

**Study on the Effect of Thermal Stability of Corrosion Inhibitor**

by

Aminah Syahidah Bt Mohd Yusuff

Dissertation submitted in partial fulfilment of  
the requirements for the  
Bachelor of Engineering (Hons)  
(Mechanical Engineering)

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# CERTIFICATION OF APPROVAL

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A project dissertation submitted to the  
Mechanical Engineering Programme  
Universiti Teknologi PETRONAS  
in partial fulfillment of the requirement for the  
BACHELOR OF ENGINEERING (Hons)  
(MECHANICAL ENGINEERING)

Approved by,

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(AP Dr Othman Bin Mamat)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

September 2011

## 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.

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AMINAH SYAHIDAH BT MOHD YUSUFF

## ABSTRACT

CO<sub>2</sub> corrosion has taken place inside the pipeline transporting wet natural gas containing CO<sub>2</sub> at very high temperature and become the main threat in oil and gas industry. One way to mitigate corrosion is by adding corrosion inhibitors to the system. Many different factors influence the inhibition efficiency and it is well known that slight changes in the chemistry changes inhibitor efficiency. The objective of this project is to understand the corrosion phenomena of pipelines specifically in terms of the effect of thermal stability that may influence the effectiveness of corrosion inhibitor on corrosion test using X-52 carbon steel sample. The thermal stability of the corrosion inhibitor will be evaluated by heating the inhibitor in temperature controlled oven at 90°C, 100°C, 120°C and 140°C for four days. The inhibitor used is from AMTECH which is generic imidazoline based inhibitor. The data for corrosion rate and efficiency calculation can be obtained by corrosion test experiments in a glass cell filled with 1L of deionized water and 3% wt NaCl at pH=4.0, T=25°C, purged with CO<sub>2</sub> at atmospheric pressure. Different inhibitor concentrations (25ppm and 50ppm) are added during each experiment. From the experiment, it is found that the efficiency of inhibitor increased when temperature increases from room temperature 25°C to 100°C. However, after 100°C, the inhibitor efficiency decreased when the temperature is increased. This is possibly due to the effect of the corrosion product film formation that governed the protection of metal surface at the higher temperature. The recommendation is to investigate the formation of corrosion product film on the steel surface at higher temperature of corrosion inhibitor or at high temperature surrounding.

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## ABBREVIATIONS AND NOMENCLATURES

<b>ASTM</b>	American Society for Testing and Materials
<b>ASME</b>	American Society of Mechanical Engineers
<b>NACE</b>	National Association of Corrosion Engineers
<b>API</b>	American Petroleum Institute
<b>CO<sub>2</sub></b>	Carbon Dioxide
<b>H<sub>2</sub>CO<sub>3</sub></b>	Carbanic Acid
<b>NaCl</b>	Sodium Chloride
<b>LPR</b>	Linear Polarization Resistance
<b>CAPCIS</b>	Corrosion And Protection Centre, Industrial Services
<b>FTIR</b>	Fourier Transfer Infrared Spectroscopy
<b>LC-MS</b>	Liquid Chromatography-Mass Spectroscopy
<b>XPS</b>	X-Ray Photoelectron Spectroscopy
<b>OM</b>	Optical Microscope
<b>SEM</b>	Scanning Electron Microscope

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# CHAPTER 1

## INTRODUCTION

This section will provide some background information of the project, discuss on the problem statement that leads to the establishment of the project and put forward the objective and scope of study of the project.

### 1.1 BACKGROUND OF STUDY

Corrosion is one of the most common, costly and widespread industrial problems in today's modern world. Corrosion comes from the Latin word "*corrodere*" which means "*to gnaw away*" [1]. Corrosion can be defined as the degradation of a material due to a reaction with its environment while degradation implies deterioration of physical properties of the material. This can be a weakening of the material due to a loss of cross-sectional area. It can be the shattering of a metal due to hydrogen embrittlement, or it can be the cracking of a polymer due to sunlight exposure. [2] Carbon dioxide ( $\text{CO}_2$ ) corrosion results when  $\text{CO}_2$  dissolves in water to form carbonic acid ( $\text{H}_2\text{CO}_3$ ). The acid may lower the pH and sufficient quantities may promote general corrosion and/or pitting corrosion of carbon steel. The partial pressure of  $\text{CO}_2$ , pH and temperature are critical factors. Increasing partial pressures of  $\text{CO}_2$  will result in lower pH condensate and higher rates of corrosion and increasing temperatures increase corrosion rate up to the point where  $\text{CO}_2$  is vaporized. Corrosion occurs in the liquid phase, often at locations where  $\text{CO}_2$  condenses from the vapour phase. [3] Metal corrosion can cause extensive problems, but there are ways to prevent this by increasing the durability and wear of a product with metal parts. One of it is by using corrosion inhibitor.

Corrosion inhibitors are chemical compounds added to the corrosive medium to reduce the rate of its attack on the metal or alloy. The chemicals which can act as corrosion inhibitors may be inorganic or organic. The inorganic compounds

such as chromates inhibit the corrosion process via formation of passive oxide film on the metal surface and thus prevent the corrosive medium to attack the base metal. On the other hand, the organic compounds adsorb on the metal surface forming a barrier between the metal and the corrosive environment. Some structural features of the organic compounds help them to do so. These include the presence of oxygen, nitrogen or sulphur atoms as well as presence of double bonds. The lone pair electrons of the mentioned atoms facilitate the adsorption process. Some criteria should be considered when making a choice of chemical compounds for inhibition of corrosion. Inhibition of metallic corrosion is mainly an economical process. Therefore, the first criterion must be fulfilled by the used inhibitors is their prices. The other very important criterion should be considered when dealing with corrosion inhibitor is its effect on the human and environment. Unfortunately, most of the effective corrosion inhibitors are synthetic chemicals with high cost. At the same time, the use of such synthetic compounds can cause harm to human and environment. Most of the naturally occurrence substances are safe and can be extracted by simple and cheap procedures. Many of these naturally occurring substances proved their ability to act as corrosion inhibitors for the corrosion of different metals and alloys in different aggressive media. [4]

The effectiveness of the corrosion inhibitor is a function of many factors like fluid composition, quantity of water, flow regime and temperature. If the correct inhibitor and quantity is selected then it is possible to achieve high efficiency up to 90-99%. Some of the mechanisms of its effect are formation of passivation layer which is a thin film on the surface of the material that stops access of the corrosive substance to the metal, inhibiting either the oxidation/reduction part of the reduction corrosion system (cathodic/anodic inhibitors).

## **1.2 PROBLEM STATEMENT**

### **1.2.1 Problem Identification**

Corrosion in the oilfield is a dynamic and complex chemical process occurring over a wide range of conditions. A major component of a corrosion management program is the use of corrosion inhibitors for steel pipes in a CO<sub>2</sub> environment. In using a corrosion inhibitor, a chemical or chemical mixture is injected into the system at a low concentration (typically parts per million). High temperature CO<sub>2</sub> corrosion of carbon steel, in conjunction with a high production rate affects the kinetic balance of formation and deterioration of the protective passivation layer [5]. A high performance corrosion inhibitor which improves performance and film life at high temperature and high shear will increase reliability of corrosion inhibitor and less reliance on expensive corrosion resistant alloy [6]. Investigation can be conducted to obtain the baseline performance data at high temperature for the corrosion inhibitor through the corrosion test method.

### **1.2.2 Significant of the Project**

The experimental project is significant in obtaining the most suitable condition of corrosion inhibitor in terms of thermal stability and effect of temperature on the formation of protective film and its morphology for X-52 carbon steel that usually used in piping system. This study may help further research for the improvement of corrosion inhibitor efficiency in order to develop a better protection of pipelines in oil and gas industry in the future.

## **1.3 OBJECTIVE**

The objective of this study is to investigate the effect of thermal stability that may influence the effectiveness of corrosion inhibitor on corrosion test using X-52 carbon steel.

#### **1.4 SCOPE OF STUDY**

The thermal stability of the corrosion inhibitor will be evaluated by heating the inhibitor in temperature controlled oven at 90°C, 100°C, 120°C and 140°C for four days. The inhibitor used is from AMTECH which is generic imidazoline based inhibitor. The data for corrosion rate and efficiency calculation can be obtained by corrosion test experiments in a glass cell filled with 1L of deionized water and 3% wt NaCl at pH=4.0, T=25°C, purged with CO<sub>2</sub> at atmospheric pressure. Different inhibitor concentrations (25ppm and 50ppm) will be added during each experiment.

#### **1.5 RELEVANCY OF PROJECT**

By doing this research, there are few advantages that noticeably will help for future enhancement especially for pipeline system. By getting the result from this experimental project, we can identify which condition of corrosion inhibitor and the thermal stability that is suitable for reducing the corrosion rate therefore can lessen the possibility of the pipeline system from being exposed to more severe CO<sub>2</sub> corrosion.

#### **1.6 FEASIBILITY OF PROJECT**

Final year project for mechanical engineering students is obligatory to be completed within 2 semesters. The project commences with study and research work in the four months time of the first semester (FYP 1) following by experimental work in four months time of the second semester (FYP 2). It is assumed that the project is feasible within the scope and time frame regardless of no issues with regard to equipment function and material availability and the project should be successfully done. The proposed Gantt chart with the milestone and expected due date is shown in Chapter 3.

## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Overview of CO<sub>2</sub> Corrosion

Carbon Dioxide (CO<sub>2</sub>) corrosion is one the most studied form of corrosion in oil and gas industry. This is generally due to the fact that the crude oil and natural gas from the oil reservoir / gas well usually contains some level of CO<sub>2</sub> (and H<sub>2</sub>S – hydrogen sulfide). The major concern with CO<sub>2</sub> corrosion in oil and gas industry is that CO<sub>2</sub> corrosion can cause failure on the equipment especially the main down hole tubing and transmission pipelines and thus can disrupt the oil/gas production [7]. Figure 2.1 shows the model of CO<sub>2</sub> corrosion of a crude oil pipeline made of mild steel.

The study of CO<sub>2</sub> corrosion rate and FeCO<sub>3</sub> film formation are essential to enhance the understanding and modelling the kinetics of FeCO<sub>3</sub> precipitation process [7]. The presence of CO<sub>2</sub> in solution would initiate the CO<sub>2</sub> corrosion process. It would produce a weak carbonic acid (H<sub>2</sub>CO<sub>3</sub>) which is corrosive to carbon steel or low alloy steel and it is presented by equation (2.1) below:



The reaction process will continue with three cathodic reactions (reduction) and one anodic reaction (oxidation). The cathodic reactions in CO<sub>2</sub> solutions are:

1. Reduction of carbonate acid into bicarbonate ions.



2. Reduction of bicarbonate ions into carbonate ions.



3. Reduction of hydrogen ions.



There are some main factors that can affect the severity of CO<sub>2</sub> Corrosion Rate (CR). The first factor is CO<sub>2</sub> partial pressure: higher partial pressure of CO<sub>2</sub>, CR will be higher. The second is temperature: higher temperature, higher CR while the third is pH: lower pH will result in higher CR. Flow velocity also can affect the severity of CO<sub>2</sub> corrosion: consequence of higher velocity is higher CR.

