Study on Corrosion Inhibitor Efficiency for Carbon Steel in CO$_2$ Corrosion

by

Wan Normimi Roslini Binti Abdullah

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

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Approved by,

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(Ir.Dr.Mokhtar Che Ismail)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

January 2009

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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WAN NORMIMI ROSLINI BINTI ABDULLAH

ABSTRACT

CO_2 corrosion is the main threat in oil and gas industry. In order to reduce the corrosion of carbon steel pipelines in CO_2 environment, inhibitors are added to control corrosion rate to an acceptable level. However, the successful of the corrosion inhibitor injection depends not only on the good inhibitor formulation used but also operational parameters such as temperature, pH and flow conditions. The objective of the project is to investigate the main operational parameters that influence the efficiency of the corrosion inhibitor for carbon steel in CO_2 environment. Temperature and pH are varied from 25^0C to 80^0C and pH 5 and 6, respectively. The corrosion inhibitor used is AMTECH and dosage is set at 500 ppm. The test medium is 3% NaCl solution saturated with carbon dioxide gas at 1 bar. From the experiment, it is found that the efficiency of inhibitor increased when temperature increases from room temperature 25^0C to 80^0C. The highest efficiency of inhibitor is at temperature 80^0C with the value of 97.4% at pH 5. However, at pH 6, the inhibitor efficiency decreased when the temperature is increased. The efficiency of inhibitor for pH 6 at 80^0C is 46.2%. This is possibly due to the effect of the corrosion product film formation that protects the metal surface at pH higher than 5. Therefore, the inhibitor efficiency will decrease for higher pH 6. The recommendations are to do further testing to simulate the turbulence flow condition and to investigate the formation of corrosion product film at higher pH that affects the efficiency of the inhibitor.

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Last but not least, I would like to thank my family's and friends's unconditional support. This FYP is completed successfully, thanks to everyone that was mentioned above. Thank you once again.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

CO_2 corrosion has been one of the most common corrosion problems in oil and gas industry which could result in general corrosion and severe localized corrosion. Carbon steel pipelines are commonly used in the transport of oil and gas. However, carbon steel piping and the process equipment are subject to corrosion caused by the presence of water and acidic gases such as carbon dioxide (CO_2), hydrogen sulfide (H_2S) and/or acetic acid (CH_3COOH). CO_2 corrosion would give rise to the failure of pipelines and equipments and result in great economic loss and catastrophic accidents. Leakage of crude oil due to CO_2 corrosion would induce fire accident, water resource and environmental pollution.

Several possible mitigation methods have been developed to reduce the corrosion rate in such pipelines to acceptable levels. One of the corrosion prevention methods is the corrosion inhibitor and is widely used in various applications. In order to reduce the corrosion of carbon steels in the oil and gas industry, inhibitors are frequently added to the produced fluid to control corrosion.

Many works and researches have been done for last fifty years in order to study the effectiveness of the inhibitor and also the mechanism of the inhibitor. There are also many techniques that have been used to study the effectiveness and mechanism of inhibitor. Many factors contribute to the mechanism and effectiveness of the inhibitor. Particularly, the effectiveness of the corrosion inhibitor is affected by the temperature, pH, flow of the system, exposure time and many more.

1.2 Problem Statement

The problem is to investigate the efficiency of the corrosion inhibitor by measuring the corrosion rate of the inhibited corrosion rate and the uninhibited corrosion rate. The efficiency of corrosion inhibitor maybe reduced in the presence of corrosion product film. The inhibitor efficiency is a function of many factors such as fluid composition, quantity of water and flow regime. Temperature, pH and concentration of corrosion inhibitor also affected the effectiveness of the inhibitor. Some of the mechanism of corrosion inhibitor effect is formation of a passivation layer which is a thin film on the surface of the material that stops access of the corrosive substance to the metal. The other mechanism is inhibiting either the oxidation or reduction part of the redox corrosion and last mechanism is scavenging the dissolved oxygen. However, the exact mechanisms of action for the inhibitors are not fully understood and remain unclear.

1.3 Objectives and Scope of study

The objective is to study the efficiencies of the corrosion inhibitor used in reducing the corrosion rate. The efficiencies can be determined using inhibited corrosion rate and uninhibited corrosion rate.

Several variables such as temperature and pH are varied in order to study the efficiencies of the corrosion inhibitor in the different environment. The different concentration of corrosion inhibitor also applied to see the effect of different concentration to the corrosion rate. The temperatures used for this study are 25^0C and 80^0C meanwhile the pHs are 5.0 and 6.0. The concentrations of corrosion inhibitor used are 0 ppm and 500 ppm.

The inhibitor used is AMTECH and the test medium is 1L 3% NaCl solution saturated with carbon dioxide gas at 1 bar by continuous purging with carbon dioxide. The material used is mild steel EN24.

Different temperature, pH and inhibitor concentration will affect the efficiency of the inhibitor. Therefore, this study is mainly to investigate the effect of the parameters to the efficiency of inhibitor. Also, the study also focuses on the mechanism which probably been applied by the inhibitor to reduce the corrosion rate based on the experimental works and also previous works regarding the corrosion inhibitor by other researchers.

CHAPTER 2

LITERATURE REVIEW

2.1 Carbon Dioxide Corrosion in Oil and Gas Industry

Corrosion of carbon steel is a significant problem in the oil and gas production and transportation systems and causes significant losses. The majority of oil and gas pipelines failures result from CO_2 corrosion of carbon and low alloy steel and occurs at all stages of production from downhole to surface equipment and processing facilities. The impact of corrosion in oil and gas industry will impact the capital expenditure, operational expenditure, health, safety and environment. According to M.B.Kermani [1], the cost of corrosion is 30 cents (USD) for the production of each barrel of oil production. CO_2 corrosion had caused increases in cost and safety issues. According to him also, the mechanism of carbon dioxide corrosion is a complicated process that is influenced by many factors and conditions.

Carbon steels and low alloy steels in the aqueous CO_2 environment could be susceptible to general corrosion and localized attack. When carbon dioxide dissolves in the presence of a water phase, carbonic acid forms, which is very corrosive to carbon steel. Numerous studies have been carried out to investigate the corrosion mechanism of carbon steel immersed in de-ionized water and brine solutions saturated with carbon dioxide. Most of the experiments in stirred beakers and small diameter flow loops.

The overall corrosion process could be divided into four steps. The first step is the dissolution of carbon dioxide in the aqueous solution to form the various reactive species, which takes part in the corrosion reaction. The second step is the transportation of these reactants to the metal surface. The third step involves the electrochemical reactions (anodic and cathodic) taking place at the metal surface. The fourth step is the

transportation of the corrosion products to the bulk of the solution. These can be shown as:-

1) Formation of reactive species in the bulk

$$CO_2 + H_2O \rightarrow H_2CO_3$$

$$H_2CO_3 \rightarrow HCO_3^- + H^+$$

$$HCO_3^- \rightarrow CO_3^{2-} + H^+$$
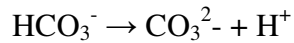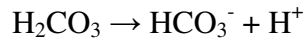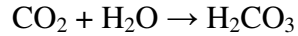
These 2 dissociation steps above are very fast compared to all other processes occurring simultaneously in corrosion of mild steel, thus preserving chemical equilibrium.

2) Transportation of reactants (bulk to surface)

$$H_2CO_3 \text{ (bulk)} \rightarrow H_2CO_3 \text{ (surface)}$$

$$HCO_3^- \text{ (bulk)} \rightarrow HCO_3^- \text{ (surface)}$$

$$H^+ \text{ (bulk)} \rightarrow H^+ \text{ (surface)}$$
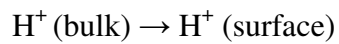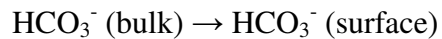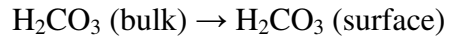
3) Electrochemical reactions at the surface

$$2H_2CO_3 + 2e^- \rightarrow H_2 + 2HCO_3^-$$

$$2HCO_3^- + 2e^- \rightarrow H_2 + 2CO_3^{2-}$$

$$2H^+ + 2e^- \rightarrow H_2$$

$$Fe \rightarrow Fe^{2+} + 2e^-$$
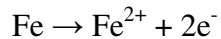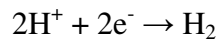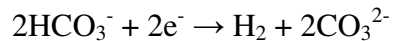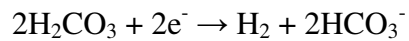
4) Transportation of products (surface to bulk)

$$Fe^{2+} \text{ (surface)} \rightarrow Fe^{2+} \text{ (bulk)}$$

$$CO_3^{2-} \text{ (surface)} \rightarrow CO_3^{2-} \text{ (bulk)}$$
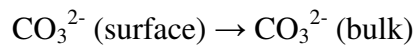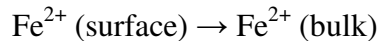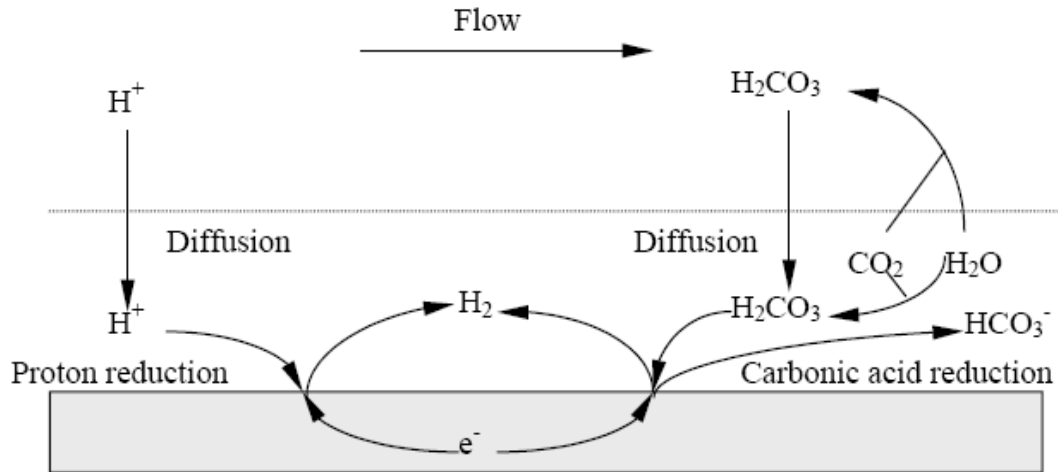
Figure 2.1: Simple model for CO_2 corrosion model [2]

Figure 2.1 above is a simplified model for carbon steel corrosion under multiphase flow conditions. The protons have to diffuse from the bulk region through the boundary layer to the metal surface, while the transport flux of carbonic acid needs to reflect both diffusion of H_2CO_3 and hydration of CO_2 in the boundary layer. The diffusion of hydrogen ions and carbonic acid is the rate-determining step.

In CO_2 corrosion when the concentrations of Fe^{2+} and CO_3^{2-} ions exceed the solubility limit, they combine to form solid iron carbonate layers according to:

$$Fe^{2+} + CO_3^{2-} \rightleftharpoons FeCO_3 \ (s)$$
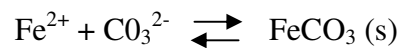
The protectiveness of solid iron carbonate will depend on the rate of precipitation (which is a strong function of temperature and supersaturation) and on the underlying corrosion rate. For high precipitation rates, and low corrosion rates, the protective iron carbonate is obtained and vice versa, low precipitation rates and high corrosion rates lead to formation of unprotective iron carbonate layers.

