

CORROSION INHIBITION OF 6061 ALUMINUM ALLOY IN ACIDIC
MEDIA BY HONEY

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JUDUL: **CORROSION INHIBITION OF 6061 ALUMINUM ALLOY
IN ACIDIC MEDIA BY HONEY**

SESI PENGAJIAN: 2010/2011

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CORROSION INHIBITION OF 6061 ALUMINUM ALLOY IN ACIDIC MEDIA BY
HONEY

MOHD AZZIZUL BIN CHAMINGAN

Report submitted in fulfillment of the requirements
for the award of the degree of
Bachelor of Mechanical Engineering

Faculty of Mechanical Engineering
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DECEMBER 2010

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Date : 6th DECEMBER 2010

Dedicated, truthfully for supports,
encouragements and always be there during hard times, to
my beloved family.

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ABSTRACT

The corrosion inhibition of aluminum and its alloys in acidic solution is the subject of the tremendous technological importance due to the increased in industrial applications of these materials. Aluminum and its alloy, however are reactive material and prone to corrosion. Inhibitor is the most practical method to protect metal against corrosion. Most of the corrosion inhibitors are synthetic chemicals, expensive and very hazardous to environment. Therefore, it should be reduced use to protect environmentally and human in the future. For this project, 6061 Aluminum Alloy in 5% H₂SO₄ was evaluated by using weight loss method and electrochemical techniques. This study investigated the effect of variation concentration of honey as inhibitor to the corrosion behavior of 6061 Aluminum alloy in the acidic solution. In weight loss method, the samples were immersed in the solution for the duration of 28 days. The samples were weighted before exposure and through the cleaning process using Nitric Acid after the immersion test. The mass loss after cleaning cycle was analyzed in term of corrosion rates. The microscopic examination was conducted by using metallurgical microscope. Potentiodynamic polarization was employed for electrochemical measurement by using WonATech potentiostat. The samples were mounted by resin before it attached as working electrode. The results showed that addition of natural honey retards the rate of dissolution and hence inhibits the corrosion of the 6061 Aluminum Alloy in 5% H₂SO₄ solution. The inhibitor efficiency increased with increasing of honey concentration for both methods. It was found that, the uniform corrosion occur to the surface of Aluminum alloy in acidic solution. The tafel plot from the potentiodynamic polarization also showed that the honey significantly decrease the corrosion potential (E_{corr}) and current density (i_{corr}) and concurrently, increase the surface coverage. The adsorption of natural honey on the metal surface obeys Langmuir adsorption isotherm.

ABSTRAK

Penghalang karatan terhadap aluminium aloi didalam larutan asid merupakan teknologi penting yang unik akibat daripada peningkatan dalam aplikasi industri terhadap bahan tersebut. Aluminium dan aloi, bagaimanapun adalah bahan reaktif dan ketahanan terhadap karatan. Penghalang adalah kaedah yang paling praktikal untuk melindungi logam terhadap karatan. Sebahagian besar penghalang karatan adalah bahan kimia sintetik, mahal dan sangat berbahaya kepada alam sekitar. Oleh kerana itu, penggunaannya perlu dikurangkan untuk melindungi persekitaran dan manusia di masa hadapan. Penghalang karatan daripada madu terhadap aluminium aloi 6061 di 5% asid sulfurik dinilai dengan menggunakan kaedah perubahan berat dan teknik elektrokimia. Kajian ini meneliti pengaruh pelbagai kepekatan madu sebagai bahan penghalang terhadap perilaku karatan pada aluminium aloi 6061 dalam larutan asid. Dalam kaedah perubahan berat, sampel direndam dalam larutan selama 28 hari. Sampel di timbang sebelum didedahkan terhadap larutan dan dicuci menggunakan asid nitrik selepas perendaman. Perubahan berat selepas proses cucian dianalisis berpandukan kadar karatan. Pemeriksaan mikroskopik dilakukan dengan menggunakan mikroskop metalurgi. Potensiodinamik polarisasi digunakan dalam pengukuran elektrokimia dengan menggunakan potensiostat WonATech. Spesimen dilindungi dengan resin sebelum dijadikan sebagai elektrod kerja. Keputusan kajian menunjukkan bahawa penambahan madu asli mengurangkan kadar karatan dan menghalang hakisan daripada berlaku terhadap aluminium aloi 6061 dalam larutan 5% asid sulfurik. Keberkesanan penghalang meningkat dengan meningkatnya kepekatan madu bagi kedua-dua kaedah. Didapati bahawa, karatan berlaku secara seragam pada permukaan aluminium dalam larutan asid. Graf Tafel dari polarisasi potenciodinamik juga menunjukkan bahawa madu secara signifikan menurunkan voltan korosi (E_{corr}) dan ketumpatan arus (i_{corr}) serta meningkatkan liputan molekul pada permukaan. Penyerapan madu asli pada permukaan logam mematuhi serapan isotherm Langmuir.

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LIST OF SYMBOLS

mol L^{-1}	Amount of substance per liter
pH	Potential of hydrogen
Al_2O_3	Aluminum oxide
$\text{Al}(\text{OH})_3$	Hydroxide
AlOOH	Oxyhydroxide
Al^{3+}	Aluminum dissolves to 3 electron
H^+	Hydrogen dissolve to 1 electron
H_2O	Water
mm y^{-1}	Milimiter per year
$^{\circ}\text{C}$	Celcius
K	Kelvin
I_{eff}	Inhibition efficiency
R_0	Corrosion rate of metal without inhibitor presence
R_i	Corrosion rate of metal with inhibitor presence
-NH	Amine
-SH	Mercapto
-OH	Hydroxyl
-COOH	Carboxyl
-PO ₃	Phosphate
C	Carbon
O	Oxygen
H	Hydrogen
S	Sulphur

M	Molarity
Hz	Hertz
V	Volt
AC	Alternating current
E_{corr}	Corrosion potential
H_2SO_4	Sulphuric acid
RPM	Revolution per minute
CS	Cutting speed
D	Diameter
A	Area
d	Diameter of mounting hole
t	Thickness
ppm	Part per million
n	Corrosion rate a material in solution
K	Corrosion constant
W	Mass loss
T	Time of exposure
E	Potential
i	Current
b_a	Anodic beta tafel constant
b_c	Cathodic beta tafel constant
EW	Equivalent weight

LIST OF ABBREVIATIONS

Al	Aluminum
Al Mn	Aluminum manganese
Al Mg	Aluminum magnesium
Al Si	Aluminum silicon
ANSI	American national standard institute
ASTM	American society for testing and material
C-steel	Carbon steel
ed	Edition
EIS	Electrochemical impedance spectroscopy
Eoc	Open circuit potential
FTIR	Fourier transform infrared
FYP	Final year project
Inh	Inhibitor
LPR	Linear polarization resistance
NAVOSH	Navy occupational safety and health
No	Number
PP	Potentiodynamic polarization
SEM	Scanning electron microscope
sp gr	Specific gravity
UMP	Universiti Malaysia Pahang

CHAPTER 1

INTRODUCTION

1.1 PROJECT BACKGROUND

Aluminum alloys are alloys in which aluminum is the predominant metal. Typical alloying elements are copper, zinc, manganese, silicon, and magnesium. Aluminum alloys are widely used in engineering structures and components where light weight or corrosion resistance is required (Polymer, I. J. 1995). It's also important materials due to their high technological value and wide range of industrial applications, especially in aerospace, household industries, and commonly used in marine applications as well. In addition, they are justified by low price, high electrical capacity and high energy density.

Many researchers were devoted to study the corrosion of aluminum in different aqueous solutions, and research into their electrochemical behaviour and corrosion inhibition in wide variety of media. Corrosion is defined as the destruction or deterioration of a material because of reaction with environment and has been classified in many different ways such as wet corrosion and dry corrosion (Fontana, M.G. 1987). The most important feature of aluminum is its corrosion resistance due to the present of a thin, adherent and protective surface oxide film. This oxide film does not offer sufficient protection against aggressive anions. The solubility of oxide film is increases above and below pH 4.0–8.5 range and aluminum exhibits uniform attack (Yurt, A. et al. 2006).

The use of inhibitors (inorganic or organic) is the one of the most practical method for protection against corrosion. The inorganic inhibitors act as anodic

inhibitors while the organic inhibitors form protective film through the adsorption of their molecules on the metal surface being responsible for the corrosion resistance. Many researchers have found that a honey is good corrosion inhibitor for most of the metal in alkaline media solution. Based on the chemical contained in honey, it shows that natural honey should be good corrosion resistant for aluminum alloy refer to their organic compound containing in the polar group. For this project, natural honey was selected as inhibitor by different concentration to examine its ability to prevent corrosion of aluminum alloy in acidic solution.

1.2 PROBLEM STATEMENT

Corrosion inhibitors are widely used in industry to reduce the corrosion rate of aluminum and its alloys in contact with aggressive environment. Most of the corrosion inhibitors are synthetic chemicals, expensive and very hazardous to environment. Therefore, it needs to reduce its use to protect environmentally and human in the future. In other words, natural honey can be safe inhibitor. For example in food industrial, to protect corrosion by lime or orange juice (acidic solution) against beverage container made from aluminum, natural inhibitor shall be used instead of using chemical to protect corrosion to ensure consumer health guaranteed. Natural honey was examined and proved to have tremendous potential for industrial usage. Unlike the pure synthetic product that requires enormous investment scale, natural honey can be produced with smaller cost. As such, natural honey can be easily made available for general population, especially in the third world. Furthermore, the potential usage of natural honey discussed in this project, is in line with the recent trend of the environment-friendly concept.

1.3 SCOPES OF STUDY

The scopes of this study include:

- (i) Designing the sample preparation based on experiments.
- (ii) Cleaning procedure before and after exposure.
- (iii) Weighing sample before and after weight loss experiment.

- (iv) Exposure of sample with inhibitors fills in for 28 days.
- (v) Electrochemical testing using potentiodynamic polarization technique
- (vi) Surface examination using Metallurgical Microscope.

1.4 OBJECTIVE OF THE PROJECT

The objectives of this study are:

- (i) To study the honey as corrosion inhibitor
- (ii) To investigate the effects of variation concentration of honey as inhibitor to the corrosion behavior of 6061 Aluminum Alloy.

1.5 SUMMARY

Chapter 1 has been discussed briefly about project background, problem statement, objective and scope of the project on role play in experimental and analysis by determining corrosion rate, corrosion behavior and inhibitor efficiency to achieve the objective mentioned. This chapter is as a fundamental for the project and act as a guidelines for project research completion.

CHAPTER 2

LITERATURE RIVIEW

2.1 INTRODUCTION

This chapter focuses on research and collection data from various sources such as journals, books, website and others. Priority this chapter is to provide knowledge and understanding of the project before it begins. The data that was collected should be review whether it is appropriate to make references, so that this project can be carried out smoothly. Research should be conducted based on the facts and authenticity of a resource that has been recognized.

2.2 OVERVIEW OF CORROSION ENGINEERING

Corrosion Engineering is a specialist in disseminating knowledge, natural laws and physical resources in order to design and implement materials, structures, devices, systems and procedures to manage the natural phenomenon known as corrosion. Corrosion engineering groups have formed around the world in order to prevent, slow and safe manage the effects of corrosion on material. While many oxidation-reduction (redox) reactions are extremely important and beneficial to society (for example, those that are used to make batteries), the redox reactions involved in corrosion are destructive. In fact, close to \$200 billion dollars is spent in the United States each year to prevent or repair the damage done by corrosion to structures such as pipelines and bridges. Economically, one of the most important metals to corrode is iron and one of its alloys, steel. Almost 20% of the iron and steel produced in the United States each year is used to replace objects that have corroded. Therefore, corrosion engineers are very important to solve the problem.

