value of impedance, $|Z|$ calculated as follows:

$$|\mathrm{Z}| = \sqrt{(Z_{real})^2 + (Z_{img})^2} \tag{9}$$

2.7 Weight Loss Coupons

Weight loss coupons or corrosion coupons are one of the oldest and most used corrosion monitoring method being used in the industry [14]. It is a straightforward, direct indication of the corrosivity of the systems. ASTM G1, ASTM 31 and ASTM G46 will be referred for the evaluation of the coupons.

To measure the weight loss, there are two types of results obtained from the coupons that is:

$$Scale\ weight = W_2 - W_3 \tag{10}$$

$$Mass\ Loss\ (M) = W_1 - W_3 \tag{11}$$

The scale weight and mass loss values units are mg/cm^2 and in terms of weight per unit exposed surface are of the coupons. To measure the corrosion rate is by equation below:

$$Corrosion\ rate\ (mpy) = \frac{534\ (M)}{D.A.T} \tag{12}$$

Where, D = density of the alloy (g/cm^3)

A = coupon exposed surface area (in^2)

T = Exposure time (hour)

From ASTM G31, the calculation for corrosion rate of immersion test is as shown below.

$$corrosion\ rate\ \left(\frac{mm}{yr}\right) = \frac{k \times \Delta w}{A \times T \times \rho} \tag{13}$$

Δw = Weight loss (g)

K = Constant for unit conversion (8.76 x 10^4)

ρ = Density of mild steel (7.86g/cm^3)

A = exposed surface area (cm^2)

The corrosion rate values can be converted from mpy to millimetres per year(mmpy) by dividing the values by 40.

CHAPTER 3

METHODOLOGY

3.1 Project workflow

Figure 3.1 is showing the project work flow chart.

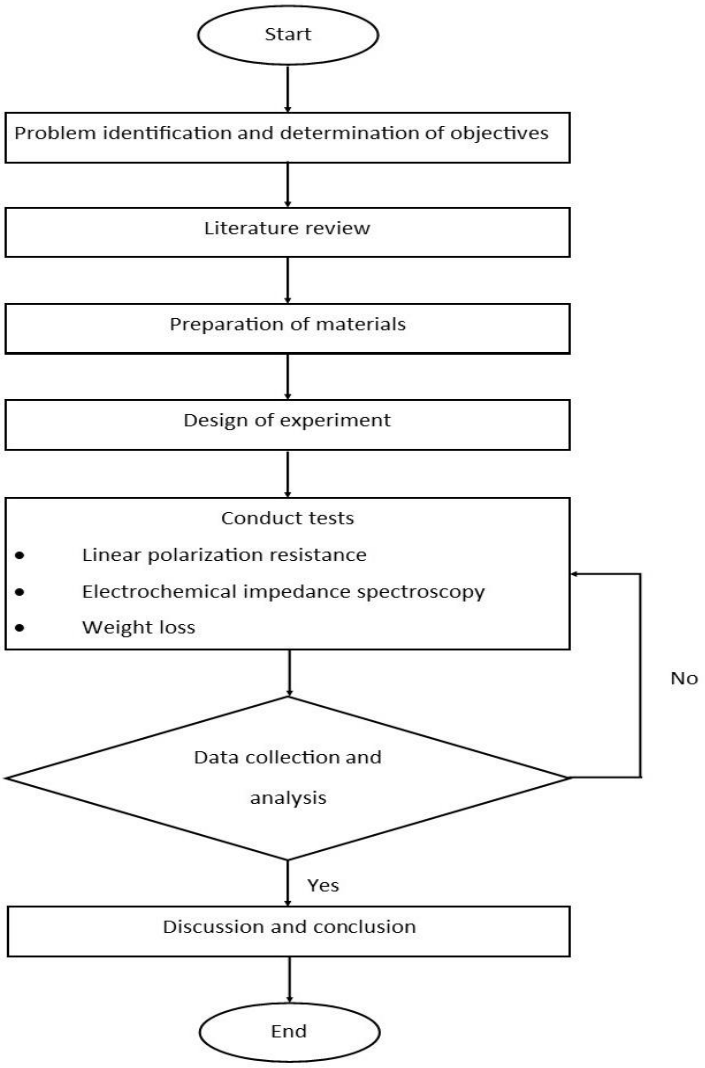


FIGURE 3.1 Flow Chart for FYP

3.2 Experimental Details

Figure 3.2 showing the design of experiment for project.

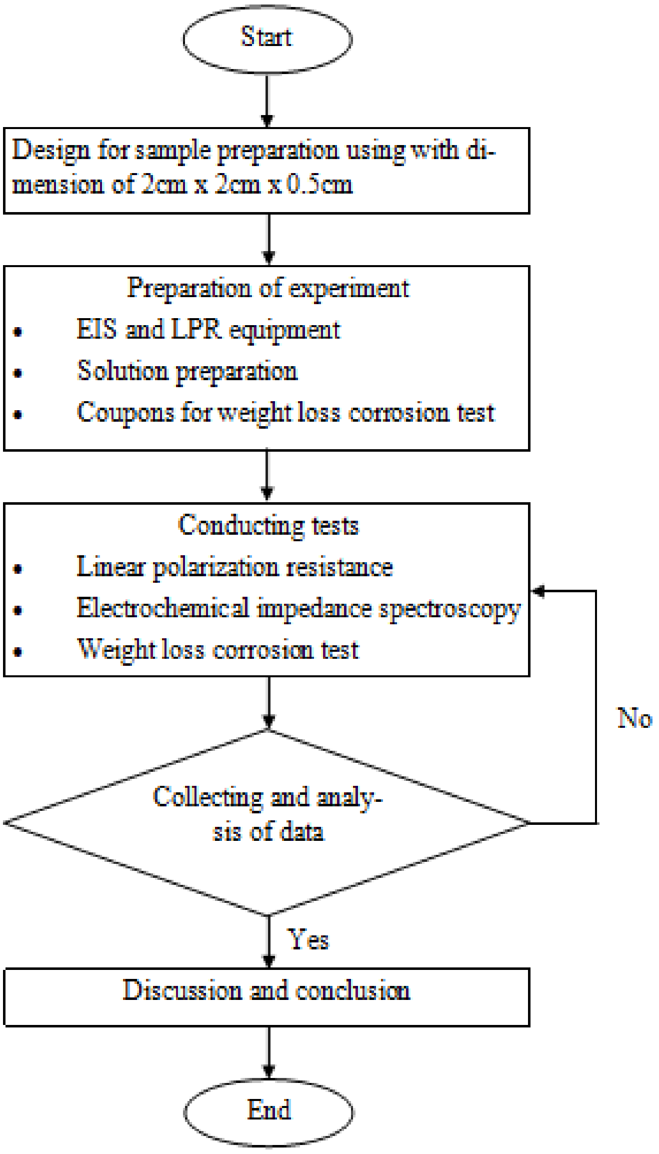


FIGURE 3.2 Design of Experiment

Test matrix for the whole experiments in conducting this project are shown in the Table 3.1 below.