As a conclusion, when CO_2 dissolves into water, carbonic acid will form, which is more corrosive to carbon steel than a completely dissociated acid (such as HCl) at the same pH value. Several mechanisms have been proposed for the dissolution of carbon steel in

CO_2 containing aqueous solution. The main cathodic process can be summarized by four reactions. At a lower pH, H^+ reduction is the dominant cathodic process because of the high concentration of H^+.

$$2H^+ + 2e^- \rightarrow H_2$$
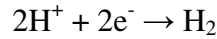
When pH is increased to 4-6, the direct reduction of $HCO3^-$ and H_2CO_3 become important.

$$2H_2CO_3 + 2e^- \rightarrow H_2 + 2HCO_3^-$$

$$2HCO_3^- + 2e^- \rightarrow H_2 + 2CO_3^{2-}$$
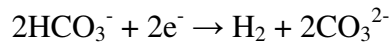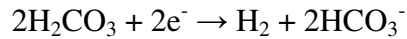
At a high overpotential, the dominant cathodic reaction changes to direct reduction water:-

$$2H_2O + 2e \longrightarrow 2OH^- + H_2$$
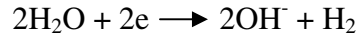
The anodic reaction is mainly the dissolution of iron. During these corrosion processes, a corrosion scale ($FeCO_3$) would form on the surface of the carbon steels. The properties and morphology of the scales would influence the corrosion rate significantly.

2.2 Corrosion Inhibitor

In the oil and gas exploration or production and also processing industries, carbon steel represents the commonly used construction material for pipelines. However, they are very susceptible to corrosion in environments containing CO_2. In order to improve their performance, corrosion inhibitors are frequently used. Specifically, a corrosion inhibitor is a chemical compound which is added to the fluid phase so that it has an effect on the metal surface. The corrosion inhibitor treatment program is often the most cost-effective option to ensure the integrity of the system over the lifetime of the asset [3]. Amine and its salts have been used successfully in the oil and gas field application.

The most widely used inhibitors in the petroleum industry are nitrogen containing compounds such as amines, amides, quaternary ammonium salts and specially imidazolines and their derivatives [4]. Corrosion inhibitor falls below three categories which are anodic inhibitor, cathodic inhibitor and mixed inhibitor. Chromate is an anodic inhibitor which forms a passivation layer on aluminium and steel surfaces which prevents the oxidation of the metals. Nitrite is another anodic inhibitor which used at low concentration can actually aggravate pitting corrosion as they form a nonuniform layer with local anodes. Example of cathodic inhibitors is Zinc Chloride, which retards the corrosion by inhibiting the reduction of water to hydrogen gas and if oxidants such as oxygen are excluded, the rate of the corrosion can be controlled by the rate of water reduction. Mixed inhibitors are the inhibitors act in a combination of anodic inhibitors and cathodic inhibitors manner. The imidazoline derivative acts as a mixed-type inhibitor from the indication of decreasing in corrosion rate associating with a shift of both cathodic and anodic branches of polarization curves towards lower current densities, together with a slight positive shift in corrosion potential [5].

Selection of corrosion inhibitor in the earlier time was based on a trail and error basis. The most significant criteria involved in the selection of the inhibitors are hydrophobicity, molecular structure, and electron density at the donor atom of the inhibitor and solubility or dispersibility of the inhibitor. One of the examples of selection method for corrosion inhibitor is BP corrosion inhibitor selection study. The BP corrosion inhibitor selection study is as follows: solubility /dispersibility screening, bubble test screening, rotating screening if there are still a large number of candidates for corrosion inhibitor and flow loop screening. The summary for the processes can be seen from Figure 2.2 [6].
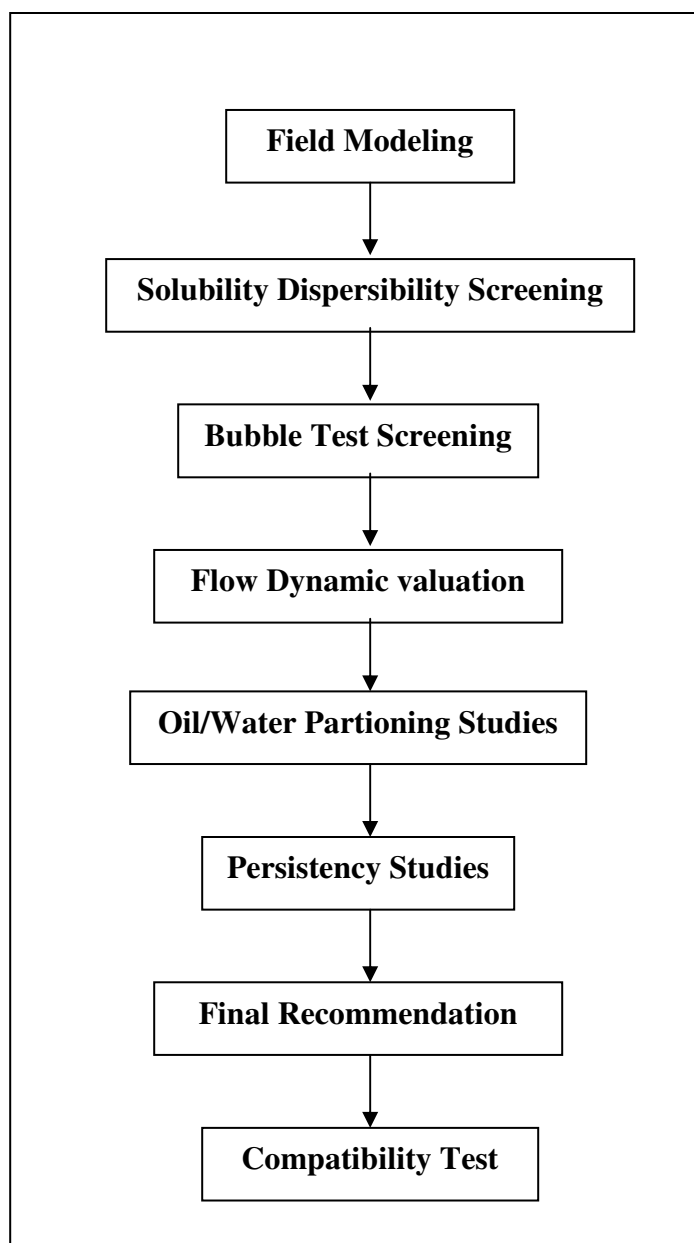
Figure2.2: Flow Chart of corrosion inhibitor selection process. [6]

Several mechanisms have been proposed for the dissolution of iron in aqueous, deareated CO_2 solutions. The main corrosion process can be summarized by three cathodic reactions (**2a**, **2b** and **2c**) and one anodic reaction (**3**). At pH 6, the main process occurring in the cathode is the reduction of H_2CO_3 and HCO_3^-.

$$2H_2CO_3 + 2e^- \rightarrow H_2 + 2HCO_3^- \quad \textbf{(2a)}$$

$$2HCO_3^- + 2e^- \rightarrow H_2 + 2CO_3^{2-} \quad \textbf{(2b)}$$

$$2H^+ + 2e^- \rightarrow H_2 \quad \textbf{(2c)}$$

$$Fe \rightarrow Fe^{2+} + 2e^- \quad \textbf{(3)}$$
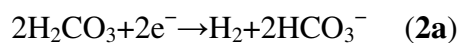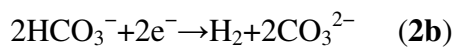
Due to these processes, a corrosion layer is formed on the steel surface. The properties of these layers and their influence on the corrosion rate are important factors to take into the consideration when studying the corrosion of steels in CO_2 aqueous solutions. The composition of corrosion product layers varies with the inhibitor concentrations and the thickness of corrosion product layer decreased due to the addition of inhibitor.

EIS results, showed that imidazoline is a very effective CO_2 corrosion inhibitor which forms a chemically bonded film on the metal surface which has a multi-layered structure which is a combination of an inner layer and many outer layers of inhibitor molecules. It is suggested that the corrosion inhibitor performance is dependent on exposure time besides other parameters such as temperature and pH. The corrosion inhibitor has a good performance of corrosion protection by forming more compact inhibitor film on the metal surface at longer exposure time [7].

Experimental results also show that, the low corrosion resistance can be resulted from high turbulence flow and the inhibitor film is damaged and washed away from the metal surface due to this flow. This will lead to the low corrosion inhibitor effectiveness because the corroding surface metals are not protected by the molecules of inhibitor.

Studies are also carried out using the ASTM substitute saltwater and carbon dioxide gas. The system temperature and pressure are maintained constant at 40°C and 0.136 MPa for all experiments. The pH value of the saltwater solution is about 5.6 and the conductivity of saltwater is around 0.046 Ω^{-1} cm^{-1}. The inhibitor used in this work is an imidazoline based inhibitor formulated with the commercial grade imidazoline and dimer–trimer acid. The inhibitor concentration is 100 ppm. The inhibitor is injected into

the flow loop system and fully mixed with the test solution before the EIS probe is installed into the system.

Nitrogen-based organic surfactants, such as imidazolines or their salts have been successfully as corrosion inhibitor of protecting the severe internal corrosion of carbon steel pipelines in the oil and gas industries because of their effectiveness and availability [8 & 11]. Since imidazoline-based inhibitors have excellent inhibition ability in the acidic media, they are widely used to protect oil well, gas well and pipelines from CO_2 corrosion in the oil and gas industry.

2.3 Laboratory works related to Corrosion Inhibitors for the Measurements of Efficiencies and Mechanisms of Corrosion Inhibitors

Extensive basic studies on corrosion inhibitor and the factors governing their effectiveness have only been in progress for the last fifty years. The effectiveness of an inhibitor is determined not only by the properties of the gas and liquids contents of the pipeline and by the properties of the inhibitor itself, but also the way it is added to the pipeline and the operating conditions of the system such as temperature, flow rate and pressure [9]. The effectiveness of the inhibitor is affected by the other operating parameters such as temperature, pH, flow, corrosion inhibitor concentration, exposure time, and many more factors.

Modern instrumental techniques such as X-ray photoelectron spectroscopy (XPS), Auger electron spectroscopy (AES) and secondary ion mass spectroscopy (SIMS) coupled with electrochemical techniques which measure the polarization curves, polarization resistance, electrochemical noise and electrochemical impedance have been proven to be of dominant importance in the explanation of corrosion inhibition mechanisms.

EIS, Electrochemical Impedance Spectroscopy, is a powerful technique to study about the corrosion processes and inhibitor performance in different environments. EIS also is a powerful technique for the corrosion study in various corrosion and protection fields

such as organic coatings, passive films and corrosion scales analysis. It can provide the information on corrosion and protection mechanism, especially when an adsorbed film or an applied organic coating is present. EIS had been widely applied in the monitoring of inhibitor film persistency and in the study of inhibitive mechanisms of inhibitors. EIS data were used to calculate corrosion related electrochemical parameters and was shown to be a very useful tool for studying corrosion inhibitor mechanisms.

In the present work, both EIS and some standard direct current measurements which are LRP and E_{corr} were employed to study the corrosion process in carbon steel with two different microstructures such as annealed and quenched and tempered, also known as Q&T, as well as the effect of the heat treatment on the efficiency of benzimidazole as a corrosion inhibitor in CO_2 saturated brine media.

Corrosion tests to investigate the effectiveness and mechanism corrosion inhibitor should be reproducible and reliable. Corrosion tests may be classified as the simulated laboratory tests and field/plant tests. Laboratory tests may be either long-term or accelerated short-term tests. Long-term laboratory tests involve typical model apparatus using simulated field or plant conditions. These tests are usually use for the selection of materials. In accelerated short-term tests, one or several factors affecting the corrosion rate are made severe to speed up the corrosion process. This type of test is done in controlled conditions and is useful in quality control of materials or protective coatings.

In earlier studies, inhibitors were tested by agitation of the samples in inhibitor containing solutions and the effectiveness of the inhibitors was determined by the loss in weight of the samples [10]. A paper referenced in *Chemical Abstracts* in 1909 states that the inhibitive power of some pigments on iron and steel were tested by agitating in water with a current of air and the loss in weight due to the rusting was determined.