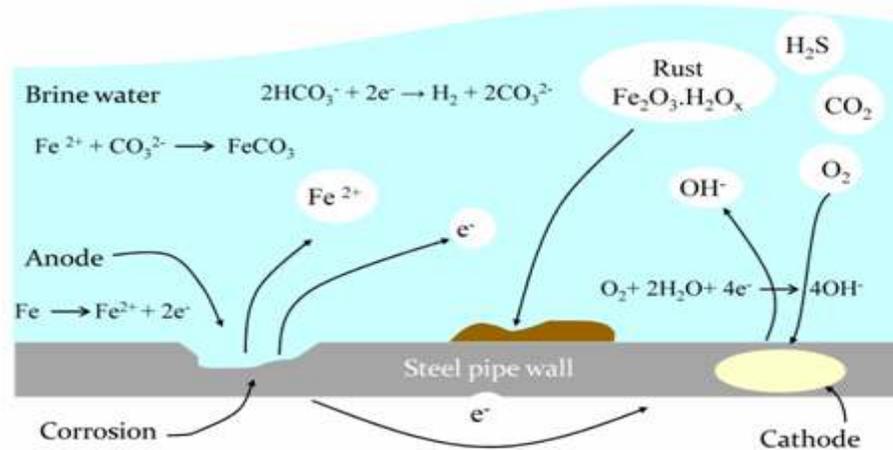


Figure 2.1: Model of CO<sub>2</sub> corrosion of a crude oil pipeline made of a mild steel.

## 2.2 Factors Affecting CO<sub>2</sub> Corrosion

There are several important factors that would affect CO<sub>2</sub> corrosion. Eventually, from these factors the formation of protective corrosion product would also be affected, which affect the corrosion rate of metal. The parameters comprise pH, temperature, CO<sub>2</sub> partial pressure, Fe<sup>2+</sup> concentration and fluid velocity.

## 2.3 Corrosion Inhibitor

Corrosion inhibitors are used to protect oil and gas pipelines made of carbon steel that transport CO<sub>2</sub> or H<sub>2</sub>S containing wet hydrocarbons. [6] Corrosion inhibitors and especially film-forming corrosion inhibitors are most frequently used for protection of oil, condensate, and gas production lines. Film-forming corrosion inhibitors work by forming a protective layer that physically prevents corrosive chemicals such as water and chloride ions from penetrating to the metal surface.

This means that the effectiveness is partly determined by the strength of its adsorption to the metal surface.

Many film-forming inhibitors are organic surfactants with a polar head group and a hydrophobic tail. The head group is designed to interact with the steel surface and the hydrophobic tails attract liquid hydrocarbons which will form an oil barrier. If for some reason part of the pipe is left unprotected by film-forming inhibitors, localized corrosion may occur.

There are also study that the interaction between inhibitors and protective film is efficient when either the species alone. With varies concentration of inhibitors, the experiments has successfully indicate that neither species dominates the adsorbed film; however a synergistic relationship has occurred to decrease the corrosion effect. [8]

#### **2.4 Corrosion control by organic corrosion inhibitors**

The corrosion of metals cannot be stopped completely, but it can be controlled by decreasing the rate of corrosion. Corrosion control functions by eliminating or reducing the effectiveness of one or more of the corrosion cell components. Corrosion control methods in oilfield systems include cathodic protection, protective coatings, chemical inhibitors, plastic or cement liners, use of special alloys, solids removal and removal of corrosive gases.

In general, cathodic protection is an approach where the metal surface to be protected is made into the cathode of a corrosion cell. Since corrosion and material loss occurs at the anode, this approach protects the metal. Protective coatings can be used to protect tubing, downhole equipment, wellhead components and pressure vessels. Coatings work by reducing the cathodic area available for the corrosion reaction. The use of organic corrosion inhibitors is the most effective way of protecting internal corrosion of carbon steel pipelines for oil product transportation.

Inhibition is used internally with carbon steel pipes and vessels as an economic corrosion control alternative to stainless steels, coatings and non-metallic composites. A particular advantage of the corrosion inhibitor is that it can be introduced *in-situ* without disrupting the transportation process and it adsorbs into the hard-to-reach surfaces inside the pipes. The major industries using corrosion inhibitors are oil and gas exploration and production, petroleum refining, chemical manufacturing, heavy manufacturing, water treatment and the product additive industries [9].

## 2.5 Mechanism of Corrosion Inhibitor

The corrosion inhibitors used in oilfield applications are organic or ionic compounds that are employed in small concentrations (less than 0.1 wt.%). They are often categorised as mixed inhibitors as they adsorb on the steel surface and inhibit both anodic and cathodic reactions. Almost all organic molecules used in oilfield corrosion inhibitor packages are strongly polar, with many being based on nitrogen, such as the amines, amides, imidazolines or quaternary ammonium salts and compounds containing P, S and O elements. Molecular structures for some of the most commonly used organic corrosion inhibitors in the oilfield system are given in Figure 2.2. The organic corrosion inhibitors are typical surface-active agents due to the presence of hydrophilic and hydrophobic moieties within the same molecule.

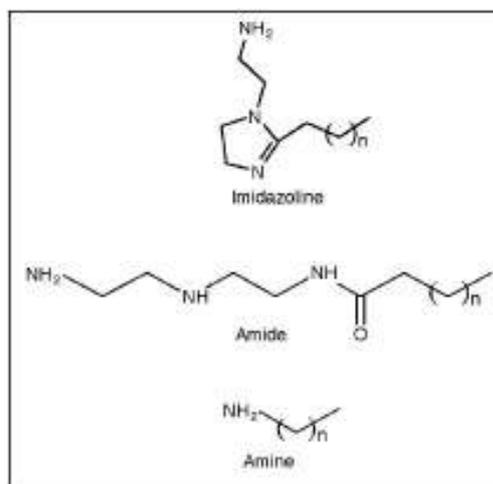


Figure 2.2: Basic molecular structures of oil field corrosion inhibitors

Typically, the molecules have a hydrocarbon chain attached to the polar group, the length of which varies. The mechanism by which the organic corrosion inhibitor used to reduce the corrosion is not fully understood so far. The polar group of the molecule provides the functionality that displaces the water molecules from the surface (Figure 2.3). The adsorption of the corrosion inhibitor from aqueous solution onto the metal surface is driven by both of the polar head group and the hydrocarbon tail group. The concentration of inhibitor has a profound effect upon corrosion inhibition.

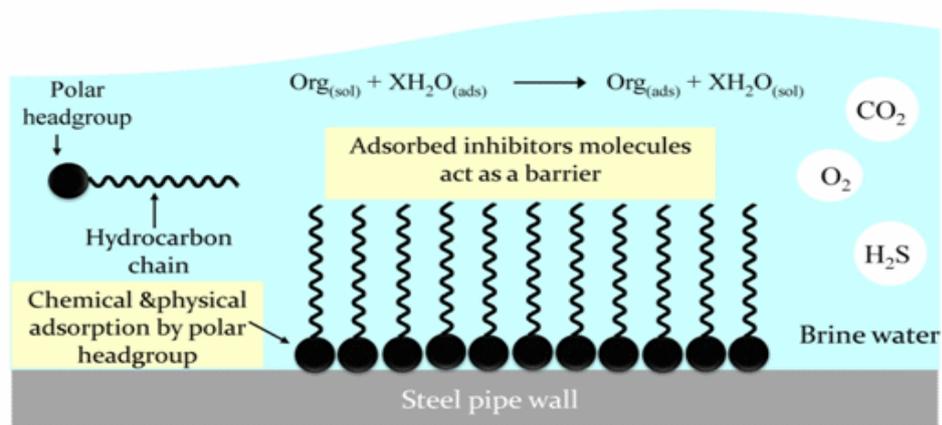


Figure 2.3: Schematic of action of oilfield corrosion inhibitor

At low concentrations, the inhibitor adsorbs parallel or tilted onto the steel surface. As the bulk concentration increases, the hydrophobic tail groups begin to protrude into the aqueous phase to accommodate more surfactant molecules, which increases the surface coverage. At the critical micelle concentration (cmc), monolayer coverage is achieved and the tail groups are parallel to each other and perpendicular to the metal surface [10] which becomes hydrophobic. Therefore, the adsorbed corrosion inhibitor molecules are believed to act as a waterproof barrier between the corrosive aqueous phase and the steel pipe.

An investigation into the inhibition of iron corrosion by a series of imidazoline derivatives by Ramachandran *et al.* [11, 12] suggests a self-assembled monolayer mechanism for corrosion inhibition. The model suggests the following criteria for an efficient corrosion inhibitor:

- I. Adequate solubility and rate of transport of the inhibitor from solution to the surface.
- II. Strong binding of the surfactant headgroups to the metal surface.
- III. Self-assembly of headgroups to form a dense and ordered layer.
- IV. Self-assembly of hydrocarbon tails to form a hydrophobic barrier.

Commercially available oilfield corrosion inhibitors usually contain up to six surface active organic compounds dissolved in a carrier solvent. The carrier solvent can be water or an alcohol or a hydrocarbon. A low freezing point solvent (e.g. ethylene glycol) is required for products used in very cold conditions. Demulsifier species may also be included in order to reduce any impact on water-oil separation in the field. [13]

## **2.6 Factors Affecting the Efficiency of Corrosion inhibitor**

The most important criteria for selection of corrosion inhibitors includes corrosion inhibition efficiency, oil/water partitioning characteristics and emulsion forming tendencies. The effectiveness of the inhibitor is affected by the other operating parameters such as temperature, pH, flow, corrosion inhibitor concentration and exposure time.

## **2.7 Linear Polarization Resistance Test**

LPR technique has been used for measuring the corrosion rate directly in real time. The method generates a plot of current (I) versus potential over a small potential range. The polarizing voltage of 10mV has been chosen to obtain the linear relationship between  $I_{corr}$  and  $\Delta E/\Delta I$ . The value is sufficiently small as to cause no significant or permanent disruption of the corrosion process, so that the measurements would valid for the entire experiments. [14]

This potential perturbation is usually applied step-wise, starting below the free corrosion potential and terminating above the free corrosion potential. The polarization resistance is the ratio of the applied potential and the resulting current response. This "resistance" is inversely related to the uniform corrosion rate.

In order to calculate a corrosion rate with the LPR technique, several fundamental assumptions must be made. [14] These include the corrosion damage in the uniform (general) mode and a particular, relatively simple, kinetic model (known as activation control) for the anodic and cathodic reactions, also a single anodic and a single cathodic reaction. A negligible solution resistance must be assumed hence the technique is most suited to solutions of relatively high conductivity and free corrosion potential is stable.

## **2.8 Summary of Journals**

Table 2.1 - 2.5 list the findings and reviews found from some journals and reports that are useful to the project:

Table 2.1: Journal 1

REF. NO	JOURNAL / PREVIOUS STUDY
[5]	<p><b><i>Author(s) &amp; Title:</i></b>  H. Sun, M. Sujatmiko, M. Aulia, J. Davis, R. Hudgings, "<b>Study of Corrosion and Inhibition of Carbon Steel in CO<sub>2</sub> Containing Natural Gas at High Temperature</b>", Paper No. 11271, NACE International Conference &amp; Expo (2011)</p> <p><b><i>Findings:</i></b>  <u>Objective:</u> The study was initially intended to solve corrosion issue for a field that produced large amount of condensate and wet natural gas at high pressure and temperature. The high gas production rate generates a very high flow gas flow velocity inside the pipelines and results in a very corrosive environment inside the pipelines. The authors have investigated the effective corrosion inhibition plan for the pipelines and the paper reveals the data of effect of temperature on the formation of protective film and its morphology.</p> <p><u>Materials &amp; Method:</u> The testing material used was X-65 carbon steel for <i>Bench Top Autoclave</i> (BTA) and C-1018 for <i>Rotating Cylinder Autoclave</i> (RCA) tests. All specimens were polished to a #600 grit finish and were properly degreased and weighed to 0.1 mg accuracy. The testing solution was synthetic brine and deoxygenated with CO<sub>2</sub> for 30 minutes. The testing conditions were 138°C. CO<sub>2</sub> partial pressure is calculated using 5.2% mole CO<sub>2</sub> and a total pressure of 11MPa. Test duration was 96 hours.</p> <p><u>Results:</u> Corrosion declines significantly when the pH value is increased. Corrosion rates decrease when temperature rises above 80°C. Increased corrosion inhibitor resulted in reduced protection. The more corrosion inhibitor added, the easier the layer could be removed.</p> <p><b><i>Relevancy to my FYP:</i></b>  The purpose and methodology of the experiment from this journal is somewhat similar to my project. The method of preparing the sample can be followed in my project with some modification.</p>