TABLE 3.1 Test Matrix

Material	Mild steel
Dimension (Length x Width x Thickness, cm)	1cm x 1cm x 0.5cm
Number of samples	3
Solution (electrolyte)	i. 30% water cutting ii. 50% water cutting iii. 70% water cutting
Test method	i. Electrochemical Impedance Spectroscopy ii. Linear Polarization Resistance iii. Weight Loss Coupon Test
Test Environment	Carbon dioxide
Test duration (hours)	168 hours
Result Evaluation	Graph analysis, corrosion rate calculation and comparison

3.2.1 Sample Preparation

For LPR and EIS experiment, there are three electrodes required that are working electrode, auxiliary electrode and reference electrode. Carbon steel will be used as the working electrode. The carbon steel will be prepared based on Table 3.1. It will be connected to copper wire in certain length and will be mounted in epoxy resin by cold mounting. After that, the exposed surface area will be grinded and polished using silicon carbide (SiC) paper. The grade of silicon carbide paper that will be used is 60, 240, 400 and 600 grit SiC paper.

3.2.2 Solution Preparation

The solution used was the mixture of brine solution and Isoparaffinic Hydrocarbon (Isopar M) together. The following steps were:

1. 105 g of sodium chloride was weighed using a weighing balance.

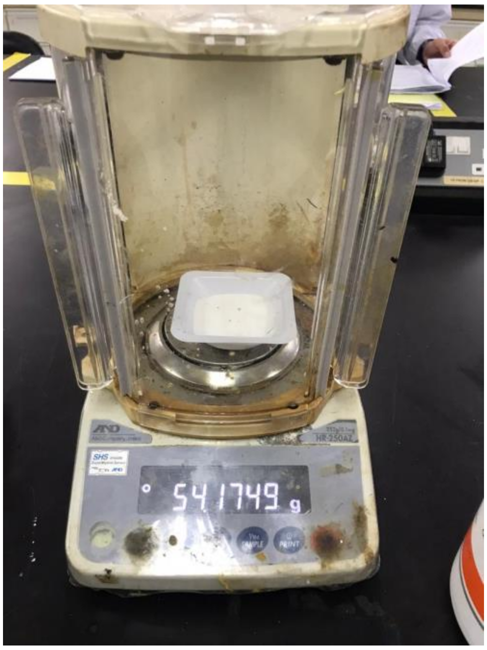


FIGURE 3.3 Sodium chloride being weighed

2. 3000 mL of deionized water poured into a glass beaker.
3. 105 g of NaCl were poured into the 3000mL of deionized water and stirred until all the NaCl fully dissolved.
4. Using a filter funnel, poured the brine solution into three separate separatory funnels with 600mL, 1000mL and 1400mL each.
5. Measured the isopar M with different volume of 1400mL, 1000mL and 600mL using the measuring cylinder.

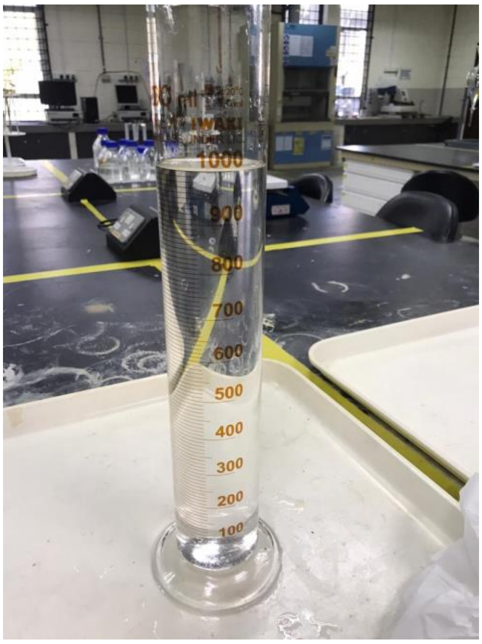


FIGURE 3.4 Isopar-M being measured by measuring cylinder.

6. Poured the 1400mL Isopar M into separatory funnel that contained 600mL of brine solution.
7. Then, 1000mL of Isopar M were put into the separatory funnel which held 1000mL of brine solution.
8. After that, poured 600mL of Isopar M into the separatory funnel with 1400 mL of brine solution.

FIGURE 3.5 Isopar-M and brine solution inside separatory funnel

9. The separatory funnels would then be shaken vigorously for two minutes to mix the solution.
10. The solutions were let to rest for 24 hours.
11. After 24 hours, the mixture of brine solution and Isopar-M were separated leaving the remaining Isopar-M in the separatory funnel.

FIGURE 3.6 Solution separating process

3.3 Linear Polarization Resistance Test

Figure 3.7 shows the flow chart of LPR test that will be conducted. Table 3.2 shows the test matrix for LPR test.

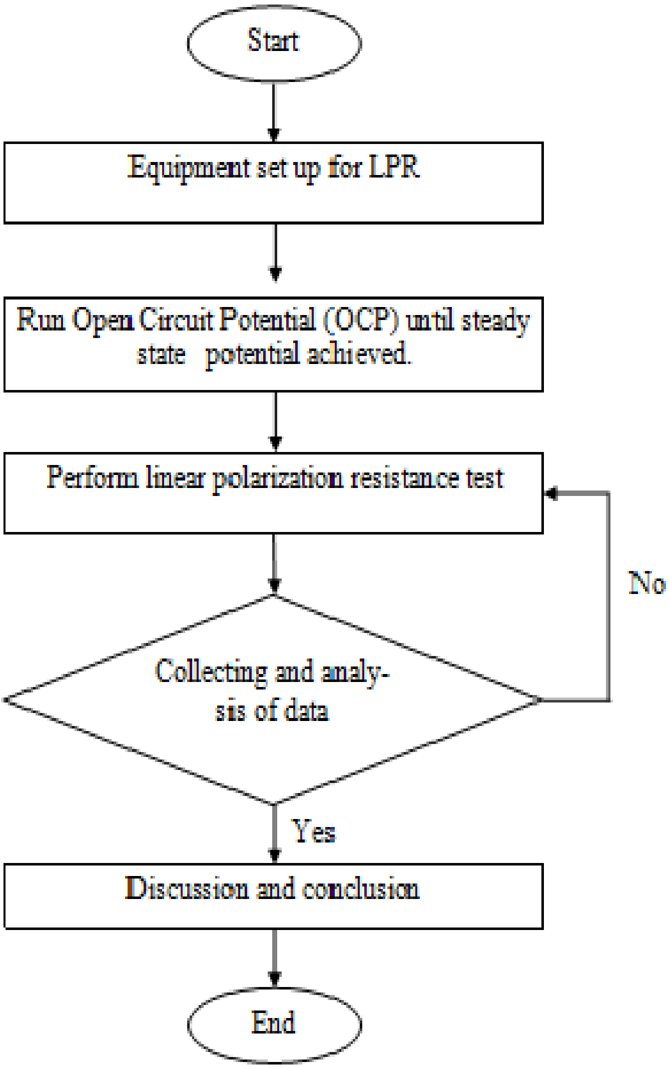
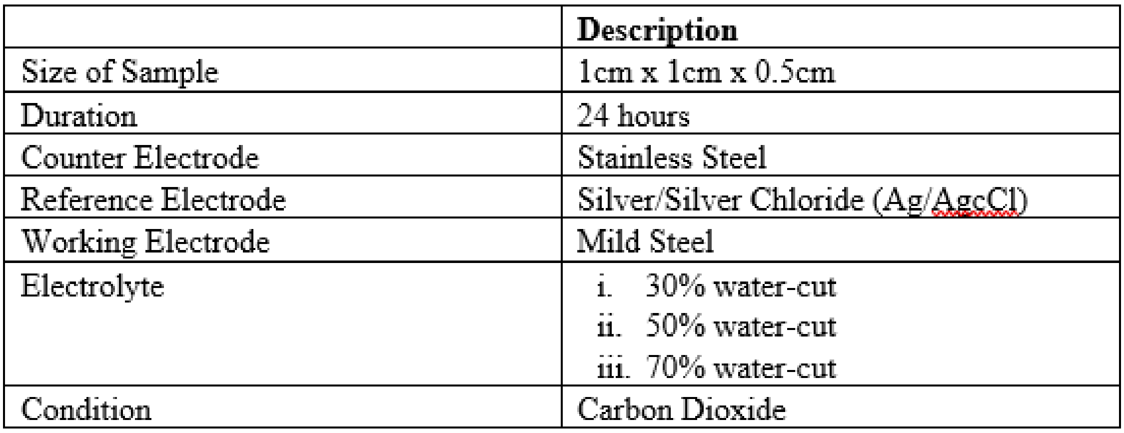


