

**The Effect of Enhanced Oil Recovery (EOR) Surfactant on Pipeline
Corrosion in CO₂ Environment**

by

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13873

Dissertation submitted in partial of
the requirements for the
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(Mechanical)

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Mechanical Engineering Programme
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BACHELOR OF ENGINEERING (Hons)
(MECHANICAL)

Approve by,

(AP. Ir. Dr. Mokhtar Bin Che Ismail)

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May 2014

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.

(W. NUR LIYANA BINTI W. ZAKARIA)

ABSTRACT

The effect of Enhanced Oil Recovery (EOR) surfactant on the pipeline corrosion in CO₂ environment is not clear although the usage of EOR surfactant becoming more popular. The objective of the project is to evaluate the characteristics of EOR surfactant towards corrosion behavior in CO₂ environment. The characteristic of the commercial EOR surfactant used in this project was investigated using FTIR and the corrosion behavior was analyzed by electrochemical Linear Polarization Resistance (LPR) technique by varying the concentration of EOR surfactant from 50 to 5000 ppm. The surface morphology was examined by Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray (EDX). In general, the additional of EOR surfactant reduces the corrosion rate in range of 70% to 97% depending on the concentration of EOR surfactant. The highest reduction of 97% was achieved with the additional of 5000 ppm and reducing the corrosion rate from 1.86 mm/year to 0.06 mm/year. Based on the results, the additional of EOR surfactant react with the sample surface similar to corrosion inhibitor as shown by SEM images. In conclusion, the EOR surfactant behaves similar to corrosion inhibitor and able to reduce effectively the corrosion rate of the pipeline in CO₂ environment.

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“In the name of Allah, the most Beneficent, the most Merciful”

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ABBREVIATIONS AND NOMENCLATURE

V	Potential difference (Volts)
I	Current (Amperes)
R	Resistance (Ohms)
β_a	Anodic Tafel curve
β_c	Cathodic Tafel curve
l	litre
ppm	Parts per million
mg	Milligrams
MSDS	Materials Safety Data Sheets
API	American Petroleum Institute
ASTM	American Society for Testing and Materials
SSW	Soften Sea Water

CHAPTER 1

INTRODUCTION

1.1 Background Study

Enhanced Oil Recovery (EOR) is the tertiary method to maximize the production of oil from unrecovered oil volume. EOR can be classified into three major groups which are thermal, chemical and miscible gas. Among the EOR major, the possibility to trigger corrosion is the injection of chemical EOR. In this project, Chemical EOR (CEOR) was chosen to predict any corrosion occur at pipeline in CO₂ environment. Application of surfactant is one of chemical EOR and the function of surfactant is to lower the surface tension between the solid and liquid. The property of surfactant is thought to be similar to corrosion inhibitor properties due to the formation of micelles which consists of hydrophilic and hydrophobic hence; produce protective film inside the pipeline as corrosion resistance. Currently, there are a lot of oil fields that produce high content of carbon dioxide, CO₂. As known, the CO₂ is very corrosive agent especially dissolved in polar solvent such as water. The objective of the project is to access whether the additional of surfactant could trigger corrosion in CO₂ environment. In general, the information about EOR surfactant in CO₂ environment was limited due to the new methods in oil recovery and there a few oil field which already apply the EOR method. Several tests will be conducted to evaluate corrosion condition toward metals use and the test setup were followed the parameter accordingly.

1.2 Problem Statement

The usage of EOR surfactant reduce CO₂ corrosion rate based on the surfactant behavior which resemble corrosion inhibitor characteristic. However, the potential of EOR surfactant is not well characterized.

1.3 Objectives

There are three objectives to achieve the aims of the project, which are:

- 1) To identify the EOR surfactant characteristic based on analytical method.
- 2) To evaluate the effect of various concentration chemical EOR surfactant on the corrosion of pipeline in CO₂ environment
- 3) To access the corrosion interactive mechanism of EOR surfactant in CO₂ environment

1.4 Scope of Study

Corrosion test scope of study includes the following:

- 1) Conduct Linear Polarization Resistance (LPR) using soften seawater (3.0% NaCl), CO₂ environment and desired temperature and pressure.
- 2) Conduct Linear Polarization Resistance (LPR) using soften seawater (3.0% NaCl), CO₂ environment and desired temperature and pressure with present of chemical EOR surfactant at certain concentration.
- 3) Analyze the trend of corrosion rate from the LPR software program.
- 4) Conduct morphological test which consist of Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray (EDX).

CHAPTER 2

LITERATURE REVIEW

There are several literature reviews that meet the objectives of the project. Below shows, the related literature reviews for understanding the purpose of the project.

2.1 Exploration Production (Brown Field)

Brown field refer to existing of oil and gas area that need expansion and revamping operation such as upgraded the processes and utilities and vigorously occur at upstream of oil and gas activity (LinkedIn, 2013 and Consulting, 2012). According to Consulting (2012), the field can be categorized as brown field over a period 20 years to 30 years. For example, the production of UK's Continental Shelf (UKCS) for past four decades, was 40 billion barrels of oil equivalent (boe) for the first production and the uncovered oil between 14 to 24 billion more (UK, 2011). Actually the brown field has their value and need the right equipment to extract remaining oil from the well. Based on Consulting (2012), "...admitted that the capital expenditure (CAPEX) to decommission a plant and decontaminated the site may be very close to the construction cost". So that way the Enhanced Oil Recovery, EOR were introduce because of the existing of equipment and required some improvement to adapt the new systems.

There are several oilfield that had been conducted the EOR injection such as polymer flooding, PF at Daqing Oilfield, located at Songliao Plain in northeastern China and the largest production in China (Wang et. al., 2013). Other than that, Pubei Oilfield one of the oil field at Daqing area had done the pilot test using same method and both experimentally results shows the incremental of oils using PF around 10%

(Wang et. al., 2013). Surfactant flooding also used in Norne E- segment located at Germany and proves that after water flooding around 75% oils came out from well and even more if inserting the surfactant into the reservoir (Emegwalu, 2009). According to the Abdelhady (2007), there are a lot of abandoned oil wells in Egypt and the application of Surfactant Polymer Flooding, SPF could enhance oils from mature oil fields because the results show that around 15% to 25% recovery from application of PF of original oil in place, OOIP and up to 28% more recovery when injecting the SPF or reservoir oils.

2.2 Enhanced Oil Recovery (EOR) – Surfactant

The production of oil can be categorized in three phases which are primary is normal injection, secondary is water flood and tertiary phase is EOR. The purpose of EOR is to maximize the production of oil in the well. The amount of unrecoverable oil especially in Malaysia is 2 billion barrels which is an attractive target. Hence, the incremental oil potentially recoverable could be realized when the EOR process is applicable in the oil and gas field. The usage of EOR can be apply either tertiary or enhanced secondary projects. There are three major of EOR methods which are thermal, miscible gas and chemical. For miscible gas, carbon dioxide, CO₂ and methane, CH₄ are the most popular injection into the well (Theodorou, 2013).

The statistic below, Figure 1 show why most of most of country need an investment on EOR method (Sgaurav, 2010). Other than that, the amounts of oil recovery are huge when applying the EOR method as shown at Figure 2 below (Dover, n.d.). Chemical EOR also involve in this third phase like the application of alkaline, surfactant and polymer. The chemical EOR can be combined such as alkaline & polymer, alkaline & surfactant and alkaline, surfactant & polymer.

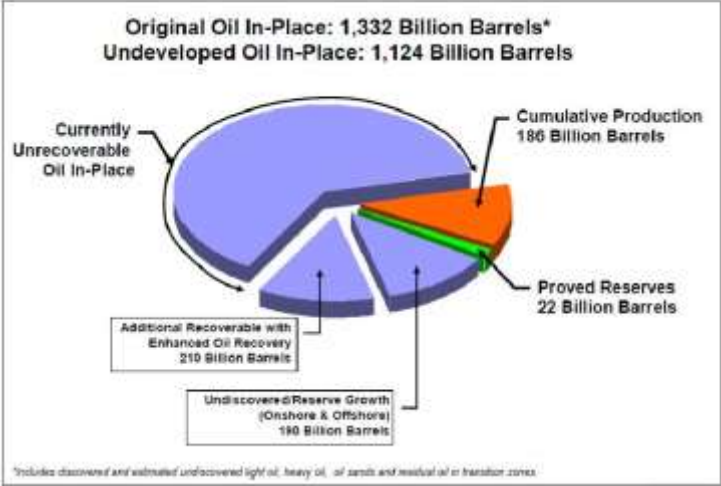


Figure 1: Oil In-Place for world-wide shortage of petroleum (Sgaurav, 2010)

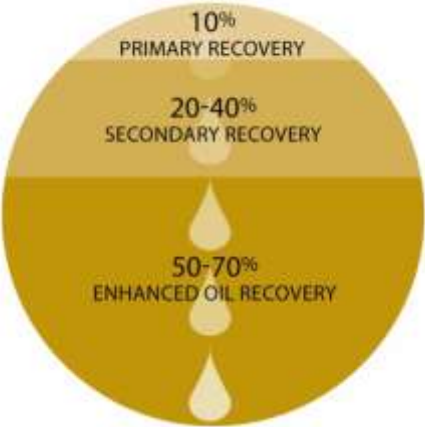


Figure 2: Percentage of oil recovery (Dover, 2013)

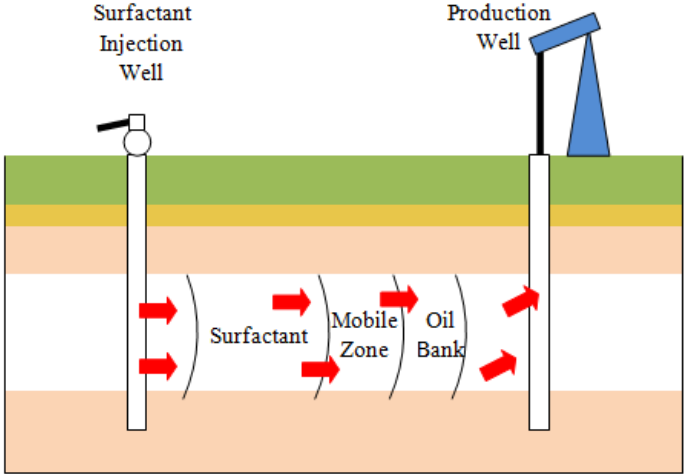


Figure 3: Illustration of surfactant injection into reservoir (Emagwalu, 2009)

One of the EOR approaches is to increase the production of oil which the application of surface-active compound or so called surfactant as shown in Figure 3 above. There are a lot of advantages using surfactant such as increase the inhibition efficiency, cheap, low toxicity and the procedure to produce surfactant are easy (Malik et. al., 2011). Surfactant acts as adsorption process to the metal surface. According to Malik, the definition of adsorption is “the ability to aggregate to form micelles”.

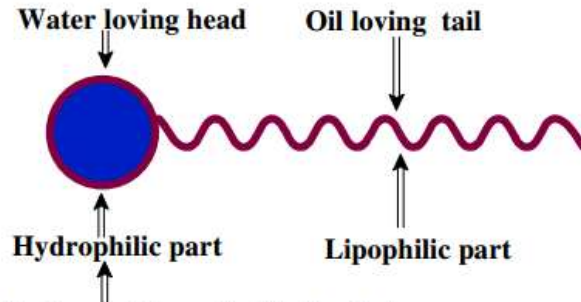


Figure 4: Schematic diagram of surface-active molecule (Malik et. al., 2011)

Figure 4 above shows, one of the molecules of surface-active (surfactant) which consist of hydrophilic head and hydrophobic tail. Malik stated that, “The surfactant have the tendency to accumulate at the interface of immiscible fluids with a marked influence resulting in decrease in free energy which is reflected in a corresponding lowering of interfacial tension that facilitates emulsification of the immiscible fluids and hence such compounds are also known as emulsifiers.”