Table 2.2: Journal 2

REF. NO	JOURNAL / PREVIOUS STUDY
[15]	<p><b>Author(s) &amp; Title:</b>            Mari Sparr "<b>Influence of Test Conditions and Test Methods in the Evaluation of Corrosion Inhibitors used in Pipelines – A Review</b>", Paper No. 11267, NACE International Conference &amp; Expo (2011)</p> <hr/> <p><b>Findings:</b>  <u>Objective:</u> This paper provides details about the test conditions that will influence the measured effectiveness of the inhibitor and the effect of operation parameters and laboratory methods.</p> <p><u>Method:</u> Validation of Inhibitor- The proposed laboratory method for evaluation of corrosion inhibitor are;  <i>Static Test:</i> Coupons are exposed in fluids with and without inhibitors and evaluate with weight losses.  <i>Wheel test:</i> Coupons placed in a bottle with field fluids, purged with CO<sub>2</sub> and H<sub>2</sub>S capped. Bottle is agitated first with and without inhibitor before weight loss is measured.  <i>Kettle test or Bubble test:</i> A sealed container filled with a corrosive media that simulate the corrosion conditions and monitored by linear polarization resistance electrode or other monitoring techniques.  <i>Flow Loop Test and Autoclave Test.</i></p> <p><u>Results:</u> Factors that influence the corrosion rate are flow velocity and fluid chemistry, temperature, pressure, erosion and abrasion, water content and water salinity. The inhibiting effects of organic corrosion inhibitors are caused by adsorption and the surface conditions and velocity will influence the performance. Oil/water content, pH, chloride content, wetting conditions and trace amount of copper, nickel, lead, antimony and oxygen affect the inhibitor performance.</p> <hr/> <p><b>Relevancy to my FYP:</b>            Understanding of the suitable test conditions before conducting the experiment is vital towards the accomplishment of my project, as it affects the progress and results of the project. The methodology involved can serve as basic fundamental guidelines to conduct my research project.</p>

Table 2.3: Journal 3

REF. NO	JOURNAL / PREVIOUS STUDY
[16]	<p><b>Author(s) &amp; Title:</b>  Huey J. Chen, Tao Hong and W. Paul Jepson, "<b>High Temperature Corrosion Inhibition Performance of Imidazoline and Amide</b>", Paper No. 00035, NACE International Conference &amp; Expo (2000)</p> <hr/> <p><b>Findings:</b>  <u>Objective:</u> The authors' objective for this paper was to investigate and obtain the baseline performance data at high temperature/pressure for an imidazoline and its precursor amide through the conventional weight loss method. The paper summarizes and compares the inhibition performance of imidazoline and amide at high temperature and high pressure (300°F and 3000psi).   <u>Materials &amp; Method:</u> Two inhibitors used are HJC_13238, an imidazoline and its amide precursor, HJC_A196. To facilitate the performance evaluation these two inhibitors are formulated with acetic acid in isopropyl alcohol and water at 25% active. For high temperature and high pressure testing, inhibitors were tested at higher concentration (100, 400, 1000 ppm) of the formulated sample as opposed to 10, 25 and 100 ppm used in conventional wheel test. The tests done by the testing lab are;  <i>Continuous Wheel Test at 150°F:</i> To evaluate the inhibitor performance simulating continuous treating in the field.  <i>Continuous Treatment High Temperature/High Pressure Wheel Test Calculation of Inhibitor Efficiency and Corrosion Rate.</i>   <u>Result:</u> The blank corrosion rate increases with increased exposure time and corrosion rate calculated decreases with increasing exposure time at both temperatures investigated in the test. The decreasing corrosion rate is due to corrosion product formed on the surface which gives a certain degree of protection, thus, the corrosion rate decreases with time. The blank corrosion rate increases with increasing temperature. Corrosion product formed at high temperature is more protective than that obtained at low temperature.</p> <hr/> <p><b>Relevancy to my FYP:</b>  The objective of the experiment from this journal is similar to my project. This paper gives detailed description and comprehension on the method for testing the corrosion inhibitor.</p>

Table 2.4: Journal 4

REF. NO	JOURNAL / PREVIOUS STUDY
[17]	<p data-bbox="448 389 1399 551"><b>Author(s) &amp; Title:</b> V. Jovancicevic, S. Ramachandran, P. Prince "<b>Inhibition of Carbon Dioxide Corrosion of Mild Steel by Imidazolines and Their Precursors</b>", Journal Paper (ID: 99050449), NACE International (1999)</p> <p data-bbox="448 600 1399 846"><b>Findings:</b> <u>Objective:</u> To carry out systematic investigation of the mechanism of inhibition of mild steel by imidazoline and study the effect of hydrocarbon chain length, thickness of the inhibitor film, and hydrolysis of imidazoline on overall corrosion inhibition under field conditions such as high shear stress</p> <p data-bbox="448 896 1399 1435"><u>Materials &amp; Method:</u> The working electrode was a type 1018 mild steel (UNS G10180) cylinder ([S] = 3 cm<sup>2</sup>). The counter and reference electrodes were two Hastelloy cylinders (S = 10 cm<sup>2</sup>). The use of a Hastelloy C-276† (UNS N10276) reference electrode. The LPR experiments were run in synthetic brine (Prudhoe Bay), pH 6.3. All tests were carried out in a CO<sup>2</sup>-saturated brine (1 atm [100 kPa]) at T = 66 °C (150°F) with a rotation speed of 6,000 rpm (shear stress ~ 45 N/m<sup>2</sup>). Throughout the corrosion tests (4 h-5 h), a magnetic stirrer was used to ensure a uniform inhibitor concentration in brine solution. Inhibitor concentration varied from 3 ppm -50 ppm, depending on the activity of the inhibitors, until the corrosion rate reached a steady-state plateau (1 mpy to 2 mpy). Two different types of inhibitor treatment (addition) were used: constant concentration and variable (slug) concentration treatments.</p> <p data-bbox="448 1485 1399 1731"><u>Results:</u> Activity of imidazoline and amide-based products is similar. The effect of the hydrocarbon chain length of imidazolines on their corrosion inhibition was quite significant. With the hydrophobic chain length &lt; 12, no corrosion inhibition was observed. The pronounced effect of the hydrophobic group on corrosion inhibition of imidazolines could be related to their high admicelles bilayer cohesive energies.</p> <p data-bbox="448 1780 1399 1980"><b>Relevancy to my FYP:</b> Although this journal does not really relate to the project's objective, it does give a detailed understanding about the method of using LPR techniques and handling of experiments in getting the proper result for my project.</p>

Table 2.5: Journal 5

REF. NO	JOURNAL / PREVIOUS STUDY
[18 ]	<p><b><i>Author(s) &amp; Title:</i></b>  A. Crossland, R. Woollam, Jose Vera "<b>Corrosion Inhibitor Efficiency Limits and Key Factors</b>", Paper No. 11062, NACE International Conference &amp; Expo (2011)</p> <hr/> <p><b><i>Findings:</i></b>  <u>Objective:</u> The study was intended to develop a risk based methodology that could be used in new projects to provide guidance on the performance limits of CO<sub>2</sub> corrosion inhibition and the potential risks to achieving the desired levels of inhibitor efficiency at the expected operating conditions.   <u>Materials &amp; Method:</u> A total of 114 test results were considered, from more than 50 different sets of field conditions. The conditions examined cover the ranges:  pp CO<sub>2</sub> : 0.13 – 7.2 bar  Temperature : 15 – 120 °C  WSS 5 (bubble test assumed value) – 320 Pa  pH : 3.8 – 7  TDS : 1,000 – 310,000 mg/L  The majority of the tests were carried out with 5 element weld electrodes, and a large number were carried out in flow loops or with <i>rotating cylinder electrode (RCE)</i> systems, pressurised if necessary. In most cases each test involved a single dose of corrosion inhibitor although some were carried out with sequential inhibitor additions.   <u>Results:</u> From the laboratory data the key parameters in determining the technical possibility of effective inhibition and required concentration are total dissolved solids, temperature, shear stress and partial pressure of carbon dioxide. Of the 114 tests, 69 contained weldment electrodes. The pass/fail split from these for the weld electrodes was 34 passes and 35 fails, the same ratio as observed from the results of the parent electrodes.</p> <hr/> <p><b><i>Relevancy to my FYP:</i></b>  Although this journal does not really relate to the project’s objective, it does give a detailed understanding about corrosion inhibitor efficiency limits and helped in explaining the fundamental of it.</p>

## CHAPTER 3 METHODOLOGY

### 3.1 RESEARCH METHODOLOGY

The overall flow of research methodology and project activities planned for the project is shown in Figure 3.1 below.

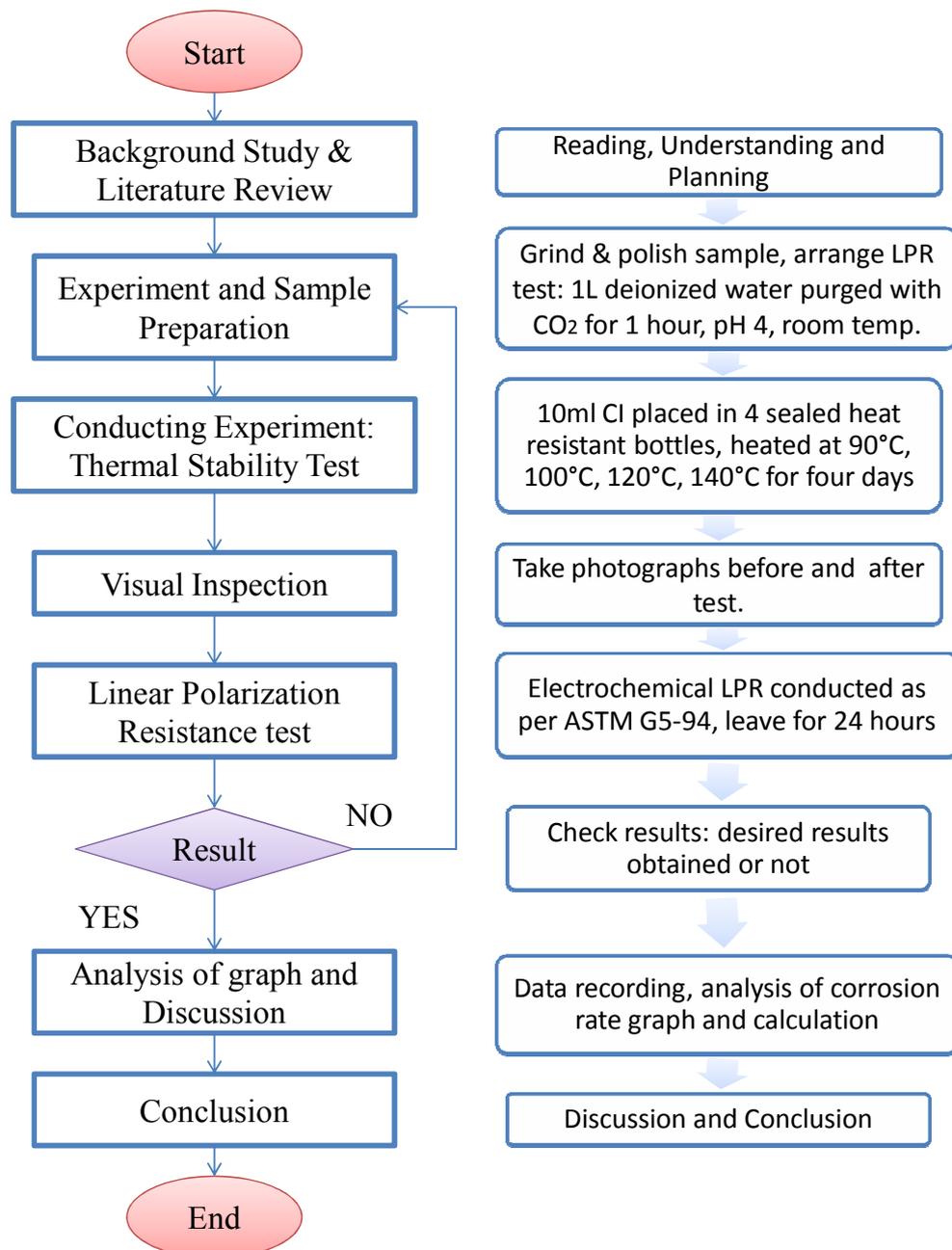


Figure 3.1: Flow chart of the project and the description.