FIGURE 3.7 LPR test process flow chart

TABLE 3.2 Test Matrix for linear polarization resistance



	Description
Size of Sample	1cm x 1cm x 0.5cm
Duration	24 hours
Counter Electrode	Stainless Steel
Reference Electrode	Silver/Silver Chloride (Ag/AgcCl)
Working Electrode	Mild Steel
Electrolyte	i. 30% water-cut ii. 50% water-cut iii. 70% water-cut
Condition	Carbon Dioxide

To run LPR, potentiostat will be connected to the three electrodes with a computer control system. Potentiostat are used to verify the electric potential difference. The open circuit potential (OCP) were recorded before running LPR test. It will run after 24 hours.

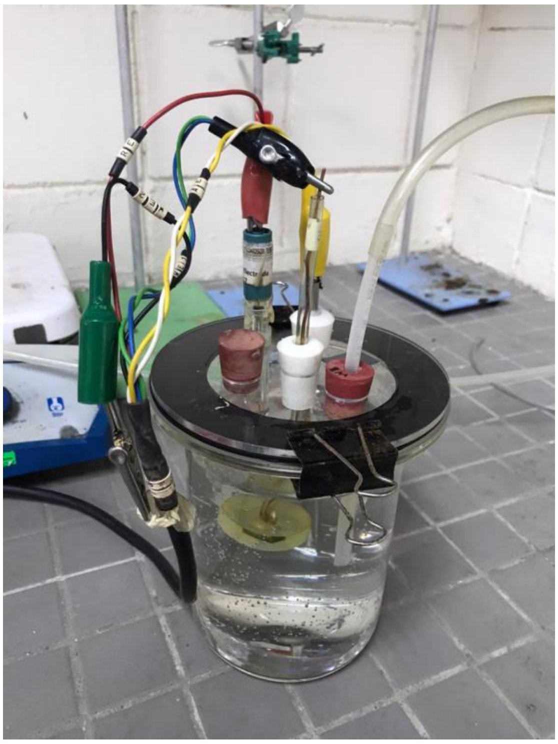


FIGURE 3.8 EIS experiment set-up

3.4 Electrochemical Impedance Spectroscopy Test

The equipment setup for EIS test are same as LPR test. Open circuit potential (OCP) will be run before the reading starts to ensure the results are correct. The reading will be taken after 24 hours. Figure 3.9 shows the process for EIS experiment that will be performed.

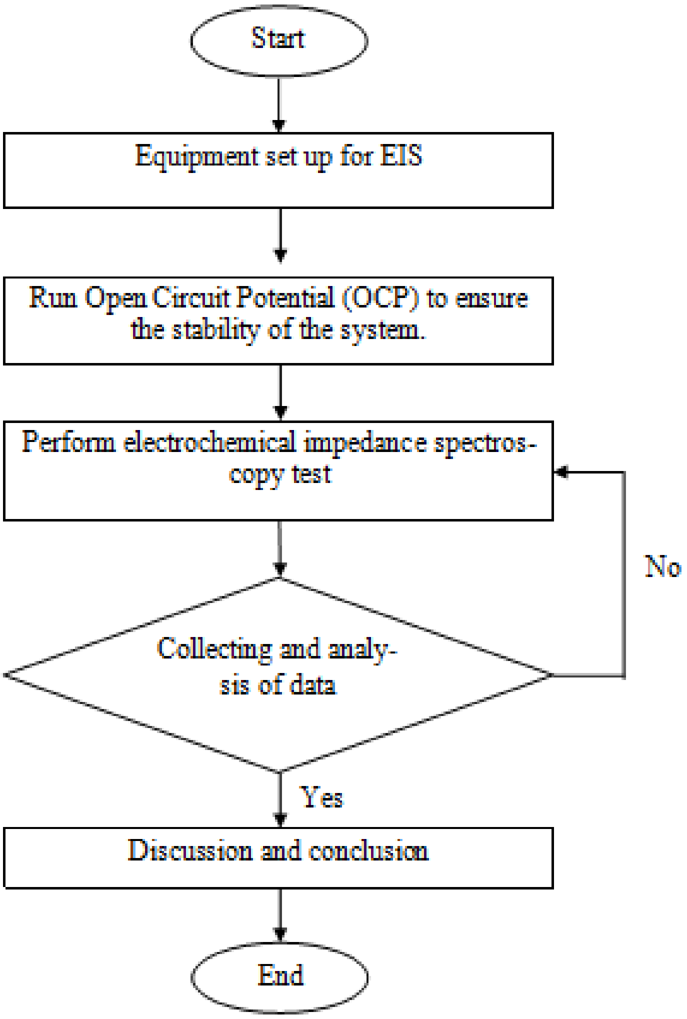


FIGURE 3.9 EIS test process flow chart

3.5 Weight Loss Coupon Test

Figure 3.10 shows the weight loss test flow chart.

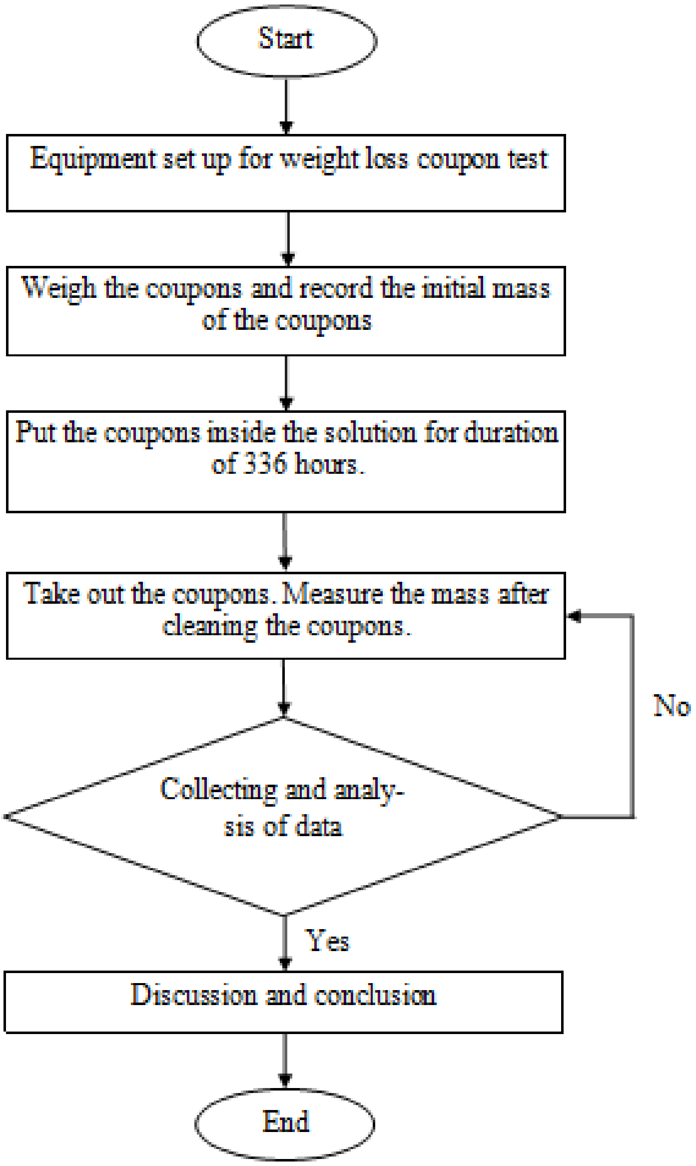


FIGURE 3.10 Weight loss coupon test process flow chart

The sample preparation for weight loss coupons are as below:

1. Grind all the surface area using the grinding and polishing machine with the silicon carbide paper of grade 60, 80, 120, 240, 400 and lastly 600.
2. Record the weight of each samples using the weighing balance.