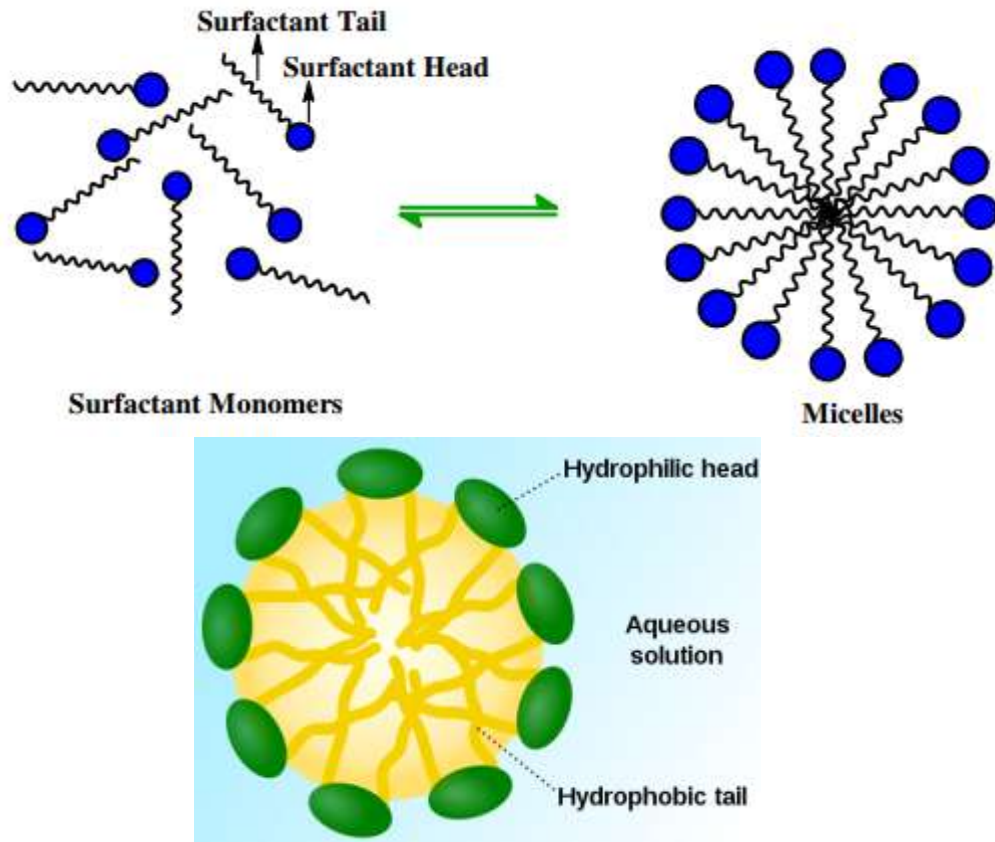


Figure 5: Schematic illustration of the reversible monomer-micelle thermodynamic equilibrium (Malik et. al., 2011) and (Wikipedia, 2013)

Figure 5 above show, a bunch of amphilic surfactant monomers together to assemble micelle where the hydrophobic tails part huddle in the middle of micelle, while the hydrophilic head were accordingly arranged outwards. According to Wikipedia, the interaction of oil is greater than water which makes the lipophilic part remain inside the micelle and on the other hand the “polar” refer to the heads acts as coating molecules for the micelle and automatically produce barrier (hydrophilic) outer layer between the micelle. The other property of micelles is constantly disintegrating and can be reform.

As mention before, the surface tension reduces when introducing surfactant. It can be shown by Figure 6 below:

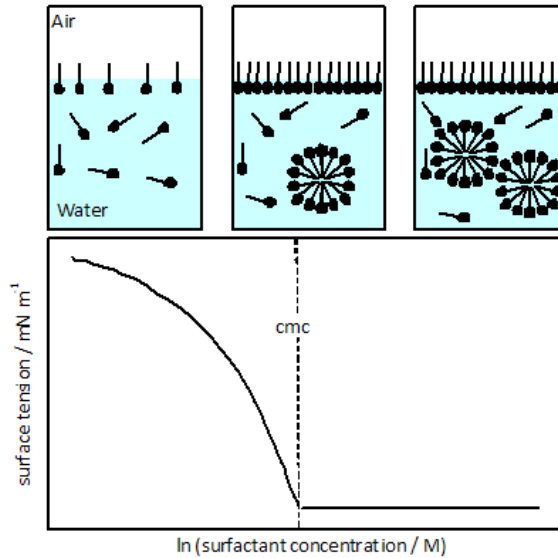


Figure 6: The effect of surfactant concentration on surface tension (Ibrahim, 2011)

When the concentration of surfactant increase, the volume of aggregation totally increase which lower the polarity between air and water hence, decrease the surface tension until reaching the critical micelle concentration, CMC (Ibrahim, 2011).

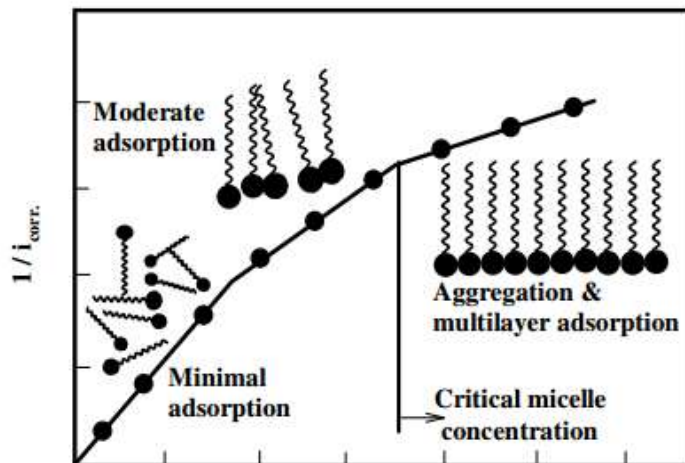


Figure 7: Effect of surfactant concentration on corrosion inhibition (Malik, 2011)

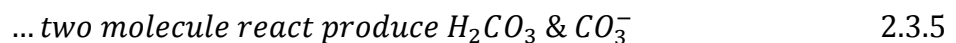
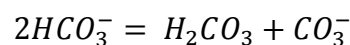
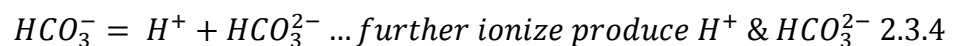
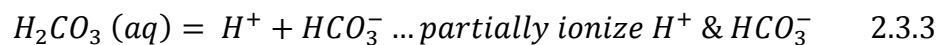
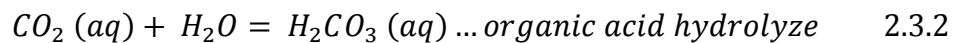
Based on Malik, “Consequently, the critical micelle concentration (CMC) in Figure 7 is a key indicator in determining the effectiveness of surfactants as corrosion inhibitor. Below the CMC, individual surfactant molecules or monomer tend to absorb on exposed interfaces, so interfacial aggregation reduces surface tension and is related

to corrosion inhibition. Above the CMC, the surface becomes covered with more than one monolayer and forms a protection layer on the metal surface. Thus, any additional surfactant added to the solution above the CMC will lead to the formation of micelles or multiple adsorbed layers on surfaces. Consequently, the surface tension and corrosion current density are not altered significantly above the CMC. Therefore, an excellent surfactant inhibitor is one that aggregates or adsorbs at low concentration. In other words, surfactants with low CMC values are desirable because they adsorb at low concentrations.”

2.3 Carbon Dioxide (CO₂) Corrosion

Combinations of carbon dioxide, CO₂ with polar solvent such as water, produce weak carbonic acid which leads to carbon dioxide corrosion or sweet corrosion (Li, 2009). According to Fontana, one of the corrosive agents is carbon dioxide with organic acid that contribute to the corrosion attack. Based on N’Guessan, corrosion cannot occur if using the dry CO₂ because it is noncorrosive towards metal and alloy, but in present of water, the carbonic acid was formed and very corrosive.

According to Li, the formation of weak carbonic acid leads to CO₂ corrosion or as “acid corrosion” which release the hydronium ions (H⁺) through several chemical reactions below and CO₂ in aqueous state experimentally showing that, the corrosion rate is higher compared with the strong acid solution even though they have the same pH value.



Continuous reactions and repeated from 2.3.2 until soluble

Usually, the corrosion type under CO₂ environment are uniform (general) and/or localized (pitting) form and carbon monoxide may develop other type of corrosion which is stress corrosion cracking (SCC) (Kowaka and Nagata, 1976; Brown, Harrison and Wilkins, 1977; Berry and Payer, 1979).

2.4 Linear Polarization Resistance (LPR)

Linear Polarization Resistance (LPR) method is to determine the corrosion rate of the specimen by using electrolytic test cell (Natarajan, n.d. & Mancio et. al., 2008). LPR also using direct current (DC) when conducts the test. According to Natarajan, there are two types of resistance which are Tafel extrapolation and polarization resistance which can determine the corrosion rate (CR). LPR work best in aqueous solution and had been proven to have rapid response technique (Caproco, n.d.).

Tafel polarization can be determine in Figure 8 below, which always show linear behavior that consists of Potential (E) at y-axis and Log Current (Log *i*) at the x-axis (Gasem, n.d.). The intersection point can be determined by extrapolate the anodic and cathodic Tafel curve and the values of E_{corr} and i_{corr} are obtained (Natarajan, n.d.). While, the polarization resistant refer to Figure 9 which obtained the value of *Resistance* (R_p) from the Ohm's Law below:

Equation 1: Ohm's Law

$$\text{Ohm's Law}; V = IR$$

Where; $V = \text{Potential difference (volts)}$

$I = \text{Current (amperes)}$

$R = \text{Resistance (ohms)}$

After getting the R_p , the value will be inserted into the i_{corr} equation which also involves the Stern-Geary coefficient:

Equation 2: i_{corr}

$$i_{corr} = B/R_p$$

Equation 3: Stern-Geary

$$\text{Where; } B = \frac{\beta_a \beta_c}{2.3(\beta_a + \beta_c)}$$

the value of β_a and β_c can be calculated using Tafel slope

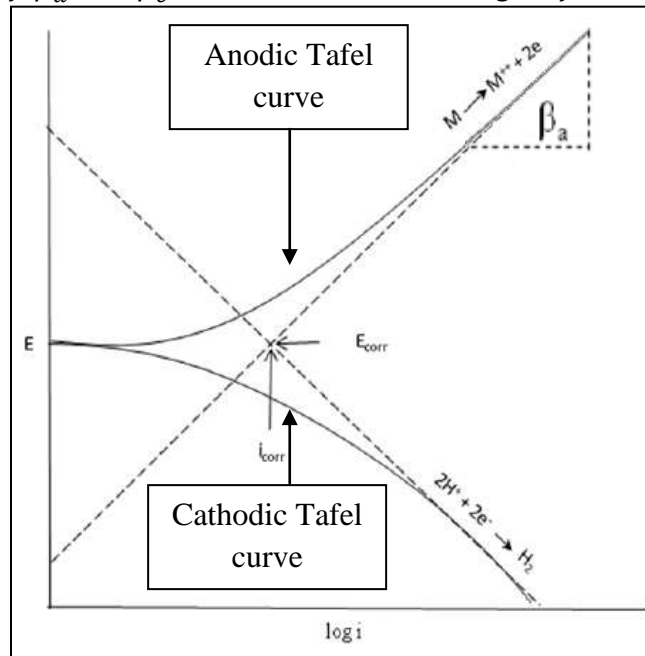


Figure 8: Tafel extrapolation (Fontana, 1986)

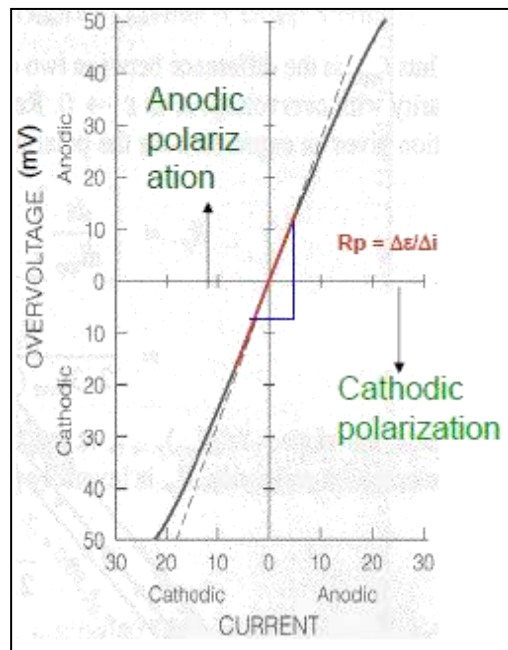


Figure 9: Polarization resistance (Fontana, 1986)

2.5 Corrosion inhibitor

Corrosion inhibitor is one of the chemical based that generally apply to the pipeline to reduce the corrosion rate of material (Rimpo, 2011). The injection of corrosion inhibitor is, to provide the passive film profile inside the pipeline but, since there was an aggressive anions such as Chloride, therefore the passive film unable to form (Branzoi, 2002).