2.3 DEFINITION OF CORROSION

Corrosion is defined as the destruction or deterioration of a material because of reaction with its environment. Some insist that the definition should be restricted to metals, but often corrosion engineering must consider both metals and non-metals for solution of a given problem. For example, deterioration of paint and rubber by sunlight or chemicals, fluxing of the lining of a steelmaking furnace, and attack of a solid metal by another molten metal (liquid metal corrosion) are all considered to be corrosion (Polymer, I. J. 1995). Corrosion of metals is the most common type of corrosion and is a process involving an exchange of electrons between two substances, one of them being the metal. In this process, the metal usually loses electrons, becoming oxidized, while the other substance gains electrons, becoming reduced. For this reason, corrosion is classified as an oxidation-reduction (redox) reaction.

2.4 ALUMINUM

Aluminum is an amphoteric metal where it can be used in the presence of water only because of its ability to form a protective layer of aluminum oxides. The potential-pH diagram in Figure 2.1 shows the aluminum alloy is stable only at low potentials. For Al^{3+} concentration of $10^{-6} \text{ mol L}^{-1}$, the stability region of aluminum oxide ranges between pH 4 and 8.5. In this range, aluminum can be successfully used for technical applications. When using potential-pH diagram, it is important to bear in mind that metal ion concentration rarely reach more than $10^{-6} \text{ mol L}^{-1}$ (Stratmann, M. and Frankel, G.S. (ed.). 2003).

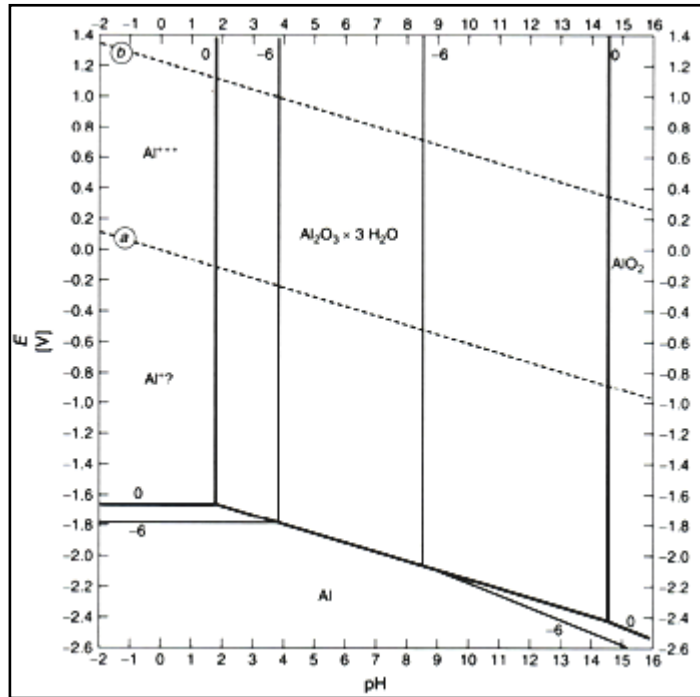


Figure 2.1: Potential-pH diagram of aluminum

Source: Stratmann, M. and Frankel, G.S. (ed.). 2003

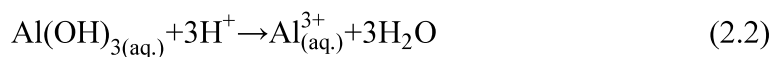
2.4.1 Corrosion of Aluminum Alloy in Acid

Aluminum and its alloys are not resistant in sulfuric acid solution since they are attacked to a greater or lesser degree, and to an increasing degree as temperature and concentration rise. The passive layer can consist of different modification of the oxide Al_2O_3 , hydroxide $Al(OH)_3$, or the oxyhydroxide $AlOOH$ (\rightarrow passivity).

The pH limits for the successful use of aluminum depend on various factors, for example, temperature, the specific oxide modification at the surface, and whether substances are present, which could form complexes or insoluble salt which aluminum. In acids, aluminum dissolves to Al^{3+} ions according to the following reaction:



The oxide layer dissolves and Al^{3+} ions are formed.



The use of aluminum in acids is very limited. Pure aluminum alloy shows the highest corrosion resistance and could be used at room temperature in up to 25% sulfuric acid, in which the dissolution rates is between 0.18 to 0.3 mm yr^{-1} . With increasing content of alloying components, the corrosion resistance can be seen in Figure 2.2.

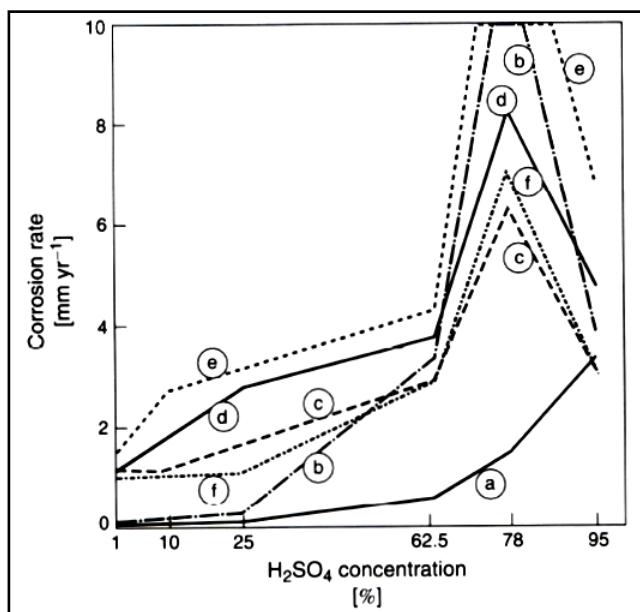


Figure 2.2: Corrosion of aluminum and aluminum alloy in sulfuric acid at 293 K

Source: Stratmann, M. and Frankel, G.S. (ed.). 2003

Refer to the figure 2.2, where:

- (i) Al 99.98 R
- (ii) Al 99.5
- (iii) Al Mn alloy
- (iv) Al Mg 3
- (v) Al Mg 7

(vi) AlSi 12

Nearly all acids attack aluminum, but concentrated nitric acid does not. In Figure 2.3, the corrosion rate of aluminum in nitric acid of difference concentrations and temperatures in the region of 0-30°C is shown. The corrosion rate increases with decreasing acid concentration. Increasing flow velocity of the medium increases the corrosion rate, especially if it is two-phase flow.

In sulfuric acid, chromic acid, or mixtures of the two, the thickness of the oxide layer can be increased by anodic polarization. Homogenous dissolution of aluminum is promoted and oxides are formed at the surface. The rate of dissolution of the oxides is lower than the formation of fresh oxide, the layers that are formed are very homogenous. This anodizing process produces protective layers, which increase the corrosion resistance. A further improvement of the corrosion resistance against atmospheric corrosion could be realized by water vapor oxidation (Stratmann, M. and Frankel, G.S. (ed.). 2003).

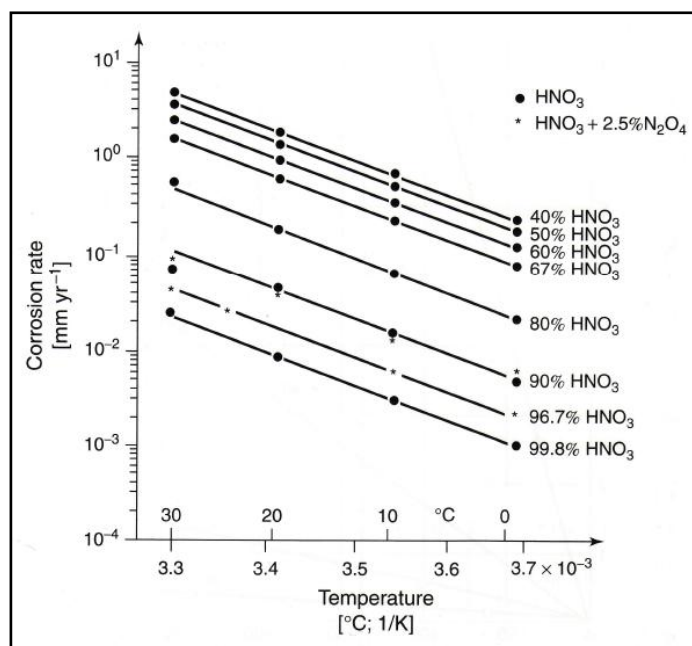


Figure 2.3: Corrosion of Al 99.5 in static nitric acid

Source: Stratmann, M. and Frankel, G.S. (ed.). 2003

2.4.2 Types of Corrosion on Aluminum

Different types of corrosion, more or less visible to the naked eye, can occur on aluminum, such as uniform (generalized) corrosion, pitting corrosion, stress corrosion, etc. The predominant type of corrosion will depend on a certain number of factors that are intrinsic to the metal, the medium and the conditions of use. There are several types of corrosion on Aluminum such as (Vargel, C. 2004):

2.4.2.1 Uniform Corrosion

This type of corrosion develops as pits of very small diameter, in the order of a micrometer, and results in a uniform and continuous decrease in thickness over the entire surface area of the metal. With aluminum, this type of corrosion is observed especially in highly acidic or alkaline media, in which the solubility of the natural oxide film is high. The dissolution rate of the film is greater than its rate of formation. However the ratio of both rates can change over time. The dissolution rate can vary from a few micrometers per year up to a few micrometers per hour, depending on the nature of the acid or base.

2.4.2.2 Pitting Corrosion

This localized form of corrosion is characterized by the formation of irregularly shaped cavities on the surface of the metal. Their diameter and depth depend on several parameters related to the metal, the medium and service conditions. Aluminum is prone to pitting corrosion in media with a pH close to neutral, which basically covers all natural environments such as surface water, seawater, and moist air. Unlike other metals, corrosion of aluminum is always eye-catching because the corrosion pits are covered with white, voluminous and gelatinous pustules of alumina gel $\text{Al}(\text{OH})_3$. These pustules are much bigger than the underlying cavity. Pitting corrosion occurs when the metal is put into permanent or intermittent contact with aqueous media such as water, seawater, rain water, and humidity.

2.4.2.3 Exfoliation Corrosion

Exfoliation corrosion is a type of selective corrosion that propagates along a large number of planes running parallel to the direction of rolling or extrusion. Between these planes are very thin sheets of sound metal that are not attacked, but gradually pushed away by the swelling of corrosion products, peeling off like pages in a book and hence the term “exfoliation corrosion”. The metal will swell, which results in the spectacular aspect of this form of corrosion. This corrosion may be intercrystalline in 2000 and 5000 alloys exhibiting a long-grained structure parallel to the direction of transformation. It can also be found in 7000 alloys with or without copper. It results from the precipitation of parallel stripes of intermetallic phases such as Al_6Mn , $\text{Al}_{12}\text{CrMn}$, AlFeMn , etc., which are cathodic with respect to the solid solution, and between which there is an anodic zone depleted in Fe and Mn.

2.4.2.4 Filiform Corrosion

Filiform corrosion is specific to lacquered metal. This is mainly an alteration of surface appearance. The underlying metal only suffers a very superficial attack, not exceeding a depth of a few tens of microns. It develops as narrow filaments, about 0.1–0.5 mm wide and a few millimeters long, which propagate at the metal–lacquer interface. Swelling of corrosion products deforms the lacquer film and appears as very narrow wires which progress like mole tunnels underneath the lacquer film. Filiform corrosion always starts at coating defects, such as scratches, and weak points: beads, cut edges or holes. It can be seen after several years of service. Coil-coated strip in aluminum alloys is not prone to this type of corrosion.

2.4.2.5 Crevice Corrosion

Crevice corrosion is a localized corrosion in recesses such as overlapping zones for riveting, bolting or welding, zones under joints, and under various deposits (sand, slag, precipitates, etc.). These zones, also called crevices, are very tiny and difficult to access for the aqueous liquid that is covering the rest of the readily accessible surfaces. This type of corrosion is also known as deposit attack. As soon as an electrochemical

reaction occurs in this confined volume, the composition of the contained liquid will change. As a consequence, the dissolution potential becomes more electronegative, and the surface in the recess becomes anodic with respect to the rest of the structure.