3. Measure the dimension of the samples to calculate the area of each sample.

FIGURE 3.11 Measuring the dimension of the sample

4. Samples will be immersed inside the solution for duration of one week.

For cleaning process, the samples were cleaned using the ultrasonic bath method. The samples were weighed, and new weight were recorded. the new weight would be taken at least five times. The weight loss was calculated.

3.6 Project Management

3.6.1 Gantt Chart

Table 3.3 shows gantt chart for research and project planning for FYP 1

TABLE 3.3 Gantt Chart

Activity	**Week**													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Research														
Identification of problem statement and objectives		🟨	🟨	🟨										
Literature review on electrochemical measurement method and microemulsion condition		🟨	🟨	🟨	🟨	🟨	🟨	🟨	🟨	🟨 ▲				
Preparation for proposal defence presentation						🟨	🟨	🟨 ▲	🟨 ▰	🟨				
Project Planning/Methodology														
Design of experiment • Electrochemical Impedance Spectroscopy • Linear Polarization Resistance • Weight Loss Test						🟨 ▰	🟨	🟨	🟨	🟨	🟨	🟨	🟨	
Design for preparation of materials • Sample preparation • Solution preparation								🟨	🟨	🟨	🟨 ▰	🟨	🟨 ▲	
Equipment and materials for experiment • To ensure the availability of equipment needed for experiments											🟨	🟨	🟨	🟨 ▲
Interim report preparation						🟨	🟨	🟨	🟨	🟨	🟨	🟨	🟨	🟨 ▰

Legends:

Project Milestone	▲	FYP Milestone	▰

3.6.2 Project and FYP Key Milestone

Table 3.4 below show the key milestone for both project and FYP 1.

TABLE 3.4 Project and FYP 1 Key Milestone

	Week	Date		FYP 1 ACTIVITIES
		Start	End	
FYP MILESTONE	6	7/10/2019	13/10/2019	Submission of Progress Assessment 1
	9	28/10/2019	03/11/2019	Proposal Defense Presentation
	11	11/11/2019	17/11/2019	Submission of Progress Assessment 2
	14	02/12/2019	06/12/2019	Submission of Interim Report
PROJECT MILESTONE	8	-	27/10/2019	Completion of proposal defense preparation
	10	-	10/11/2019	Completion of experimental test matrix
	13	-	25/11/2019	Completion of design of experiment
	14	-	06/12/2019	Completion of equipment and materials gathering

3.6.3 Gantt Chart

Table 3.5 shows gantt chart for research and project planning in FYP 2.

TABLE 3.5 Gantt Chart for FYP2

Activity	**Week**													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Research														
Literature review for experiment procedure and result analysis	▨	▨	▨	▨	▨	▨	●							
Preparation for pre-SEDEX	▨	▨	▨	▨	▨	▨	▨	▨	▨	●				
Project Planning/Methodology														
Preparation for experiment • Safety briefing • JSA, HIRARC and lab booking	▨	▨	▨	▲										
Preparation of materials • Sample preparation • Solution preparation				▨	▨	▨	▨	▨	▲	▨	▨	●		
Experimental Testing • LPR • EIS • Weight loss					▨	▨	▨	▨	▨	▨	▨	▲		
Dissertation preparation						▨	▨	▨	▨	▨	▨	▨	▨	●

Legends:

Project Milestone	▲	FYP Milestone	●

3.6.4 Project and FYP Key Milestone

Table 3.6 below show the key milestone for both project and FYP.

TABLE 3.6 Key Milestone for FYP 2

	Week	Date		FYP 1 ACTIVITIES
		Start	End	
FYP MILESTONE	7	17/02/2020	21/02/2020	Submission of Progress Report
	10	09/03/2020	13/03/2020	Pre-SEDEX
	11	16/03/2020	20/03/2020	Submission of Draft Final Report
	12	23/03/2020	27/03/2020	Submission of Dissertation (soft copy)
	12	23/03/2020	27/03/2020	Submission of Technical Paper
	14	06/04/2020	10/04/2020	Viva
	15	13/04/2020	17/04/2020	Submission of Project Dissertation (Hard Bound)
PROJECT MILESTONE	4	-	31/01/2020	Completion of safety briefing and lab booking
	9	03/02/2020	06/03/2020	Completion of experimental testing
	12	-	25/11/2019	Completion of test result analysis

CHAPTER 4
RESULTS AND DISCUSSION

4.1 Experimental Result by Electrochemical Impedance Spectroscopy

The result obtained after three different samples were immersed in three different water-cut percentage which were 30%, 50% and 70% water-cut after being exposed for 24 hours. Figure 4.1 and Figure 4.2 shows bode plot and Nyquist plot of 30% water-cut. Corrosion rate were calculated when corrosion current density, i_{corr} had been measured. Polarization resistance obtained from Nyquist plot were used to measure the i_{corr}.

TABLE 4.1 Corrosion Rate of EIS.

Solution	**R_p (ohm)**	**Icorr(μA/cm^2)**	**Corrosion rate (mm/yr)**
30% Water-cut	126.61	205.36	2.38
50% Water-cut	118.04	220.26	2.55
70% Water-cut	112 .87	230.35	2.672

To measure corrosion rate, equation (3) had been utilised with density of 7.8 g/cm^3 and equivalent weight of 27.92 gm.

Based on Figure 4.1, the impedance decreased when the frequency increased. The impedance decreased from 75.122 ohm.cm^2 to 1.5513 ohm.cm^2.