### 3.2 GANTT CHART

Table 3.2 shows the Gantt Chart and key milestone for FYP 2 with expected due date of the project.

Table 3.2: Gantt Chart of the project (FYP 2)

Task / Month	Nov-11				Dec-11				Jan-12			
	6	7	8	9	10	11	12	13	14	15	16	
Background Study & Literature Review	■											
Resources Collection & Analyzing Sample	■	■										
Experiment Preparation - Thermal Stability Test			■									
Conducting Thermal Stability Test			■	■	■							
Submission of Progress Report			■									
Due Date: 18th Nov			▲									
Preparation - Corrosion Test				■	■							
Linear Polarization Resistance Test					■	■						
Draft Report Preparation						■	■					
Due Date: 16th Dec							▲					
Result: Analysis & Calculation							■	■				
Discussion & Conclusion of Project							■	■				
Submission of Dissertation (softbound)								■				
Due Date: 23rd Dec								▲				
Technical Paper Preparation								■				
Due Date: 23rd Dec								▲				
Assessment preparation									■			
Oral Presentation									■			
Final Report Preparation									■	■		
Submission of Project Dissertation (Hardbound)										■	■	
Due Date: 13th Jan											▲	



### 3.3 MATERIALS, EQUIPMENT AND TOOLS REQUIRED

All the materials and tools used for this project are available in the material laboratory at Block 17 and Block I (Corrosion Research Center) in UTP. Table 3.3 shows the materials, equipment and tools required to do the experiment for the project:

Table 3.3: Materials, equipment and tools required

<b>Materials, Equipment &amp; Tools</b>	
 <p>Figure 3.3.1: Temperature Controlled Oven -To heat the corrosion inhibitor and control the temperature</p>	 <p>Figure 3.3.2: Grinding Machine -To grind the sample to get smooth surface</p>
 <p>Figure 3.3.3: Magnetic Stirrer Machine -To stir the deoxygenized water and NaCl until properly dissolved</p>	 <p>Figure 3.3.4: Weight Scale -To measure the weight of NaCl to put into solution</p>



Figure 3.3.5: Potentiostat and Computer  
-To control the three electrode cell and run the experiments



Figure 3.3.6: Micropipette  
-To measure the accurate volume of the corrosion inhibitor into the solution.



Figure 3.3.7: Deionized water and Ethanol



Figure 3.3.8: AMTECH Corrosion Inhibitor

### 3.4 LABORATORY SET-UP

The set-up for the laboratory test using electrochemical measurement method of Linear Polarization Resistance experiments is showed in Figure 3.4 and Figure 3.5. The test assembly consists of one-litre glass cell bubbled with CO<sub>2</sub> gas. The electrochemical measurements are based on a three-electrode system, using a commercially available potentiostat with a computer control system.

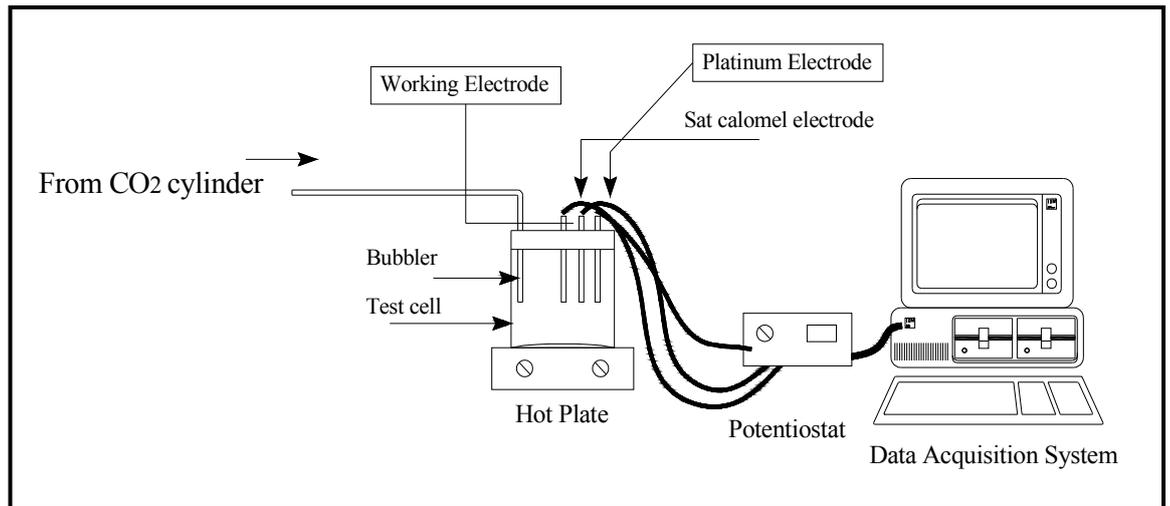


Figure 3.4: Schematic diagram for static experimental set-up

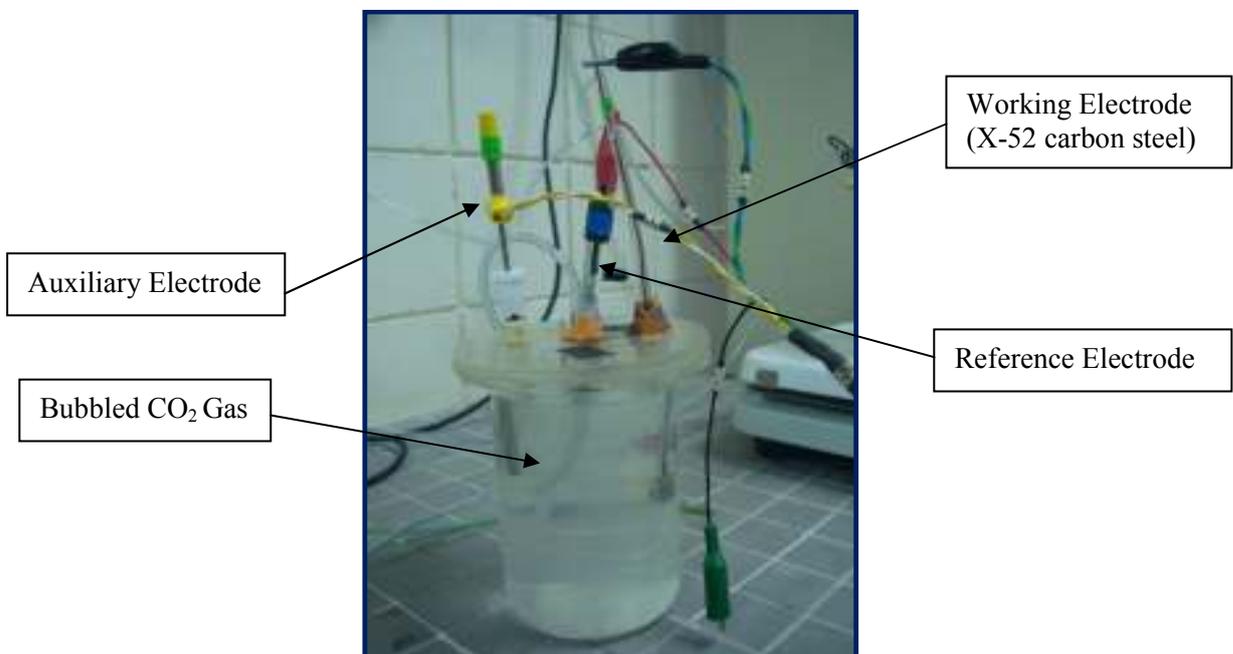


Figure 3.5: Static bubble test using Linear Polarization Resistance test set up in the laboratory.

### 3.4.1 Software Overview

The device used for measuring the LPR is Gill AC from ACM Instruments. The Gill AC is a high specification automated Potentiostat; Galvanostat; Zero Resistance Ammeter and Frequency Response Analyser in one neat enclosure. [19] A Gill 12 is housed in a robust fully screened metallic case with an internally screened mains supply. Internal circuit is a one double-sided board with a minimum of wire links for improved reliability and noise rejection.

At the heart of an ACM system is a Sequencer and Core Running application. Working in unison, Sequencer setups a sequence of techniques as seen in Figure 3.6 and Core Running in Figure 3.7 collects data from a sequence of techniques. The Sequencer is easy to use, with an intuitive interface, one that is common across the range from Data Collection to Analysis which is shown in Figure 3.8.



Figure 3.6: Sequencer - available techniques are displayed to the right, and added to the sequence list on the left.

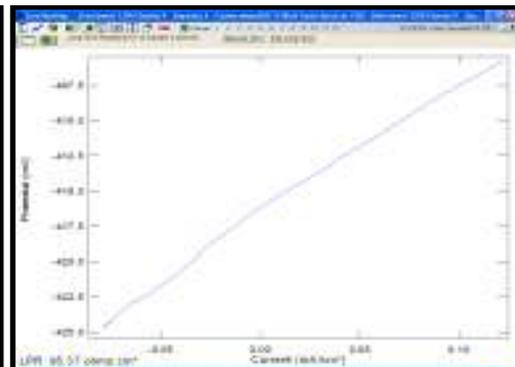


Figure 3.7: Core Running - data collection control.



Figure 3.8: Analysis – display multiple plots on same graph, smooth, delete points, label, zoom, all catered for.

### 3.5 LPR TEST MATRIX

Table 3.5 below shows the table of all LPR experiments according to different temperature and concentration of corrosion inhibitor.

Table 3.5: Test matrix for the laboratory works for Corrosion Inhibitor study

Thermal Stability	LPR Test				
	0 ppm	25 ppm		50 ppm	
Temperature (°C)	CR	CR	efficiency	CR	efficiency
as-received	Exp. 1	Exp. 2		Exp. 3	
90	Exp. 1	Exp. 4		Exp. 5	
100	Exp. 1	Exp. 6		Exp. 7	
120	Exp. 1	Exp. 8		Exp. 9	
140	Exp. 1	Exp. 10		Exp. 11	

### 3.6 EXPERIMENT PROCEDURES

In this study, there are several laboratory tests which have to be conducted by varying the parameters. Table 3.6 below shows the experiment parameters and their values. The procedures of each experiment are nearly the same. Experiment procedures are as per described in this section.

Table 3.6: Experiment parameters

Parameter	Value
Steel Type	X-52 Carbon steel
Solution	3% NaCl
De-oxygenation Gas	CO <sub>2</sub>
pH	4.0
Temperature (°C)	25 or room temperature
Corrosion Inhibitor Type	Ethoxylated Imidazolines by Amtech
Corrosion Inhibitor (ppm)	25, 50
Rotational Velocity (rpm)	0 or stagnant
Measurement Technique	LPR

The experiment is started by performing the thermal stability test first followed by Linear Polarization Resistance test before getting the corrosion rate and efficiency of the corrosion inhibitor.

### **3.6.1 Thermal Stability Test**

The thermal stability test was based on the test procedure used by Corrosion And Protection Centre, Industrial Services Unit (**CAPCIS**). The corrosion inhibitor used in this project was AMTECH 1557 from AMTECH 1500 Series. It is used in wet gas production and transportation and also for gas lift systems to combat against corrosion associated with acid gases and inorganic salts. It comprised of ethoxylated imidazolines which is use to allow the product to be stable in high temperature and pressure system [20]. A summary of the test protocol is shown below.