2.4.2.6 Cavitation

Cavitation occurs when the hydrodynamic pressure exceeds the vapor pressure of a moving liquid. Gas bubbles form within the liquid, which thus becomes a two-phase system. These bubbles will be crushed against the metal surface at high speed, an attack that leads to cavities with rounded contours. This degradation is caused by the combination of a mechanical effect and corrosion of the metal. The natural oxide film is destroyed and the aluminum is attacked there is a competition between tearing off the film and reforming it. It is not possible to estimate the individual contributions of the mechanical effect and corrosion. Cavitation develops quickly within a few days, and even within a few hours.

2.4.2.7 Erosion

Corrosion by erosion occurs in moving media. This type of corrosion is related to the flow speed of the fluid. It leads to local thinning of the metal, which results in scratches, gullies, and undulations, which are always oriented in the same direction, namely the flow direction.

2.5 CORROSION INHIBITORS

A corrosion inhibitor is a chemical compound that is added to environment in order to reduce the rate of metal corrosion. The inhibition efficiency, I_{eff} indicates to what extent the corrosion rate is slow down by presence of the inhibitor:

$$I_{\text{eff}} = \frac{R_0 - R_i}{R_0} \times 100\% \quad (2.3)$$

where,

I_{eff} = efficiency of inhibitor, %

R_0 = corrosion rate of metal without inhibitor present;

R_i = corrosion rate of metal with inhibitor present,

R_0 and R_i can be determined by electrochemical and weight loss measurement.

2.5.1 Types of Inhibitors

There are several types of inhibitors available to the industries now. They are:

- (i) Volatile
- (ii) Passivating (Anodic)
- (iii) Precipitation
- (iv) Cathodic
- (v) Organic
- (vi) Inorganic
- (vii) Mixed

2.5.1.1 Volatile Inhibitors (Roberge, P.R. 2000)

This type of inhibitors is also known as vapor phase inhibitors. As corrosion can also occur in vapor environment, it is useful to carry corrosion inhibitors into the system, which then need to be, themselves, volatile. When the inhibitor molecules in the vapor come into contact with the surface of a metal, the adsorption of the inhibitor will occur. The moisture then hydrolyses it, hence protective ions may be released. These include species like amines and nitrites for inhibition of ferrous metal.

2.5.1.2 Passivating (Anodic) Inhibitors (Roberge, P.R. 2000)

Passivating inhibitors are anodic inhibitors. They cause the anodic curve of polarization to shift such that less current flows. They have the ability to passivate the metal surface. There are two categories of passivating inhibitors, namely oxidizing anions and non-oxidizing anions. Oxidizing anions such have the ability to

passivate metal in the absence of oxygen. Typical oxidizing anions are chromate, nitrite and nitrate. Non-oxidizing ones such as phosphate, tungstate and molybdate require oxygen to perform passivation. This kind of inhibitor is by far the most widely used and possesses higher efficiency than others. However, one major drawback of it is that in order to maintain sufficient passivation of the metal and thus providing sufficient inhibition, the concentration of the inhibitor must be kept well above a critical or minimum concentration. If the concentration is below the minimum value, it is likely that the metal, which is to be protected in to the first place, will suffer from localized corrosion such as pitting.

2.5.1.3 Precipitation Inhibitors (Roberge, P.R. 2000)

These inhibitors are often film-forming in nature, for instance silicates and phosphates. They are effective at blocking anodic and cathodic sites. They precipitate on the metal surface, forming a protective barrier. Hard water is rich in magnesium and calcium. When these salts precipitate on the metal surface, for example at the cathode where the pH is higher, they establish a protection layer on the metal. Film-forming type of inhibitors is often distinguished by two classes. The first one works by slowing down the corrosion without stopping it completely. The second ceases the attack completely. However the efficiency of this inhibitor depends on the pH value and saturation index. The saturation index is then in turn determined by the water composition and temperature.

2.5.1.4 Cathodic Inhibitors (Roberge, P.R. 2000 and Revie, R.W. and Uhlig, H.H. 2008)

Cathodic inhibitors reduce the rate of cathodic reaction namely oxygen reduction in near neutral environments and hydrogen evolution in acid solutions respectively. These form species that precipitate on cathodic sites and thus increase the surface impedance and lower the diffusion rate. The inhibiting action of cathodic inhibitors work by three mechanisms:

(i) Cathodic poisons

In this case, the cathodic reduction process is suppressed, for example by impeding the hydrogen recombination and discharge. But it may increase the tendency of the metal to be susceptible to hydrogen induced cracking. Hydrogen may absorb into the metal during this process. It is essential to keep hydrogen in atomic form. Typical types of cathodic poisons are arsenic and antimony.

(ii) Cathodic precipitates

Compounds such as calcium, magnesium will precipitate as oxides to form a protective layer which acts as a barrier on the metal surface.

(iii) Oxygen scavenger

This mechanism functions by the removal of oxygen in the system to reduce corrosion. The compounds of oxygen scavenger react with oxygen presenting the system to form a product.

2.5.1.5 Organic Inhibitors (Roberge, P.R. 2000)

This type of inhibitors is, too, film-forming in nature. They form a hydrophobic layer on the surface of the metal to prevent dissolution of metal. The efficiency of this inhibitor depends upon the chemical composition and molecular structure of the inhibitor as well as their affinity with the metal. They are used often when environmental issues are taken into account. They are classified into organic anions and cations. Inhibitors like sulphonates and phosphonates fall into the anions category. Chemicals with active groups such as aliphatic and aromatic and positively charged amine groups are organic cations. This type of organic inhibitor is often in the form of liquid or wax-like.

2.5.1.6 Inorganic Inhibitors (Revie, R.W. and Uhlig, H.H. 2008)

The common inorganic inhibitors used are crystalline salts, for instance, sodium chromate and molybdate. The only active groups of these compounds that functions to reduce corrosion are the negative anions they carry.

2.5.1.7 Mixed Inhibitors (Revie, R.W. and Uhlig, H.H. 2008)

Corrosion inhibitors are rarely used as a single compound only. The formulation can be composed of 2 or more inhibitors which all carry different characters. This is due to three factors:

- (i) A single inhibitor can only inhibit a few numbers of metals. When the environments involve multi-metal system, the inhibitive action may sometimes cause jeopardizing effects to other metals.
- (ii) Advantages from anodic and cathodic inhibitors can be combined and optimised for best performance.
- (iii) Addition of halide ions improves the action of organic inhibitor in acid solutions.

2.5.2 Inhibitor for Acid Environment

A large number of organic substances, most often aromatic compound or macromolecules with linear or branched chains, serve as corrosion inhibitors for acid environment. These inhibitors adsorb onto the surface of the metal and thus slow of the rate of corrosion and in some cases film formation can occur. The inhibition efficiency in a given application depends on the molecular structure of their inhibitor and on their concentration (Landolt, D. 2007).

An organic inhibitor molecule typically includes a hydrophobic non-polar part that contain mostly carbon and hydrogen atom, and one or several hydrophilic functional group such as $-NH_2$ (amine), $-SH$ (mercapto), $-OH$ (hydroxyl), $-COOH$ (carboxyl), $-PO_3$ (phosphate) or their derivative. The polar functional groups permit

dissolution of the inhibitor in water and link with the inhibitor molecule to the metal surface. The non-polar part, which takes up a much greater volume, partially blocks the active surface area such as Figure 2.4.

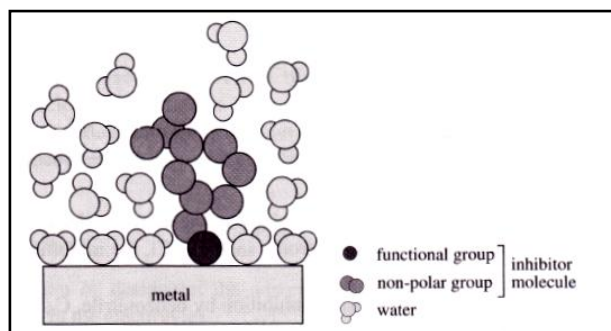


Figure 2.4: Adsorption of an organic compound onto metal surface in aqueous environment

Source: Landolt, D. 2007

Inhibitor for acid environments generally chemisorb on the surface. The effectiveness of the inhibitor therefore is the better, the greater tendency of the functional groups to form bond with the metal by acting as electron donors. For organic molecules that differ only by their functional groups, the inhibition efficiency varies inversely with the electronegativity of the functional atoms. It increases in the following order:



The molecular structure of the non-polar part can also influence the ability of the functional atoms to donate electrons. By placing a methyl group, $-\text{CH}_3$, at the 3 or 4 position of pyridine molecule, the electron density at the nitrogen atom is increased, thus promoting a bond with the metal, the reason being that methyl group repels electrons (nucleophilic group). The inhibition efficiency of pyridine derivatives thus increases in the following order such as Figure 2.5.

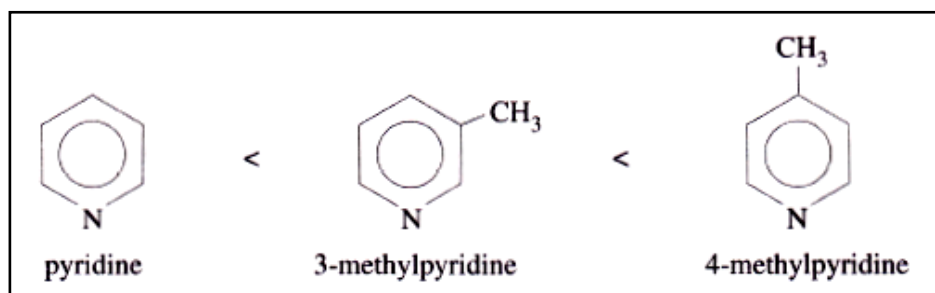


Figure 2.5: Reaction of electrons in pyridine molecule

Source: Landolt, D. 2007

If, on the other hand, instead of a methyl group the above molecules contain an electrophilic substituent that attracts electrons, such as $-\text{Cl}$, the inhibition efficiency decreases (Landolt, D. 2007).

2.5.3 Honey as Inhibitor

Honey is a natural sweet substance produced by bees from the nectar of blossoms or from the secretions of living parts of plants. Besides that, honey can also be produced from excretions which bees collect, transform and combine with specific substances of their own, store and leave in the honey comb to ripen and mature.

Chemical composition of the honey shows differences depending on many factors. The most important of these factors is the natural combination of the nectar and secretion. Water content in honeys is important only for determining the quality and fermentation and it should be less than 20%. Besides, temperature and humidity where honey is stored can cause in the moisture content of honey.

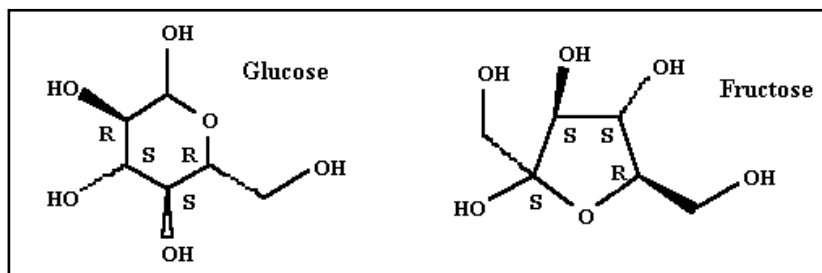


Figure 2.6: Fructose and Glucose composition in honey

Source: Loveridge, J. 2001

Basic composition of honey is carbohydrate is about 82%. Fructose and sucrose are most commonly found and there are generally around 38.2% fructose and 31% sucrose such as Figure 2.6. There are also some oligosaccharides present (4.2%), including erlose, theanderose and panose, formed from incomplete breakdown of the higher saccharides present in nectar and honeydew. Honey also contains organic acids such as acetic, butanoic, formic, citric, succinic, lactic, malic, pyroglutamic and gluconic acids, and a number of aromatic acids shown in Figure 2.7. The main acid present is gluconic acid, formed in the breakdown of glucose by glucose oxidase. Honey also contains hydroxymethylfurfural, a natural product of the breakdown of simple sugars below pH 5.