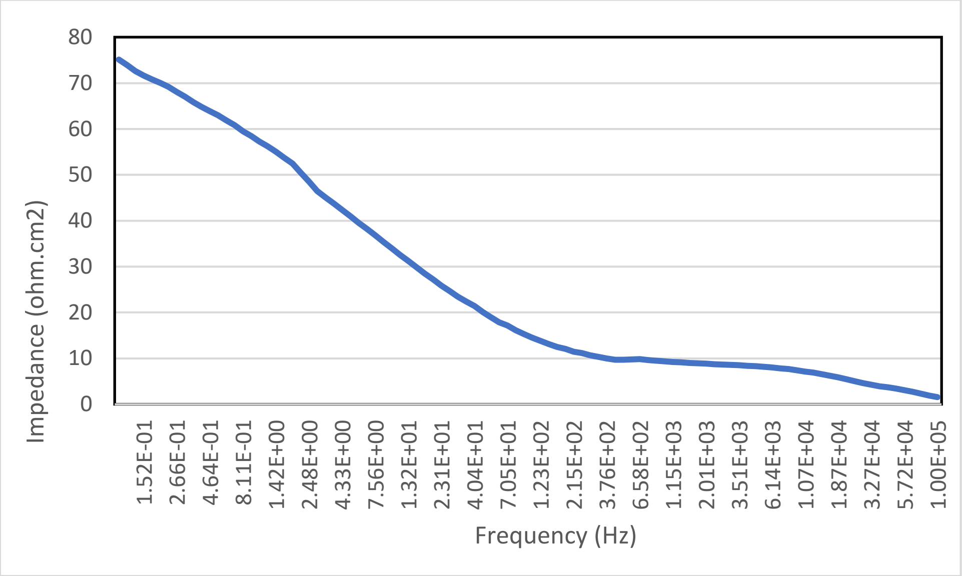


FIGURE 4.1 Bode Plot of 30% Water-cut

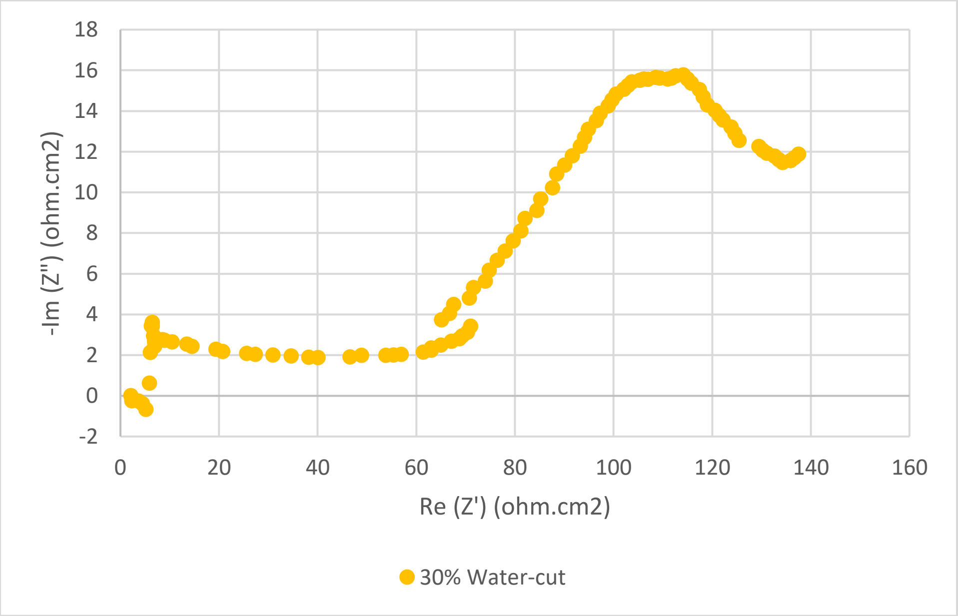


FIGURE 4.2 Nyquist Plot for 30% Water-cut

Figure 4.3 and Figure 4.4 plotted the bode plot and Nyquist plot for 50% water-cut. The impedance decreased from 44.331 ohm.cm^2 to 1.0642 ohm.cm^2.

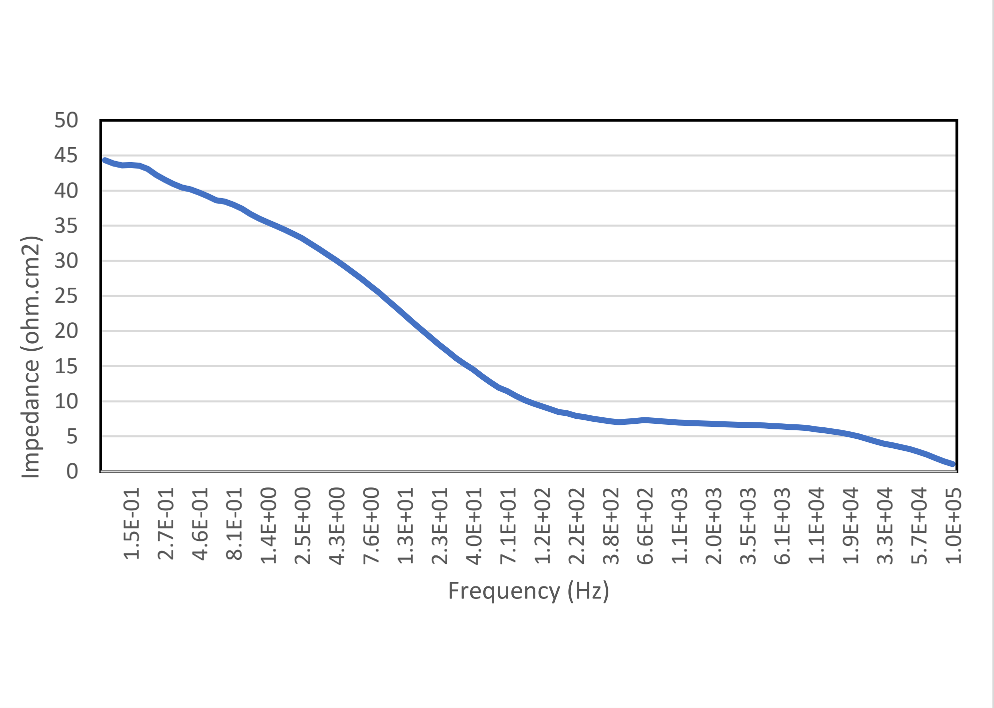


FIGURE 4.3 Bode Plot of 50% Water-cut

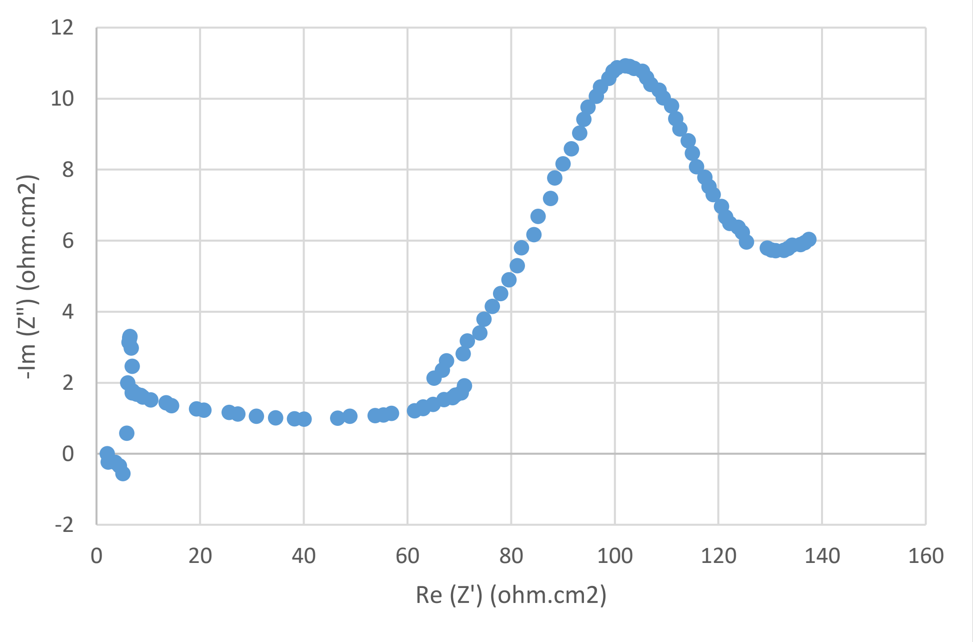


FIGURE 4.4 Nyquist Plot for 50% Water-cut

Followed with Figure 4.5 and Figure 4.6 that illustrated the bode plot and Nyquist plot for 70% water-cut. Impedance had decreased from 73.246 ohm.cm^2 to 2.0699 ohm.cm^2.