Most of the studies on the inhibition mechanism of imidazoline based inhibitors have been conducted in laboratory scale systems, such as rotating cylinder electrode cell or the laboratory scale flow loop, under a water or a water-oil phase [7]. Under stagnant

conditions, copper wire was attached to the back of the specimen, which was mounted in an epoxy resin leaving an area of $1cm^2$ exposed to the solution. The five holes distributed at the cover of the container for CO_2 gas entry, working electrode, reference electrode (saturated Ag/AgCl), counter electrode (graphite) and condesator. All experiments under flowing condensation were conducted in the modified rotating disc electrode system. It is well known that the corrosion rate in pipelines is strongly related to flow condition [7].

Hausler [9] defined three criteria by which a laboratory methodology can be judged relative to the information that it provides:

- **Uniqueness** (Corrosion rate must be obtained and interpreted in terms of corrosion kinetics).
- **Relevancy** to the field for which the inhibitor is being evaluated; and
- **Predictive** capability of failure mechanism

Liu Et al, [9] developed relationship between rotating disk, pipe flow and corrosion by considering equality in corrosion rate as the basis criterion when corrosion occurs without forming a product film and the equal wall shear stress as the basic criterion when a corrosion product films form on the surface. Meanwhile, Chen Et al, [9] concluded that the corrosion rate measured in all hydrodynamics system is independent of the geometry involved. Denpo and Ogama, [9] compared the corrosion rate of steel pipe with different RCE (Rotating Cylinder Electrode) speeds. The pipe loop experiments were carried out for 96h and corrosion rate was determined by weight loss; The RCE experiments were carried out for 60minutes and corrosion rate was determined by electrochemistry. As the conclusion, the corrosion rate of the rotating electrode obtained chemically was used to predict the corrosion rate of the pipe at equivalent velocity. Therefore, the predicted corrosion rate was in agreement with the measured corrosion rate.

Abayarathna et al, [9] studied the performance of corrosion inhibitor in the laboratory using RCE, rotating cylinder in autoclave (RCA), and flow loop, and in the field using

weight loss and electrical resistance probes. During the RCE tests, the chemicals were tested at low concentration since the conditions being stimulated were relatively mild. Meanwhile, Dawson et al, [9] obtained identical results from the rotating cylinder electrode and from the jet impingement from the same wall shear stress. Based on the results, they concluded that the shear stress can be used as a fundamental test parameter for inhibitor evaluation under turbulent flow conditions.

The other experimental work regarding the corrosion inhibitor is the inhibition and adsorption of 2-unde-cyl-ethylamino imidazoline (2UEI) in CO_2 saturated 3% NaCl solution was investigated using potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) as well as SEM observation [9]. From the result and discussion, for the *potentiodynamic polarization measurements*, 2-Undecyl-1-ethylamino imidazoline (2UEI) inhibits the corrosion of N80 mild steel in CO_2-saturated 3% NaCl solution and the extent inhibition is dependent on 2UEI concentration, temperature and exposure time. 2UEI mode of inhibition is due to the active sites blocking effect in the absence of corrosion products and geometric blocking effect in the presence of corrosion products.

The corrosion behavior of API X65 steel exposed to CO_2 saturated 5% NaCl solution without or with different concentration of inhibitor were studied by potentiodynamic polarization at ambient temperature. The result of the *potentiodynamic polarization* indicate that this imidazoline derivative, as a mixed-type inhibitor, inhibits both cathodic and anodic processes, together with a slight positive shift in corrosion potential (*Ecorr*). The adsorbed inhibitor on the surface of electrode affects the kinetic process of anodic and cathodic reactions and increases the reaction activation energy.

Factors that make the laboratory evaluation of corrosion inhibitor for application in oil and fields difficult, include the large number of laboratory methodologies that are available, the several correlations that can be used to convert corrosion rate and hence the inhibitor efficiency from one geometry to another, the vast variation of field operating conditions and the impossibility of reproducing in the laboratory all field

operating conditions. Therefore, Uniform International standards should be developed by organizations such as NACE, ASTM and ISO in tandem. The development and usage of such standards will benefit all those involved as a result of increased effectiveness of CI, lower cost, fewer field failures and also increased safety.

2.4 Protocol to test corrosion inhibitor in Laboratory

According to the A.J.Mohan [6], written in his papers, "Round Robin" Validation of Test Methods and Bubble Test Protocol, a set of standard operating procedures are important to establish confidence in the repeatability and reproducibility of test methods. The standard procedures should cover all aspects of the corrosion test from steel quality, specimen preparation, solution preparation, environmental conditions, flow regimes, through to corrosion monitoring method.

"Round Robin" Validation of test methods
In BP Round Robin protocol consists of uninhibited and inhibited test under the stimulation condition. Equivalent of the hydrodynamic conditions are used in each type of apparatus. A J McMahon stated that in order to produce solution, the quantities of salt cannot be added straight into the distilled water of 1 liter because it will lead greater volume of water more than 1 liter. To prevent scaling and precipitation, chloride have to dissolve first and follow by dissolution of the carbon dioxide and finally bicarbonate.

Standard steel is important because high sulfur content of carbon steel thus S element will act as corrosion inhibitor and affects corrosion rate. The active surface preparation as stated in this protocol. Cleanliness of the equipment also is important to obtain reliable data. The recommended cleaning after inhibitor are deionsed water rinse, toluene rinse, petroleum ether rinse, acetone rinse and deionised water rinse at least 5 times.

Corrosion measurement in the testing of corrosion inhibitor can use weight loss measurements and Linear Polarization Resistance (LPR) to monitor the corrosion rates.

In LPR, the working electrode in three electrode system is wept from 0-10mV at 300 MV/min. The polarization resistance is converted to the corrosion rate using Stern-Geary constant of 27.3mV.

Bubble Test Protocol

"Bubble test" is a simple test which can be set up reasonably quickly and is ideal for rapidly carrying out a large number of tests. This test is also conducted in the first stage of corrosion inhibitor selection, or for screening a wide range of field conditions. The main limitation of the bubble test is shear stresses in the stirred solution are significantly lower than experienced in the pipeline. The operating procedure for bubble test is very crucial during the cleaning of the cell or called vessel.

Standard Test method for conducting Potentiodynamic Polarization Resistance Measurement

Polarization resistance measurement is used to determine the corrosion rate of metal in a specific environment. ASTM 59 described the experimental procedure for polarization resistance measurements which can be used for calibration of equipment and verification of experimental technique.

The test method can be utilized to verify the performance of polarization measurements equipments. Polarization resistance can be related to the rate of general corrosion for metals at or near their corrosion potential, it is an accurate and rapid way to measure the general corrosion rate. This method also can be used as a way to rank inhibitor in the order of resistance to general corrosion.

The test procedures standard included are:-
- Test solution should be prepared and the standard test cell requires 900ml of test solution where the temperature must be maintained at 30^0C within 1 celcius.

- Test cell must purge at 150cm^3 /min with an oxygen free gas. The purge is started at least 30 minutes before the specimen immersion and continue through out the test.

- Working electrode is prepared, and experiment must be conducted within 1 hour of the preparing electrode. Preparation including sequential wet polishing with 240 grit and 600 grit SiC paper. Surface area of the specimen is determined to the nearest of 0.01 cm^2 and subtract the area under the gasket.

- Prior to the immersion of the specimen, it is degreased with a solvent such as acetone and rinsed with distilled water. The time delay between rinsing and immersion should minimal.

- The test specimen is transferred into the test cell and position the Luggin probe tip to 2 to 3 mm from the test electrode surface. The diameter of the tip must be not more than 1 mm.

CHAPTER 3

METHODOLOGY

3.1 Laboratory Simulation Test

Laboratory simulation test is conducted to determine the effect of corrosion inhibitor to the corrosion rate of carbon steel. In this laboratory test corrosion inhibitor dosage used is 500 ppm that affects the corrosion rate. Concentration of corrosion inhibitor will be injected into the testing environment solution and the corrosion rate of each test will be recorded down to determine the effect of the corrosion inhibitor dosage on the corrosion rate. The variables such as temperature and pH besides concentration of corrosion inhibitor will be varied for each different laboratory tests. The exposure time also important for this study to determine the mechanism which inhibitor been applied. Methodology of the experiment is as describe in the flow chart shown in the Figure 3.1.

```
           ┌─────────────────────────────────────┐
           │   Preparation of Working Electrode   │
           └─────────────────────────────────────┘
                            │
                            ▼
           ┌─────────────────────────────────────┐
           │     Preparation of Sodium Chloride   │
           │                Solution              │
           └─────────────────────────────────────┘
                            │
                            ▼
   ┌──────────────────────────────────────────────────────┐
   │      Linear Polarization Resistance Test Procedure     │
   └──────────────────────────────────────────────────────┘
```

Temperature	Inhibitor with different dosage	pH

- 25°C
- 80°C

- 0 ppm
- 500 ppm

- 5.0
- 6.0

Analysis of Efficiency of CI
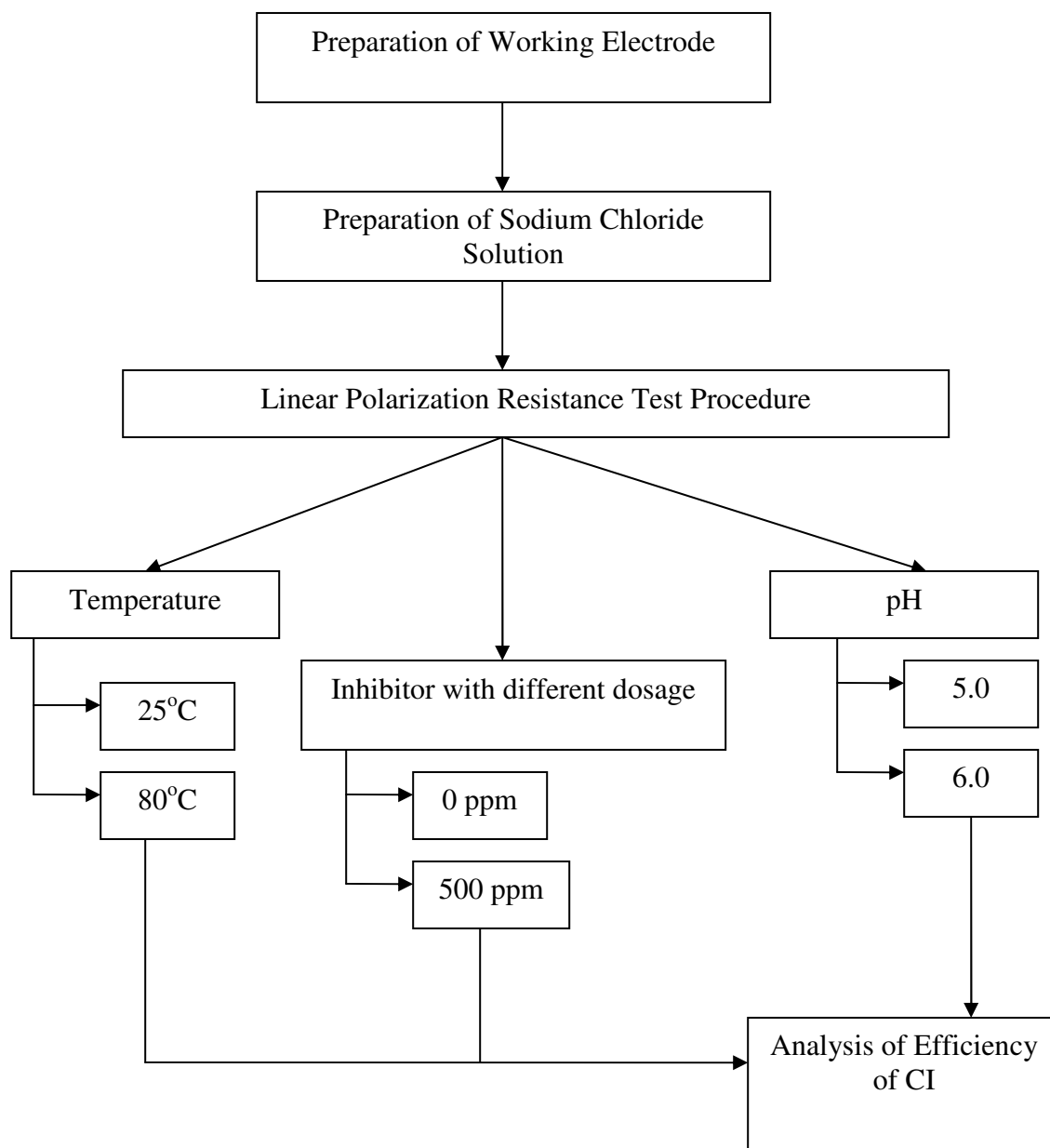
Figure 3.1: Flow chart of the methodology involve in the study of CI for CO_2

3.1.1 Test Matrix

Table 3.1 below shows eight experiments which have been done according to the different temperature and pH for 0 ppm of corrosion inhibitor and 500 ppm of corrosion inhibitor.