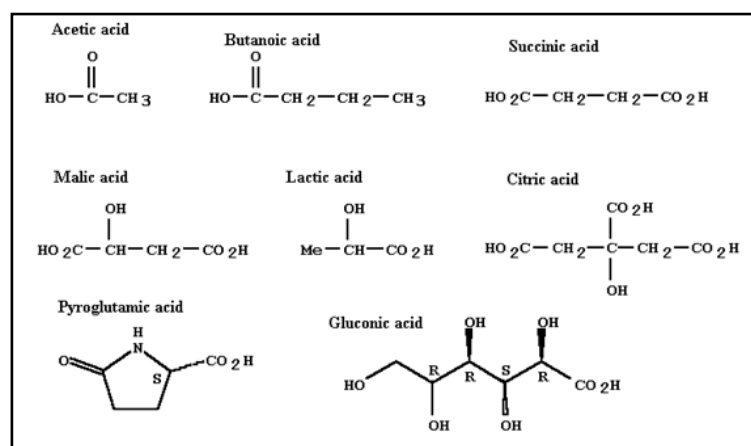


Figure 2.7: Some acid in honey

Source: Loveridge, J. 2001

The inhibition performances of honey could be explained as follows FTIR spectrum in Figure 2.8 and it was demonstrates that honey is a mixture of various compounds containing carbon (C), oxygen (polyphenols), nitrogen and sulphur (glucosinolates) which all can be adsorbed on the corroded metal. The bands at about 1055.3 cm^{-1} and 1418.1 cm^{-1} consists of C, O, H and N atoms, meanwhile the peak at 1255.6 cm^{-1} is due to sulphur (S). A band appearing near 2935.7 cm^{-1} proves the existence of C, O and H atoms in natural honey. A band located at 3355.1 cm^{-1} corresponds to O, N and H atoms. The adsorption of natural honey onto the surface of aluminum alloy may take place through all these functional groups. The simultaneous adsorption of the four functional groups forces the natural honey molecule to be horizontally oriented on the surface of aluminum alloy. (R. Rosliza et al, 2009).

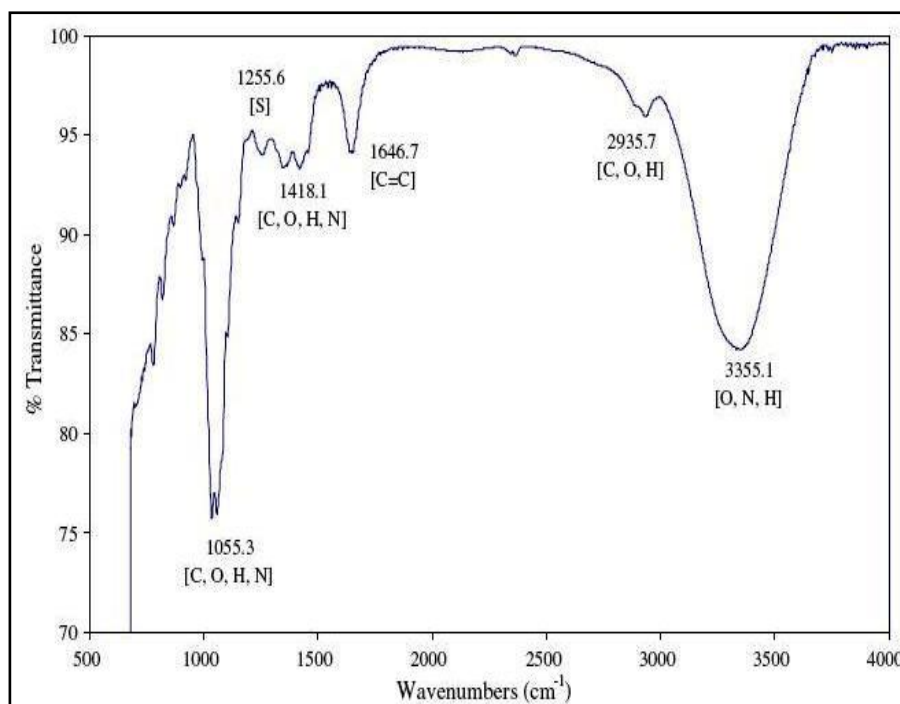


Figure 2.8: Fourier transform infrared (FTIR) spectrum of natural honey

Source: R.Rosliza et al, 2009

2.6 PREVIOUS RESEARCH

2.6.1 The Effect of Inhibitor on the Corrosion of Aluminum Alloys in Acidic Solutions (R.Rosliza et al, 2008)

The corrosion inhibitions of aluminum alloys in acidic solutions were evaluated by using weight loss, polarization and electrochemical spectroscopic (EIS) measurements. The selected aluminum alloys AA6061 and AA6063 were employed for this study to investigate corrosion effect in sulfuric acid 0.1M and acetic acid 0.5M with inhibitor used was sodium benzoate (NaBz).

In weight loss experiment, the material was cut into small samples and mechanical polished before exposure. They were weighed for the original then hung in test solution for 30 days. After 30 days, the corroded samples were removed from the solutions, and then were cleaned to remove corrosion products. The cleaned samples were weighed again in order to measure the mass loss before and after exposure. The results showed that the additional of sodium benzoate in various solution concentrations changed the corrosion rates of aluminum alloys in acidic solution. The inhibition efficiency increases with the increase the immersion time in acetic acid however it displays a different behavior in sulfuric acid.

Due to electrochemical experiment, the potentiodynamic current-potential curves were recorded by changing the electrode potential automatically from -100 mV to +100 mV with the scanning rate of 10 mVs⁻¹. Electrochemical impedance spectroscopy (EIS) measurements were carried out using AC signal of impedance measurements and were conducted at the corrosion potential with a frequency range of 10³ Hz to 10⁻¹ using 10 mV amplitude of sinusoidal voltage and 10 mVmin⁻¹ of scanning rate. Electrochemical studies showed that there was significant increase in overall resistance after addition of sodium benzoate, when compared to the case without inhibitor. Langmuir adsorption isotherm fits well with experimental data.

2.6.2 Anti-corrosive Properties of Natural Honey on Al-Mg-Si Alloy in Seawater (R.Rosliza et al, 2009)

The experiments were performed to evaluate anti-corrosive properties of natural honey on Al-Mg-Si alloy in seawater that was collected from Pantai Telok Kalong, Kemaman, Terengganu in the port area. Electrochemical measurement was selected by evaluated potentiodynamic polarization (PP), linear polarization resistance (LPR) and electrochemical impedance spectroscopy (EIS) measurements. The scanning electron microscope (SEM) was used to determine differences of surface morphology before and after immersed in seawater. The corrosion resistance used was natural honey collected freshly from the nest of bees with the concentration range from 200 to 1000 ppm.

The samples for each concentration were immersed in a 100 mL beaker containing the respective solution for 30 min. All electrochemical measurements were obtained using Autolab frequency response analyzer (FRA) coupled to an Autolab potentiostat connected to a computer. The potentiodynamic current-potential curves were recorded by changing the electrode potential automatically from -250 mV to $+250$ mV related to the open circuit potential with the scanning rate of 10 mVs⁻¹. Since the LPR measurements were carried out from -10 mV to $+10$ mV versus E_{corr} at scan rate of 1 mVs⁻¹. Electrochemical impedance spectroscopy (EIS) measurements were carried out using AC signal of impedance measurements and were conducted at the corrosion potential with a frequency range of 5×10^5 Hz down to 5×10^3 .

Polarization data indicated that natural honey is a mixed-type corrosion resistant. LPR and EIS studies showed that there were significant increases in the overall resistance after the addition of natural honey. The adsorption of natural honey on the metal surface obeys Langmuir's adsorption isotherm. The analysis of morphology studies confirmed the formation of precipitates of natural honey on the metal surface, which reduced the overall corrosion reaction.

2.6.3 Natural Honey and Black Radish Juice as Tin Corrosion Inhibitors (Radojic, I et al, 2008)

The influence of natural honey (chestnut and acacia) and natural honey with black radish juice, on corrosion of tin in aqueous and sodium chloride solutions was evaluated by using weight loss method and polarization techniques. The two monofloral honey types used in experiments are dark-colored acacia honey and light-colored acacia honey. The 3% NaCl solution was used with addition concentration of honey from 200 ppm to 1000 ppm. The another solution is black radish juice was mixed with honey and dissolved in 3% NaCl solution to prepare solution concentration from 200 ppm to 1000 ppm.

The inhibition efficiency of acacia honey was reported lower than that of chestnut honey, while the addition of black radish juice increased the inhibition efficiency of both honey varieties. The process of inhibition was attributed to the formation of multilayer adsorbed film on the tin surface. The adsorption of natural honey and honey with black radish on tin was found to follow the Langmuir's adsorption isotherm.

2.6.4 Natural Honey as Corrosion Inhibitor for Metal and Alloys. II. C-Steel in High Saline Water (Et-Etre, A.Y. and Abdallah, M. 2000)

The inhibitive action of natural honey on the corrosion of C-steel, which used in manufacturer of petroleum pipelines in high saline water was evaluated by using weight loss measurements and potentiostatic polarization technique. The Carbon steel of type A-106 was used with surface area of 1.0 cm^2 in weight loss experiment. The experiments were performed in water that obtained from the oil field (lower Baharya) which related to (Qarun) Petroleum Company. The inhibitors used in this study were natural honey from citrus, trefoil and cotton flowers.

It was found that, natural honey exhibited a very good performance as inhibitor for steel corrosion in high saline water. The inhibition efficiency increases with an increase in natural honey concentration. After some time, the inhibition efficiency decreased due to the growth of fungi in the medium. The adsorption of natural honey on the C-steel was found to follow the Langmuir adsorption isotherm.

CHAPTER 3

METHODOLOGY

3.1 INTRODUCTION

This chapter described the methods that were used to measure the efficiency of natural honey as inhibitor for 6061 Aluminum Alloy in H_2SO_4 solution. In corrosion testing, the methods commonly used to measure the corrosion rate are weight loss method and electrochemical technique. All procedures to conduct both experimental were based on American Society for Testing and Material (ASTM) standard for metal corrosion.

The procedures included in this study are sample's preparation, experimental setup, result evaluation and calculation as well as reporting the corrosion rates. The sample, 6061 Aluminum Alloy was weighed by using micro weighing scale before and after weight loss experiment to calculate the corrosion rate and the duration to immerse the samples in acidic solution is 28 days. The differential weight before and after immersed in H_2SO_4 solution were collected to measure the efficiency of inhibitor. Metallurgical microscope was used to examine the micro surface before and after immersed in acidic solution.

The other method to investigate the corrosion rate is electrochemical technique. This was obtained by using potentiostat/ galvanostat electronic instrument (model WPG 100i from WonATech Co. Ltd) and was connected to the computer. In electrochemical technique, corrosion rates and corrosion behavior can be easily determined by using IVMAN software. It was measured by changing the electrical currents or voltages to react with chemical solution and transfer the electrons.

3.2 WEIGHT LOSS METHOD

The experimental flow chart below is a visual representation of the sequence of the weight loss method. A completed flowchart organizes the topic and strategies done to ensure smooth working flow of this experiment. Figure 3.1 illustrated a simple flow chart shows the flow processes of this experiment. As illustrated, the first step is material preparation that involve cutting and cleaning process and this experiment go through to the others step such as experiment setup, cleaning after experiment and lastly corrosion analysis.