#### Parameter:

Solution Composition : Amtech Corrosion Inhibitor (Imidazolines based)

Volume : 10 ml per glass bottle

Temperature : 90°C, 100°C, 120°C, 140°C

#### Procedure:

1. 10ml corrosion inhibitor was injected in a sealed heat resistant bottle inside a temperature controlled oven.
2. Temperature was set to (90°C, 100°C, 120°C, 140°C) and left for 96 hours (four days) for each bottle.
3. The bottle was taken out from oven and visual inspection was done.
4. Evaluation and comparison were made to observe the changes.

### 3.6.2 Linear Polarization Resistance Test

Standard LPR techniques were used to measure the instantaneous corrosion rate at the open-circuit potential in a NaCl solution over time based on the **ASTM G5-94**. The chemical composition of X-52 carbon steel (based on **API Spec 5L**) is shown in Table 3.7.

Parameter:

Solution	: Deionized water with 3% wt NaCl
Volume	: 1 L
Temperature	: Room Temperature (25°C)
Pressure	: 1 bar (atmospheric pressure)
pH	: 4.0
Material	: Working electrode – X-52 carbon steel Auxiliary electrode – graphite electrode Reference electrode – Ag/AgCl

Table 3.7: API 5L X-52 chemical composition

Carbon	Manganese	Phosphorus	Sulfur	Others
0.2	1.4	0.025	0.015	c,d

<sup>c</sup>Columbium [niobium], vanadium, titanium, or combinations thereof may be used at the discretion of the manufacturer.

<sup>d</sup>The sum of the columbium [niobium], vanadium, and titanium contents shall not exceed 0.15%

The preparations of the working electrode are as follow:

1. X-52 carbon steel sample was spot welded with copper wire.
2. After that, it was mounted with epoxy by cold mounting and then polished to 600-grade finish using silicon carbide paper.
3. Then it was degreased and rinsed with deionized water and ethanol.

Procedure:

1. 3% NaCl was prepared with 30g of NaCl mixed into 1L of deionized water.
2. Purging of the CO<sub>2</sub> gas started and continuous purging for at least an hour until the CO<sub>2</sub> is saturated in the solution. pH meter was used to indicate pH 4.0 of the solution.
3. The first experiment consists of 3% NaCl at room temperature which is 25°C with natural condition (corrosion inhibitor is not added) and left for 24 hours.
4. The next experiment, 25 ppm of as-received inhibitor was injected and left for 24 hours followed by 50 ppm the next day.
5. Step 4 was repeated with aged 90°C, 100°C, 120°C and 140°C inhibitor.
6. Once the chemicals and electrodes added into the solution, access the data acquisition system, with a computer connected to the ACM Instruments Version 5, run Gill 12 Weld Tester Serial No. 1350 –Sequencer and the Core Running software.
7. Key in all the parameters that set for the measurement of the experiment into the Sequencer software.
8. Run the ACM Instruments and data is gathered automatically into the ACM Analysis Version 4, where they record down the Linear Polarization Resistances and calculate the corrosion rate using the formula that will be discussed in Chapter 4.

## CHAPTER 4

### RESULT AND DISCUSSION

This section will show all the findings gathered from this project and the analysis of the results. The first part will show the result of thermal stability test and in the second part, Linear Polarization Resistance (LPR) test for corrosion rate measurement will be discussed and analyzed.

#### 4.1 THERMAL STABILITY TEST

After the corrosion inhibitor is heated in the oven, visual examination was done for changes in colour, layer formation, and viscosity. Based on visual observation, the inhibitor did not degrade at 90°C and 100°C but started to give sign of apparent deposit and become more viscous at 120°C and 140°C. Higher temperature resulted in darker colour change of inhibitor and increasing the viscosity. Figure 4.1.1 until 4.1.5 show the visual observation of thermal stability test.

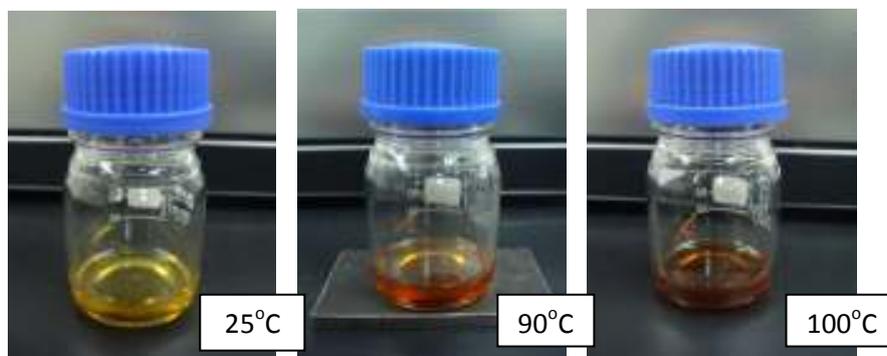


Figure 4.1.1: Before thermal test

Figure 4.1.2: Thermal test at 90°C

Figure 4.1.3: Thermal test at 100°C

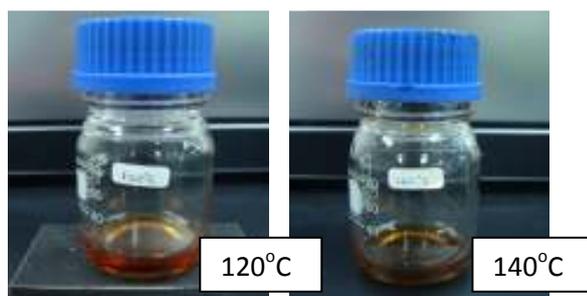


Figure 4.1.4: Thermal test at 120°C

Figure 4.1.5: Thermal test at 140°C

## 4.2 LINEAR POLARIZATION RESISTANCE TEST (LPR)

LPR test or Potentiodynamic Polarization measurements results consist of the result for the system of 3%NaCl with 25°C and pH 4.0 for 0 ppm, 25 ppm and 50 ppm corrosion inhibitor that has been heated at 90°C, 100°C, 120°C and 140°C respectively.

All the experiments had been done in 24hours with one reading had been taken every half an hour. The mean corrosion rate is the average from 24 readings of corrosion rates after immersion of 24 hours. The result for each experiment can be seen from all the figures below.

### 4.2.1 The system of 3%NaCl solution with pH 4.0 at 25°C and corrosion inhibitor at room temperature (25°C).

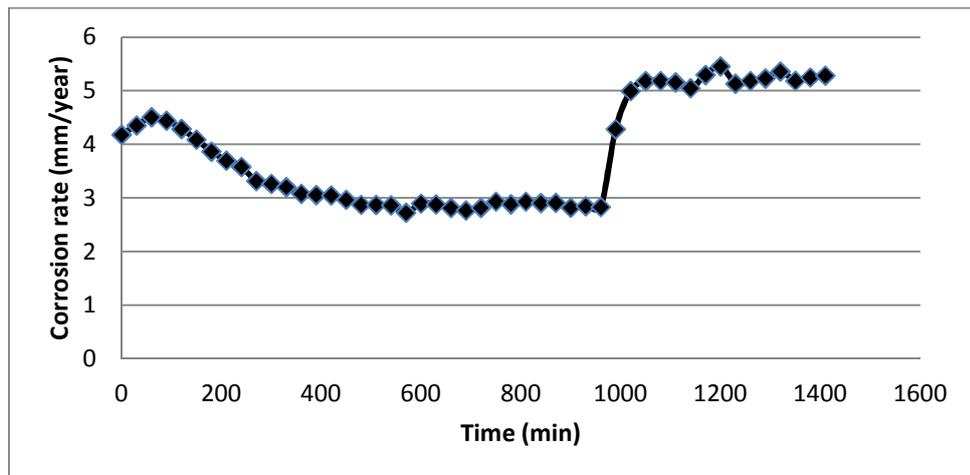


Figure 4.2: The trend of uninhibited corrosion rate for the system of 3%NaCl

From Figure 4.2, the uninhibited mean corrosion rate for 3%NaCl with temperature 25°C and pH 4 is 3.849 mm/year. The graph shows the trend of the corrosion rate which is decreasing and increased suddenly at 1000min. The reduction of the corrosion rate is due to the formation of  $\text{FeCO}_3$  film.

At 25 ppm:

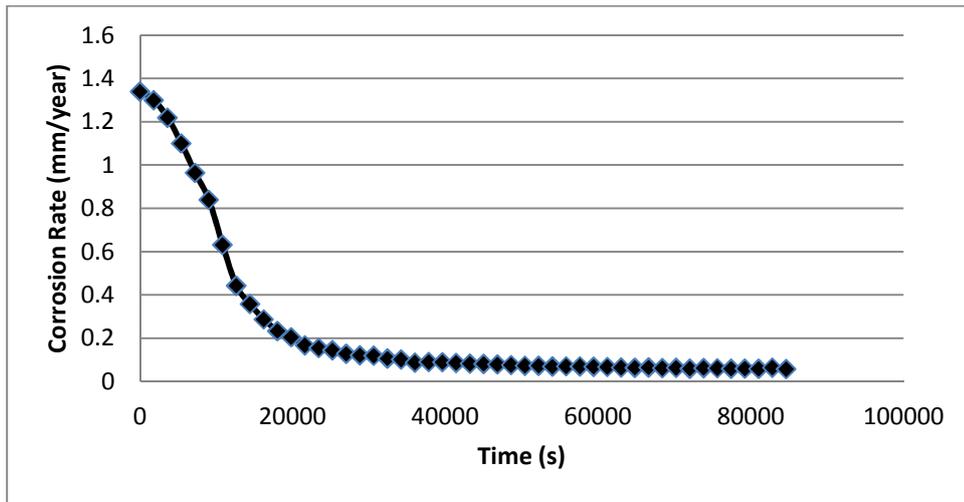


Figure 4.2.1(a): The trend of corrosion rate for 25 ppm corrosion inhibitor heated at 25°C.

At 50 ppm:

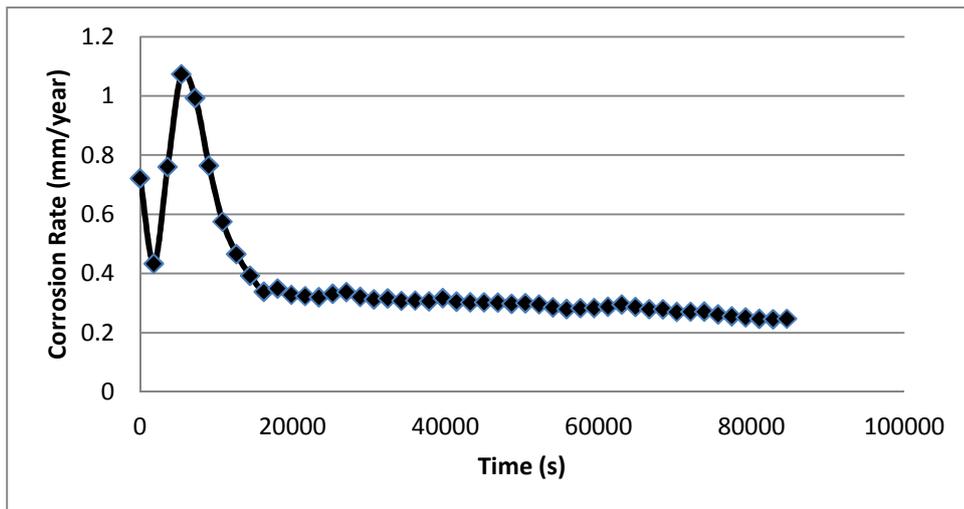


Figure 4.2.1(b): The trend of corrosion rate for 50 ppm corrosion inhibitor heated at 25°C.