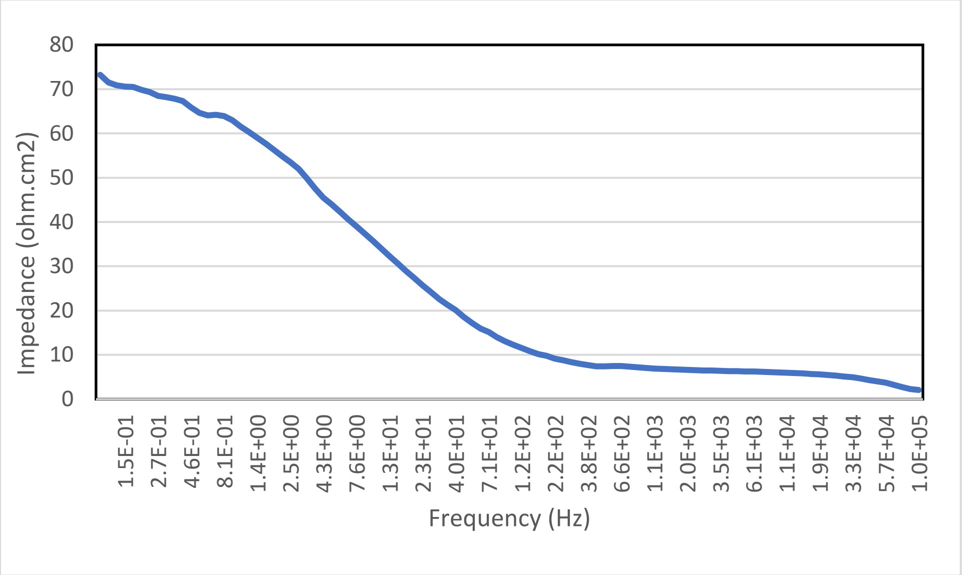


FIGURE 4.5 Bode Plot of 70% Water-cut

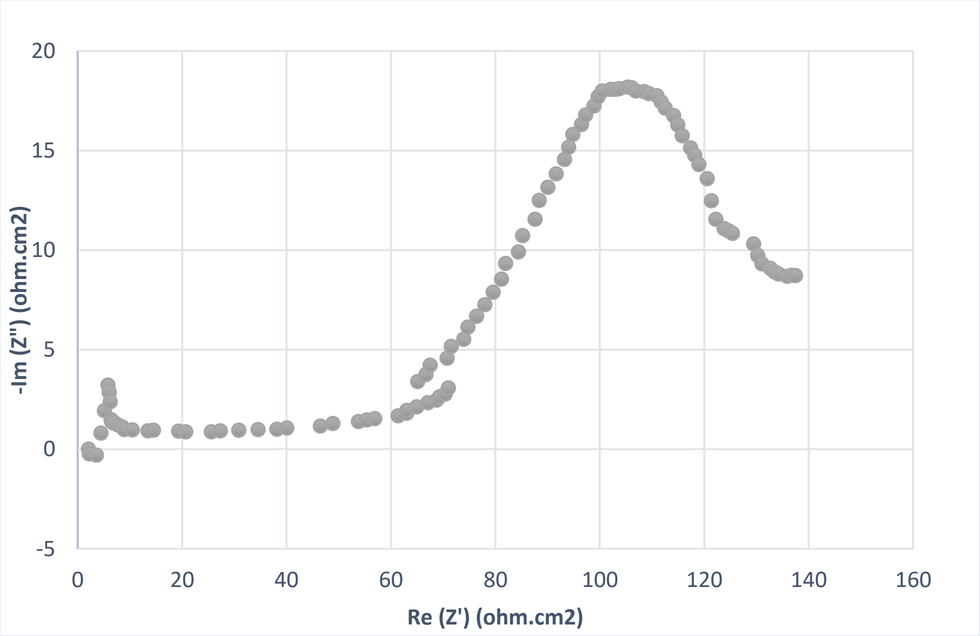


FIGURE 4.6 Nyquist Plot for 70% Water-cut

4.2 Experimental Result by Linear Polarization Resistance

Linear Polarization Resistance (LPR) result had been shown below. The test had been done after 24 hours exposure in the three different solution with CO_2 environment. Figure 4.7 showed the corrosion rate versus exposure time for 30% water-cut. Average corrosion rate is 1.8902 mm/yr. Rate of corrosion of mild steel in 50% water-cut and 70% water-cut are 3.8962 mm/yr and 2.703 mm/yr respectively (Figure 4.8 and Figure 4.9).