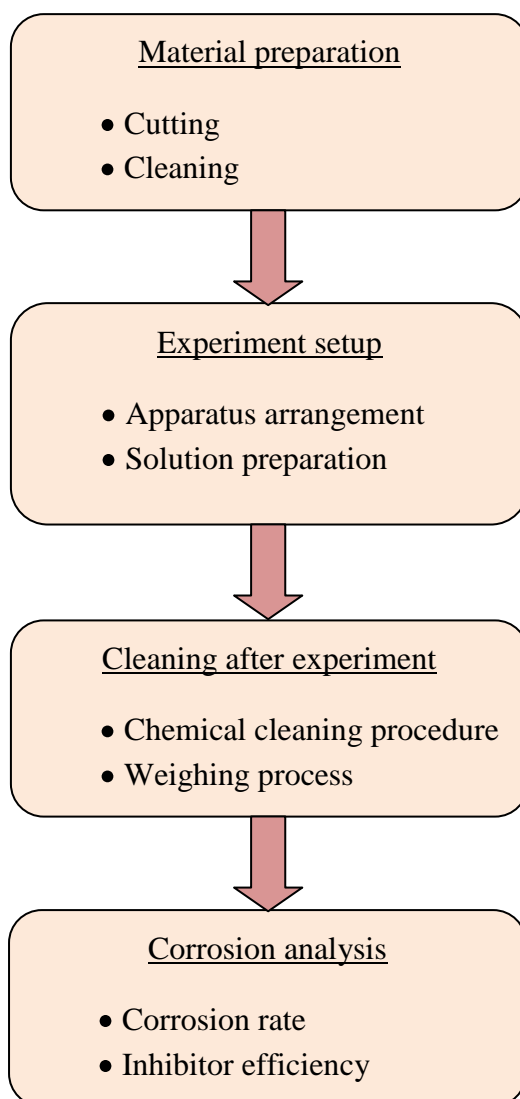


Figure 3.1: Experimental procedure for weight loss method

3.2.1 Samples Preparation

This experiment used six samples with same dimension for all concentrations of inhibitor from 0 ppm to 1000 ppm. Round bar with 32 mm diameter of 6061 Aluminum Alloy was cut into small sample is about 30 mm diameter and 5 mm thickness. Lathe machine was used to reduce the diameter to be 30 mm such as Figure 3.2. The feed rate and cutting speed were set up to 0.004 mm/rev and 2820 feet/minute respectively to ensure the surface roughness to be smooth. Both parameters were set up based on carbide cutting tool that was used and the rotational spindle speed is to be 955.04 revolution per minute, calculated by following equation:

$$RPM = \frac{CS \times 4}{D} \quad (3.1)$$

where:

RPM = revolution per minute

CS = cutting speed in feet/minute

D = diameter of part in inch

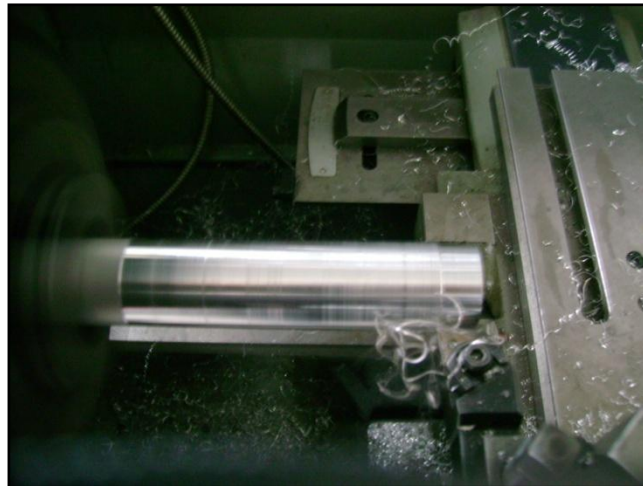


Figure 3.2: Turning process

Sectioning cut-off machine shown in Figure 3.3 was used after finished turning process to make 5 mm thickness for all samples. The thickness 5 mm was measured

carefully before it is clamped and the rotational cutting tool is 3000 rev/min was set up based on sample's material. The pressure coolant was generated and placed near to the cutting tool before it started. The cutting process was moving slowly guiding by rotational hand spindle to avoid the cutting tool from damage and make the cutting surface became smooth.



Figure 3.3: Sectioning cut-off machine

Then, all samples were drilled 2.5 mm diameter hole at the side of the surface samples to be hanged by nylon before immersed in acid sulfuric solution such as Figure 3.4. The selection of drilling size was based on standard from American National Standard Institute (ANSI). The total surface area of a circular sample is 1914.41 mm given by following equation:

$$A = \frac{\pi}{2} (D^2 - d^2) + t\pi D + t\pi d \quad (3.2)$$

where:

t = thickness of sample,

D = diameter of sample, and

d = diameter of mounting hole.

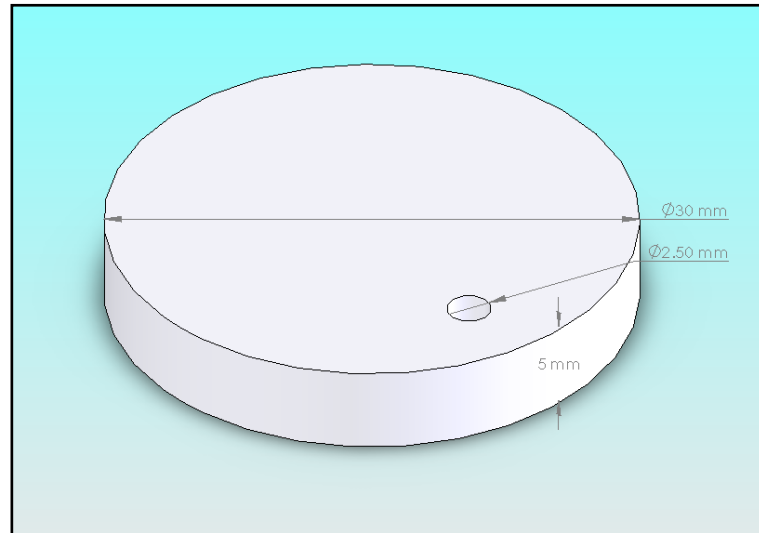


Figure 3.4: Dimension of sample

After that, the samples were mechanically grinded using 320 followed by 400 and 600 grit size emery papers and lubricated using distilled water to clean the surface of samples from any scratches. The polishing process was operated by using polishing machine shown in Figure 3.5 and the sequences operation was based on the following Table 3.1. The samples were cleaned with acetone and washed using distilled water. Then it's were dried using air compressor and the result show the samples became smooth and shining such as Figure 3.6. It was stored in desiccators until exposure and finally was weighed using micro weighing scale shown in Figure 3.7.



Figure 3.5: Polishing machine

Table 3.1: Polishing process

SiC Grit Size	Time (sec)	Wheel Speed (RPM)	Pressure (psi)
1 micron diamond compound/ red felt cloth/ microid extender	300	250	30
Colloidal silica/ Imperial cloth (wetted)	60	150	15

**Figure 3.6:** The samples after polishing process**Figure 3.7:** Weighing before exposure

3.2.2 Experiment Setup

The apparatus arrangement needed for this experiment should be follow ASTM standard G 31 (Laboratory immersion corrosion testing of metal) as shown in Figure 3.8. A glass reaction beaker has size more than 1000 mL was used in this experiment to prevent the glass react with the chemical solution. The purpose of opening in flask is to provide ventilation cycle required in corrosion process. The beaker shall be closed to avoid the solution from evaporation and contamination.

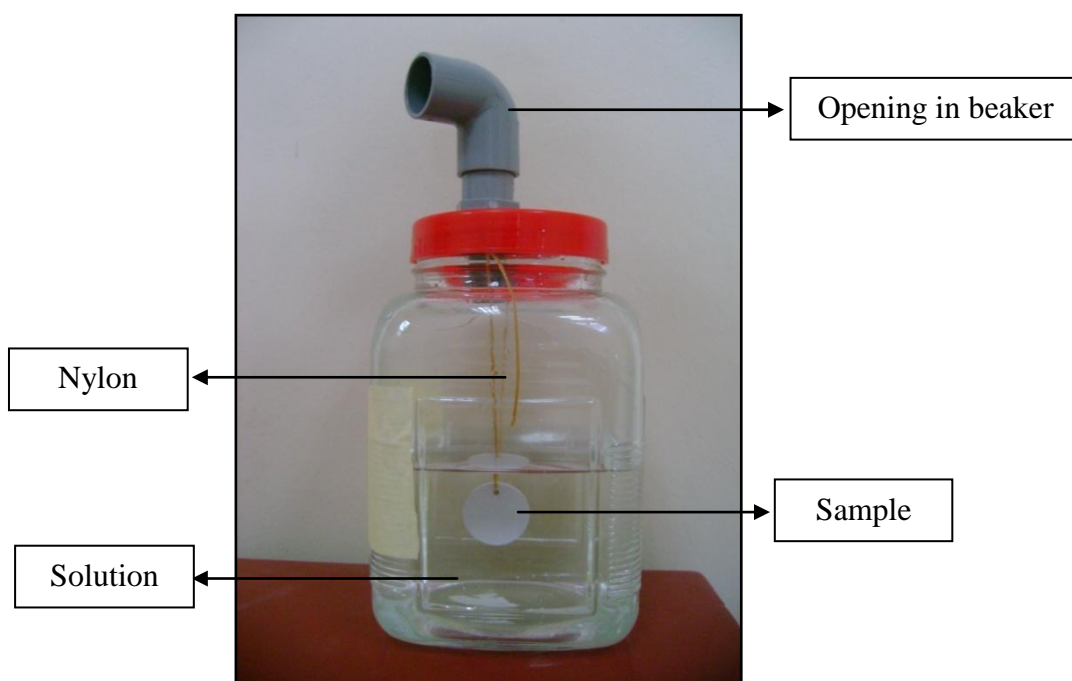


Figure 3.8: Apparatus arrangement

This experiment were divided into six and one of the experiment was used as reference experiment without inhibitor and other experiments run with inhibitor addition refer to the concentration of inhibitors that were produced from 200 ppm to 1000 ppm concentration. All experiments were used 5% H_2SO_4 concentration and conducted in room temperature (25°C). The volume of the test solution should be large enough to avoid any appreciable change in its corrosivity during the experiment, either through exhaustion of corrosive constituents or by accumulation of corrosion products that might affect further corrosion. The minimum solution volumes to sample area ratio are

0.2 mL/mm² and 0.4 mL/mm² of surface. Preparations of solutions were based on the following Table 3.2 below and shown in Figure 3.9:

Table 3.2: Solution preparation

Experiment no.	Inhibitor concentration	Quantity of Honey	Remarks
1	Without inhibitor	0 mg	
2	200 ppm	200 mg	Mixed with 1000 mL H ₂ SO ₄ 5% concentration
3	400 ppm	400 mg	
4	600 ppm	600 mg	
5	800 ppm	800 mg	
6	1000 ppm	1000 mg	



Figure 3.9: Solution preparation

The prepared solution is then was evaluated by using pH meter as shown in Figure 3.10. In order to show the effectiveness inhibitors, the pH value of all solution with and without addition of natural honey was determined to make sure the acidic behavior of H₂SO₄ solution with inhibitor presence doesn't changed too much with the range of 5% H₂SO₄ solution. The other reasons are to know the type of acidic solution either high acidic media, medium acidic media or low acidic media.

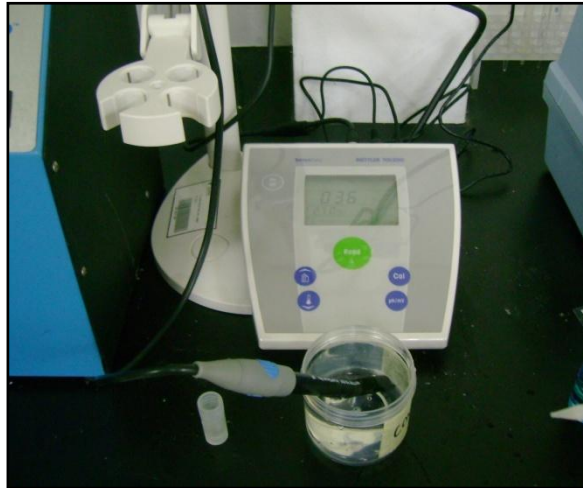


Figure 3.10: Solution testing using pH meter

The samples were mounted into nylon and fully immersed in solution as shown in Figure 3.8. The beaker should be enclosed and placed in constant room temperature around 25°C and away from interference as shown in Figure 3.11. The experimental were conducted for 28 days depending on corrosivity occur on the samples. The calculation for minimum period to immerse the sample in solution as following this equation:

$$\text{Hours} = \frac{2000}{n} \quad (3.3)$$

where:

n = (mpy) mils per year for corrosion rate a material in solution.