From Figure 4.2.1(a) and 4.2.1(b), the mean corrosion rate for 3%NaCl with temperature 25°C and pH 4 at 25 ppm is 0.249 mm/year while at 50 ppm is at 0.369 mm/year.

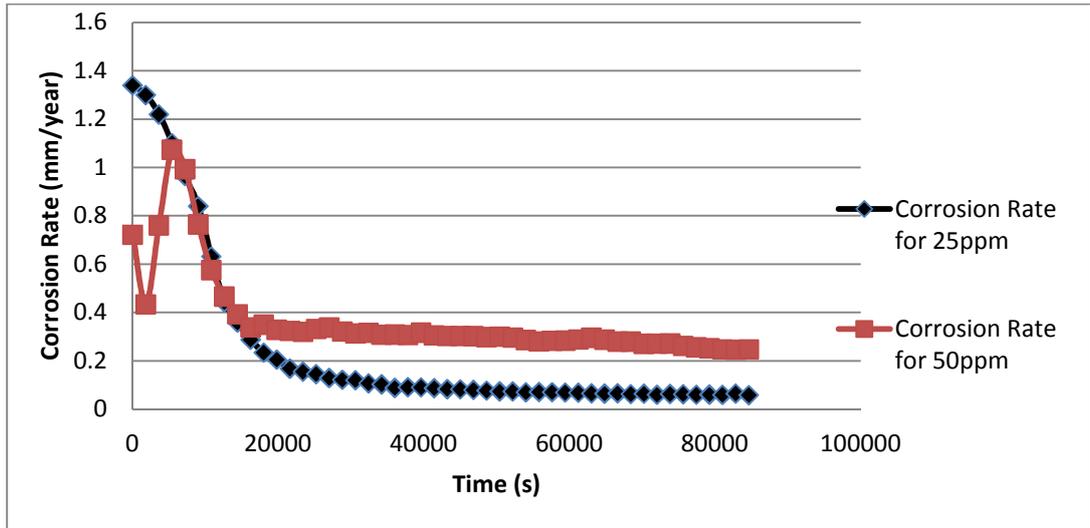


Figure 4.2.1(c): The trend of CR with 25ppm and 50ppm CI versus Time for CI heated at 25°C

From the Figure 4.2.1(c) above, there are two plots of lines which are corrosion rate with 25 ppm and 50 ppm corrosion inhibitor for temperature 25°C which the corrosion inhibitor is not heated. The mean corrosion rate decrease from 0.249 mm/year with efficiency of 93.53% to 0.369 mm/year with efficiency 90.41%. The efficiency of corrosion inhibitor for this system is decreasing. The reduction of the corrosion rate is due to the formation of FeCO<sub>3</sub> film.

**4.2.2 The system of 3%NaCl solution with pH 4 with corrosion inhibitor heated at 90°C.**

At 25ppm:

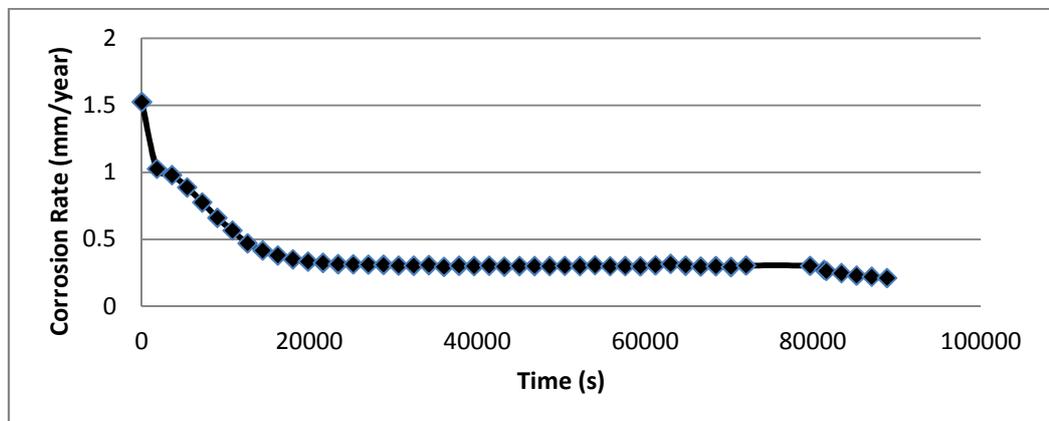


Figure 4.2.2(a): The trend of corrosion rate for 25 ppm corrosion inhibitor heated at 90°C.

At 50 ppm:

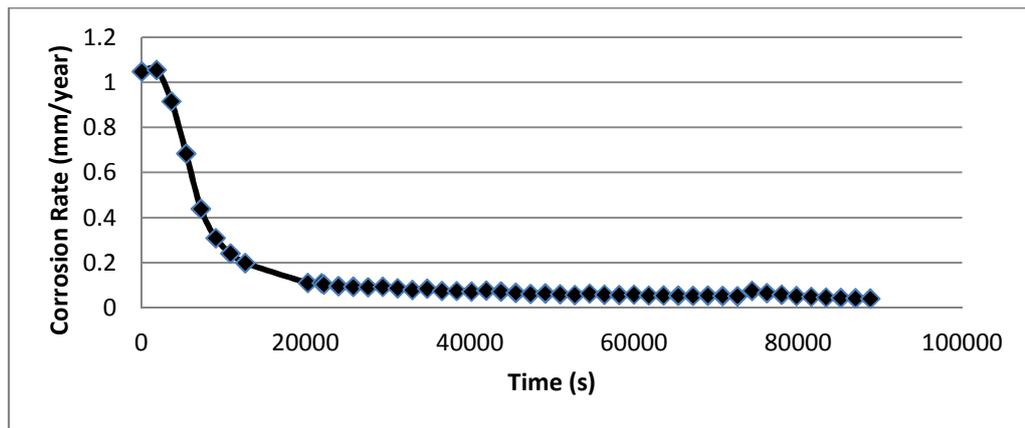


Figure 4.2.2(b): The trend of corrosion rate for 50 ppm corrosion inhibitor heated at 90°C.

From Figure 4.2.2(a) and 4.2.2(b), the mean corrosion rate for 3%NaCl with temperature 25°C and pH 4 at 25 ppm is 0.396 mm/year while at 50 ppm is at 0.159mm/year.

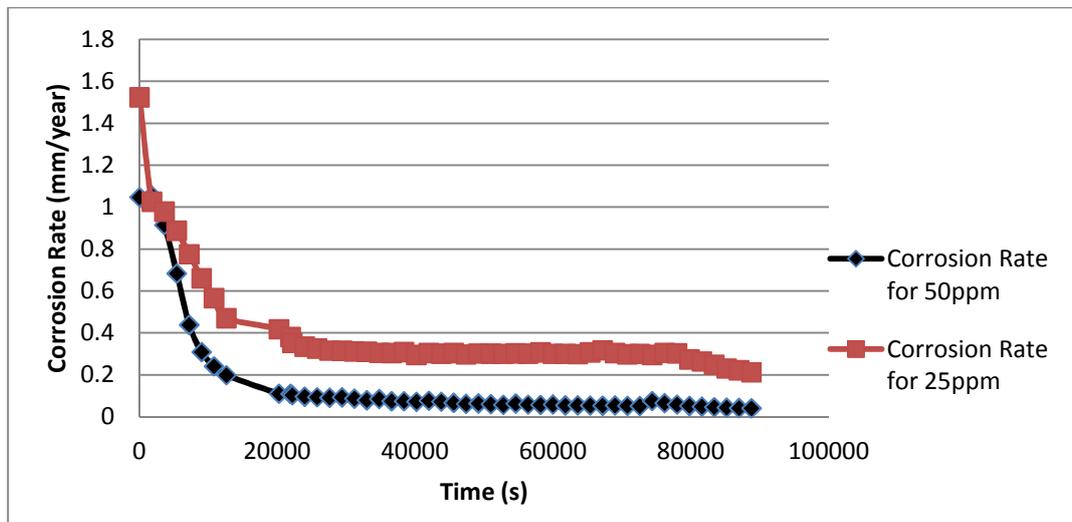


Figure 4.2.2(c): The trend of CR with 25ppm and 50ppm CI versus Time for CI heated at 90°C

From the Figure 4.2.2(c) above, there are two plots of lines which are corrosion rate with 25 ppm and 50 ppm corrosion inhibitor for temperature 90°C. The mean corrosion rate decrease from 0.396 mm/year with efficiency of 89.71% to 0.159 mm/year with efficiency 95.87%. The efficiency of corrosion inhibitor for this system is increasing.

**4.2.3 The system of 3%NaCl solution with pH 4 with corrosion inhibitor heated at 100°C.**

At 25 ppm:

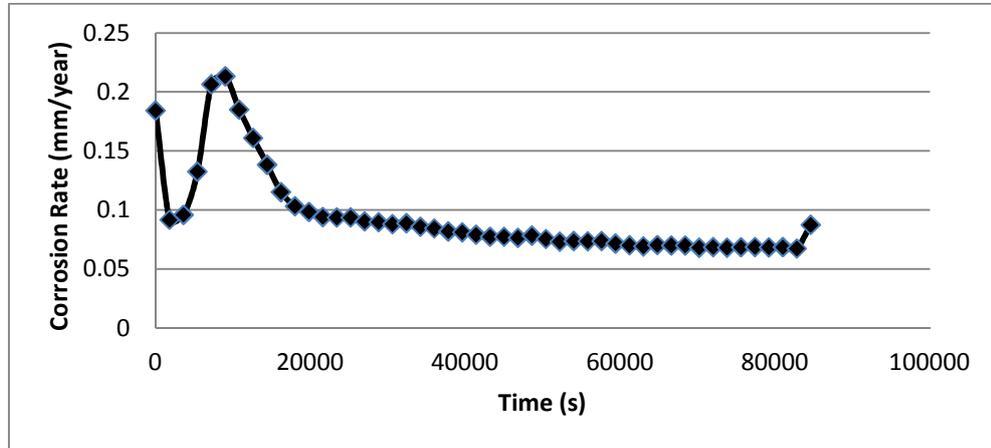


Figure 4.2.3(a): The trend of corrosion rate for 25 ppm corrosion inhibitor heated at 100°C.

At 50 ppm:

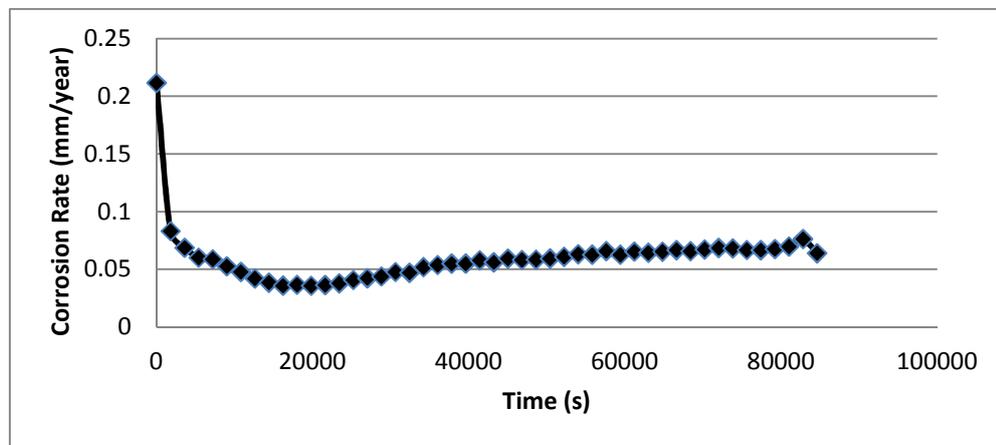


Figure 4.2.3(b): The trend of corrosion rate for 50 ppm corrosion inhibitor heated at 100°C.