Table 3.1: Test matrix for the laboratory works for Corrosion Inhibitor study

pH	25^0C Uninhibited	25^0C (500 ppm CI)	80^0C Uninhibited	80^0C (500 ppm CI)
5.0	EXPERIMENT 1	EXPERIMENT 2	EXPERIMENT 3	EXPERIMENT 4
6.0	EXPERIMENT 5	EXPERIMENT 6	EXPERIMENT 7	EXPERIMENT 8

Each pH will be used in different temperatures, which are 25^0C and 80^0C. The first experiment will be started using 0 ppm of corrosion inhibitor dosage with the sample of carbon steel in the environment of 3% NaCl and CO_2. The next experiment will be continued for the other temperature using the same dosage of corrosion inhibitor. After all the readings have been taken for 0 ppm of corrosion inhibitor, then the experiment procedure will be repeated for 500 ppm corrosion inhibitor. The experiment will be preceded under different pH, 5.0 and 6.0, also under the temperature of 25^0C and 80^0C. Same as the first experiment, the accurate reading or result will be taken from the average of the 21 readings.

3.1.2 Laboratory Set-up

The set-up for the laboratory test using electrochemical measurement method of Linear Polarization Resistance experiments is showed in Figure 3.2 and Figure 3.3. The test assembly consists of one-liter glass cell bubbled with CO_2 gas. The required test temperature is set through the hot plate. The electrochemical measurements are based on a three-electrode system, using a commercially available potentiostat with a computer control system. The reference electrode used is a saturated calomel electrode (SCE) and the auxiliary electrode is a platinum electrode.
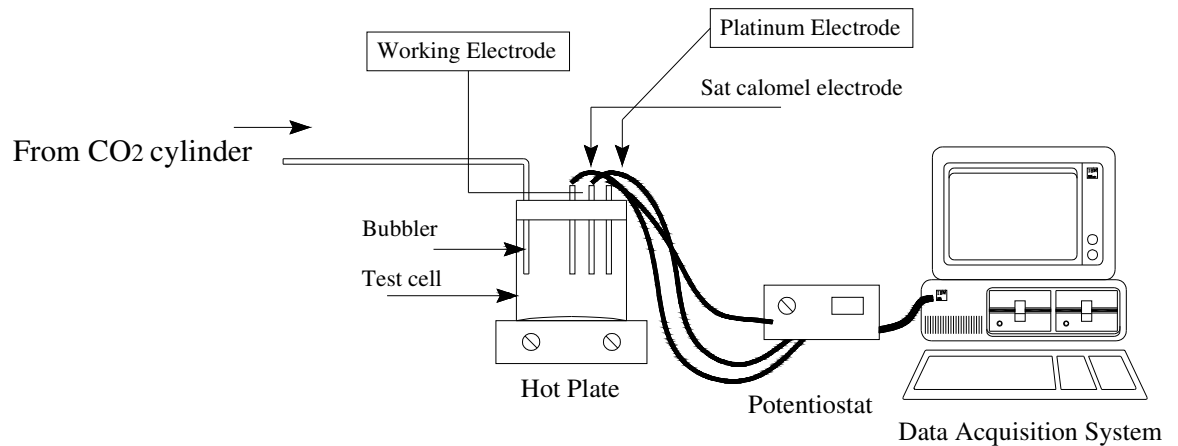
Figure 3.2: Schematic diagram for static experimental set-up

Figure 3.3: Real experiment set up in the laboratory

Corrosion rate is measured by linear polarization resistance method carried out is based on the ASTM G59-97, Standard Method for conducting potentiodynamic polarization resistance measurement.

3.2 Material

The working electrode or sample in this experiment is mild steel (EN 24). The composition of the mild steel EN 24 as shown below: [12]

Table 3.2: Composition of Plain Carbon Steels

Samples	Plain Carbon Steels	
Composition	Min (%)	Max (%)
Carbon	0.35	0.45
Silicon	0.05	0.35
Manganese	0.60	1.00
Sulphur		0.06
Phosphorus		0.06

The preparations of the working electrode are as follow:
1. The samples were spot welded with copper wire.
2. After that, it was mounted with epoxy by cold mounting and then polished to 800-grade finish using silicon carbide paper.
3. Finally, it was degreased and rinsed with deionizer water and ethanol. The working electrode is shown in Figure 3.5.
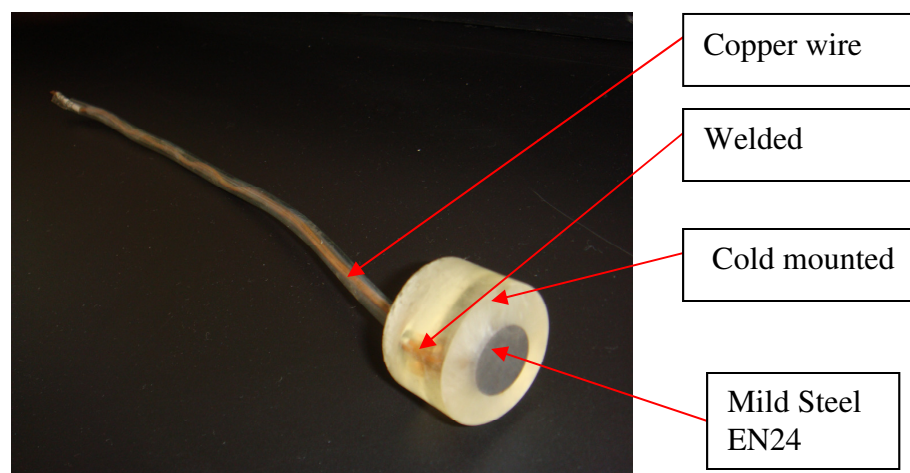
Figure 3.4: Photo of EN24 working electrode.

3.3 Preparation of Solutions

The solutions were prepared from the 3% NaCl solution is saturated with CO_2 by purging for at least one hour prior to the exposure of electrode. The pH of the solution could be adjusted by adding an amount of 1M $NaHCO_3$. The pH value is checked by microcomputer pH-meter METTLER-TOLEDO Model 320, which had been calibrated using standard buffer solutions.

3.4 Experiment Environment

The environment for the laboratory had being set to different conditions with different variables. The values of pH which had been varied in the study are 5.0 and 6.0 while temperatures are 25^oC and 80^oC and lastly the dosage of the corrosion inhibitor used is 500 ppm. 1 bar of carbon dioxide purge in the solution through out the experiment to provide the environment of CO_2 corrosion.

3.5 Addition of Corrosion Inhibitor

Corrosion Inhibitor used in this experiment is **AMTECH**, manufactured by AMTECH Sdn. Bhd comprised of imidazolines and amines. Concentration of corrosion inhibitor injected into the cell for the experiment is according to reference from a few papers published by NACE International. Micropipette is used to measure the accurate volume of the corrosion inhibitor into the solution.

The volume of corrosion inhibitor added into the solution is base on parts per million (ppm) according to the volume of solution used in the experiment. For this experiment, the volume of the 3% NaCl used is 1 liter. Thus 1ppm of corrosion inhibitor in this experiment is equivalent to 1μL.

3.6 Experiment Procedures

In this study, there are several laboratory tests which have to be conducted by varying the temperature, concentration of corrosion inhibitor and pH. The procedures of the each experiment are nearly the same. Experiments procedures are as per described below:

1. Solution medium of sodium chloride 3% prepared, 30g of sodium chloride is mixed into the distilled water of 1 liter.

2. Working electrode prepared as per describe in the section 3.2. And Setting up of the equipment for the laboratory test as per described in section 3.1.

3. Purging of the carbon dioxide gas started and continuous purging for half an hour until the carbon dioxide is saturated in the solution. The indication of the cell is saturated with carbon dioxide can be tested with the pH meter when it indicate the reading of pH nearly 3.8.

4. The first experiment consists of temperature at room temperature which is $25^{o}C$ and sodium bicarbonate is added into the solution to increase the pH of the solution to 5. Once the environment of the experiment achieve.

5. For the first section of laboratory test using pH 5.0, corrosion inhibitor is not added into the solution, thus proceed to the step 7 once the working electrode is placed in the cell. The experiment was running for 21 hours. The second experiment is adding 500 ppm of inhibitor into the sodium chloride solution. The third and fourth experiments are by increasing the temperature of the solution to $80^{0}C$ with the concentration of inhibitor 0 ppm and 500 ppm, respectively.

6. For second pH, which is 6, for the first laboratory test, same as the pH 5.0, corrosion inhibitor is not added into the solution, thus proceed to the step 7 once the working electrode is placed in the cell. The experiment was running for 21

hours. The second experiment for solution pH 6 is adding 500 ppm of inhibitor into the sodium chloride solution. The last 2 experiments are by increasing the temperature of the solution to 80^0C with the concentration of inhibitor 0 ppm and 500 ppm, respectively.

7. Once the chemicals and electrodes added into the solution, access the data acquisition system, in this laboratory is computer connected to the ACM Instruments Version 5, run Gill 12 Weld Tester Serial No. 1350 –Sequencer and the Core Running software.

8. Key in all the parameters that set for the measurement of the experiment into the Sequencer software.

9. 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 discuss in the Section 3.2.7.
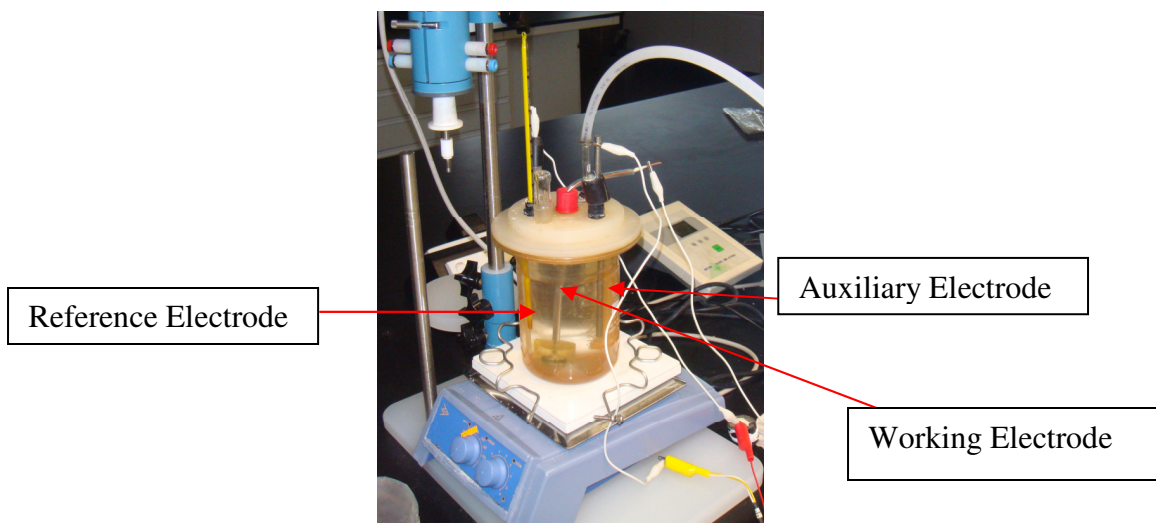
Figure 3.5: Static bubble test using Linear Polarization Resistance method set up in the laboratory.