Figure 3.11: Weight loss experimental

3.2.3 Cleaning Samples After Experiments

Generally, the cleaning procedure after the experimental should remove all corrosion products from samples with minimum removal of sound metal. The cleaning methods should be follow ASTM standard G1 (preparing, cleaning, and evaluating corrosion test samples). The procedure of cleaning samples after experimental were shown in Table 3.4 and Figure 3.12.

Table 3.3: Chemical cleaning procedures for removal of corrosion products

No.	Solution	Time	Temperature	Remarks
1	<ul style="list-style-type: none"> • 50 ml phosphoric acid (H_3PO_4, sp gr 1.69) • 20 g chromium trioxide (CrO_3) • Reagent water to make 1000 ml 	5 to 10 minute	90° to boiling	If the corrosion product film remain, rinse then follow with nitric acid procedure (No.2)
2	<ul style="list-style-type: none"> • Nitric acid (HNO_3, sp gr 1.42) 	1 to 5 minute	20° to 25°C	Remove extraneous deposits and bulky corrosion products to avoid reactions that may result in excessive removal of base metal.

Source: ASTM G 1



Figure 3.12: Cleaning process via Nitric acid by light brushing

The cleaning procedure should be repeated on samples several times and determined the weight loss after each cleaning by weighing the samples. The accurate values of weight loss were determined by chosen the lowest slope of BC as shown in Figure 3.13. At this point, the value of weight loss will remain constant and it show that the corroded sample was successfully eliminated.

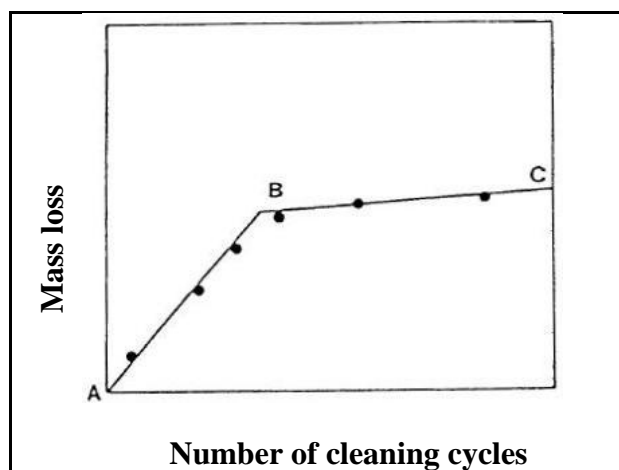


Figure 3.13: Mass of corroded samples resulting from repetitive cleaning cycle

Source: ASTM G 1

3.2.4 Metallurgical Microscope Procedure

The metallurgical microscope, often referred to as the light microscope, is a type of microscope which uses visible light and a system of lenses to magnify images of small samples. The surface samples can be captured in micrometer or nanometer depending on the power of lens and equipment.

The procedures required for Metallurgical microscope are:

- (i) Samples preparation
- (ii) Magnification range expected to be the most effective
- (iii) Depth of focus needed
- (iv) Choose working distance, aperture size, lens power



Figure 3.14: Metallurgical microscope

3.2.5 Experimental Analysis

After corroded samples were cleaned, the corrosion rate of every sample can be calculated. The samples should be weighing before and after experimental and initial

total surface area are determined. The average corrosion rate may then be obtained as follows:

$$\text{corrosion rate} = \frac{K \times W}{A \times T \times D} \quad (3.4)$$

where:

$K = 3.45 \times 10^6$ mils per year (mpy)

W = mass loss in grams (g)

A = total area in cm^2

T = time of exposure in hours

$D = 2.70 \text{ g/cm}^3$ for 6061 Aluminum Alloy

The corrosion rate for reference experiment (without inhibitor) is used to compare with the others experiment with inhibitor at various concentrations. The inhibitor efficiency can be expressed as below:

$$I_{\text{eff}} = \frac{R_0 - R_i}{R_0} \times 100\% \quad (3.5)$$

where:

I_{eff} = efficiency of inhibitor, %

R_0 = corrosion rate of metal without inhibitor present;

R_i = corrosion rate of metal with inhibitor present,

3.3 ELECTROCHEMICAL TECHNIQUE

Electrochemical technique is another way to measure the corrosion rate of 6061 Aluminum Alloy based on their ability to give a chemical reaction involving the transfer of electrons. It is also a chemical reaction which involves oxidation and reduction. This method more focuses on computerized calculation and analysis. The general procedure for this method was shown in Figure 3.15 below:

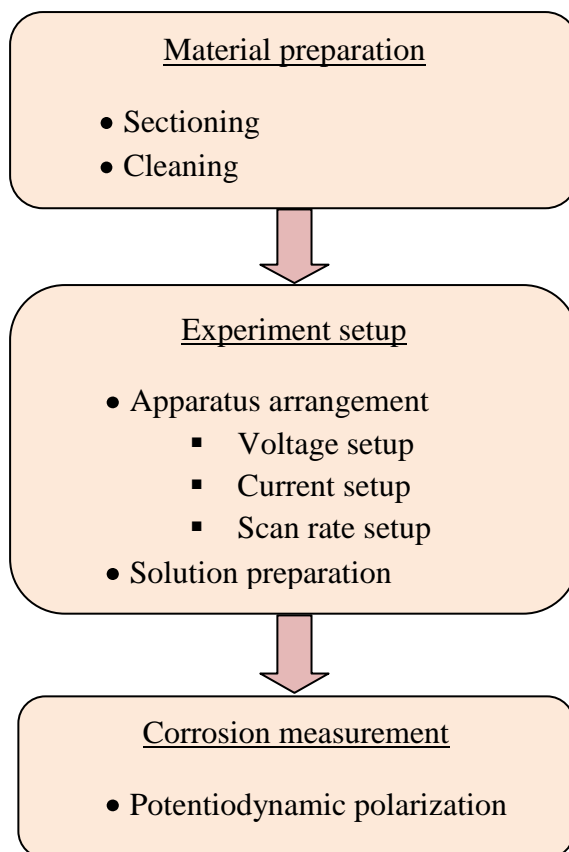


Figure 3.15: Electrochemical experiment flow chart

3.3.1 Samples Preparation

Samples preparation for electrochemical technique were based on ASTM standard G1 (preparing, cleaning and evaluating corrosion test samples) and type of material is 6061 Aluminum Alloy. Six difference experiments were performed in this method refer to inhibitor concentration and every experiments were used different sample but form of each samples used are same as shown in Figure 3.16 below:

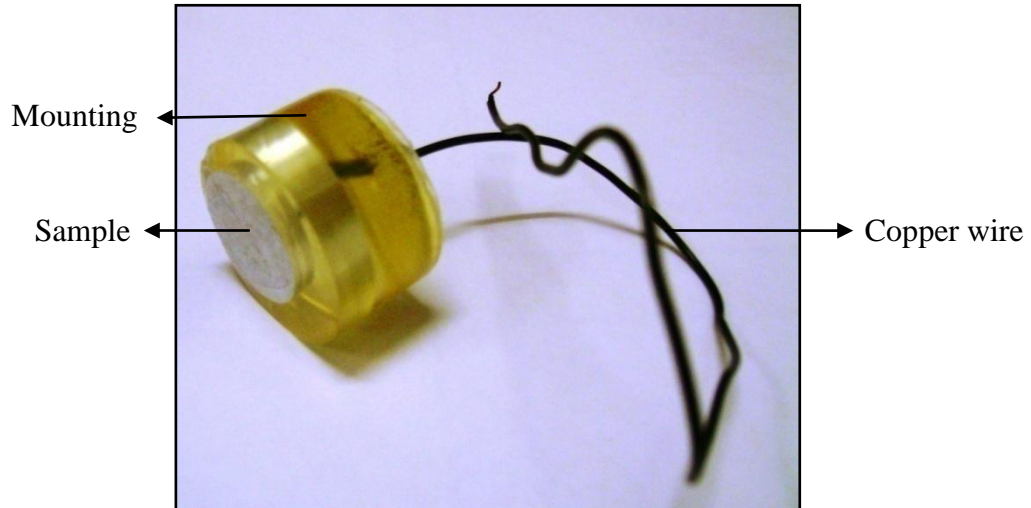


Figure 3.16: Mounted sample

The round bar of 6061 Aluminum Alloy was cut into small samples same as procedure to prepare samples for weight loss method. Then the small samples were mounted by using Cold Mounting machine around 20 minutes with the ratio of powder transparent and liquid are 1:1 respectively. After finished the process, all samples were through the cleaning process as listed below:

- (i) Mechanically polished using 320, 400 and 600 emery paper.
- (ii) Rinse with distilled water
- (iii) Polishing process (same as before).
- (iv) Dried in air and stored in desiccators before use

The total surface area of samples was collected for purpose in experimental. The total surface area of sample is 3.142 cm^2 by following this equation:

$$A_T = \frac{\pi D^2}{4} \quad (3.6)$$

where:

D = diameter of sample.

3.3.2 Experiment Setup

Apparatus this experiment was developed and setup by WonATech Co. Ltd and was connected to computer. The samples were installed as working electrode and immersed in sulfuric acid solution shown in Figure 3.17. The other electrodes are Graphite Counter electrode and Saturated Calomel electrode (reference electrode). The reaction occur from working electrode to counter electrode is used to measure current and reaction between reference to working electrode is to apply potential. The end of lugging capillary shall be 1 mm closed to the sample. It was used to position the sensing point of a reference electrode to a desired point in a cell.

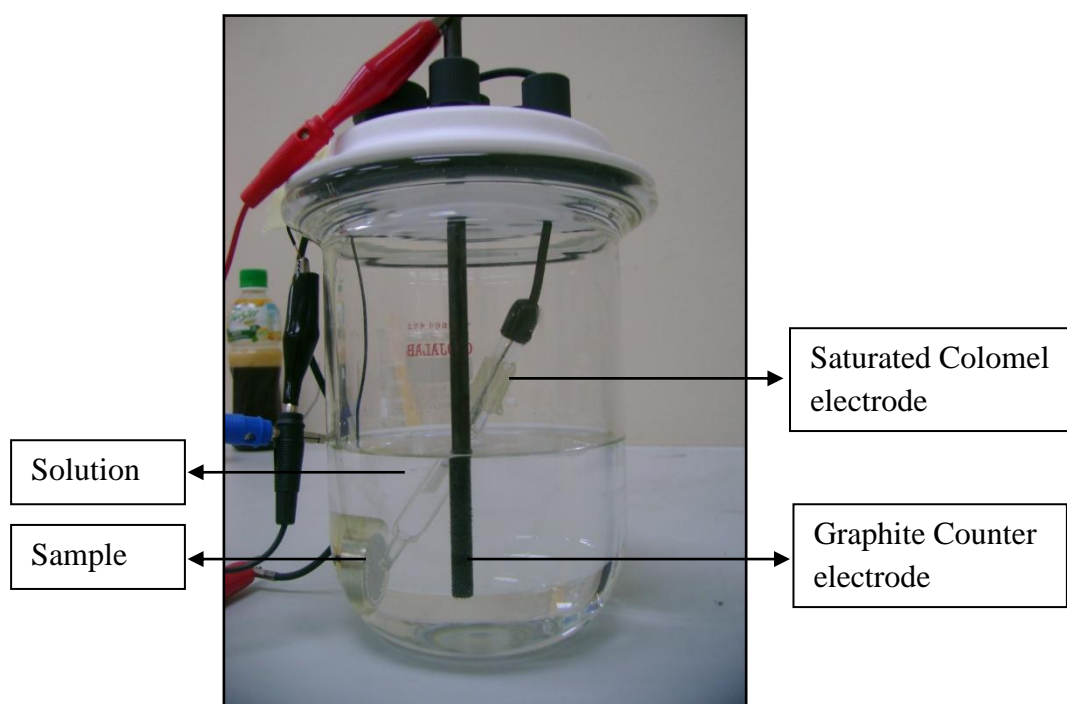


Figure 3.17: Apparatus arrangement

Electrochemical technique was divided into six types of experiment and the first experiment represented the solution without inhibitor and the rest were based on inhibitor concentration from 200 ppm to 1000 ppm. The main solution was used is 5% H_2SO_4 concentration for whole experiments. All experiments were conducted in constant room temperature (25°C). The preparation of solution is same as before (weight loss method) and based on Table 3.2.