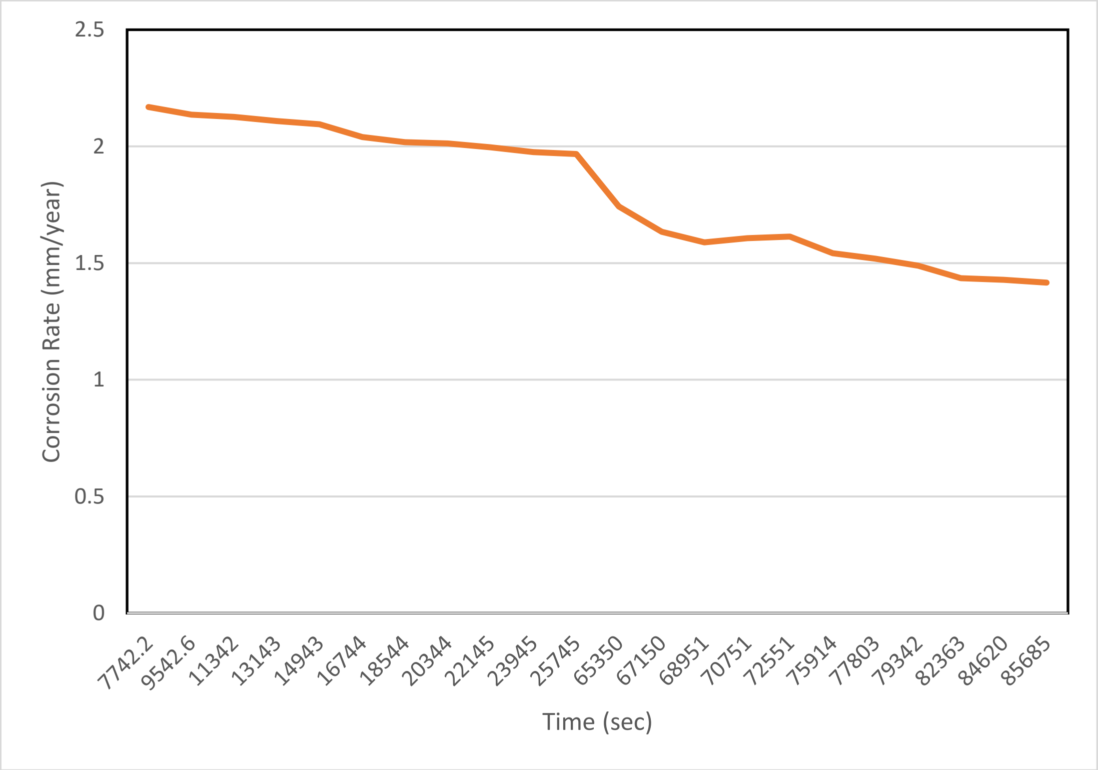


FIGURE 4.7 Corrosion Measurement for 30% Water-cut

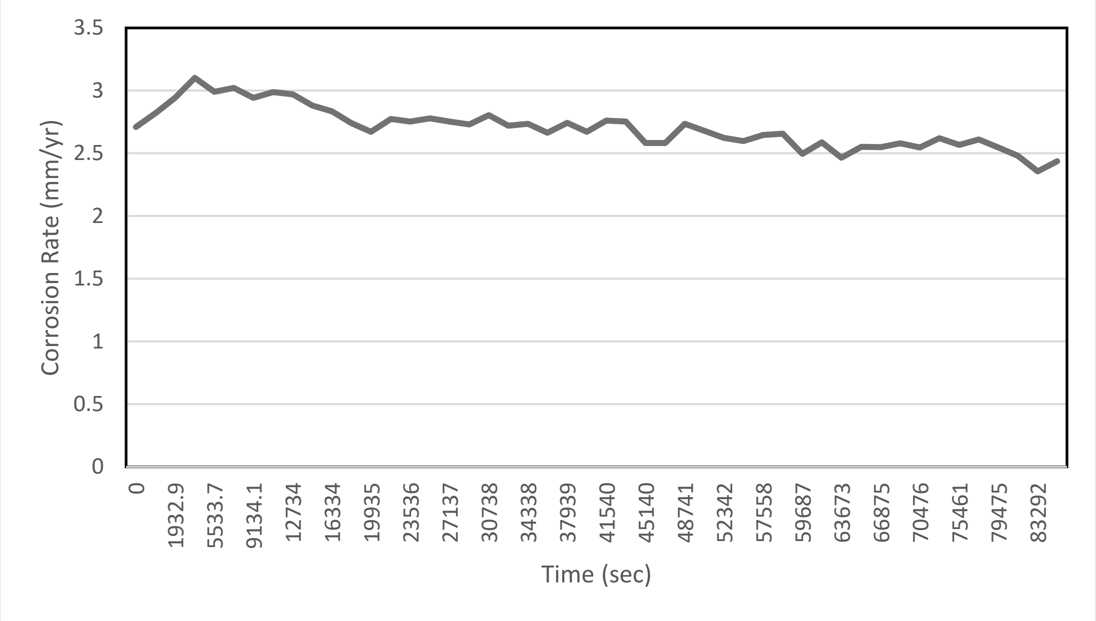


FIGURE 4.8 Corrosion Measurement for 50% Water-cut

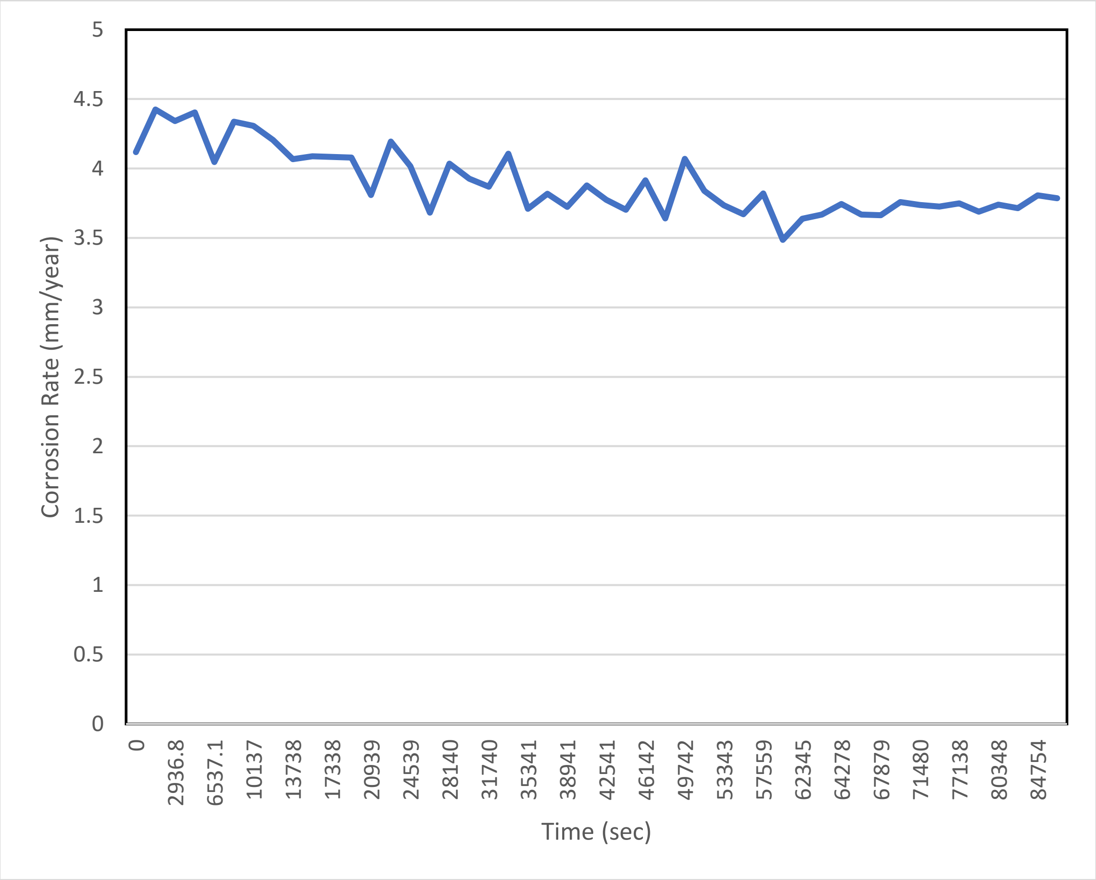


FIGURE 4.9 Corrosion Measurement for 70% Water-cut

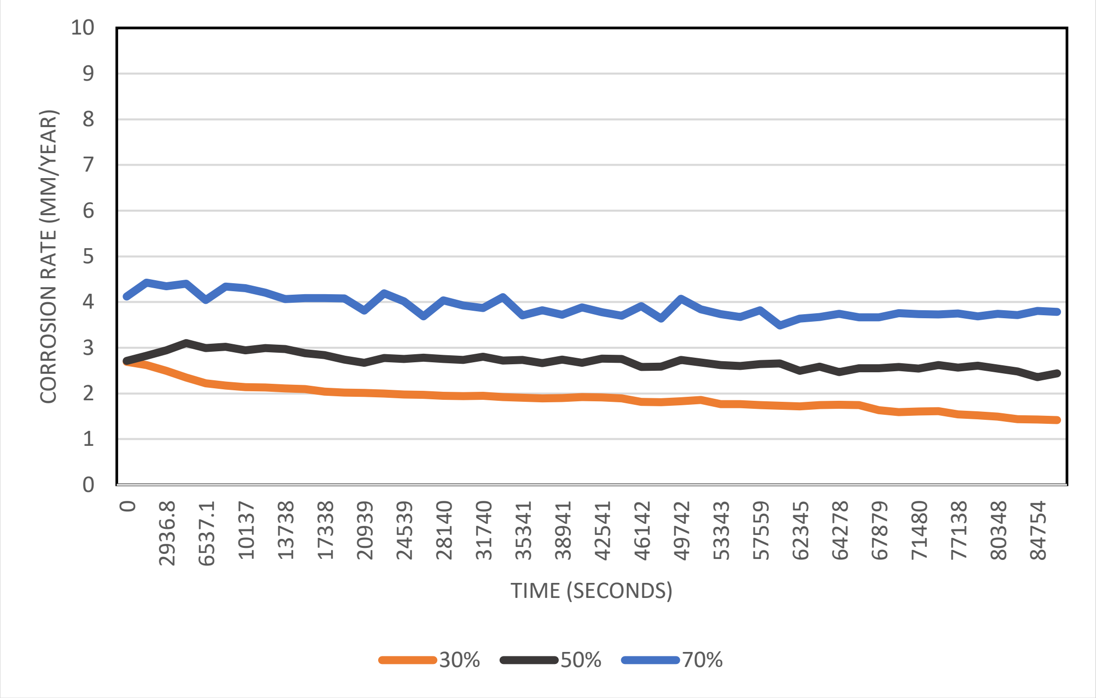


FIGURE 4.10 Corrosion Measurement for Three Different Percentage of Water-Cut

Based on Figure 4.10, overall corrosion measurement for three different water-cut decreases when time of exposure increases. 30% water-cut has lowest corrosion rate compared to the other two solutions. It shows that highest corrosion rate is from the 70% water-cut.