3.7 Theory behind calculation

From the linear polarization resistance test, we can determine the corrosion rate of the sample. The theory of the calculation for linear polarization is as shown below:

The corrosion current density is related to polarization resistance by Stern_Geary coefficient, B. The Stern-Geary Constant, B, is approximated as 25 mV for all pH.

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

The dimension of R_p is ohm-cm2, i_{corr} is mA/cm^2, and B is in V. B also can be written as:

$$B = \frac{b_a \, b_c}{2.303(b_a + b_c)}$$

Where ba, bc is the Tafel slope for cathodic and anodic reaction.

The corrosion rate, CR in mm/year can be determined from the formula shown below:

$$CR = 3.27 \text{ x } i_{corr} \text{ EW/ density of the corroding material}$$

Where, EW is the equivalent weight of the corroding species in grams and the density of the corroding material is in g/cm^3. In this case equivalent weight of iron is 27.92 g and density of the corroding material is iron, thus, iron density is 7.8 g/cm^3.

CHAPTER 4

RESULT AND DISCUSSION

4. 1 Potentiodynamic Polarization Measurements

Potentiodynamic Polarization measurements results consist of the result for the system of 3%NaCl with 25^0C and pH 5, 3%NaCl with 80^0C and pH 5, 3%NaCl with 25^0C and pH 6 and last the system of 3%NaCl with 80^0C and pH 6 for 0 ppm corrosion inhibitor and 500 ppm corrosion inhibitor. The result for each experiment can be seen from all the figures below.

4.1.1 The system of 3%NaCl solution with 25^0C and pH 5

From Figure 4.1 and Figure 4.2, there is biggest decrement between the first readings until the reading of 21. All the experiments had been done in 21hours with one reading had been taken in one hour.

Figure 4.1: The trend of uninhibited corrosion rate for the system of 3%NaCl at 25^0C with pH 5

From Figure 4.1, the uninhibited mean corrosion rate for 3%NaCl with temperature 25^0C and pH 5 is 3.17 mm/year. The mean corrosion rate is the average from 21 readings of corrosion rates after immersion of 21 hours.

Figure 4.2: The trend of inhibited Corrosion Rate with 500 ppm of CI for the system of 3%NaCl at 25^0C with pH 5

From Figure 4.2, the mean corrosion rate with 500 ppm corrosion inhibitor for 3%NaCl with temperature 25^0C and pH 5 is 0.93 m/year. The mean corrosion rate is the average from 21 readings of corrosion rates after immersion of 21 hours.

Figure 4.3: The trend of Uninhibited CR and CR with 500 ppm CI versus Time for 25^0C & pH 5

From the Figure 4.3 above, there are two plots of lines which are uninhibited corrosion rate and corrosion rate with 500 ppm corrosion inhibitor for temperature 25^0C and pH 5. There is huge decrement when the concentration of inhibitor is added. The mean corrosion rate decrease from 3.17 mm/year to 0.93 mm/year. The efficiency of corrosion inhibitor for this system is 74.5%.

4.1.2 The system of 3%NaCl solution with 80^0C and pH 5

Figure 4.4: The trend of uninhibited Corrosion Rate for the system of 3%NaCl at 80^0C
with pH 5

From Figure 4.4, the uninhibited mean corrosion rate for 3%NaCl with temperature 80^0C and pH 5 is 8.60 mm/year. The mean corrosion rate is the average from 21 readings of corrosion rates after immersion of 21 hours.

Figure 4.5: The trend of inhibited Corrosion Rate with 500 ppm of CI for the system of 3%NaCl at 80^0C with pH 5

From Figure 4.5 above, the mean corrosion rate with 500 ppm corrosion inhibitor for 3%NaCl with temperature 80^0C and pH 5 is 0.47 mm/year. The mean corrosion rate is the average from 21 readings of corrosion rates after immersion of 21 hours.

Figure 4.6: The trend of Uninhibited CR and CR with 500 ppm CI versus Time for 80^0C & pH 5

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From Figure 4.6 above, there are two plots of lines which are uninhibited corrosion rate and corrosion rate with 500 ppm corrosion inhibitor for temperature of 80^0C and pH 5. There is really huge decrement when the concentration of inhibitor is added. The mean corrosion rate decrease from 8.60 mm/year to 0.47 mm/year. The corrosion inhibitor efficiency for this system is 97.4%.

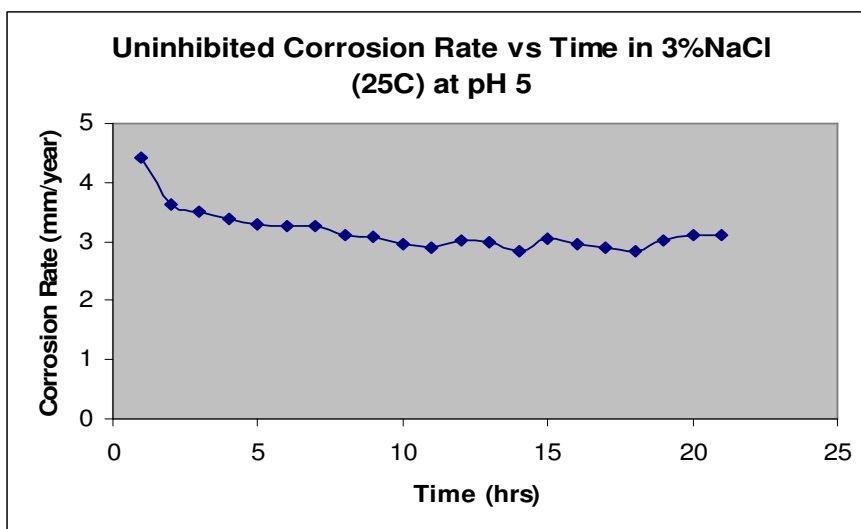**4.1.3 The system of 3%NaCl solution with 25^0C and pH 6**
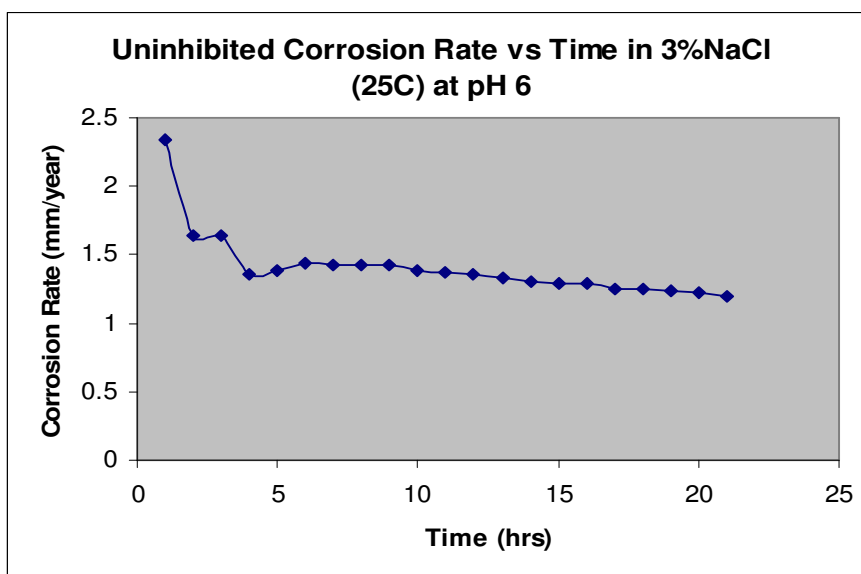
Figure 4.7: The trend of uninhibited Corrosion Rate for the system of 3%NaCl at 25^0C with pH 6

From Figure 4.7 above, the uninhibited mean corrosion rate for 3%NaCl with temperature 25^0C and pH 6 is 1.40 mm/year. The mean corrosion rate is the average from 21 readings of corrosion rates after immersion of 21 hours.
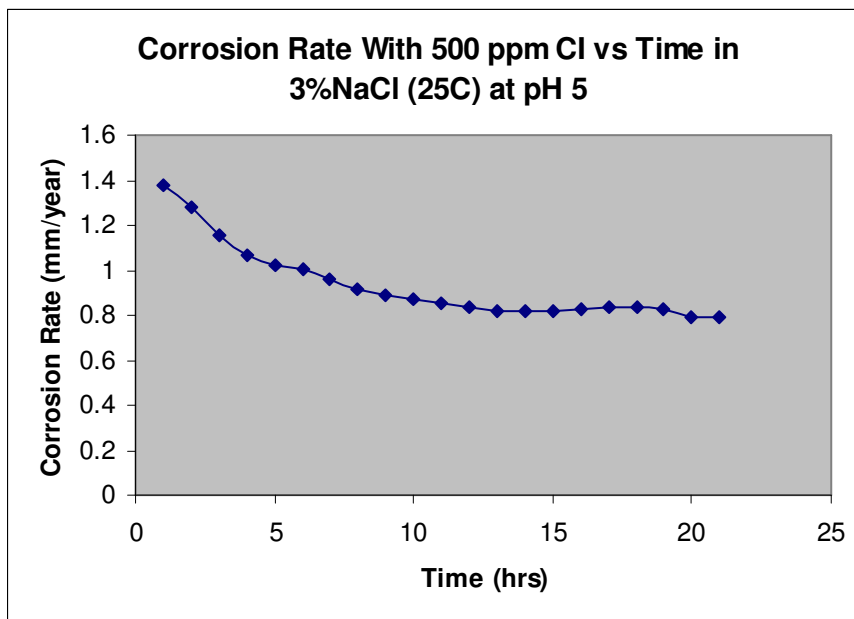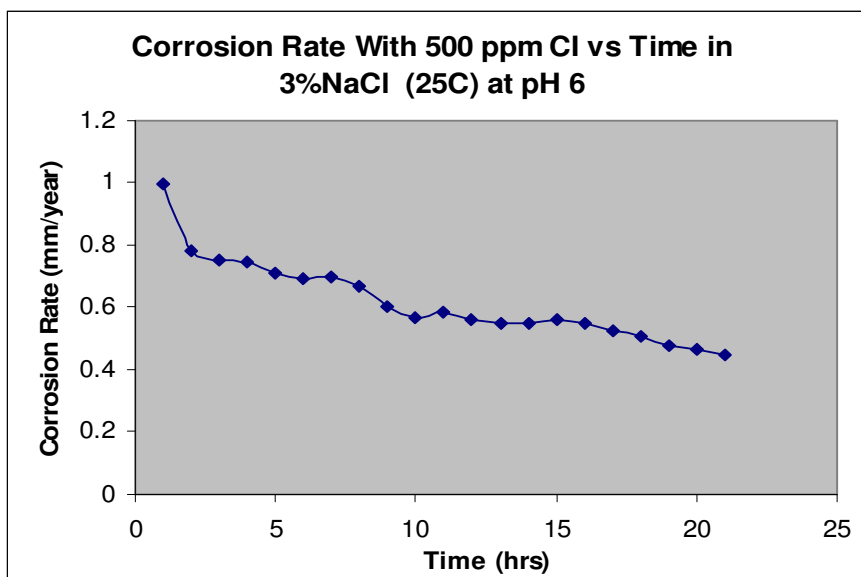
Figure 4.8: The trend of inhibited Corrosion Rate with 500 ppm of CI for the system of 3%NaCl at 25^0C with pH 6