3.3.3 Corrosion Measurement

Electrochemical corrosion rates measurements always provide results in term of electrical current. It is simple and fast method to determine the corrosion rates of metal. It also provides better result based on their ability to conduct electron and react with chemical involves oxidation and reduction. The flows of electrons (current) in the corrosion reaction can be measured and controlled electronically. In testing practice, a polarization cell is setup consisting of an electrolyte solution, a reference electrode, a counter electrode and the metal sample of interest connected to a sample holder. An electrochemical potential (voltage) is generated between the various electrodes and the corrosion potential E_{corr} is measured by potentiostat as an energy difference between the working electrode and the reference electrode. In electrochemical technique, corrosion rate can be measure by using potentiodynamic polarization with transfer the results in IVMAN software. This technique also generated tafel graph in order to calculate the corrosion rates. The parameters resulting from this technique were related each other and were used in analysis and calculation to determine the corrosion rate and corrosion behavior.

3.3.3.1 Potentiodynamic Polarization

A potentiodynamic scan is a qualitative experiment to assess the passivation tendencies of a metal sample. The relationship between current and potential at an electrode interface can be probed either controlling the potential and measuring the current or by controlling the current and measuring the potential (Stratmann, M. and Frankel, G.S. (ed.). 2003). In practice, many metals form an oxide layer on their surface as they corrode. Passivate on the metal occur when oxide layer inhibits further corrosion. In some cases, local area of passive film break down allowing significant metal corrosion to occur in a small area and it's called pitting corrosion.

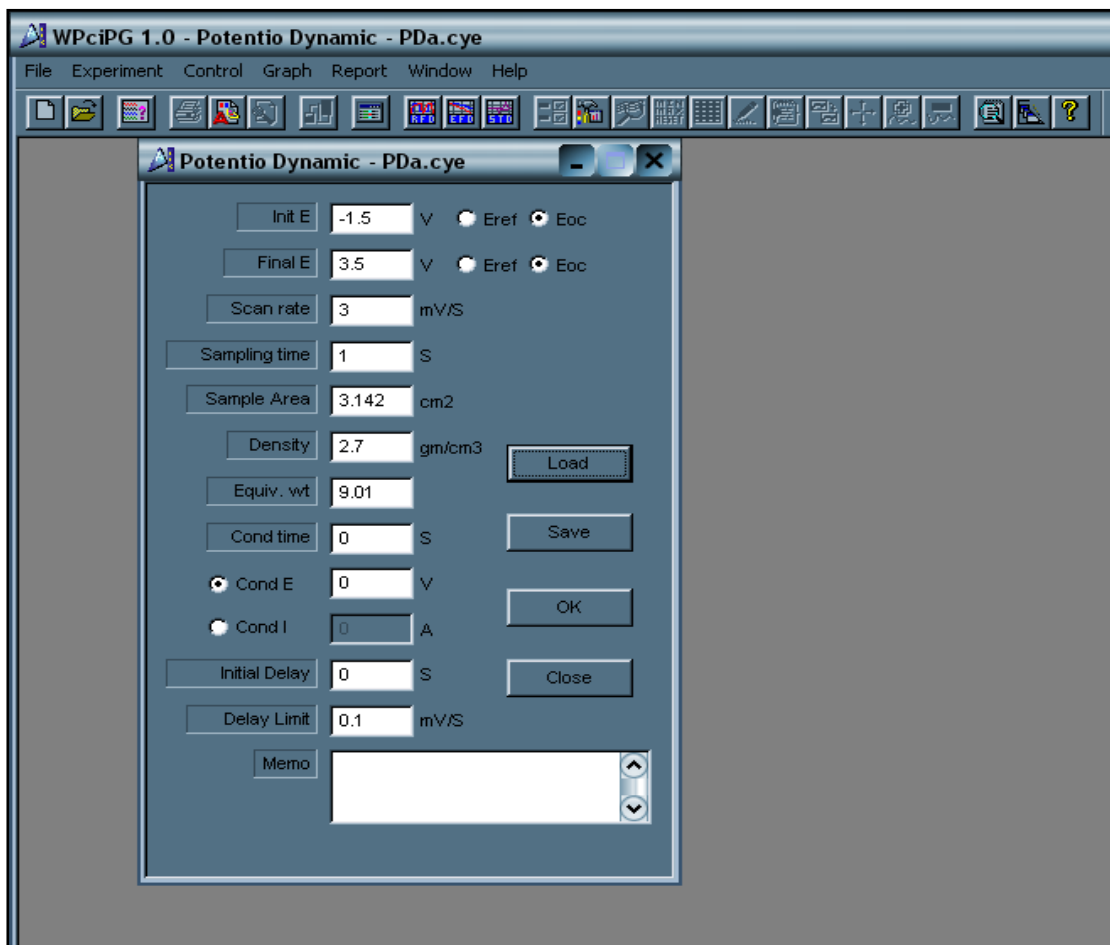


Figure 3.18: Setup window for potentiodynamic polarization

Figure 3.18 show the parameter to be filled in potentiodynamic polarization setup window before an experiment carried out. The initial electrode potential (Init E) and final electrode potential (Final E) were selected by changing automatically from -1.5 V to $+3.5$ V related to open circuit potential (Eoc) with the scanning rate of 3 mV/s. It means the scanning region takes 1667 second (27 minute 47 seconds) in cathodic and anodic region. The sample area was determined by using equation (3.6) and the density was based on ASTM G1 in Table X1.1 for 6061 Aluminum Alloy is 2.70 g/cm³. The equivalent weight of the 6061 Aluminum Alloy is 9.01 refer to ASTM G 102 in Table 1. The others parameters were set as default.

This experiment was generated the potentiodynamic polarization curve that is plotted potential versus current density such as Figure 3.19. The corrosion current

density (i_{corr}), and corrosion potential (E_{corr}) were evaluated from the intersection of linear anodic and cathodic branches of the curve. In passivation analysis, this curve was valuable to determine the reaction region occur in the surface of metal.

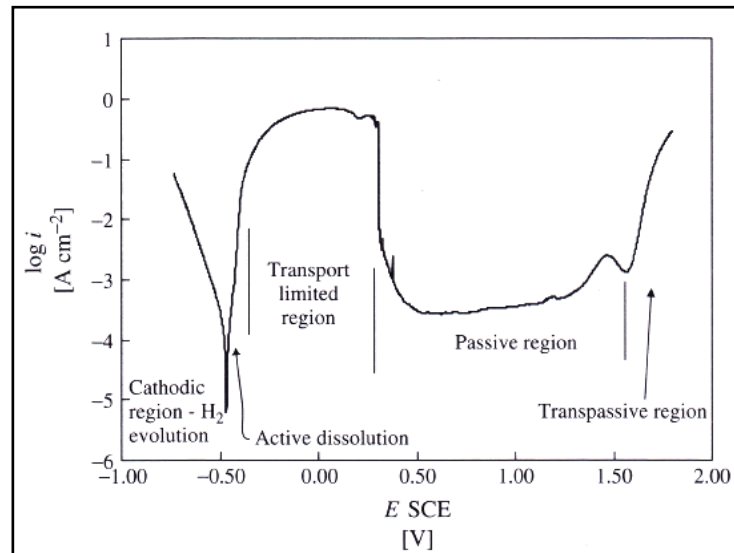


Figure 3.19: Example of potentiodynamic polarization curve for Fe in 0.5 M H₂SO₄

Source: Stratmann, M. and Frankel, G.S. (ed.). 2003

This technique also can generate Tafel plots that will allow the direct measurement of corrosion current from which the corrosion rate can be quickly calculated. This graph was performed to get the Tafel coefficient and determine the corrosion rate from IVMAN software.

The graph is plotted E (V vs SCE) versus $\log i$ to get Tafel constant in Tafel equation. Tafel analysis is performed by extrapolating the linear portions of a \log current versus potential plot back to their intersection. Based on Figure 3.20, the value of either the anodic (green line) or the cathodic (blue line) current at the intersection is called i_{corr} . In experiment, E_{corr} is as the value of E_{oc} before experiment. Information that were resulting from this technique are corrosion current (i_{corr}), cathodic beta Tafel constant (b_a) and anodic beta Tafel constant (b_c). The corrosion rate and mass loss were calculated automatically from this software but still need calculate manually to compare the result by following Faraday's Law equation:

$$CR = K_1 \frac{i_{\text{corr}}}{\rho} EW \quad (3.7)$$

$$MR = K_2 i_{\text{corr}} EW \quad (3.8)$$

where :

CR = penetration rate, in mm/yr

i_{corr} = corrosion current, in $\mu\text{A}/\text{cm}^2$

K_1 = refer to Table 3.4

K_2 = refer to Table 3.4

ρ = material density, in g/cm^3

MR = mass loss rate, in $\text{g}/\text{cm}^2\text{d}$

$EW = 9.01$

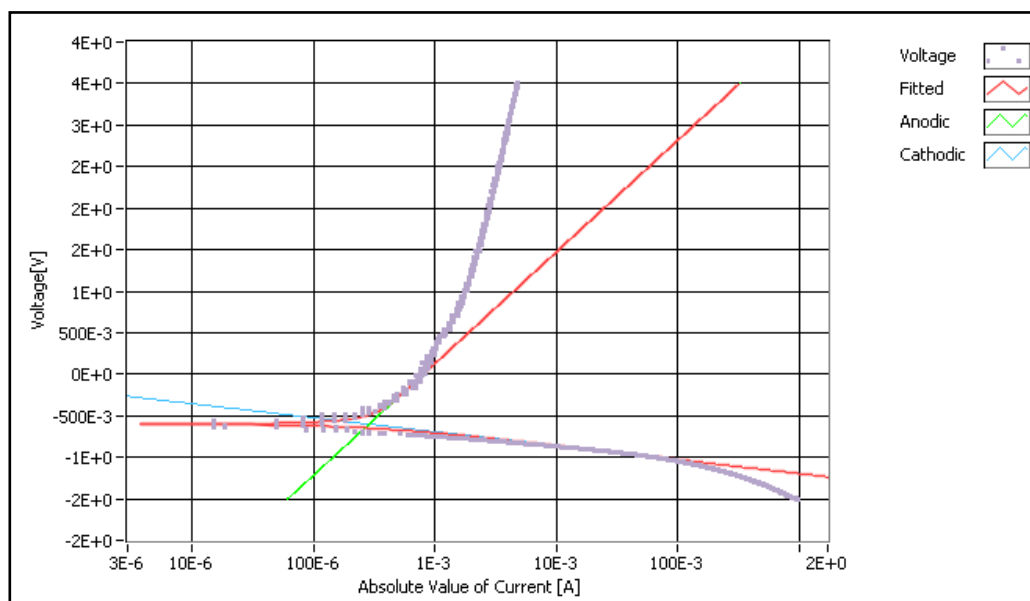


Figure 3.20: Tafel plot using IVMAN software

Table 3.4: Value of constant for use in Faraday's equation rate

A				
Penetration Rate unit (CR)	I_{corr} unit	ρ unit	K_1	Unit of K_1^A
mpy	$\mu\text{A}/\text{cm}^2$	g/cm^3	0.1288	mpy $\text{g}/\mu\text{A cm}$
mm/yr ^B	$\text{A}/\text{m}^{2\text{B}}$	$\text{Kg}/\text{m}^{3\text{B}}$	327.2	mm $\text{kg}/\text{A m y}$
mm/yr ^B	$\mu\text{A}/\text{cm}^2$	g/cm^3	3.27×10^{-3}	mm $\text{g}/\mu\text{A cm y}$
B				
Mass loss rate	I_{corr} unit	K_2	Unit of K_2^A	
$\text{g}/\text{m}^2\text{d}^{\text{B}}$	$\text{A}/\text{m}^{2\text{B}}$	0.8953	g/Ad	
$\text{mg}/\text{dm}^2\text{d}$ (mdd)	$\mu\text{A}/\text{cm}^2$	0.0895	$\text{mg cm}^2/\mu\text{A dm}^2 \text{d}$	
$\text{mg}/\text{dm}^2\text{d}$ (mdd)	$\text{A}/\text{m}^{2\text{B}}$	8.593×10^{-3}	$\text{mg m}^2/\text{A dm}^2 \text{d}$	