One of the oil fields in China, Shengli oilfield had been carried out the EOR method by using chloride corrosion inhibitor which obtained good corrosion inhibiting effect and from the static test, the concentration dosage of chloride corrosion inhibitor work best above 20 mg/l (You et. al., 2011). From the graph the corrosion rate start to decrease when the concentration of chloride CI increase. The graph and results can be shown below:

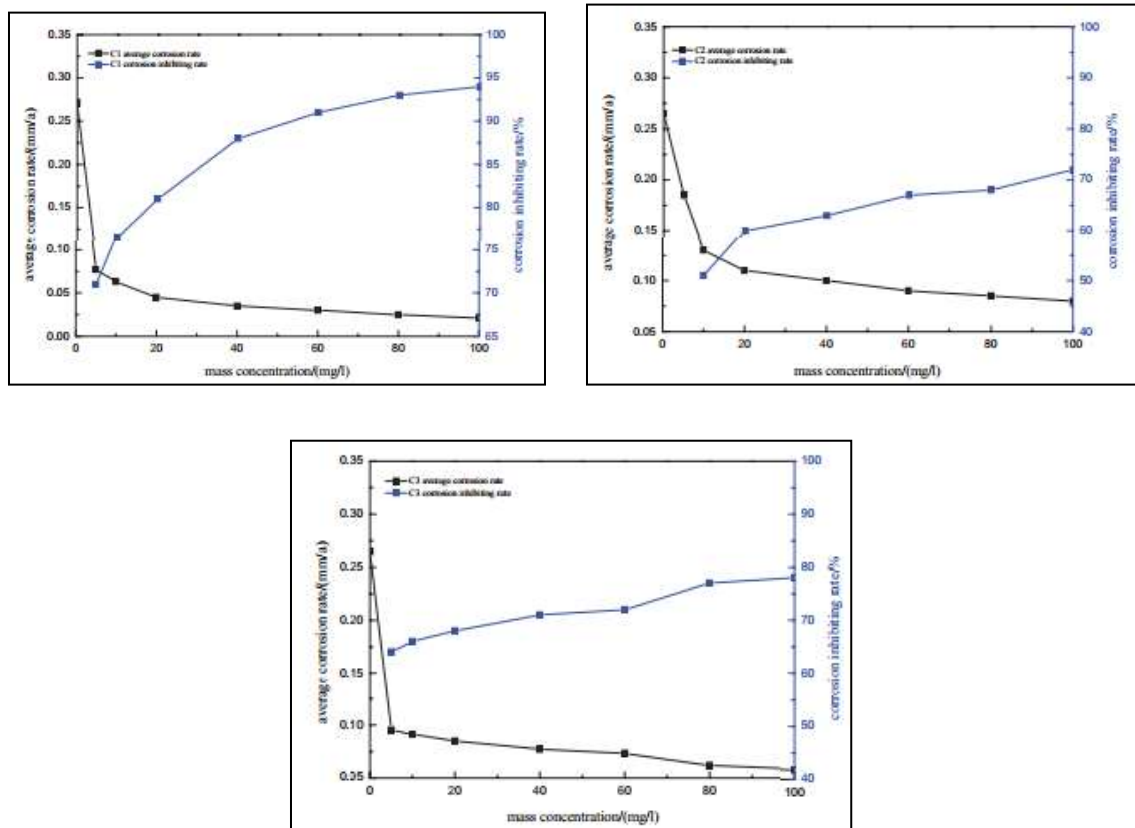


Figure 10: The corrosion inhibiting effect (You et. al., 2011)

Table 1: Electrochemical results after adding corrosion inhibitor (You et. al., 2011)

mass concentration/(mg/l)	natural corrosion potential/mV	corrosion current density/($\mu\text{A}/\text{cm}^2$)	corrosion inhibiting rate/%
0	-704.2	121.6	—
5	-719.2	19.7	83.8
10	-685.0	13.6	88.9
20	-651.9	9.8	91.9
40	-637.8	7.1	94.2
60	-620.3	5.4	95.6

CHAPTER 3

METHODOLOGY

3.1 Research Methodology

Research methodology below shows the overall process required to complete this project.

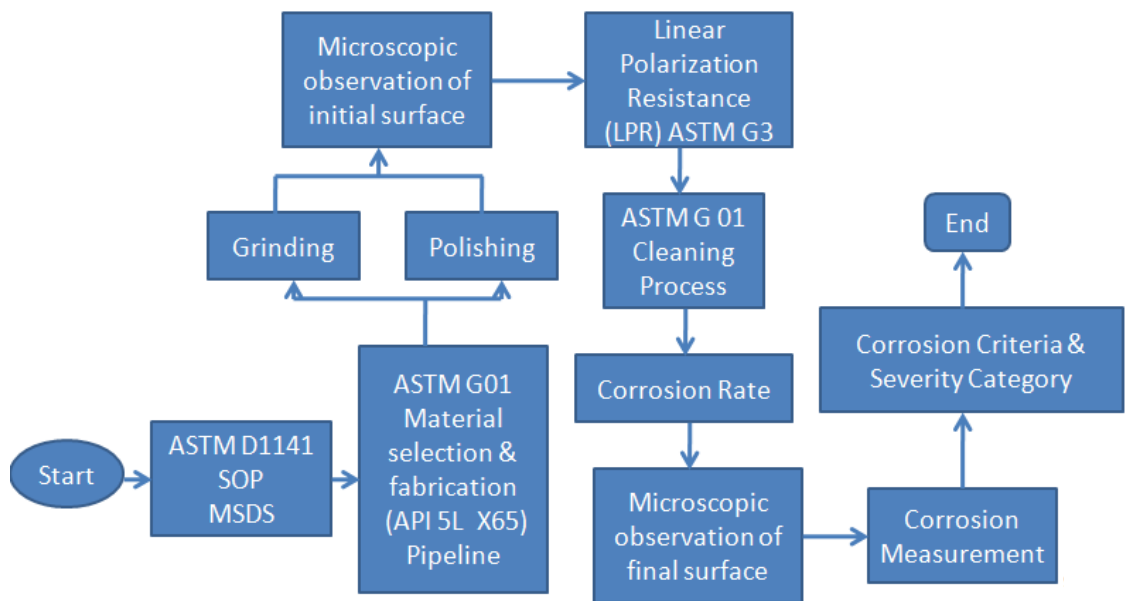


Figure 11: Research Methodology

3.2 Test Parameters

In order to conduct LPR test, parameters are required which mimic with the original reservoir condition. Table 2 below shows, the test matrix required to accomplish the LPR test.



Table 2: Test Matrix

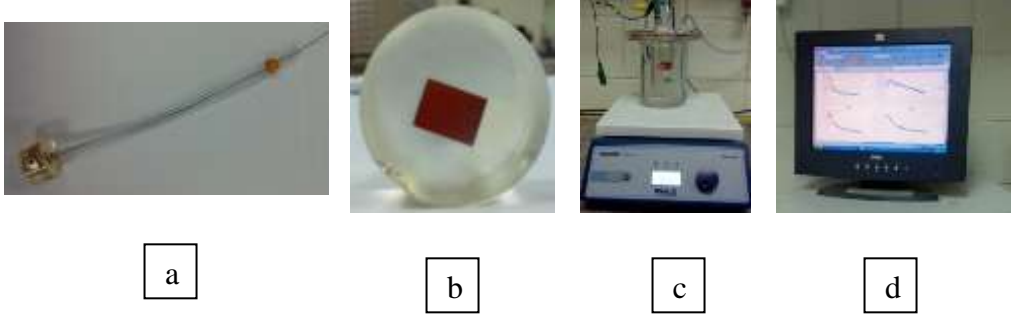
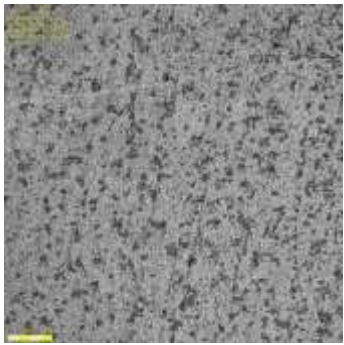
Test Matrix	
Standard(s)	<ul style="list-style-type: none"> • ASTM G1 • ASTM G3 • ASTM G31 • ASTM 102
Temperature (°C)	25
Pressure (bar)	1
Material	Carbon Steel API 5L X65
Exposure Time (Hours)	24
pH	4
Concentration surfactant (ppm)	<ul style="list-style-type: none"> • 0 • 50 • 100 • 300 • 5000
Tools	<ul style="list-style-type: none"> • Linear Polarization Resistance (LPR) • Morphological Experiment <ul style="list-style-type: none"> - Scanning Electron Microscope (SEM) - Energy Dispersive X-ray (EDX)

3.3 Test Setup

LPR is the main experiment to achieve the objectives of the project and below (Table 3) are the steps to accomplish the procedure as well as to obtain the corrosion rate.

Table 3: Test setup and activity

No.	Activity	References	Description
1	Preparation of brine	ASTM D1141, Standard of procedure (SOP) and MSDS Surfactant	Synthetic Water <ul style="list-style-type: none"> • Softten Seawater, SSW  <p>Figure 12: 3.0% NaCl</p>
2	Selection of materials	API	Materials used as specimens for corrosion test: <ul style="list-style-type: none"> • API 5L – X 65 (Pipeline)
3	Grinding and polishing of specimen	ASTM G01	To make sure the surface of specimen free from impurities and any scratch  <p>Figure 13: Grinding process</p>
4	Linear Polarization	ASTM G3	Apparatus of LPR as shown below:

	Resistance (LPR)																								
	 <p style="text-align: center;"> a b c d </p> <p style="text-align: center;">Figure 14: a) Ribbon electrode; b) Specimen mounted into low viscosity epoxy; c) LPR test and d) LPR software program</p> <p>The magnetic stirrer was immersed inside the test cell during conducting LPR test because to blend completely the ER surfactant with the electrolyte.</p>																								
5	Morphological equipments <ul style="list-style-type: none"> • Scanning Electron Microscopy (SEM) • Energy Dispersive X-ray Spectroscopy (EDX) 	SOP	 <p style="text-align: center;">Figure 15: Microstructure view</p>																						
6	Corrosion criteria and severity category	API 581	<p style="text-align: center;">Table 4: Current rate thinning calculation</p> <table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th rowspan="2">Category</th> <th rowspan="2">Criteria</th> <th rowspan="2">Description</th> <th colspan="2">SCL Value</th> </tr> <tr> <th>Design Ck: 1 year for 30 years</th> <th>Design Ck: 8 mm for 30 years</th> </tr> </thead> <tbody> <tr> <td style="text-align: center;">Critical</td> <td style="text-align: center;">$>4\%_t$</td> <td>Critical: Corrosion might lead to immediate failure of the component</td> <td style="text-align: center;">0.02</td> <td style="text-align: center;">0.4</td> </tr> <tr> <td style="text-align: center;">Warning</td> <td style="text-align: center;">ΔX_t to $4\%_t$</td> <td>Warning: Component life might reduce to one fourth of the design life</td> <td style="text-align: center;">0.05-0.12</td> <td style="text-align: center;">1.0-1.8</td> </tr> <tr> <td style="text-align: center;">Normal</td> <td style="text-align: center;">$<4\%_t$</td> <td>Acceptable: Component will be able to achieve design life</td> <td style="text-align: center;">0.30</td> <td style="text-align: center;">8.1</td> </tr> </tbody> </table>	Category	Criteria	Description	SCL Value		Design Ck: 1 year for 30 years	Design Ck: 8 mm for 30 years	Critical	$>4\%_t$	Critical: Corrosion might lead to immediate failure of the component	0.02	0.4	Warning	ΔX_t to $4\%_t$	Warning: Component life might reduce to one fourth of the design life	0.05-0.12	1.0-1.8	Normal	$<4\%_t$	Acceptable: Component will be able to achieve design life	0.30	8.1
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Normal	$<4\%_t$	Acceptable: Component will be able to achieve design life	0.30	8.1																					

3.4 Procedure of Linear Polarization Resistance (LPR) method

Sample preparation

- 1) The specimens were cut into rectangular shape with dimension 1cm by 1cm and undergo grinding process using emery papers that have different size (Refer ASTM G31).
- 2) The orientation of specimens must be synchronize and frequently when conducting the grind and polishing process. The scratches from the previous need to remove before proceed to the finer grit.
- 3) The specimens were rinsed with distilled water and acetone and then placed in proper medium to avoid moisture from surrounding.