From Figure 4.2.3(a) and 4.2.3(b), the mean corrosion rate for 3%NaCl with temperature 25°C and pH 4 at 25 ppm is 0.0941 mm/year while at 50 ppm is at 0.0602 mm/year.

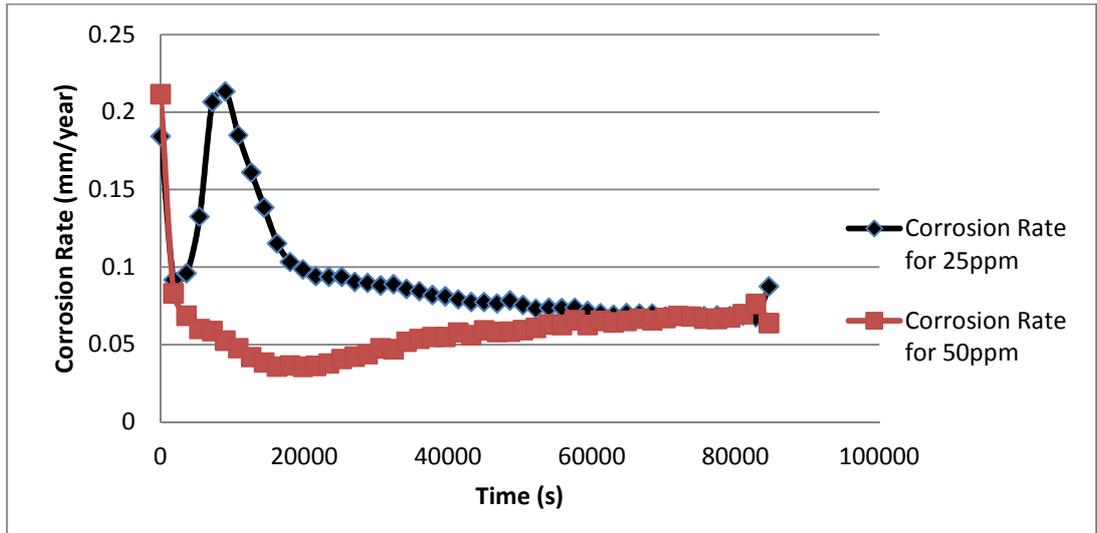


Figure 4.2.3(c): The trend of CR with 25ppm and 50ppm CI versus Time for CI heated at 100°C

From the Figure 4.2.3(c) above, there are two plots of lines which are corrosion rate with 25 ppm and 50 ppm corrosion inhibitor for temperature 100°C. The mean corrosion rate decrease from 0.0941 mm/year to 0.062 mm/year. The efficiency of corrosion inhibitor for this system is 97.55% and 98.43% which is increasing.

**4.2.4 The system of 3%NaCl solution with pH 4 with corrosion inhibitor heated at 120°C.**

At 25 ppm:

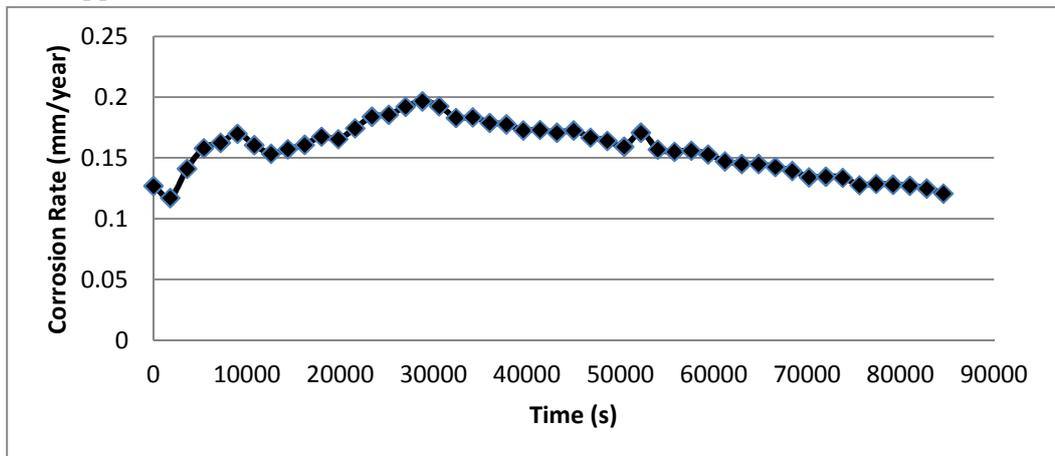


Figure 4.2.4(a): The trend of corrosion rate for 25 ppm corrosion inhibitor heated at 120°C.

At 50 ppm:

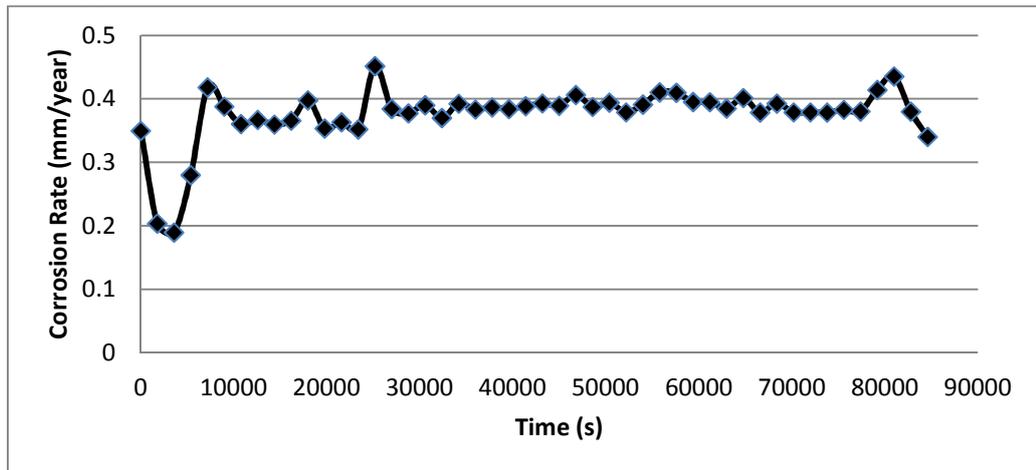


Figure 4.2.4(b).: The trend of corrosion rate for 50 ppm corrosion inhibitor heated at 120°C.

From Figure 4.2.4(a) and 4.2.4(b), the mean corrosion rate for 3%NaCl with temperature 25°C and pH 4 at 25 ppm is 0.157 mm/year while at 50 ppm is at 0.376 mm/year.

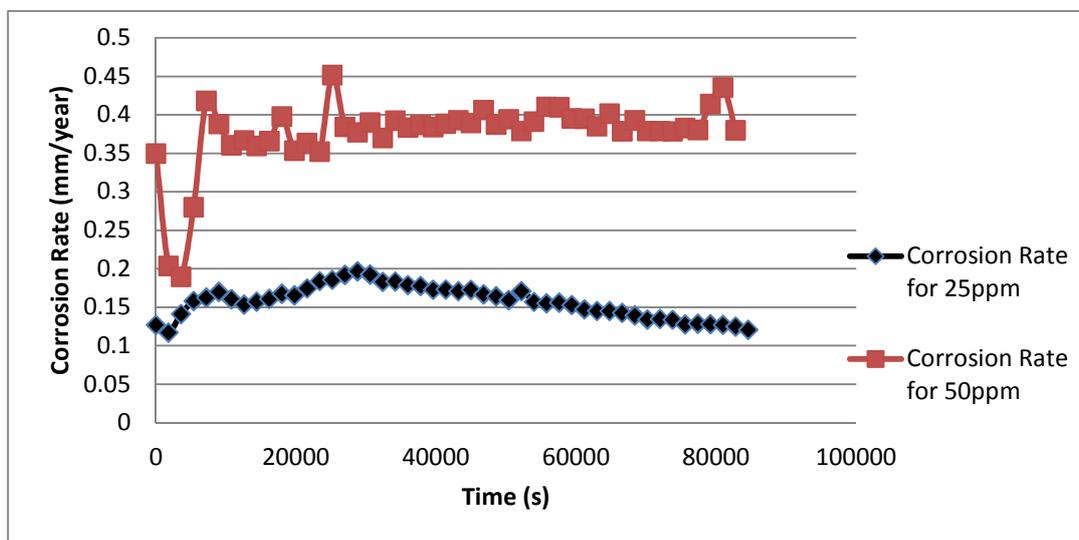


Figure 4.2.4(c): The trend of CR with 25ppm and 50ppm CI versus Time for CI heated at 120°C

From the Figure 4.2.4(c) above, there are two plots of lines which are corrosion rate with 25 ppm and 50 ppm corrosion inhibitor for temperature 120°C. The mean corrosion rate increase from 0.157 mm/year to 0.376 mm/year. The efficiency of corrosion inhibitor for this system is 95.92% and 90.23% which is decreasing.

**4.2.5 The system of 3%NaCl solution with pH 4 with corrosion inhibitor heated at 140°C.**

At 25 ppm:

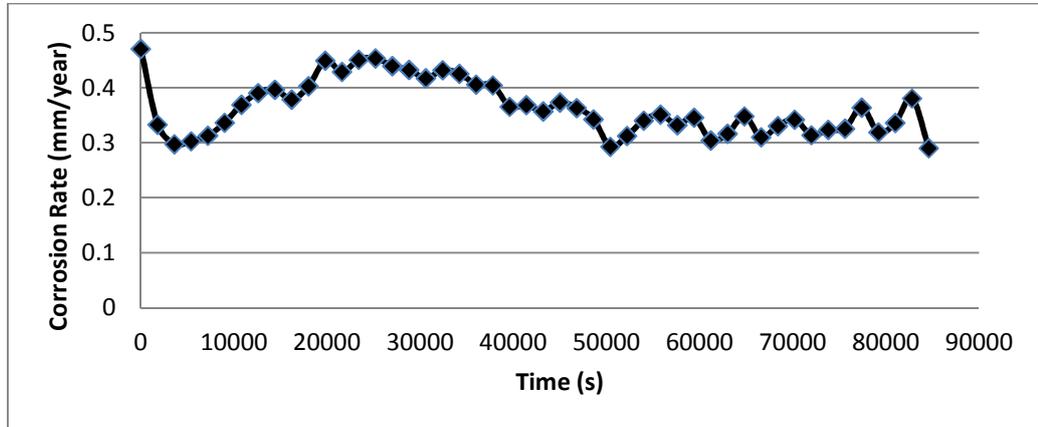


Figure 4.2.5(a): The trend of corrosion rate for 25 ppm corrosion inhibitor heated at 140°C.

At 50 ppm:

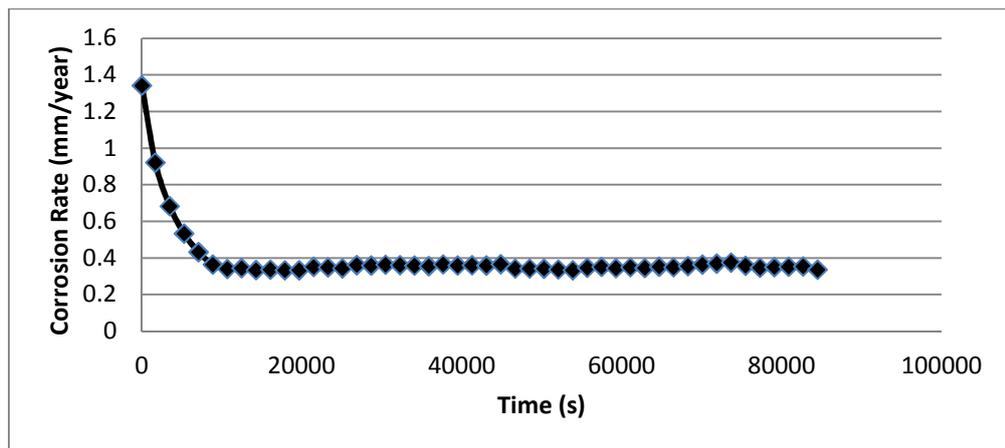


Figure 4.2.5(b): The trend of corrosion rate for 50 ppm corrosion inhibitor heated at 140°C.