4.3 Experimental Result by Weight Loss Coupon Test

Table 4.2 shows corrosion rate from weight loss coupon test. The results for weight loss coupon were taken after being immersed within duration of 168 hours (1 week) inside three different water-cut percentage. Each coupon’s weight was taken repeatedly up to five times and the average were taken as the result. The corrosion rate was calculated using Equation 12. Figure 4.11 shows differences of the corrosion rate. Corrosion rate of 50% water-cut was the highest among the three with 1.0430 mm/yr. It was then followed by 70% water-cut with 0.9581 mm/yr and lastly is 30% water-cut with 0.7203 mm/yr.

TABLE 4.2 Corrosion Rate After One-Week Immersion

Sample	**Initial Weight (g)**	**Final Weight (g)**	**Corrosion Rate (mm/yr)**
30% Water-cut	4.3357	4.2885	0.7203
50% Water-cut	5.0365	4.9668	0.9581
70% Water-cut	4.3725	4.3039	1.0430

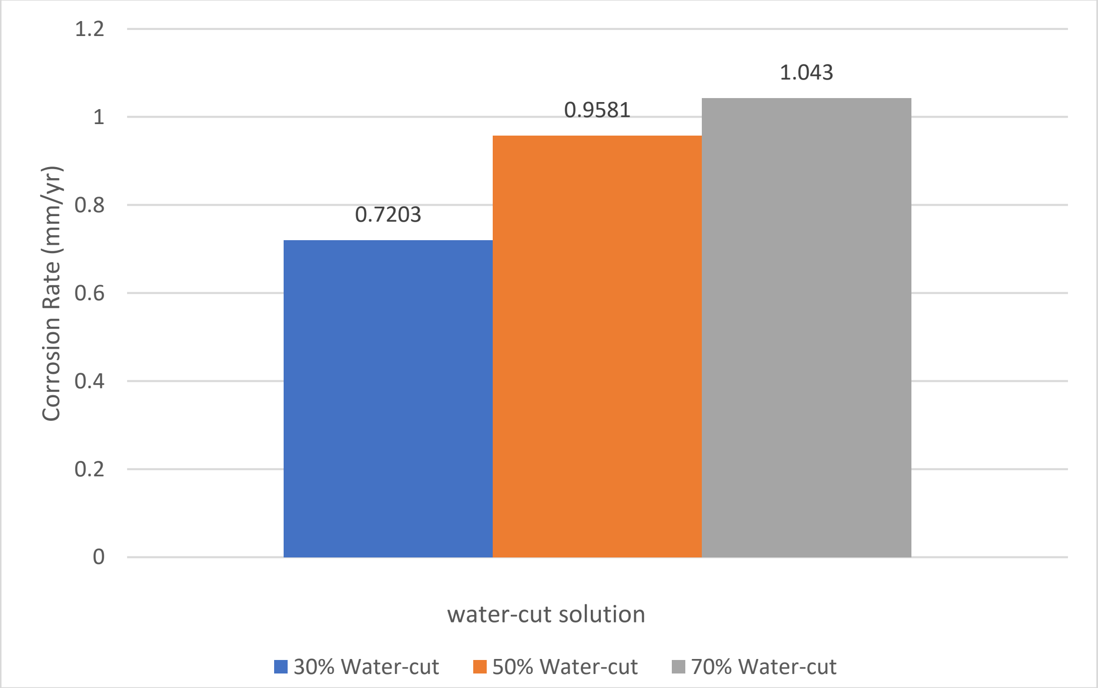


FIGURE 4.11 Comparison of corrosion rate after one-week immersion test

4.4 Comparison of three different corrosion tests

TABLE 4.3 Comparison of Corrosion Rate from Three Different Corrosion Test

Corrosion rate (mm/yr)			
Techniques / *Solution*	*Electrochemical Impedance Spectroscopy*	*Linear Polarization Resistance*	*Weight Loss Coupon Test*
30 % Water-cut	2.38	1.8902	0.7203
50% Water-cut	2.55	2.703	0.9581
70% Water-cut	2.672	3.8962	1.043

All of the experimental results had been compiled in Table 4.3. Based on Table 4.3, the corrosion rate increased with increasing water-cut. Highest corrosion rate of mild steel is in 70% water-cut while the lowest corrosion rate is in 30% water-cut. For 30% water-cut, EIS results shows that the corrosion rate is 2.38 mm/yr, LPR reading shows that it is 1.8902 mm/yr and 0.7203 mm/yr from weight loss coupon test. With 50% water-cut, EIS results for 2.55 mm/yr followed with LPR of 2.703 mm/yr and weight loss coupon test with 0.9581 mm/yr. Lastly, after being tested in 70% water-cut, EIS, LPR and weight loss test shows that the corrosion rate obtained is 2.672 mm/yr, 3.8960 mm/yr and 1.043 mm/yr respectively. There were a large different between corrosion rate obtained from the three types of test. However, the results show that the corrosion rate increases when water-cutting percentages increases.

4.5 Different Percentage of Water-cut

The different percentage of water-cut indicate different level of microemulsion in the solution. 30% water-cut have low conductivity as the brine solution mixed into 1400 of Isopar-M was only 600 ml. As for 50% water-cut, 1000 ml of brine solution were mixed with 1000 ml of Isopar-M. Lastly, 600 ml of Isopar- M were mixed with 1400 ml of brine solution to produce 70% water-cut. The conductivity of the solution varied to illustrate that different level of fluid conductivity will affect the result from different techniques of tests conducted.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Based on Table 4.3, it shows that the corrosion rates from EIS result have large gap from LPR and weight loss results. For 70% water-cut, the percentage different between the corrosion rate of EIS and LPR is 45%. In addition, the percentage different of the two techniques for 50% water-cut and 30% water-cut is 6% and 21% respectively. The percentage different is very high for 70% water-cut compared to the other water-cut percentage. Then, if comparing between EIS and LPR result with weight loss method, it can be said that percentage difference for 70% water-cut for EIS have smaller difference percentage with 61% whereas LPR with 73% difference. The reading of LPR might be inaccurate compared to EIS as LPR need electrolytes with high conductivity.

EIS is a good and sensitive method which can perform better in low conductivity condition compared to LPR. Not only that, EIS and LPR are good to measure the corrosion rate in high conductivity condition where the results can be obtained in short amount of time compared to weight loss coupons.

Weight loss coupons are one of the inexpensive methods to measure the corrosion rate. However, it can only measure the average corrosion rate on the time of exposure in the respective condition.

5.2 Recommendation

Detailed and effective analysis can be done to verify the result of the study. A good software that calculate the corrosion rate can be compared with the experimental works.

All three corrosion measurement techniques are important and have their own specialties. Exploration of the techniques are important to challenge the limit each of the techniques have. Not only that, the techniques are also important to ensure that the society can live in a comfortable and conducive environment where corrosion have been predicted and treated beforehand.

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