Solution preparation/electrolytes

- 1) The brine was prepared by adding Sodium Chloride (NaCl) only.
- 2) 1 liter of 3.0% of NaCl was used in LPR test and the calculation can be shown below:

Equation 4: Preparation of SSW

3.0% NaCl brine:

$$3.0\% \rightarrow 30,000\text{mg/l}$$

$$= \frac{30,000 \text{ mg}}{1\text{l}} \times 1\text{l}$$

$$= 30,000 \text{ mg}$$

= 30 g ... of NaCl and dilute with 970g of distilled water

Electrode preparation (Ribbon electrode)

- 1) The pre-preparation of specimen by mounted into the low viscosity epoxy resin (Figure 14a) and the exposed area was measured carefully.
- 2) Before mounting, the electric contact was made to the back surface of specimen by attaching a thin copper wire using solder.

LPR Test

- 1) The apparatus were setup according to ASTM G3 and ASTM G31.
- 2) The brine solution was measured 900ml and pour into the 1 liter beaker (Figure 14c). This is to avoid leakage when injecting the EOR surfactant.
- 3) Purging process was conducted by inserting CO₂ gas into the brine until reaching the desired pH value.
- 4) The clamp was used to tight the beaker so that no gas coming out when running the test.
- 5) There were three probe using throughout the test which were ribbon electrode, auxiliary probe and references probe.
- 6) The ribbon electrode was immersed into the brine solution and the temperature and pressure was setup accordingly. The position of ribbon electrode lowered and near to reference probe.
- 7) The LPR test was using direct current (DC) and connected with the software program (Figure 14d).
- 8) The test was monitored every hour and continuously running according to test matrix.

Injecting EOR surfactant

Equation 5: Preparation of EOR surfactant

$$\frac{\text{Parts}}{\text{million}} = \frac{\text{mg}}{\text{l}}$$
$$\frac{50 \text{ Parts}}{1,000\ 000} = \frac{x}{900 \text{ g}}$$
$$x = \mathbf{0.045 \text{ g}}$$

- 1) EOR surfactant was injected according to the test matrix after four hours running the test.
- 2) The magnetic stirrer was added into the beaker so that the surfactant was mix properly with synthetic water.

Cleaning process

- 1) The specimen was examined visually and note if there any change in appearance such as discoloration, corrosion product, pitting and etc.

- 2) The specimen was clean with distilled water and acetone and dried using hot air.
- 3) The specimen was observed, and if there is any corrosion occurs, the morphology apparatus was used to determine the maximum depth and distribution.
- 4) The photo of specimen was taken as the permanent record.
- 5) Corrosion damage was assessing according to ASTM G1.
- 6) The samples were kept inside the desiccators to avoid moisture from surrounding.

3.5 Scanning Electron Microscopy (SEM)

Sample preparation

- 1) The sample was placed on to the aluminum holder tub using double sticky carbon tape.
- 2) The sample insulated was located either carbon/gold and electrically grounded.
- 3) The sample was dry in the drying oven for 60°C for 3 hours.

Loading the specimen into the SEM holder

- 1) The two valves of nitrogen tank were open and the “Vent” button was pressed at the display panel of the Microscope table.
- 2) The lever was located at the bottom of the door and the lever was gently pulled up and opens the chamber door once click sound appear.
- 3) The mounted sample was moved by pressing the z-axis.
- 4) The sample holder stubs was placed into the mounting holes. The sample’s tall located away from the left side of chamber due to the existence of secondary electron detector. The long screwdriver was used to tight the mounting hole.
- 5) The door was closed and the rotary pump start to work after the EVAC button was pressed. The green light appeared after 2 minutes and achieve high vacuum $<5 \times 10^{-5}$ Pa.

Turning on the SEM

- 1) The filament was ON after vacuum reach proper level and the key switch and monitor were also turn ON.
- 2) 15kV of acceleration voltage was set and the spot size was set to large and 1.
- 3) The filament and high voltage were turn on. The lowest magnification (10X) was used at the beginning and TV scan mode was chosen.
- 4) The trackball was used to find the sample. The coarse focus switch was turn on and the working distance was set to be 14mm.
- 5) The sample was brought up slowly by pressing the z-axis UP key.
- 6) The coarse focus was turn off and the magnification was increased. The spot size was change to medium if the magnification above 1000X.
- 7) The VARIABLE button was pressed to open up the small variable window on the screen. The size of screen was adjusted and overlay the region of interest. The image was focused within the small screen using outer focus ring and later inner focus ring for fine focus.
- 8) The scanning speed was set to S1 which allow the electronic imagine acquisition.
- 9) The spirit icon was double click and go to file menu and select preferences. The directory folder name was selected and input the sample name and ID. The image set up icon was click, select Mapping option and normal resolution to be 1024 while the frame was 1.
- 10) The image acquire icon was click to record the image and the software control the SEM then monitor will freeze. Parameter was not change during the image recording.
- 11) The image was save as *.tif* format

Turning off the SEM

- 1) The magnification (10x) was reduce and the spot size was set to large and 1. TV scan mode was chosen and set the acceleration voltage to 15kV.
- 2) The sample was brought down by pressing the z-axis until 0.
- 3) The key switch was turn to VAC position.

- 4) The sample was taken out if the nitrogen gas was good enough. The chamber door was closed and evacuate.

3.6 Procedure of Energy Dispersive X-ray (EDX)

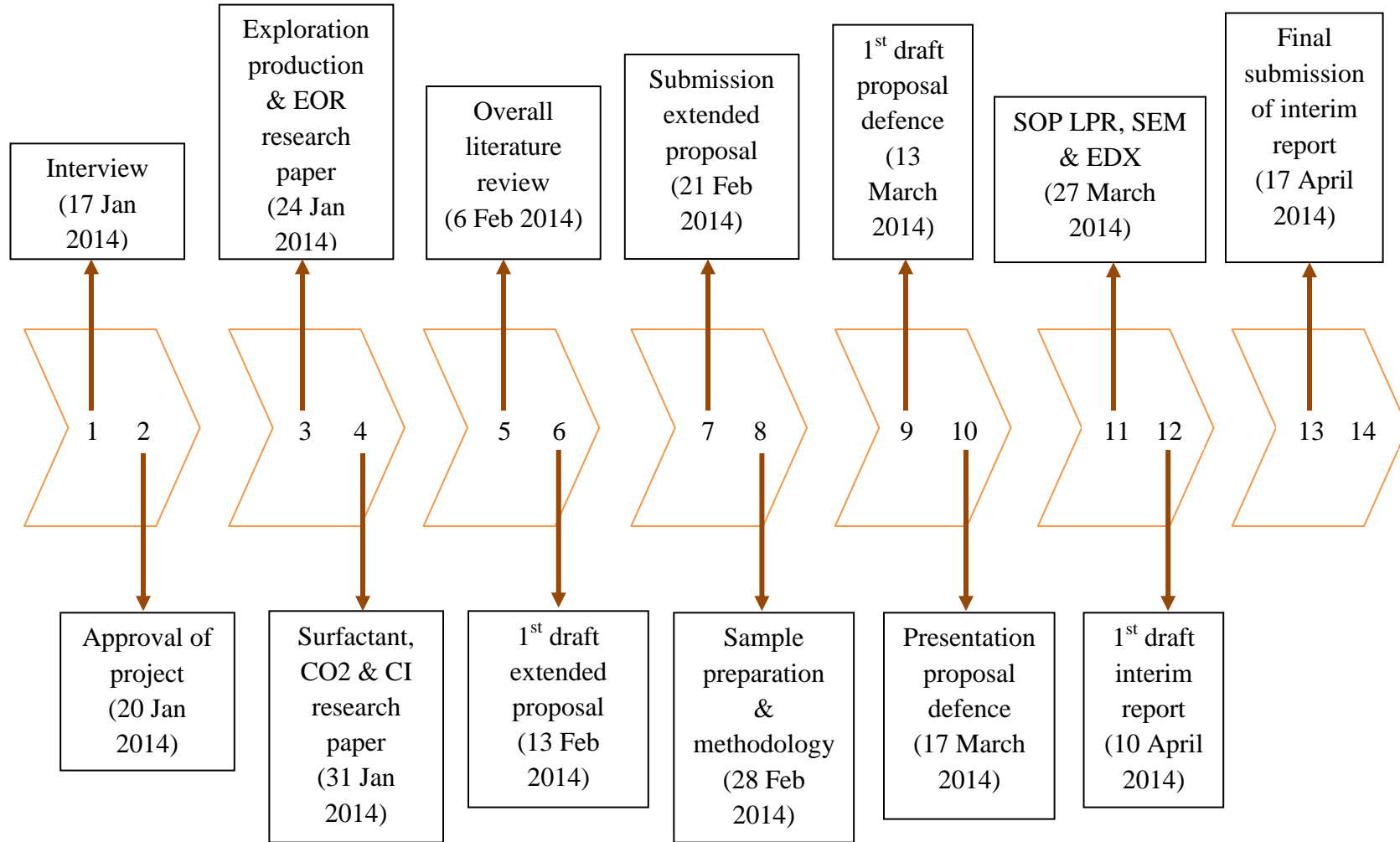
The EDX can be obtained using same tools of SEM and have slightly different before turning off the SEM.

Energy Dispersive X-ray Spectroscopic Analysis

- 1) The acceleration voltage was set up to 20 kV and working distance was 14 mm.
- 2) The detector was move down to 45 mm by rotary knob below the detector.
- 3) The sample was focused.
- 4) The X-ray setup icon was click and Enable box was check. The live time was set for 200 secs.
- 5) The X-ray icon was click and the cursor was placed at the spectrum window and read the counts per second and dead time (DT) on the bottom of spectrum window.
- 6) The DT was adjusted to 25 to 30% by changing the spot size. The larger spot size was obtained by increasing the DT and count per second.
- 7) Erase current spectrum was click and start over collecting the X-ray spectrum.
- 8) The periodic table was click to identify the peaks and the existing element was selected to match the peaks.
- 9) The spectrum was saved as *.pgt* file.

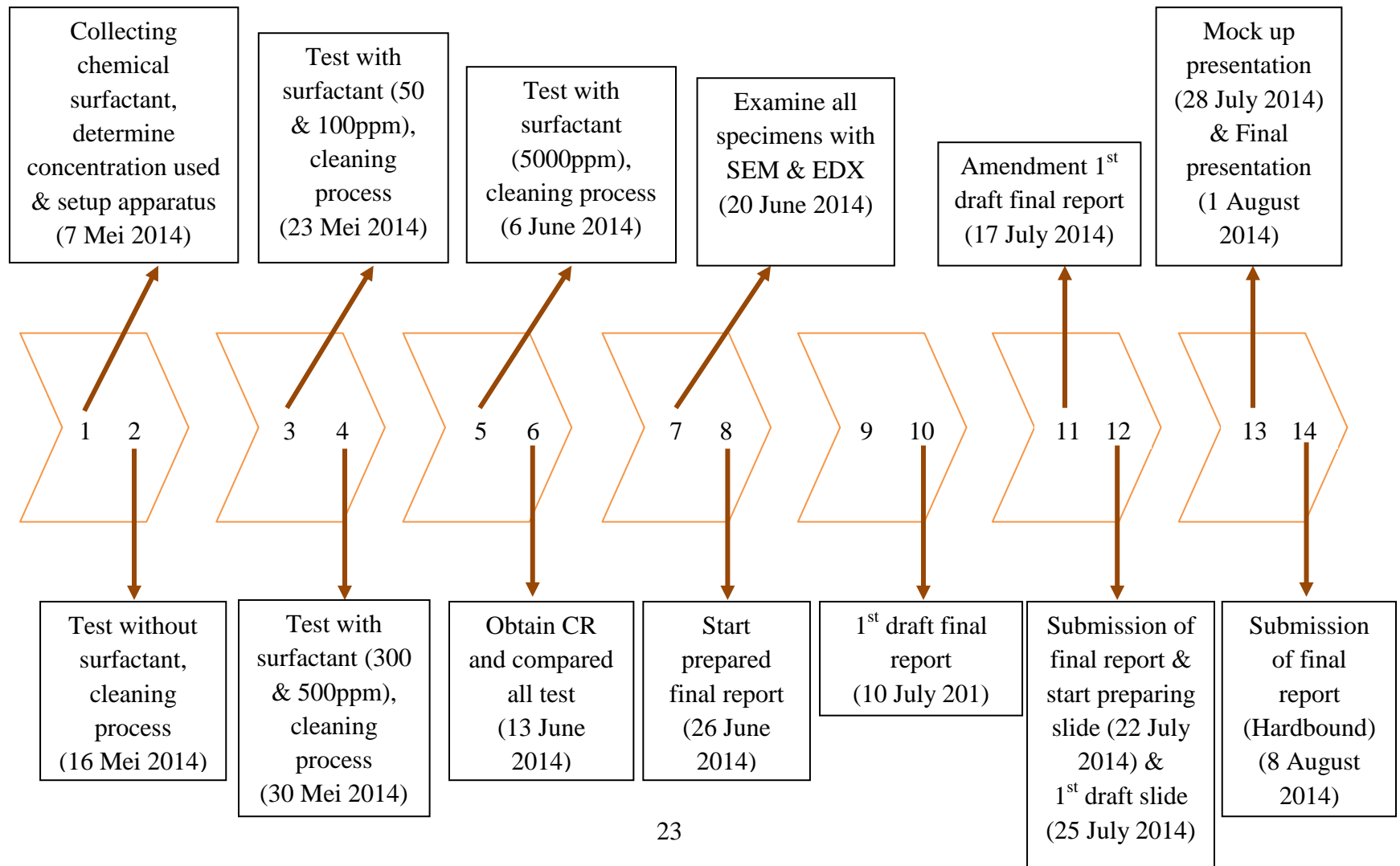
3.4 Key Milestone (January until May 2014)