From Figure 4.8 above, the mean corrosion rate with 500 ppm corrosion inhibitor for 3%NaCl with temperature 250^0C and pH 6 is 0.62 mm/year. The mean corrosion rate is the average from 21 readings of corrosion rates after immersion of 21 hours.
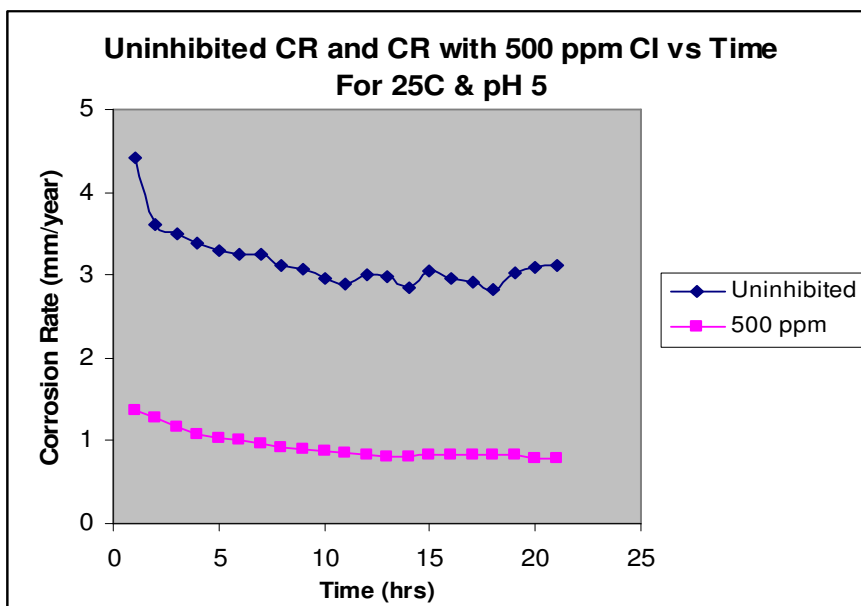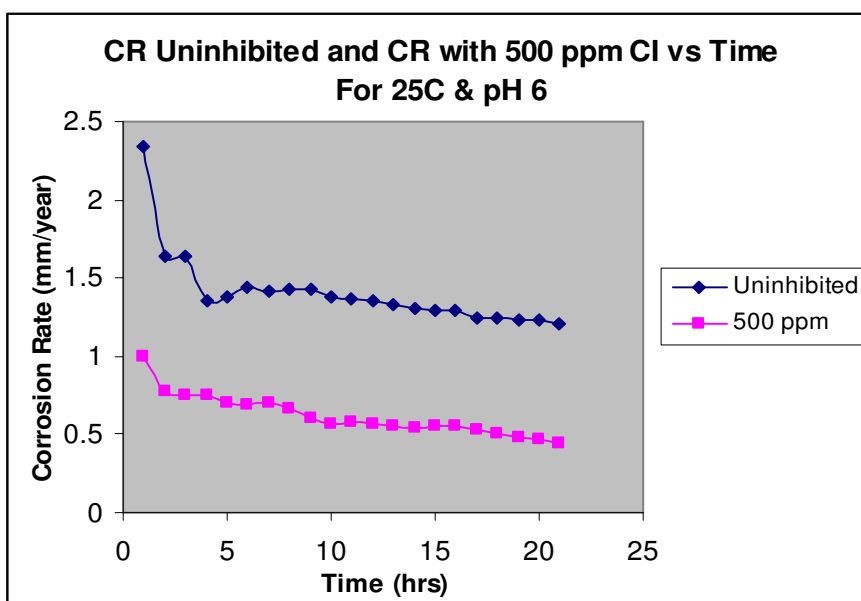

Figure 4.9: The trend of Uninhibited CR and CR with 500 ppm CI versus Time for 25^0C & pH 6

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From Figure 4.9 above, there are two plots of lines which are uninhibited corrosion rate and corrosion rate with 500 ppm corrosion inhibitor for temperature of 25^0C and pH 6. There is decrement when the concentration of inhibitor is added. The mean corrosion rate decrease from 1.40 mm/year to 0.62 mm/year. The corrosion inhibitor efficiency of this system is 62.8%.

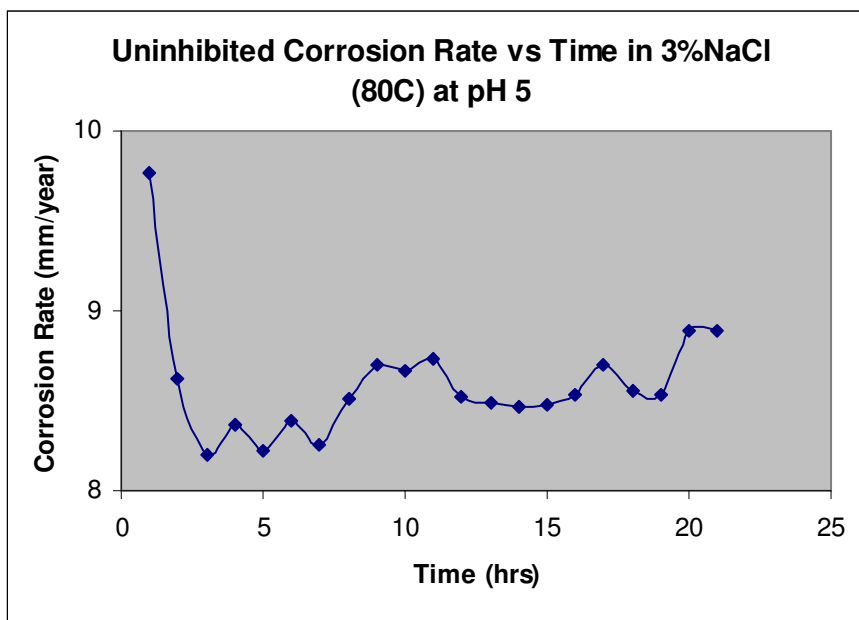**4.1.4 The system of 3%NaCl solution with 80^0C and pH 6**
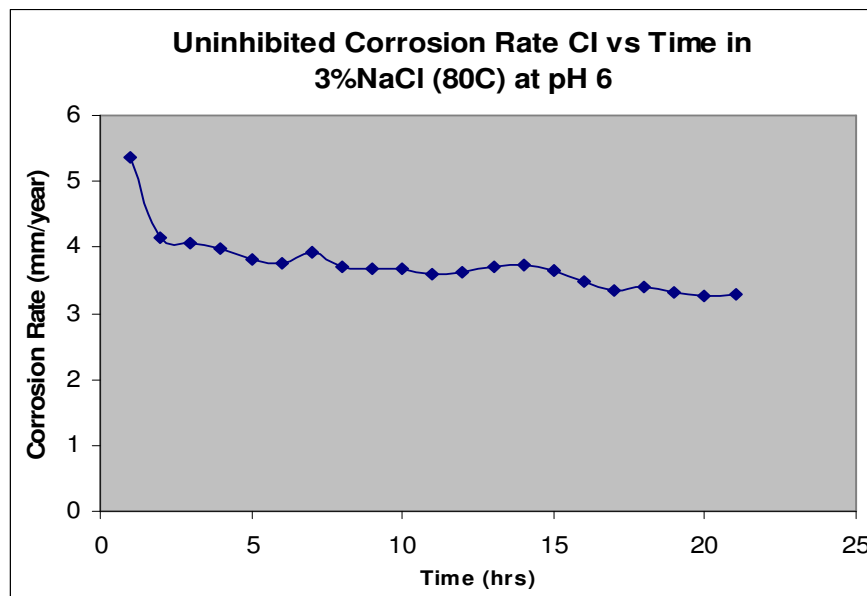
Figure 4.10: The trend of uninhibited Corrosion Rate for the system of 3%NaCl at 80^0C with pH 6

From Figure 4.10 above, the uninhibited mean corrosion rate for 3%NaCl with temperature 80^0C and pH 6 is 3.74 mm/year. The mean corrosion rate is the average from 21 readings of corrosion rates after immersion of 21 hours.
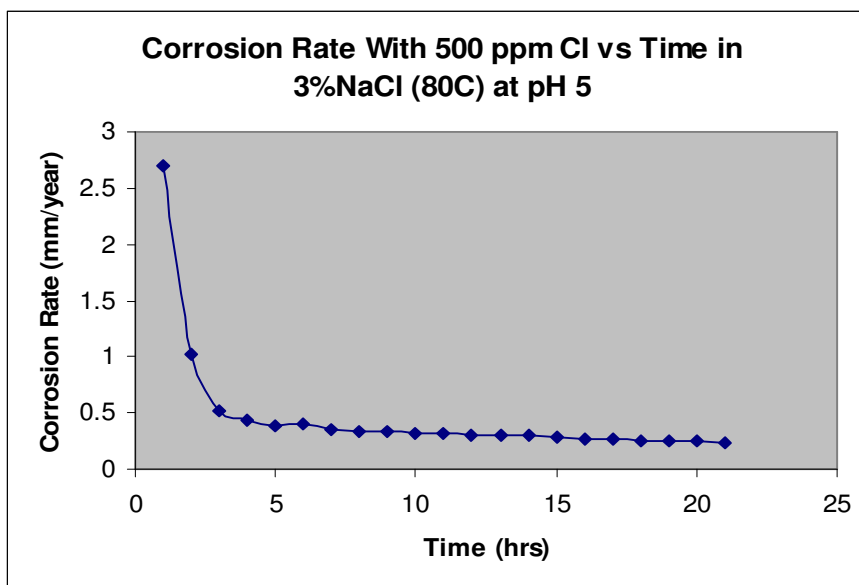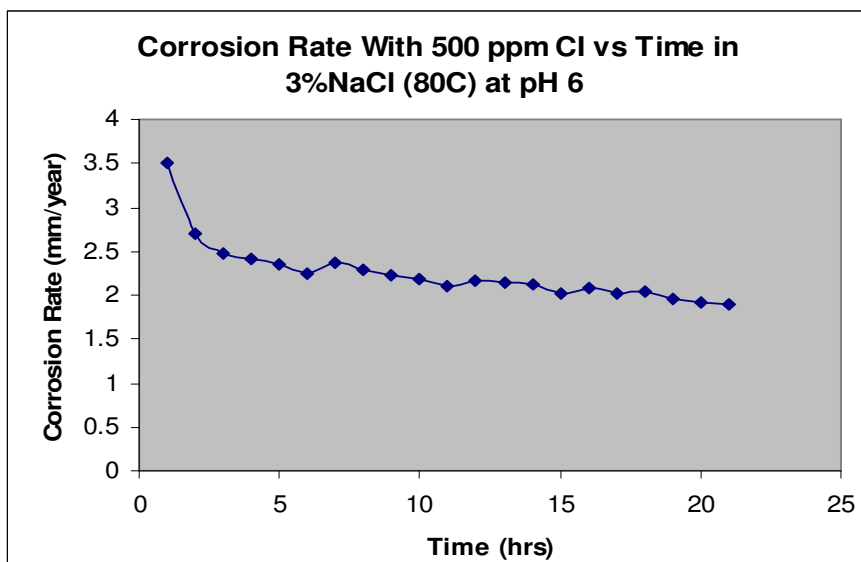
Figure 4.11: The trend of inhibited Corrosion Rate with 500 ppm of CI for the system of 3%NaCl at 80^0C with pH 6