From Figure 4.2.5(a) and 4.2.5(b), the mean corrosion rate for 3%NaCl with temperature 25°C and pH 4 at 25 ppm is 0.364 mm/year while at 50 ppm is at 0.395 mm/year.

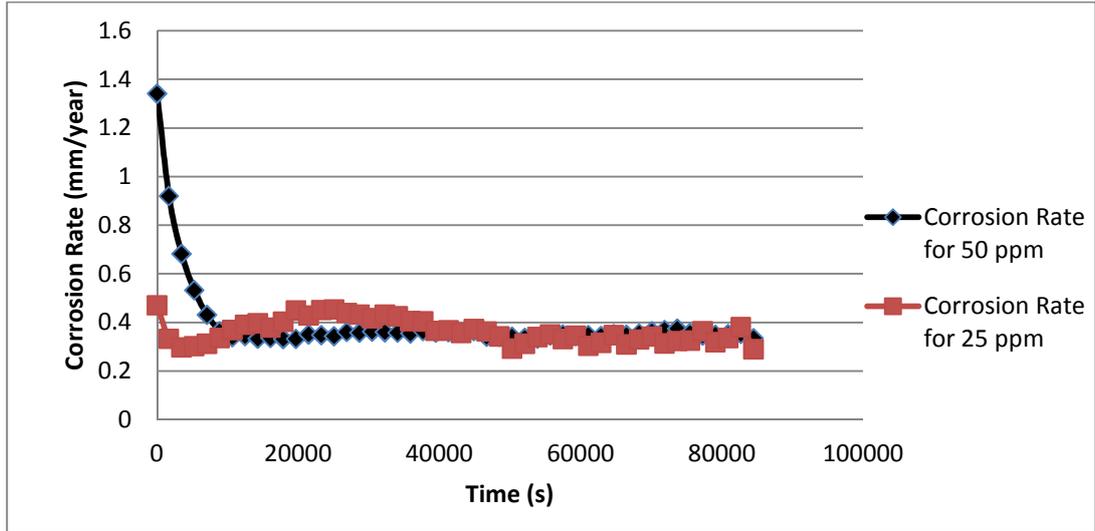


Figure 4.2.5(c): The trend of CR with 25ppm and 50ppm CI versus Time for CI heated at 140°C

From the Figure 4.2.5(c) above, there are two plots of lines which are corrosion rate with 25 ppm and 50 ppm corrosion inhibitor for temperature 140°C. The mean corrosion rate increase from 0.364 mm/year to 0.395 mm/year. The efficiency of corrosion inhibitor for this system is 90.54% and 89.74% which is decreasing.

For each system of 3%NaCl at 25°C with pH 4.0 consist of 25 and 50 ppm of corrosion inhibitor, the highest efficiency is 98.43% and the lowest is 89.71%.

### 4.3 CORROSION RATE MEASUREMENTS

#### 4.3.1 Linear Polarization Resistance

The Linear Polarization Resistance (LPR) technique was used to measure the polarization resistance  $R_p$ , which is used to calculate corrosion rate using following equations:

$$I_{corr} = \frac{b_a b_c}{R_p 2.303(b_a b_c)} \quad (4.1)$$

$$R_p = \frac{B}{i_{corr}} = \frac{(\Delta E)}{(\Delta i)_{\Delta E \rightarrow 0}} \quad (4.2)$$

$$B = \frac{b_a \cdot b_c}{2.3(b_a \cdot b_c)} \quad (4.2)$$

$$\text{Corrosion rate} = I_{\text{corr}} 3272 \text{ EW}/\rho A \quad (4.4)$$

Where:

CR : Corrosion rate (mm/y)

$I_{\text{corr}}$  : Corrosion current (amps)

K : A constant that defines the units for the corrosion rate

EW : The equivalent weight in grams/equivalent

$\rho$  : Metal density (grams/cm<sup>3</sup>)

A : Sample area (cm<sup>2</sup>)

B : Assumed 26 mV/decade

### 4.3.2 Inhibitor efficiency

By definition, a corrosion inhibitor is a chemical substance that, when added in small concentration to an environment, effectively decreases the corrosion rate. The efficiency of that inhibitor is thus expressed by a measure of this improvement:

$$\text{Inhibitor Efficiency (\%)} = \frac{(\text{CR}_{\text{uninhibited}} - \text{CR}_{\text{inhibited}})}{\text{CR}_{\text{uninhibited}}} \times 100 \quad (4.5)$$

where:

$\text{CR}_{\text{uninhibited}}$  = corrosion rate of the uninhibited system

$\text{CR}_{\text{inhibited}}$  = corrosion rate of the inhibited system

The results of the LPR test are shown in Table 4.3 below:

Table 4.3: Corrosion Rate and efficiency of corrosion inhibitor

Thermal Stability	LPR Test				
	0 ppm	25 ppm		50 ppm	
Temperature (°C)	CR	CR	efficiency	CR	efficiency
as-received: 25	3.849	0.249	93.53%	0.369	90.41%
90	3.849	0.396	89.71%	0.159	95.87%
100	3.849	0.0941	97.55%	0.0602	98.43%
120	3.849	0.157	95.92%	0.376	90.23%
140	3.849	0.364	90.54%	0.395	89.74%

At 50 ppm corrosion inhibitor heated at 100°C, the efficiency is the highest which is 98.43%. While at 25 ppm corrosion inhibitor heated at 90°C, the efficiency is the lowest which is 89.71%. Below are the discussion of the parameters that affecting the efficiency of the inhibitor. The manipulated parameters are temperature and concentration of inhibitor while the variable parameters are pH and pressure. All of these parameters give impact to the efficiency of the inhibitor.

#### 4.4 DISCUSSION

From Table 4.3, it shows that the highest inhibitor efficiency is from the solution of 3%NaCl with 50 ppm at 100°C and it explains that corrosion inhibitor can be most efficient at a temperature that is not too high. The lowest inhibitor efficiency is from the solution of 3%NaCl with 25 ppm at 90°C and it maybe because the corrosion inhibitor concentration is not enough to protect the sample in the solution.

Generally, when temperature increase, the corrosion rate would increase as well until it reaches a critical temperature. Researchers have approves that the rate of corrosion can be controlled by either increasing the pH solution or increasing the temperature [21]. This indicates that there is interrelation between these factors that affects CO<sub>2</sub> corrosion. By applying imidazoline at 25ppm and 50ppm, the inhibitor film layer form on the surface of the metal. The protective layer was responsible for reducing the general corrosion rate. The coverage of the crystal increases after testing for several hours and that explains the decreased corrosion rates from the graphs after several hours being immersed in the solution.

Efficiency of corrosion inhibitor is increased when the temperature heated on the inhibitor is increased until 100°C. For the corrosion test after 100°C, increase in temperature will result in the decrease in efficiency. The inhibitor efficiency is decreased because at higher temperature, protective layer which is corrosion product film formation tend to be very active and the existence of this film will protect the metal surface. Thus, the existence of this film will affect the effectiveness of the inhibitor.

Table 4.4 shows the relation between the corrosion potential,  $E_{corr}$  (mV<sub>SCE</sub>) and the current density,  $i_{corr}$  (mA/cm<sup>2</sup>) that can explain the behaviour of different temperature to the result of the efficiency. Increase in temperature of corrosion inhibitor was observed to have little or no effect on the anodic reaction which can be seen from the value of corrosion potential,  $E_{corr}$ . From the Table 4.4, the values for  $E_{corr}$  for both systems are quite the same.

Table 4.4: The result for  $E_{corr}$ ,  $I_{corr}$ , corrosion rate and efficiency for the systems of 3% NaCl at pH 4.

Temp (°C)	pH	Systems	Polarization		Corrosion rate (mm/year)	Efficiency $\eta\%$
			$E_{corr}$ (mV <sub>SCE</sub> )	$I_{corr}$ (mA/cm <sub>2</sub> )		
25	4	3% NaCl (natural condition)	-815.58	0.332	3.849	Uninhibited
25	4	3% NaCl + 25ppm	-647.56	0.022	0.249	93.53%
25	4	3% NaCl + 50ppm	-617.95	0.0032	0.369	90.41%
90	4	3% NaCl + 25ppm	-622.58	0.034	0.396	89.71%
90	4	3% NaCl + 50ppm	-624.83	0.014	0.159	95.87%
100	4	3% NaCl + 25ppm	-627.84	0.008	0.0941	97.55%
100	4	3% NaCl + 50ppm	-614.36	0.005	0.0602	98.43%
120	4	3% NaCl + 25ppm	-606.02	0.0136	0.1571	95.92%
120	4	3% NaCl + 50ppm	-638.09	0.032	0.376	90.23%
140	4	3% NaCl + 25ppm	-620.3	0.031	0.364	90.54%
140	4	3% NaCl + 50ppm	-631.43	0.034	0.395	89.74%

Results confirm that the most effective corrosion inhibitor does not have high temperature performance. An inhibitor product which gives high inhibition performance at a normal treatment concentration of 25 ppm would give a reduced inhibition performance at higher temperature and would not give the protection needed. Consequently, the need for developing inhibitor for high temperature corrosion inhibition is obvious and certain.

#### **4.4.1 Errors and Modifications**

While conducting the experiments, there are some errors that might be occurred and may have affected the accuracy of the results. The pH of the NaCl solution used in the experiments should be maintained at pH 4.0 but during the test, it may have been increased or decreased due to the bubbled CO<sub>2</sub> in the glass cell. To overcome this error, the pH of the solution could be adjusted by injecting an amount of 1M NaHCO<sub>3</sub>.

The corrosion rates obtained from the LPR test using the equipments in the laboratory were not too accurate and precise due to factors like insufficiency of CO<sub>2</sub> gas throughout the experiment, distraction of the electrodes by hands and the connections of the wires to connect the electrodes to the potentiostat. These errors can be encountered by carrying out the experiments under proper care and efficiently by following all the standards appropriately and checking the competency of the tools and equipments beforehand.

## **CHAPTER 5**

### **CONCLUSION AND RECOMMENDATION**

#### **5.1 CONCLUSION**

The efficiency of corrosion inhibitor depends on parameters such as temperature and pH. The efficiency of the AMTECH inhibitor increases with temperature from room temperature 25°C to 100°C and then decreases. At 50 ppm corrosion inhibitor heated at 100°C, the efficiency is the highest which is 98.43%. While at 25 ppm corrosion inhibitor heated at 90°C, the efficiency is the lowest which is 89.71%. This is possibly due to the effect of the corrosion product film formation that governed the protection of metal surface at the higher temperature. Thermal stability of corrosion inhibitor influences the effectiveness of corrosion inhibitor on X-52 carbon steel and the objective of this project has been achieved.

#### **5.2 RECOMMENDATION**

The problem with this current approach is that while inhibitor efficiencies of perhaps more than 98% can be achieved in laboratory testing, long term field monitoring often indicates efficiencies of 90% or less. One of the primary reasons is that in the field there will be periods when corrosion inhibitor is not injected due to pump failures, logistics problems and other issues.

The recommendation is to investigate the formation of corrosion product film on the steel surface at higher temperature of corrosion inhibitor or at high temperature surrounding. More useful tools and equipment can be used such as Fourier Transfer Infrared Spectroscopy (FTIR), Liquid Chromatography-Mass Spectroscopy (LC-MS) and X-Ray Photoelectron Spectroscopy (XPS) for further analyzing the corrosion inhibition and chemistry of the system. Optical Microscope (OM) or Scanning Electron Microscope (SEM) may be used to capture the micrographs of the particles at high magnification and evaluation of the microstructures can be done.

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