Final Year Project 1 (FYP 1)



3.5 Key Milestone (May until August 2014)


Final Year Project 2 (FYP 2)



3.6 Gantt Chart Final Year Project 1 (FYP 1)

Table 5: Gantt chart for FYP 1

No	Activity	Week													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14
Final Year Project 1 (FYP 1)															
1	Selection of FYP 1 title														
1.1	FYP 1st briefing														
1.2	Consolidation of Supervisor (Interview)														
1.3	Approval of project														
2	Data gathering and Analysis														
2.1	Conduct preliminary research work (Reference)														
2.2	Contact PRSB for chemical supply														
2.3	Research paper :														
	Exploration production (Brown Field)														
	Enhanced Oil Recovery (EOR)														
	Surfactant (Effect when introduce to environment)														
	Carbon Dioxide Corrosion														
	Corrosion Inhibitor														
3	Extended proposal														
3.1	1st draft														
3.2	Final and submission														
4	Preparation proposal defence														
4.1	1st draft slide presentation														
4.2	Presentation														
5	Experimental														
5.1	Sample preparation:														
	Cutting and grinding														
	Conducting lab														
5.2	Prepare SOP and detail methodology:														
	Linear polarization resistance (LPR)														
	Scanning Electron Microscope (SEM)														
	Energy dispersive X-ray (EDX)														
6	Interim report														
6.1	1st draft														
6.2	Final and submission														

 - Complete

3.7 Gantt Chart Final Year Project 2 (FYP 2)

Table 6: Gantt chart for FYP 2

No	Activity	Week													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14
Final Year Project 2 (FYP 2)															
1	Data gathering and analysis:														
	Collecting surfactant chemical at PRSB														
	Determine range concentration of surfactant														
	Findings														
	Analysis of findings														
2	Experimental														
2.1	Apparatus setup														
	Conducting LPR without introducing surfactant														
	Cleaning process														
	Examine the specimen using SEM and EDX														
	Analyze the corrosion rate														
2.2	Apparatus setup														
	Conducting LPR with introducing surfactant:														
	Concentration (50, 100, 300, 500, 5000) ppm														
	Cleaning process														
	Examine the specimen using SEM and EDX														
	Analyze the corrosion rate														
2.3	Comparison of corrosion rate of both test														
3	Final report:														
3.1	1st draft														
3.2	Final and submission														
4	Technical presentation														
4.1	1st draft presentation slide														
4.2	Mock up presentation														
4.3	Final presentation														
5	Final report submission (hardbound)														

 - Complete

CHAPTER 4

RESULTS AND DISCUSSION

The effect of Enhanced Oil Recovery (EOR) surfactant on pipeline corrosion in CO₂ environment was studied at various concentration from 50 to 5000 ppm of EOR surfactant.

4.1 Characterization of EOR Surfactant using In-Situ Fourier Transform Infra-Red Spectrometer with High Vacuum Purge System (FTIR) Test.

The characteristics of EOR surfactant was analyzed and Table 7 below show the summarization of the range of IR frequency and the bond and functional group based on the results of Figure 16, page 28:

Table 7: Most common IR frequencies and bond stretches

IR Frequency	Bond and functional group
3500–3200	O–H stretch in phenols
3400–3250	N–H stretch in amines, amides
3300–2500	O–H stretch in carboxylic acids
3330–3270	–C≡C–H (alkyne H)
3100–3000	=C–H stretch (alkene H)
3000–2850	–C–H stretch (alkane H)
2830–2695	C–H stretch in aldehydes
2260–2210	C≡N stretch (nitrile)
2260–2100	–C≡C– stretch in alkynes
1760–1665	C=O stretch carbonyls (general)
1760–1690	C=O stretch in carboxylic acids
1750–1735	C=O stretch in esters
1740–1720	C=O stretch in aldehydes
1715	C=O stretch in ketones
1680–1640	–C=C– stretch in alkenes

Graph FTIR

Figure 16: FTIR graph

Based on the graph above showed that, the common bond and functional group of EOR surfactant can be identified by referring to Table 7 which was the IR frequencies and bond stretches. Seven peaks were chosen and analyzed accordingly.

The specific peaks bond and functional group of EOR surfactant can be shown at Table 8 below:

Table 8: Identified the bond and functional group for EOR Surfactant

Region	Components	IR Frequency	Bond and Functional group
Group frequency	Main	3411	N-H stretch in amines (NH ₄)
		2931	-C-H stretch, alkane H (CH ₃)
		2872	-C-H stretch, alkane H (CH ₂)
		2115	-C≡C- stretch in alkynes
Fingerprint	Additive	1556	Nitro (NO ₂)
		1073	C-O, benzyl alcohol-primary OH
		893	Phosphorus, PH bending

4.2 The effect of EOR Surfactant on corrosion rate in CO₂ Environment

The LPR results as shown below, with all required details as well as graph for each corrosion rate in mm/year. A part of that, the corrosion rate obtained from each concentration can be examined based on corrosion criteria and severity category (Table 9).

Table 9: Corrosion criteria and severity category

Category	Criteria	Description	SOL Value	
			Design CA: 1 mm for 30 years	Design CA: 3 mm for 30 years
Critical	$>4CR_d$	Corrosion might lead to immediate failure of the component	>0.12	>0.4
Warning	$2CR_d$ to $4CR_d$	Component life might reduce to one fourth of the design life	0.06 – 0.12	0.2 – 0.4
Normal	CR_d	Component will be able to achieve its design life	0.03	0.1

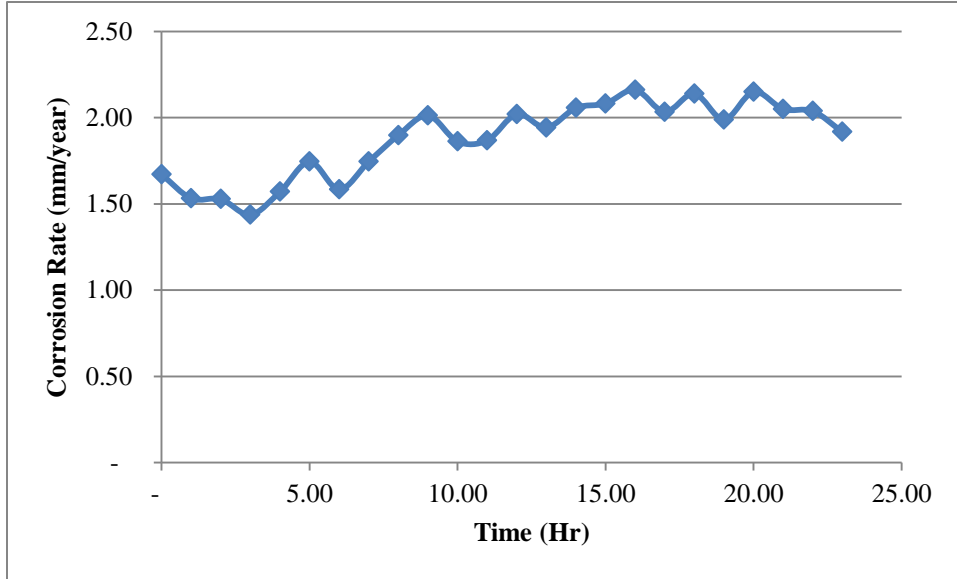


Figure 17: Corrosion rate trend for blank test at 25°C

The graph above (Figure 17) shows, the LPR test without EOR surfactant (Blank test). The blank test consists of carbon dioxide and the corrosion rate based on standard is in between 1.5 to 1.9 mm/year. The corrosion rate obtained from the blank test is 1.88 mm/year.

Figure 17 clearly shows the fluctuation and increment at the beginning and almost flat until end of test (24 hours).

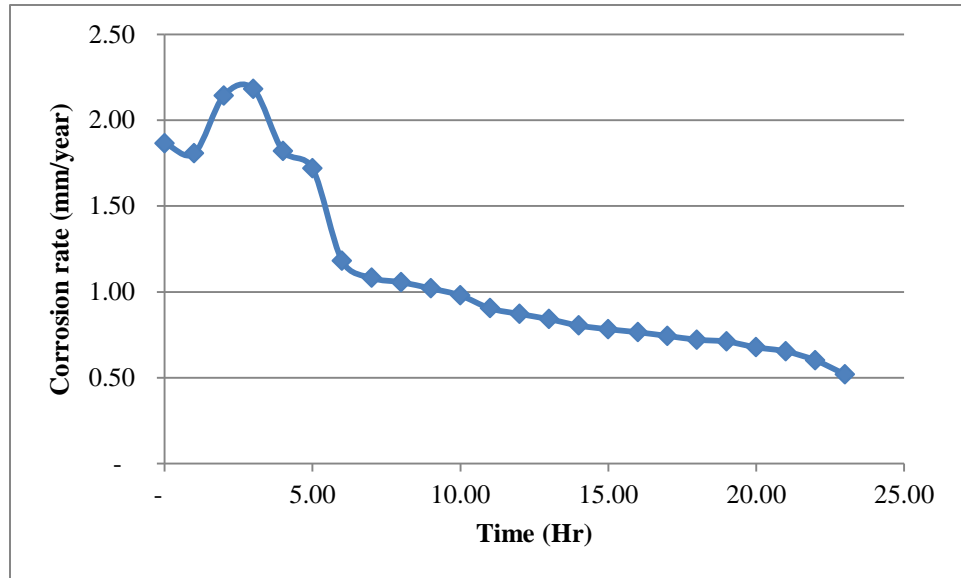


Figure 18: Corrosion rate trend for 50 ppm of EOR surfactant at 25°C

According to Figure 18 above, the corrosion rate starts to drop drastically at fifth hour of operations where the corrosion rate fall from 1.82 mm/year to 0.52 mm/year after adding 50 ppm of EOR surfactant.

An upward trend obtained at the beginning (one to third hours) and start to maintain the base line at fourth hours. After injection of 50 ppm EOR surfactant at fifth hours, the graph decreased steadily until end of test. At this stage, an assumption can be made where, the higher the concentration of EOR surfactant, the lower the corrosion rate.

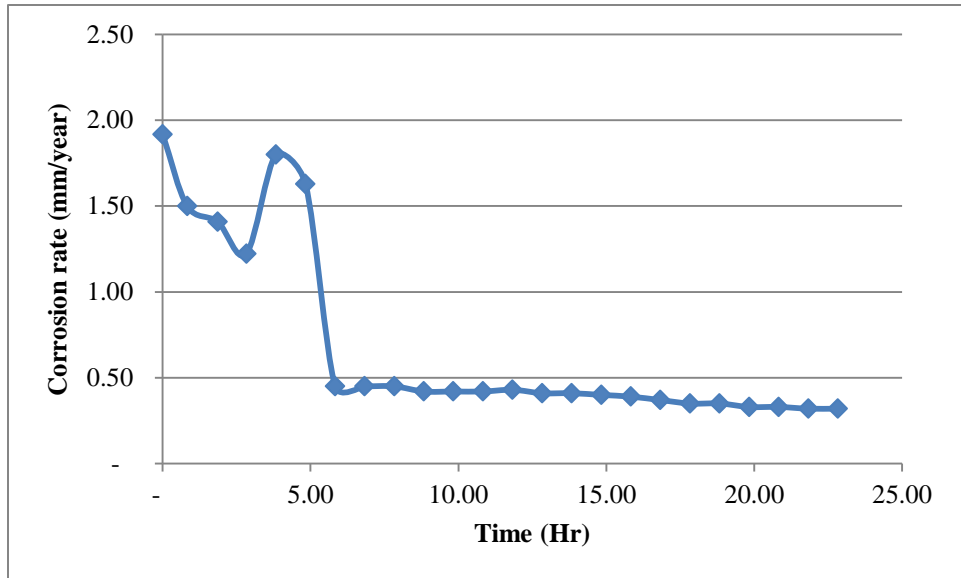


Figure 19: Corrosion rate trend for 100 ppm of EOR surfactant at 25°C

Figure 19 shows the corrosion rate trend for 100 ppm of EOR surfactant. Based on the graph above, the corrosion rate declined sharply from 1.80 mm/year to 0.32 mm/year at fifth hours of conducting the LPR test.