From Figure 4.11 above, the mean corrosion rate with 500 ppm corrosion inhibitor for 3%NaCl with temperature 250^0C and pH 6 is 2.25 mm/year. The mean corrosion rate is the average from 21 readings of corrosion rates after immersion of 21 hours
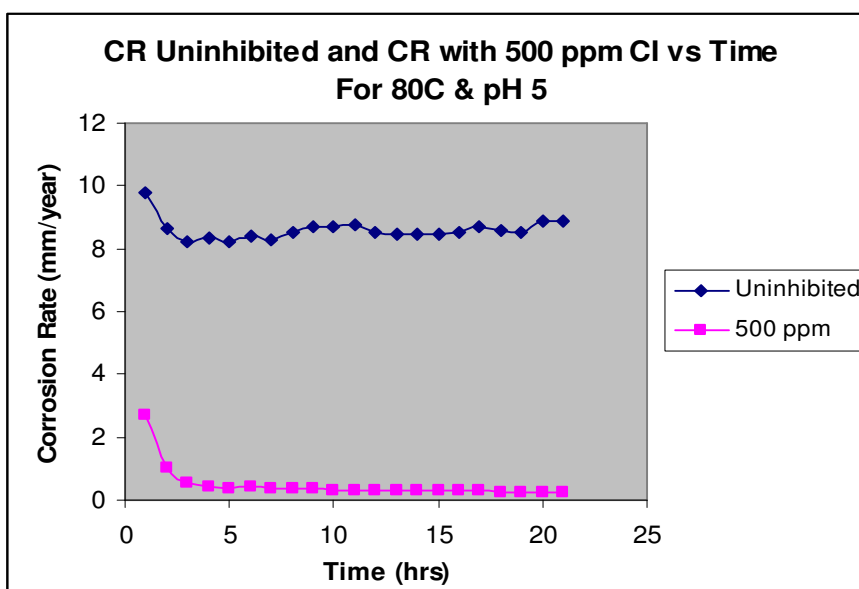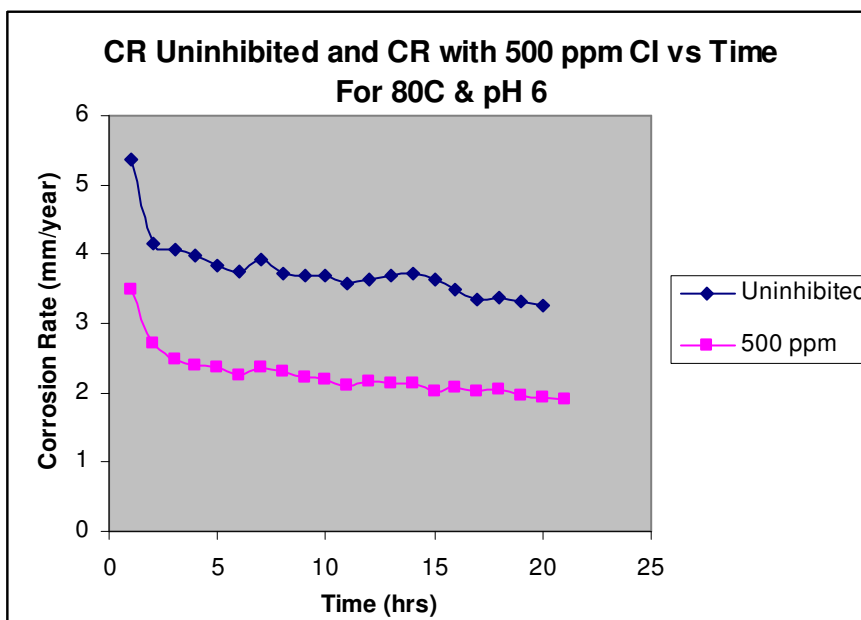

Figure 4.12: The trend of Uninhibited CR and CR with 500 ppm CI versus Time for 80^0C & pH 6

From the Figure 4.12 above, there are two plots of lines which are uninhibited corrosion rate and corrosion rate with 500 ppm corrosion inhibitor for temperature 0f 25^{0}C and pH 6. There is a slightly decrement when the concentration of corrosion inhibitor is added. The mean corrosion rate decrease from 3.74 mm/year to 2.25 mm/year. Lastly, the inhibitor efficiency for this system is 46.2%.

4.1.5 The result of the Efficiencies for each system with the current densities, *Icorr*, corrosion potential, *Ecorr* and corrosion rate.

From Table 4.1 below, the efficiency for the inhibitor is highest at system of 3% NaCl with temperature of 25^{0}C and pH 5. Noted here also that each of the result in the Table 4.1 is the result for the last readings for each experiment after 21hours or running the experiments. The efficiency of the inhibitor can be calculated with the formula below:-

$$CR = \frac{CR_{uninhibited} - CR_{inhibited}}{CR_{uninhibited}}$$

For each system consist of 0 ppm inhibitor and inhibited conditions which are 500 ppm of inhibitor, the highest efficiency is 97% and the lowest is 46%.

Table 4.1: Efficiencies and the Potentiodynamic polarization parameters for EN 24 mild steel in 3% NaCl solutions saturated with CO_2 at pH 5 and 6 for uninhibited and inhibited conditions.

| Temp (^{0}C) | pH | Systems | Polarization | | Corrosion rate (mm/year) | Efficiency η% |
			Ecorr (mV_{SCE})	Icorr (mA/cm_2)		
25	5	3% NaCl	-744.16	0.27	3.12	Uninhibited
25	5	3% NaCl + 500ppm	-631.22	0.07	0.79	**74.5%**
80	5	3% NaCl	-299.84	0.78	8.89	Uninhibited
80	5	3% NaCl + 500ppm	-198.13	0.02	0.23	**97.4%**
25	6	3% NaCl	-750.37	0.10	1.20	Uninhibited
25	6	3% NaCl + 500ppm	-631.83	0.04	0.45	**62.8%**
80	6	3% NaCl	-239.82	0.28	3.29	Uninhibited
80	6	3% NaCl + 500ppm	-178.85	0.16	1.90	**46.2%**

At pH 5, with the highest temperature, the efficiency is the highest which is 97.4%. While at the same temperature, pH 6, the efficiency is the lowest which is 46.2%. Below are the discussion of the parameters that affecting the efficiency of the inhibitor. The parameters are pH, temperature and concentration of inhibitor that had been used to run the experiments. All these parameters give impact to the efficiency of the inhibitor. The mechanism of the inhibitor can also be discussed according to the polarization measurements parameters above.

4.2 The discussion of the pH effect on the inhibitor efficiency

The affect of pH can be seen from the parameters in the Table 4.2 below. From Table 4.1 shows that the highest inhibitor efficiency is 97.4% in the solution of 3%NaCl with 80^0C at pH 5. The lowest inhibitor efficiency is 46.2% in the solution of 3%NaCl with 80^0C at pH 6. For the room temperature, also exhibits the same result which is inhibitor efficiency is lower at pH 6 compared to pH 5. Table 4.2 below also shows the relation between the corrosion potential, Ecorr (mV_{SCE}) and the current density, icorr (mA/cm^2) that can explain the behavior of different pH to the result of the efficiency.

Table 4.2: The result for Ecorr, Icorr and efficiency for the systems with 500ppm CI 3%NaCl 25^0C for pH 5 and pH 6

SYSTEM (Different pH)	CORROSION POTENTIAL, Ecorr	CURRENT DENSITY, Icorr	EFFICIENCY, η%
3% NaCl 25^0C pH 5 with 500 ppm CI	-631.22	0.07	**74.5%**
3% NaCl 25^0C pH 6 with 500 ppm CI	-631.83	0.04	**62.8%**

The discussion for the above Table 4.2 shows the parameters for mild steel in CO_2 – saturated NaCl solution obtained at 25^0C and at pH of 5 and 6 in the presence of 500 ppm inhibitor after 21hours of exposure. Increase in pH was observed to have little or no effect on the anodic reaction which can be seen from the value of corrosion potential, Ecorr. From the Table 4.2, the values for Ecorr for both systems are quite the same. However, a decrease in cathodic reaction can be seen because the value of current densities for both pH are decreasing for increment of pH. The decrement is principally due to a change in the concentration species involved in the cathodic reaction. The

inhibitor efficiency is decreased with the increment of pH because at higher pH, protective layer which is corrosion product film formation tend to be very active and the existence of this film will protect the metal surface and the effectiveness of the inhibitor will be decreased. Thus, the existence of this film will effect the effectiveness of the inhibitor.

4. 3 The discussion of the Temperature effect on the inhibitor efficiency

The effect of the temperature in anodic and cathodic reactions in the presence of AMTECH inhibitor for mild steel in 3% NaCl solution saturated with CO_2 at pH 5 and 6 is represented in the Table 4.3 below.

Table 4.3: The result for Ecorr, Icorr and efficiency for the systems with 500ppm CI 3%NaCl & pH 5 and 6 for temperature 25^0C and 80^0C

SYSTEM (Different Temperature)	CORROSION POTENTIAL, Ecorr	CURRENT DENSITY, Icorr	EFFICIENCY, $\eta\%$
3% NaCl 25^0C pH 5 with 500 ppm CI	-631.22	0.07	**74.5%**
3% NaCl 80^0C pH 5 with 500 ppm CI	-198.13	0.02	**97.4%**
3% NaCl 25^0C pH 6 with 500 ppm CI	-631.83	0.04	**62.8%**
3% NaCl 80^0C pH 6 with 500 ppm CI	-178.85	0.16	**46.2%**

From Table 4.1 and table 4.3, there is clear acceleration of both cathodic and anodic reactions with an increase in temperature. These can be proved from the result corrosion potential, Ecorr and current density, Icorr in the Table 4.3 above. There is huge increase in the current density especially for the pH 5 in the absence of inhibitor for temperature of 25^0C and 80^0C.. From Table 4.3, it can be seen that the current density increased with the temperature in the uninhibited or inhibited solutions. This is due to the acceleration of all processes involved in corrosion such as electrochemical, chemical, transport, etc.

with increase in temperature. It could also notice that increase in temperature increased the inhibition efficiencies of the inhibitor suggesting chemisorption of the organic molecule on the surface of the metal. But, at pH 6, although the temperature is increased to 80^0C, the efficiency of the inhibitor is not higher than the room temperature because the effect of the corrosion product film formation at pH 6 that protected the surface compared to the mechanism of the inhibitor.

4. 4 The discussion of the CI Concentration effect on the inhibitor efficiency

From the Table 4.3, the introduction of AMTECH inhibitor to the system at 25^0C and at pH 5, 80^0C at pH 5, 25^0C at pH 6 and lastly 80^0C at pH 6, three distinct features can be observed. The presence of inhibitor greatly increased the corrosion potential, Ecorr to a mere positive region. This can be seen through the Table 4.3 above. The large shift in the corrosion potential indicates that the inhibitor for the system is probably due to the active sites blocking effect. In the Table 4.3 also can be observed that the anodic reaction which is the value of Ecorr when the inhibitor is introduced into the solution is slightly higher than $500mV_{SCE}$ due to the large change in the corrosion potential by the inhibitor. Lastly, a pronounced effect is exerted on the cathodic process. The limiting current for hydrogen evolution is greatly induced indicating that the inhibition is confined to the hindering of the hydrogen reduction reaction. Also, the current density reduced when the inhibitor is present. We can see in the Table 4.1 that the density is reduced in all systems when the CI is introduced.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

For the blank CO_2 solution without corrosion inhibitor, the corrosion rate increases with increase in temperature and reduces with higher pH. 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 25^0C to 80^0C. The highest efficiency of corrosion inhibitor is 97.4% for 80^0C and pH 5. From this efficiency, can be concluded that the efficiency of CI is highest at temperature 80^0C. But, when pH is increased from 5 to 6, the inhibitor efficiency decreased although the temperature is increased. The efficiency for pH 6 with temperature 80^0C is the lowest efficiency with the value of 46.2%. This is possibly due to the effect of the corrosion product film formation that governed the protection of metal surface at the pH higher than 5. Therefore, the inhibitor efficiency decreases for higher pH from 5 but temperature of 80^0C will increase the efficiency of the corrosion inhibitor at pH 5.

5.2 Recommendation

The recommendation is to further investigate the effect of flow condition to the inhibitor efficiency. Flow condition is one of the factors influenced the efficiency of the inhibitor. The rotating cylinder electrode (RCE) can be used to simulate the condition of turbulence flow for the systems. The static bubble test simulates the static flow condition and does not simulate the real situation in the pipeline due to the low shear wall provided by the static bubble test. The speed of the rotation can be varied in order to determine the effect of turbulence flow condition to the efficiency of the corrosion inhibitor.