At the beginning, the corrosion rate clearly shows the fluctuation in order to get the base line. The graph almost constant after added the 100 ppm EOR surfactant throughout the test period.

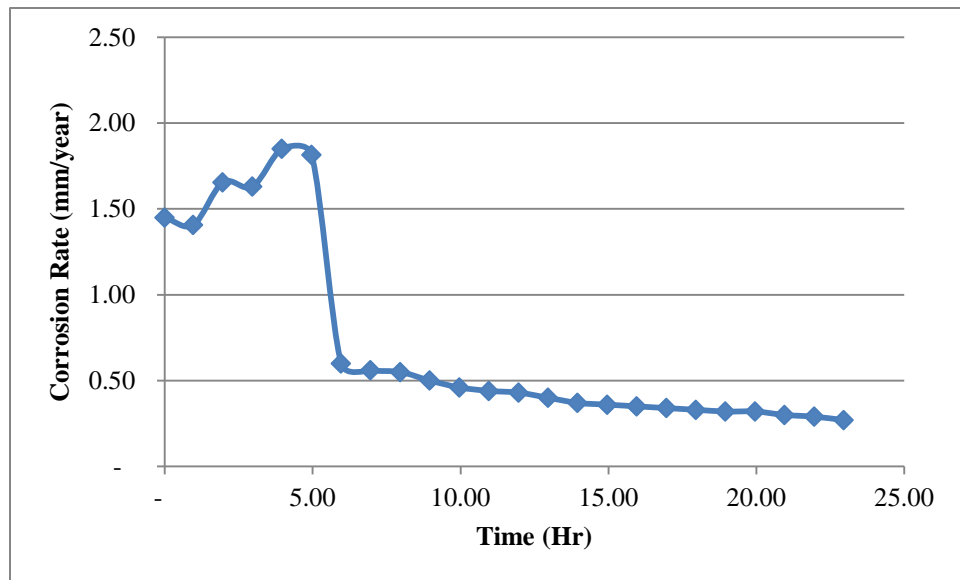


Figure 20: Corrosion rate trend for 300 ppm of EOR surfactant at 25°C

The corrosion rate for Figure 20 fell substantially starting at fifth hours from 1.85 mm/year to 0.27 mm/year after injection of 300 ppm EOR surfactant. The graph decrease constantly until the end of experiment. At the beginning the graphs increment slowly until reach the base line value.

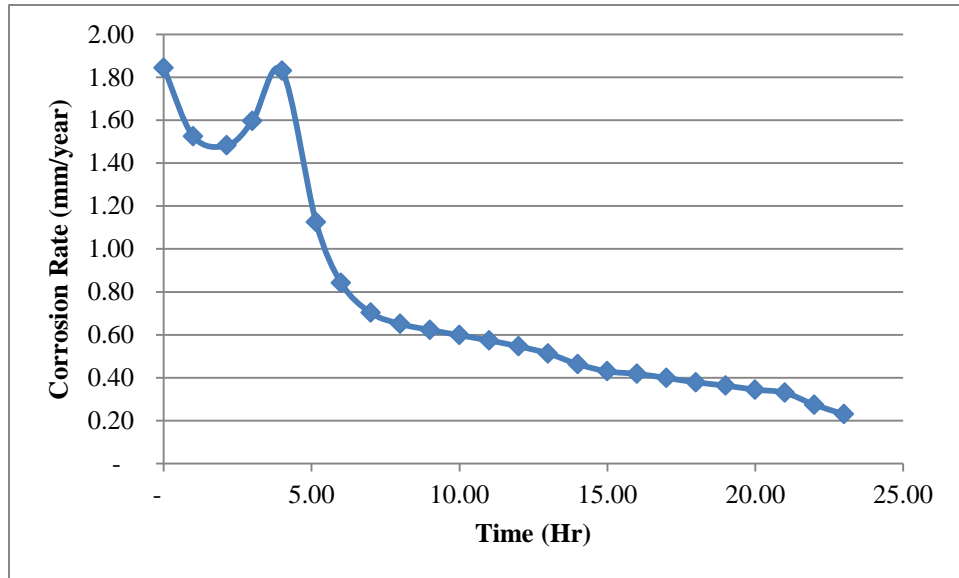


Figure 21: Corrosion rate trend for 500 ppm of EOR surfactant at 25°C

According to Figure 21 above, the corrosion rate trend reduce as the other EOR concentration before. The corrosion rate decrease from 1.83 mm/year to 0.23 mm/year. The graph fall from fifth hours of LPR test and continuously decrease until end of experiment. Compared with the 300 ppm of EOR surfactant, the differences of corrosion rate value were small only 0.04 mm/year.

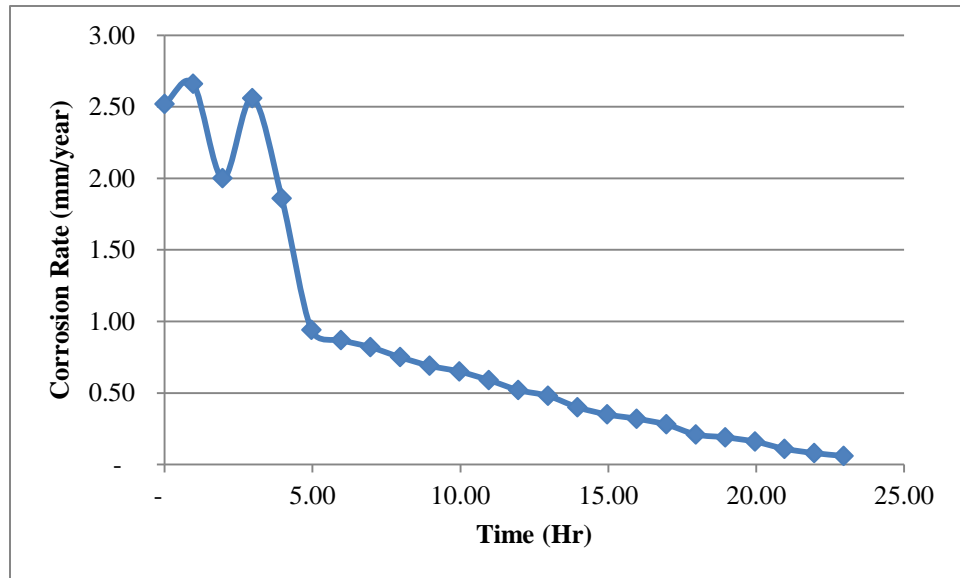


Figure 22: Corrosion rate trend for 5000 ppm of EOR surfactant at 25°C

The corrosion rate for 5000 ppm of EOR surfactant decreased from 1.86 mm/year to 0.06 mm/year. At the beginning, the fluctuations occur at first hours until fourth hours of LPR period and declined drastically after injection. The corrosion rate result was too small due to the large amount of EOR surfactant compared to the other LPR test above.

Same as before, the fluctuation at Figure 22 occur at the beginning of LPR test to obtain the base line. After injection, the graph clearly fell progressively until the LPR test done.

Based on the trend of corrosion rate above, it shows that, the additional of Enhanced Oil recovery (EOR) surfactant into the synthetics seawater reduce the corrosion rate. Below (Table 10) is the summarization of all test conducted:

Table 10: Summary of the reduction of corrosion rate with additional of EOR surfactant

No. of Test	Concentration of EOR surfactant (ppm)	Corrosion rate (mm/year)	
		Before injection	After injection
1	50	1.82	0.52
2	100	1.80	0.32
3	300	1.85	0.27
4	500	1.83	0.23
5	5000	1.86	0.06

According to (Table 9, page 30), the corrosion criteria and severity category, the highest value of corrosion rate indicated at concentration of 50 ppm which is 0.52 mm/year. By referring to Table 9, the value of corrosion rate falls to critical category for design of 3 mm for 30 years.

While the lowest corrosion rate shown at concentration of 5000 ppm which is 0.06 mm/year and the category indicate at normal conditions which design at 3 mm for 30 years. Despite of that, the sequence of corrosion rate can be shown as below:

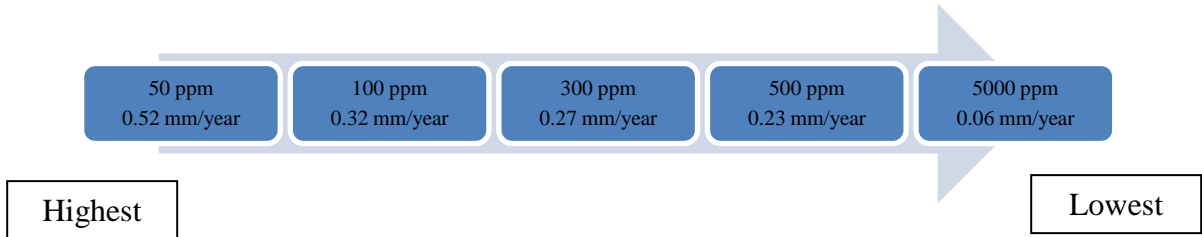


Figure 23: Sequence from highest to lowest of corrosion rate

Based on the theory of surfactant, the more concentration of surfactant injected, the more the formation of micelles which results in decreasing of corrosion rate as well as reduce the surface tension hence increase the production of oil. It is also act as corrosion inhibitor which protects the pipeline from corroding. According to the LPR test results, it is shows that, the higher the concentration of EOR surfactant, the lower the corrosion rate obtained.

Therefore, it is prove that the additional of EOR surfactant may not trigger the corrosion but also act as corrosion inhibitor.

4.3 pH Test

In order to obtain a CO₂ environment, the pH value needs to be tested in each condition, so that the solutions of synthetic seawater were rich with carbon dioxide. There were four types of conditions that need to be examined, which were pH values when diluting 3.0% NaCl with distilled water (Synthetic seawater), after inserting CO₂ gas for one hour (Purging test), injection of EOR surfactant and after leaving for one day (24 Hours). The pH values were analyzed with different concentrations of EOR Surfactant as shown below (Table 11):

Table 11: pH value for each condition

Conditions	Concentration of Enhanced Oil Recovery (EOR) Surfactant (ppm)					
	0 (Blank)	50	100	300	500	5000
Synthetic seawater	5.55	5.34	5.59	5.19	5.42	5.46
After purging test	3.94	3.85	3.83	3.85	3.86	3.92
After adding EOR surfactant	*Due to the blank test, the pH value still same, 3.94	3.97	3.94	4.03	4.03	4.02
After one day (24 Hours)	4.16	4.10	4.05	4.21	4.20	4.13

4.4 Metallography

Metallography is the process of preparing metal surface to reveal micro-structural information. In order to have the microscopy image, the suitable tool is Scanning Electron Microscopy (SEM) and Figure 24 and 25 below shows, the microscopy view for highest corrosion rate.

- Highest corrosion rate (50 ppm)

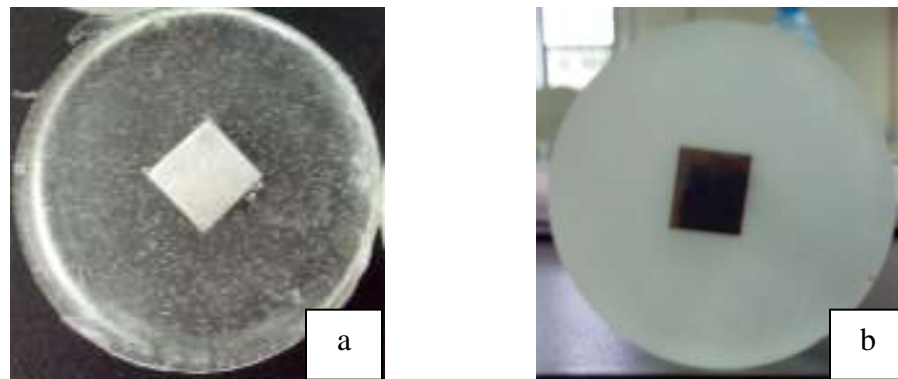


Figure 24: Sample X-65 for 50 ppm, a) Before undergo LPR test and b) After one day undergo LPR test

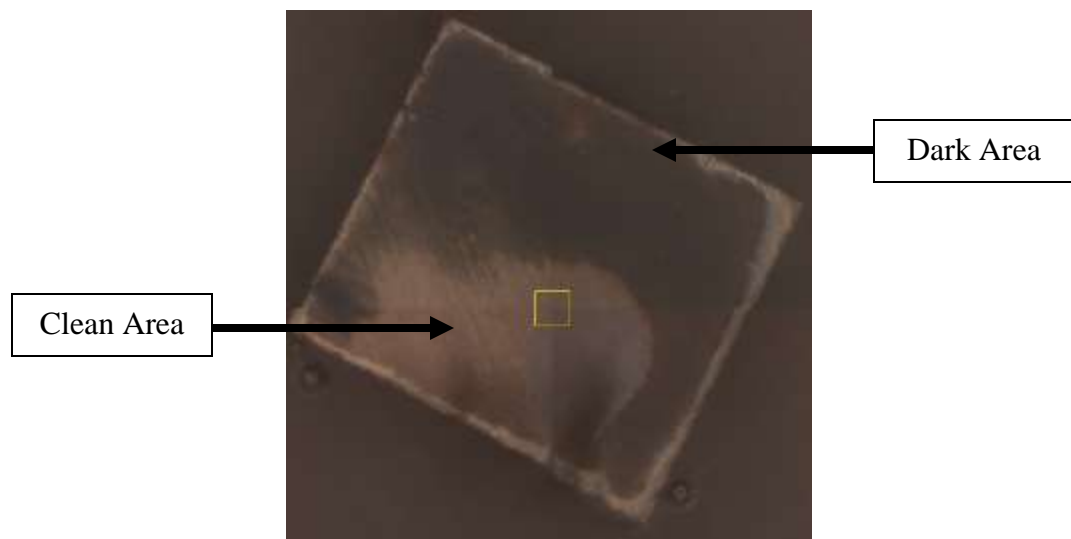
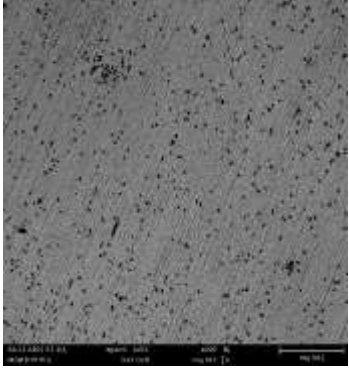
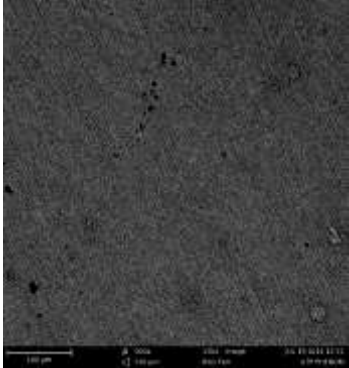
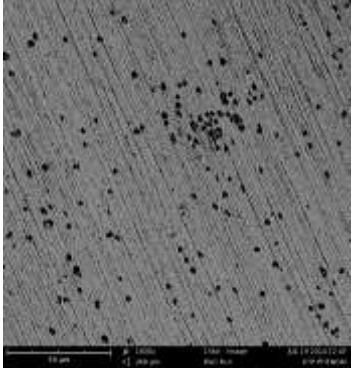
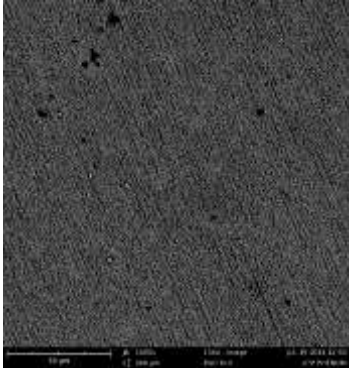
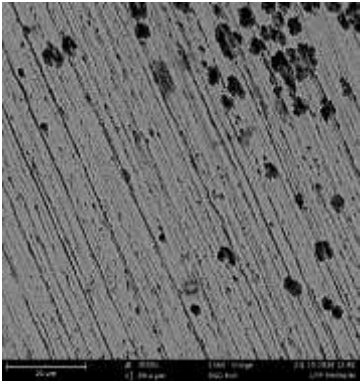
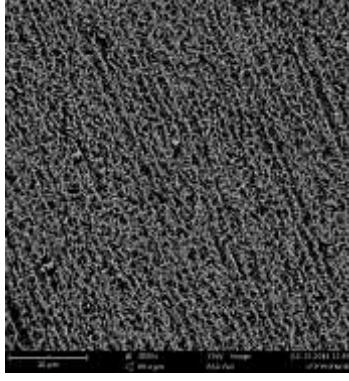


Figure 25: Overall view of sample X-65 under SEM for 50 ppm

The dark area as shown in Figure 25 above is the film that produces by the injection of 50 ppm EOR surfactant throughout the LPR test.

Table 12 below shows the microscopy image of sample X-65 by using difference magnifications.

Table 12: Surfaces of sample X-65 using SEM test for 50 ppm

Magnification	Area	
	Clean	Dark
500X		
1000X		
3000X		

Figures 26 and 27 below shows, the metallography for the lowest corrosion rate

- Lowest corrosion rate (5000 ppm)

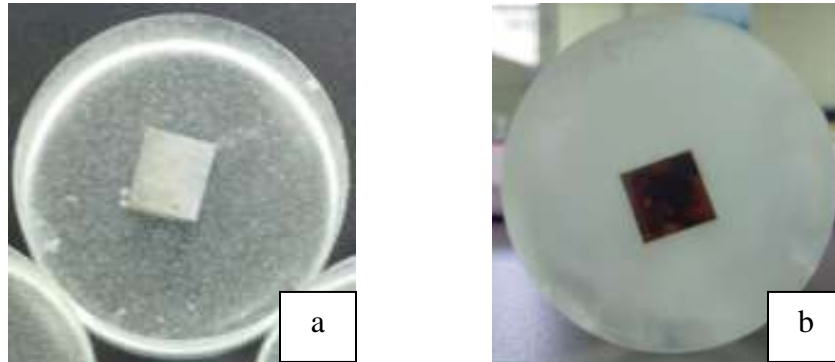


Figure 26: Sample X-65 for 5000 ppm, a) Before undergo LPR test and b) After one day undergo LPR test

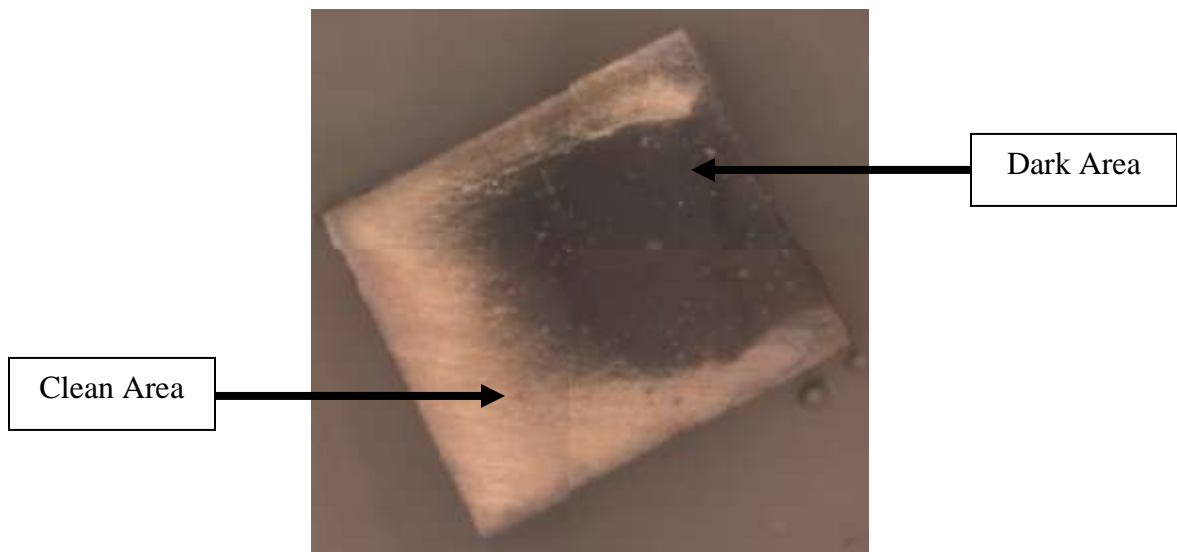
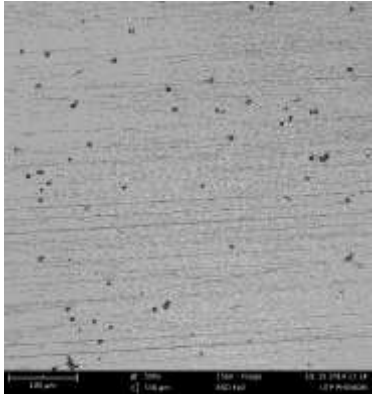
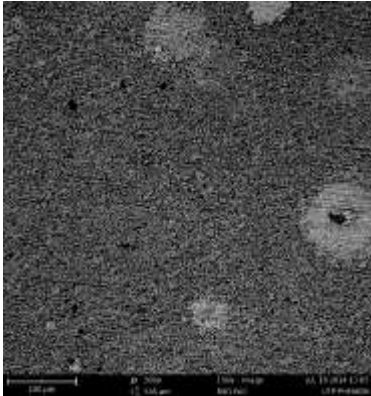
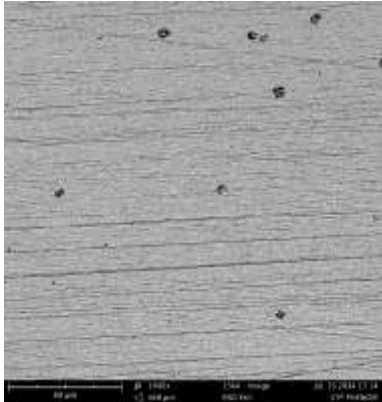
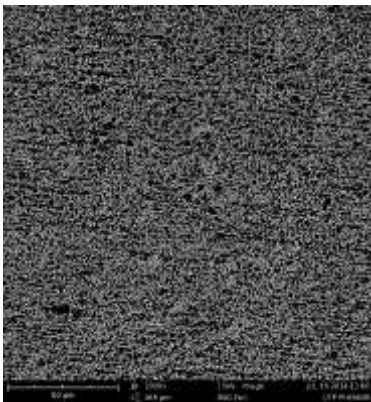
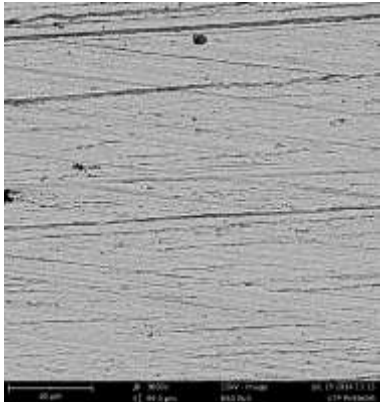
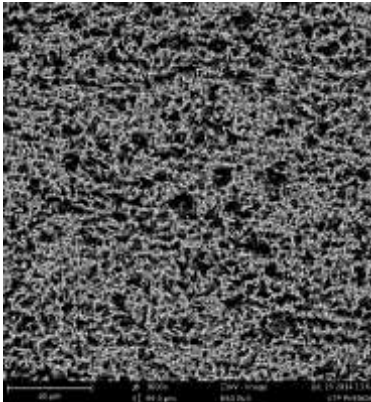


Figure 27: Overall view of sample X-65 under SEM for 5000 ppm

The dark area as shown in Figure 27 above is the film that produces by the injection of 5000 ppm EOR surfactant throughout the LPR test.

Table 13 below shows the microscopy image of sample X-65 by using difference magnifications.

Table 13: Surfaces of sample X-65 using SEM test for 5000 ppm

Magnification	Area	
	Clean	Dark
500X		
1000X		
3000X		

4.5 X-ray Diffraction

The techniques was used to identify the chemical composition of sample X-65 using x-rays and Figure 28 below show the required region to be examined.