Other recommendation is to investigate the formation of corrosion product film on the steel surface at higher pH such as pH 6. The properties of the corrosion product film and their influence on the corrosion rate are important factor that will affect the inhibitor efficiency. Scanning electron microscope (SEM) can be used to determine the properties and composition of the film. The existence of the corrosion product film will reduce the corrosion rate by protecting the steel surface and the composition of the film varies with the inhibitor efficiency and the thickness of this film decreases with addition of inhibitor.

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APPENDIXES

Linear Polarization Resistance Result

Laboratory Testing 1

Table 1: LPR Result for experiment at 25°C and pH 5.0 for 0 ppm CI

Time (Sec)	LPR (ohm.cm²)	Icorr (mA/cm²)	Corrosion Rate mm/year)	Potential (mV)
0	65.433	0.3820683	4.4281	-725.04
3600.2	80.039	0.3123464	3.62	-740.67
7200.4	82.995	0.3012214	3.4911	-740.97
10800	85.795	0.29139	3.3772	-742
14401	87.687	0.2851032	3.3043	-742.82
18001	88.861	0.2813365	3.2606	-742.56
21601	89.196	0.2802785	3.2484	-742.59
25202	93.088	0.2685622	3.1126	-743.28
28802	94.05	0.2658134	3.0807	-743.91
32402	98.139	0.2547389	2.9524	-743.85
36003	100.27	0.2493134	2.8895	-744.9
39603	96.305	0.2595911	3.0086	-744.16
43203	97.427	0.2566023	2.974	-744.49
46803	101.7	0.2458044	2.8488	-743.45
50404	95.303	0.2623197	3.0402	-743.1
54004	98.245	0.2544653	2.9492	-742.34
57604	99.636	0.2509108	2.908	-742.29
61205	102.38	0.244165	2.8298	-742.5
64805	96.047	0.2602868	3.0167	-742.68
68405	93.417	0.2676146	3.1016	-744.61
72006	92.951	0.2689578	3.1172	-744.16

Table 2: LPR Result for experiment at 25°C and pH 5.0 for 500 ppm CI

Time (Sec)	LPR (ohm.cm²)	Icorr (mA/cm²)	Corrosion Rate (mm/year)	Potential (mV)
0	210.14	0.1189654	1.3788	-628.32
3600.2	226.12	0.1105585	1.2813	-621.85
7200.3	250.47	0.0998095	1.1567	-623.27
10800	271.25	0.0921648	1.0681	-622.19
14400	283.15	0.0882897	1.0232	-620.03
18001	289.22	0.0864388	1.0018	-620.17
21601	302.49	0.0826461	0.9578678	-620.67
25201	316.85	0.0789011	0.9144639	-621.06
28801	326.74	0.0765112	0.8867649	-621.34
32401	333.31	0.0750051	0.8693094	-620.98
36002	338.37	0.0738828	0.8563013	-621.78
39602	346.54	0.0721406	0.8361101	-621.65
43202	355.25	0.0703723	0.8156145	-622.84
46802	355.61	0.0703007	0.8147847	-624.32
50403	352.85	0.0708515	0.8211684	-625.24
54003	350.11	0.0714061	0.8275967	-626.64
57603	346.51	0.0721468	0.8361811	-627.35
61203	346.46	0.0721576	0.836307	-628.28
64804	351.89	0.0710439	0.8233983	-631.5
68404	367.02	0.0681161	0.789465	-631.03
72004	365.02	0.0684893	0.7937906	-631.22

Table 3: LPR Result for experiment at 80°C and pH 5.0 for 0 ppm CI

Time (Sec)	LPR (ohm.cm²)	Icorr (mA/cm²)	Corrosion Rate (mm/year)	Potential (mV)
0	29.674	0.8424677	9.7642	-278.22
3600	33.601	0.7440247	8.6232	-290.29
7200.4	35.317	0.7078737	8.2042	-293.33
10800	34.618	0.7221501	8.3697	-294.67
14400	35.235	0.7095036	8.2231	-297.09
18001	34.531	0.7239858	8.3909	-295.87
21601	35.11	0.7120462	8.2526	-294.37
25201	34.056	0.7340681	8.5078	-292.95
28801	33.285	0.7510815	8.705	-292.18
32401	33.413	0.7481961	8.6715	-290.47
36002	33.182	0.7534122	8.732	-289.19
39602	34.003	0.7352207	8.5212	-289.4
43202	34.14	0.7322587	8.4868	-288.55
46802	34.212	0.7307277	8.4691	-288.77
50403	34.192	0.7311492	8.474	-288.37
54003	33.934	0.7367031	8.5383	-288.65
57603	33.296	0.7508214	8.702	-288.88
61204	33.881	0.7378599	8.5517	-289.63
64804	33.942	0.7365415	8.5365	-316.94
68404	32.588	0.7671381	8.8911	-305.39
72004	32.588	0.7671502	8.8912	-299.84

Table 4: LPR Result for experiment at 80°C and pH 5.0 for 500 ppm CI

Time (Sec)	LPR (ohm.cm²)	Icorr (mA/cm²)	Corrosion Rate (mm/year)	Potential (mV)
0	107.57	0.2324007	2.6935	-218.88
3600.2	285.25	0.0876411	1.0157	-209.92
7200.7	550.51	0.0454118	0.5263231	-205.08
10801	676.15	0.0369737	0.4285257	-201.97
14401	763.97	0.0327237	0.3792679	-204.26
18001	709.86	0.0352179	0.4081755	-207.35
21601	830.28	0.0301103	0.3489784	-208.81
25201	861.75	0.0290105	0.3362313	-209.45
28802	870.66	0.0287137	0.3327913	-209.49
32402	910.23	0.0274654	0.3183244	-210.09
36002	903.53	0.027669	0.3206834	-210.41
39602	945.54	0.0264399	0.3064379	-210.05
43202	962.42	0.0259761	0.3010629	-210.41
46803	980.61	0.0254942	0.2954782	-210.92
50403	998.87	0.0250281	0.2900754	-208.24
54004	1062.9	0.0235198	0.2725943	-211.28
57604	1069.8	0.0233682	0.2708369	-210.98
61205	1125.8	0.0222055	0.2573623	-199.15
64805	1181.7	0.0211544	0.2451797	-195.89
68405	1190.5	0.020998	0.2433665	-197.25
72005	1233.1	0.0202737	0.2349718	-198.13

Table 5: LPR Result for experiment at 25°C and pH 6.0 for 0 ppm CI

Time (Sec)	LPR (ohm.cm²)	Icorr (mA/cm²)	Corrosion Rate (mm/year)	Potential (mV)
0	123.63	0.2022102	2.3436	-747.07
3600	176.46	0.1416698	1.6419	-753.81
7200.1	177.12	0.141146	1.6358	-752.38
10800	214.36	0.1166223	1.3516	-751.21
14400	210.03	0.1190301	1.3795	-741.35
18000	201.7	0.1239462	1.4365	-742.56
21601	203.89	0.1226114	1.421	-743.76
25201	203.01	0.123143	1.4272	-744.89
28801	203.27	0.1229871	1.4254	-745.73
32402	209.23	0.1194823	1.3847	-746.74
36002	211.52	0.11819	1.3698	-747.19
39602	213.68	0.1169956	1.3559	-747.31
43202	218.31	0.114514	1.3272	-747.88
46802	221.83	0.1126947	1.3061	-748.41
50402	224.82	0.1111984	1.2887	-748.46
54002	224.86	0.1111793	1.2885	-748.74
57603	231.81	0.1078437	1.2499	-749.11
61203	232.09	0.1077141	1.2484	-749.11
64803	234.52	0.1065999	1.2354	-749.19
68403	236.01	0.105924	1.2276	-750.93
72003	240.86	0.1037907	1.2029	-750.37

Table 6: LPR Result for experiment at 25°C and pH 6.0 for 500 ppm CI

Time (Sec)	LPR (ohm.cm²)	Icorr (mA/cm²)	Corrosion Rate (mm/year)	Potential (mV)
0	290.87	0.0859464	0.9961192	-634.97
3600.1	370.53	0.0674698	0.7819747	-637.91
7200.6	384.82	0.0649647	0.7529404	-637.48
10801	387.86	0.0644551	0.7470343	-635.93
14401	409.3	0.0610791	0.7079073	-638.76
18001	416.61	0.0600078	0.6954908	-636.02
21601	413.3	0.0604881	0.7010568	-634.62
25202	432.67	0.0577804	0.6696746	-633.48
28802	481.54	0.0519164	0.6017105	-633.39
32402	508.98	0.0491173	0.5692691	-633.08
36002	496.92	0.0503099	0.5830915	-631.02
39602	515.72	0.0484757	0.561833	-631.13
43203	526.38	0.0474936	0.5504506	-631.97
46803	530.16	0.0471549	0.5465251	-631.01
50403	517.68	0.0482922	0.5597064	-630.28
54004	526.05	0.0475239	0.5508024	-631.43
57604	551.99	0.0452902	0.5249133	-631.5
61204	572.03	0.043704	0.5065288	-632.82
64804	608.85	0.0410605	0.4758908	-633.27
68405	620.65	0.0402801	0.4668459	-632.76
72005	647.57	0.0386055	0.4474377	-631.83

Table 7: LPR Result for experiment at 80°C and pH 6.0 for 0 ppm CI

Time (Sec)	LPR (ohm.cm²)	Icorr (mA/cm²)	Corrosion Rate (mm/year)	Potential (mV)
0	53.97	0.4632192	5.3687	-263.68
3600.1	69.867	0.3578227	4.1471	-294.66
7200.1	71.366	0.3503066	4.06	-294.71
10800	72.984	0.3425407	3.97	-292.19
14400	75.786	0.3298749	3.8232	-287.75
18001	77.205	0.323813	3.7529	-285.97
21601	73.779	0.3388481	3.9272	-283.59
25201	77.983	0.3205818	3.7155	-282.91
28802	78.515	0.3184093	3.6903	-275.24
32402	78.632	0.3179362	3.6848	-280.24
36002	80.805	0.3093862	3.5857	-278.63
39602	79.836	0.3131401	3.6292	-274.87
43203	78.28	0.3193651	3.7014	-269.56
46803	77.631	0.3220332	3.7323	-269.4
50403	79.638	0.3139198	3.6383	-267.67
54004	83.001	0.3011979	3.4908	-261.48
57604	86.678	0.2884228	3.3428	-258.06
61204	85.521	0.292323	3.388	-257.49
64805	87.231	0.286593	3.3216	-254.05
68405	88.527	0.2823986	3.2729	-249.47
72005	88.203	0.2834352	3.285	-239.82

Table 8: LPR Result for experiment at 80°C and pH 6.0 for 500 ppm CI

Time (Sec)	LPR (ohm.cm²)	Icorr (mA/cm²)	Corrosion Rate (mm/year)	Potential (mV)
0	82.815	0.3018761	3.4987	-191.59
3600.2	107.28	0.2330287	2.7008	-232.36
7200.6	116.74	0.2141339	2.4818	-220.83
10800	120.45	0.2075548	2.4055	-215.16
14401	123.13	0.2030325	2.3531	-211.52
18001	128.53	0.1945052	2.2543	-206.66
21601	122.26	0.2044798	2.3699	-199.69
25201	126.23	0.1980424	2.2953	-197.45
28801	130.2	0.192008	2.2253	-195.16
32402	132.19	0.189119	2.1918	-194.42
36002	137.3	0.1820733	2.1102	-190.88
39602	133.8	0.1868417	2.1654	-193.82
43202	134.9	0.1853115	2.1477	-196.63
46803	135.96	0.1838769	2.1311	-198.19
50403	143.53	0.1741731	2.0186	-201.3
54003	138.86	0.1800301	2.0865	-196.96
57604	143.21	0.1745591	2.0231	-195.01
61204	141.54	0.1766216	2.047	-191.55
64804	147.57	0.1694053	1.9634	-189.76
68405	150.89	0.1656759	1.9201	-184.77
72005	152.5	0.1639255	1.8998	-178.85