- Highest corrosion rate (50 ppm)

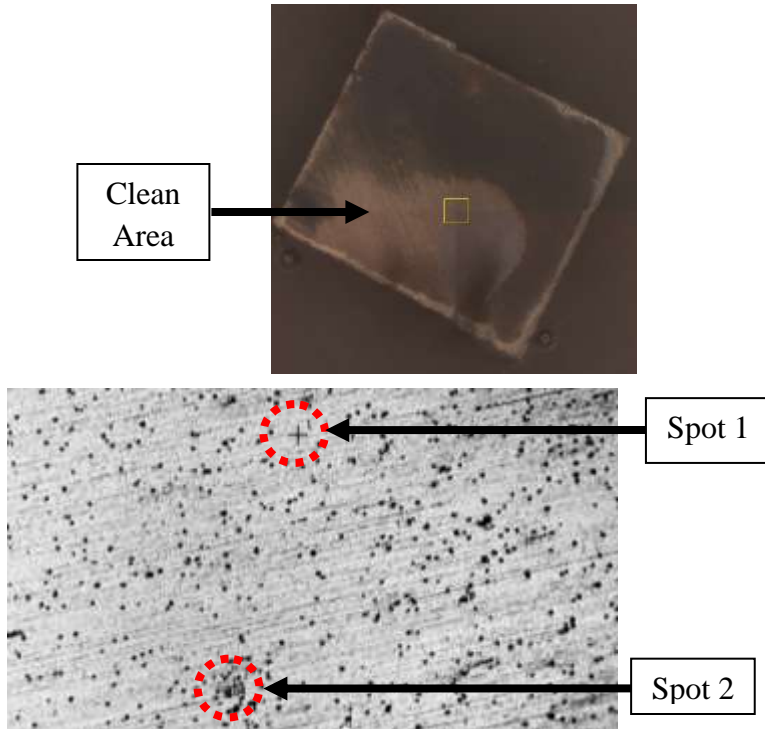


Figure 28: Spot 1 and spot 2 using EDX analysis (Clean area, 50 ppm) at 500X

Table 14 and 15 indicate that the percentage chemical composition for the selected spot at highest corrosion rate, 50 ppm (clean area).

Table 14: Percentage of each element at spot 1 (Clean area, 50 ppm)

Number at Periodic Table	Name of the element	Percentage (%)
26	Fe (Iron)	86.0
8	O (Oxygen)	10.8
6	C (Carbon)	1.4
14	Si (Silicon)	1.8

Table 15: Percentage of each element at spot 2 (Clean area, 50 ppm)

Number at Periodic Table	Name of the element	Percentage (%)
26	Fe (Iron)	80.1
8	O (Oxygen)	15.4
6	C (Carbon)	1.6
7	N (Nitrogen)	2.9

While Figure 29 shows, the sample X-65 undergo x-rays for highest corrosion rate consist of dark area.

- Highest corrosion rate (50 ppm)

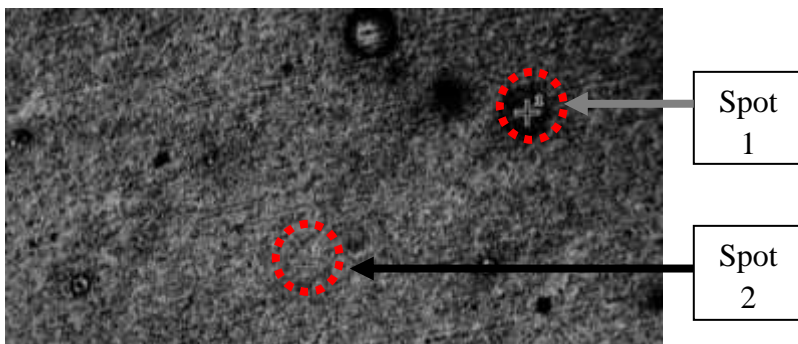
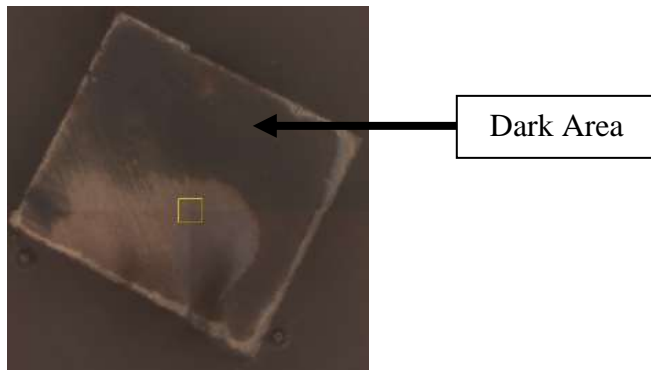


Figure 29: Spot 1 and spot 2 using EDX analysis (Dark area, 50 ppm) at 500X

Table 16 and 17 shows the percentage of chemical composition for the selected spot for highest corrosion rate, 50 ppm at dark are.

Table 16: Percentage of each element at spot 1 (Dark area, 50 ppm)

Number at Periodic Table	Name of the element	Percentage (%)
26	Fe (Iron)	77.1
8	O (Oxygen)	20.5
6	C (Carbon)	2.5

Table 17: Percentage of each element at spot 2 (Dark area, 50 ppm)

Number at Periodic Table	Name of the element	Percentage (%)
26	Fe (Iron)	84.4
8	O (Oxygen)	13.5
6	C (Carbon)	2.2

Figure 30 below shows, the sample X-65 undergo x-rays diffraction.

- Lowest corrosion rate (5000 ppm)

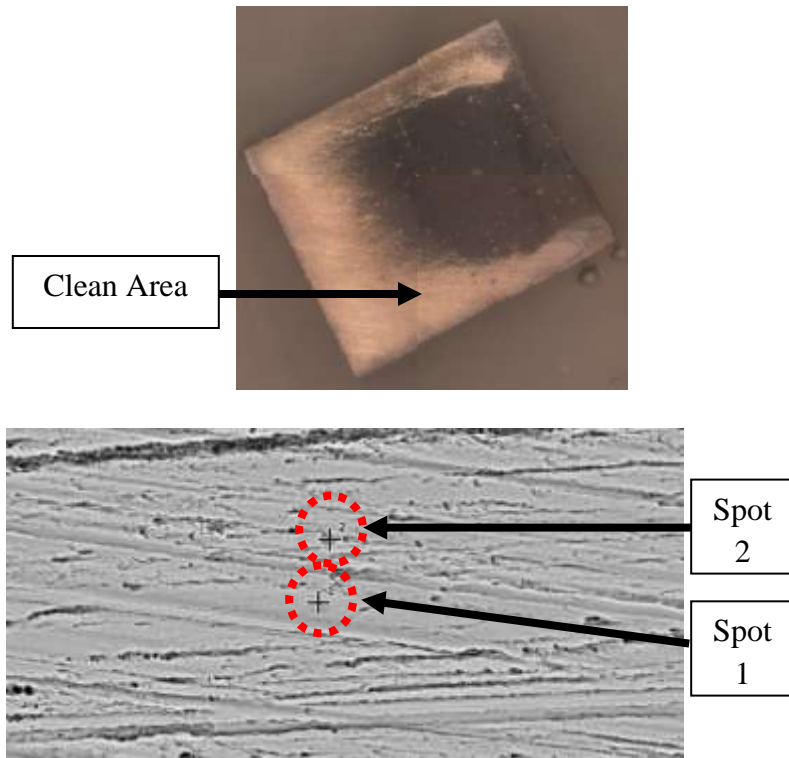


Figure 30: Spot 1 and 2 using EDX analysis (Clean area, 5000 ppm) at 500X

Table 18 and 19 shows the percentage of chemical composition for the selected spot for highest corrosion rate, 5000 ppm at clean are.

Table 18: Percentage of each element at spot 1 (Clean area, 5000 ppm)

Number at Periodic Table	Name of the element	Percentage (%)
26	Fe (Iron)	83.9
8	O (Oxygen)	10.3
14	Si (Silicon)	4.3
41	Nb (Niobium)	4.7
17	Cl (Chlorine)	4.8

Table 19: Percentage of each element at spot 2 (Clean area, 5000 ppm)

Number at Periodic Table	Name of the element	Percentage (%)
26	Fe (Iron)	87.5
8	O (Oxygen)	9.0
14	Si (Silicon)	3.5

While Figure 31 shows, the sample X-65 undergo x-rays for lowest corrosion rate consist of dark area.

- Lowest corrosion rate (5000 ppm)

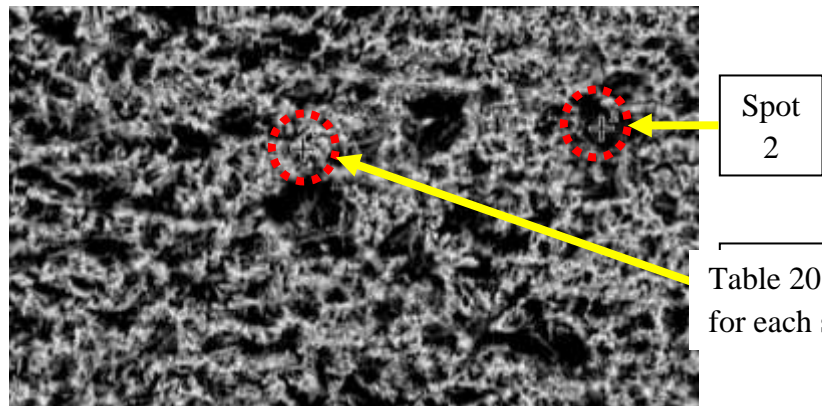
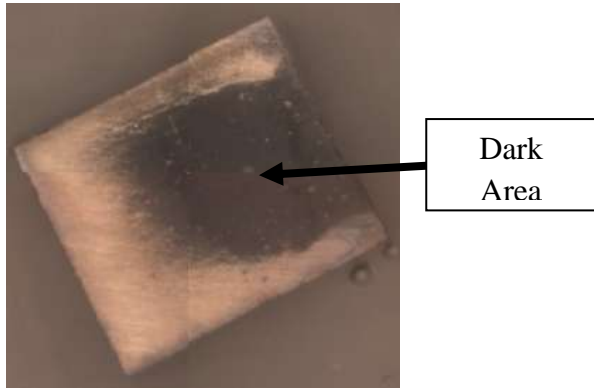


Table 20: Percentage of each element at spot 1 (Dark area, 5000 ppm)

Number at Periodic Table	Name of the element	Percentage (%)
26	Fe (Iron)	82.3
8	O (Oxygen)	14.6
6	C (Carbon)	1.2
14	Si (Silicon)	1.8

Table 21: Percentage of each element at spot 2 (Dark area, 5000 ppm)

Number at Periodic Table	Name of the element	Percentage (%)
6	C (Carbon)	11.6
26	Fe (Iron)	27.1
8	O (Oxygen)	29.5
11	Na (Sodium)	20.0
7	N (Nitrogen)	11.9

Table 20 and 21 shows the percentage of chemical composition for each spot at dark area.

Figure 31: Spot 1 and 2 using EDX analysis (Dark area, 5000 ppm) at 500X

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The function of EOR surfactant is to reduce the surface tension between solid and liquid hence increase the production of oil. EOR surfactant mainly composed of active compound such as amines (NH_4) and alkane (CH_3 and CH_2). The additional of EOR surfactant reduce the corrosion rate depending on the dosage of EOR surfactant. The additional of EOR surfactant is able to reduce the corrosion rate up to 97%. The usage of EOR surfactant reduces the CO_2 corrosion rate based on the surfactant behavior which resembles corrosion inhibitor properties. The EOR surfactant also an active-component not only increases the production of oil as well as able to reduce the corrosion rate trend.

5.2 Recommendation

Below are some recommendations that can improve the results as well as the corrosion rate

- In order to have better results, it is recommended to have test with flowing fluids (flow loop corrosion) which present of velocity and able to stimulate condition mimic to the real pipeline condition.
- Before mounting the probe, the sample must be cover with the cure tack to avoid any corrosion occur at the side of samples.
- The reference probe needs to be calibrated according to the standard and work properly.
- The reference probe must be clean before inserting into the glass cell which can make the reading become messy and fuzzy.
- Avoid any flow (CO₂ gas) pass through the mounting probe which cause bubble at the surface of mounting probe.
- Before injecting the EOR surfactant, make sure the corrosion rates are stable. The corrosion rate is stable after 15 hours running the test without inserting EOR surfactant.
- For better results, the parameters must be longer about seven days or more.
- Repetitions of tests are required to get the precise and average reading.
- To variable the results, including any temperature is recommended.
- For future study, analyze the characteristic of EOR surfactant at 5000 ppm which cause the sudden drop of corrosion rate.

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Appendices