Source: ASTM G 102

3.3.3.2 Inhibitor Efficiency

The value of the inhibitor efficiency, IE (%) from the potentiodynamic polarization test was calculated using the following equation:

$$\text{IE}_{\text{PP}} = 100 \times \left(1 - \frac{i'_{\text{corr}}}{i_{\text{corr}}} \right) \quad (3.9)$$

or

$$\text{IE}_{\text{PP}} = 100 \times \left(1 - \frac{CR'}{CR} \right) \quad (3.10)$$

where:

 i'_{corr} = corrosion current densities with inhibitor, i_{corr} = corrosion current densities without inhibitor, CR' = the corrosion rate in inhibited solution, CR = corrosion rate in uninhibited solution

3.4 SAFETY PRECAUTION

Some of the chemicals used in photography are skin irritants, and others can cause serious injuries. Chemicals should be regarded as poisons and handled with caution. The safety precautions when handling poisonous or corrosive chemical contained in Navy Occupational Safety and Health (NAVOSH) should be review and preferred to avoid any accident occur in laboratory. There is several safety items that must be worn when used a chemicals and are as follows:

1. Face shield or goggles use to protects the eyes from caustic chemicals
2. Plastic or rubber apron use to reduce the chance of chemical contamination of clothing.
3. Rubber gloves use to protect the hands and lower arms. Gloves should extend up to the elbows.
4. Long sleeve shirt is used to protect the arms

3.5 SUMMARY

This chapter was discuss briefly about process involve in both experimental. All procedure and process are referring to ASTM standard for metal corrosion. The process for sample preparation including, machining, grinding, polishing and weighing samples. Then, the samples were immersed in 5% H_2SO_4 solution with and without natural honey addition for 28 days. In electrochemical technique, the samples were tested using potentiostat electronic instrument in order to define potentiodynamic polarization curve. After go through the experimental, the result that were obtained will analyzed detailed in chapter 4. The analysis was performed by using theoretical and calculations to determine the effect of inhibitor as well as the corrosion behavior occur onto 6061 Aluminum Alloy in acidic solution.

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 INTRODUCTION

Corrosion inhibition of 6061 Aluminum Alloy in 5% H₂SO₄ solution was evaluated by using weight loss method and electrochemical technique. This chapter has described the analysis of the experiments that have been done by plotted the graph, calculation of corrosion rates, and determination of inhibitor efficiency as well as corrosion behavior. All parameters related to the experiments were collected carefully to ensure the data has been taken precise and accurately.

Analysis the weight loss measurement consists of sample's area determination, mass loss graph plotting for cleaning after exposure, calculating corrosion rate with inhibitor efficiency and corrosion behavior through the surface. In order to show the experimental accuracy, comparisons with theoretical and previous research make experiments successful and accurately.

Another method to analyse the corrosion is by using electrochemical measurement. This method can show the reaction between metal and solution environment whether the reaction in anodic site, cathodic site or both. The parameters including in this method were calculated automatically by using IVMAN software.

4.2 WEIGHT LOSS MEASUREMENT

The weight loss of 6061 Aluminum Alloy in 5% H₂SO₄ solution with and without concentration of honey were determined by weighed the samples before and after exposure. The weighed samples were cleaned several times for completed removal corrosion products with minimal dissolution of any base metal. The average mass samples before and after immersion in 28 days were collected and listed in Table 4.1 and 4.2.

Table 4.1: Mass samples before immersed

Weighing no.	Mass samples, g					
	No inh.	200 ppm	400 ppm	600 ppm	800 ppm	1000 ppm
1	9.166	9.159	9.155	9.178	9.169	9.175
2	9.165	9.158	9.155	9.177	9.169	9.176
3	9.166	9.157	9.156	9.177	9.169	9.176
Average	9.166	9.158	9.155	9.177	9.169	9.176

Table 4.2: Mass samples after immersed

Weighing no.	Mass samples, g					
	No inh.	200 ppm	400 ppm	600 ppm	800 ppm	1000 ppm
1	8.802	8.860	8.868	8.890	8.884	8.897
2	8.801	8.859	8.866	8.889	8.884	8.897
3	8.802	8.858	8.867	8.890	8.883	8.897
Average	8.802	8.859	8.867	8.890	8.884	8.897

The mass samples after immersed being used as references mass to determine the mass loss occur by corroded surface resulting from repetitive cleaning cycles. The average mass samples after cleaning due to repeated cleaning process were taken from table cleaning cycles in Appendix B then was reconstructed in Table 4.3. This table show the value of average mass sample decreasing compared to the mass sample after immersed in 5% H₂SO₄ solution. The cleaning process completely assume to be done when the average mass value each cycle constant in several time.

Table 4.3: Average mass samples after cleaning

No. of cleaning cycle	Average mass samples, g					
	No inh	200 ppm	400 ppm	600 ppm	800 ppm	1000 ppm
1	8.797	8.855	8.863	8.886	8.880	8.893
2	8.795	8.856	8.861	8.887	8.880	8.892
3	8.795	8.855	8.863	8.885	8.879	8.890
4	8.795	8.853	8.861	8.885	8.880	8.891
5	8.795	8.853	8.862	8.885	8.879	8.892

The mass losses during each cleaning cycle were determined by calculated the difference mass refer to the weight before immersed as shown in Table 4.4. The mass loss was graphed as a function of the number of equal cleaning cycles. The two lines were obtained as line AB and line BC. This letter will correspond to corrosion of metal after removal corrosion products. The mass loss due to corrosion will correspond to be assuming approximately to line BC. This line was constructed with average mass loss and the slope approximately to be zero. The Figure 4.1 shows the example graphing method of mass loss for without inhibitor presence resulting from repetitive cleaning cycle. The others results were shows in Appendix C.

Table 4.4: Mass loss

No. of cleaning cycle	Mass loss, g					
	No inh	200 ppm	400 ppm	600 ppm	800 ppm	1000 ppm
0	0.364	0.299	0.288	0.287	0.285	0.279
1	0.369	0.303	0.292	0.291	0.289	0.283
2	0.371	0.302	0.294	0.290	0.289	0.284
3	0.371	0.303	0.292	0.292	0.290	0.286
4	0.371	0.305	0.294	0.292	0.289	0.285
5	0.371	0.305	0.293	0.292	0.290	0.284

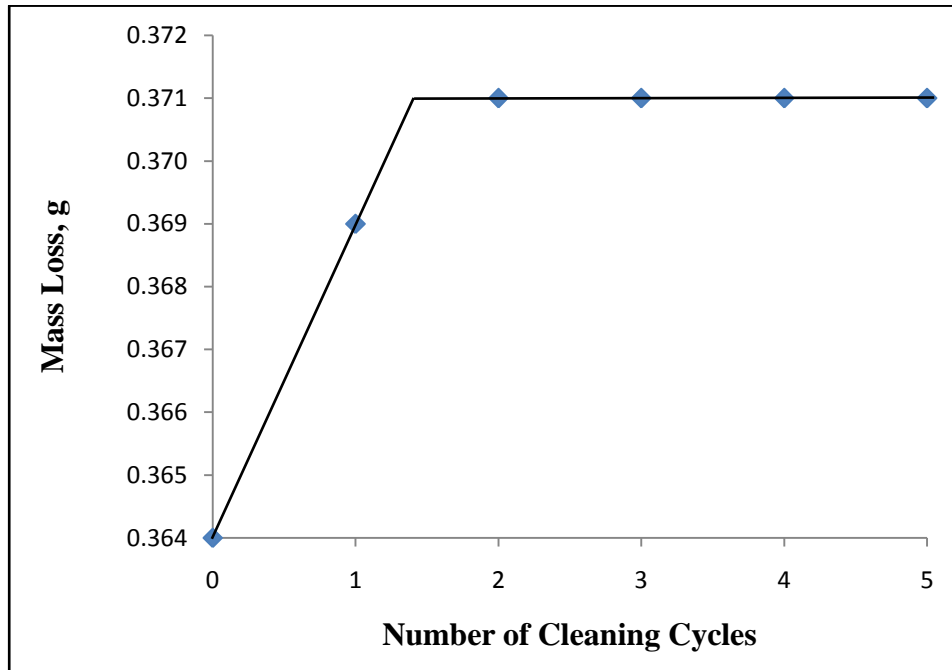


Figure 4.1: Example of mass loss for without inhibitor presence resulting from repetitive cleaning cycle

4.2.1 Corrosion Rates

The analysis of corrosion rates were performed by determined the samples area by using Equation 3.2. The scale of diameter and thickness were evaluated before exposure. The example calculation of samples area was estimated by using diameter of sample is 30 mm, diameter of mounting hole is 2.5 mm and thickness of 5.00 mm.

Example of area calculation

$$A = \frac{\pi}{2} (D^2 - d^2) + \pi D t + \pi d t$$

where,

$$t = 5.00 \text{ mm},$$

$$D = 30 \text{ mm}$$

$$d = 2.5 \text{ mm}$$

$$A = \frac{\pi}{2} [(30 \text{ mm})^2 - (2.5 \text{ mm})^2] + (5 \text{ mm} \times \pi \times 30 \text{ mm}) + (5 \text{ mm} \times \pi \times 2.5 \text{ mm})$$

$$A = 1914.408 \text{ mm}^2$$

$$A = 1914.408 \text{ mm}^2 \times \left(\frac{1 \text{ cm}}{10 \text{ mm}} \right)^2$$

$$A = 19.144 \text{ cm}^2$$

In order to determine the corrosion rates in 5% H₂SO₄ solution with and without inhibitor addition, the parameters have been analyzed by using Equation 3.4. The mass loss, samples area, density and time of exposure have to be estimated before analyses of corrosion rates have been taken. Many different units can be used to express corrosion rates depend to the value of *K* in corrosion rates equation. Table 4.5 below mentioned corrosion rates in mmpy and mg/m².day for entire sample from 0 ppm to 1000 ppm. These results were calculated refer to 28 days or 672 hours time of exposure in 5% sulfuric acid solution along with the density of Aluminum Alloy 6061 is 2.70 g/cm³ such as example below.

Table 4.5: Corrosion rates of 6061 Aluminum Alloy in 5% H₂SO₄ solution with and without inhibitor presence

c (ppm)	Area, <i>A</i> (cm ²)	Mass loss, g	Corrosion rate, (10 ⁻³ mmpy)	Corrosion rate, (mg/m ² .hour)
0		0.371	935.648	288.385
200		0.305	769.198	237.082
400	19.144	0.294	741.457	228.531
600		0.292	736.413	226.976
800		0.290	731.369	225.422
1000		0.285	718.759	221.535

Example calculation of corrosion rates with no inhibitor

$$\text{Corrosion Rate} = \frac{K \times W}{A \times T \times D}$$

where,

$$K = 8.76 \times 10^4 \text{ is constant value}$$

$$W = 0.371 \text{ g}$$

$$A = 19.144 \text{ cm}^2$$

$$T = 672 \text{ hr}$$

$$D = 2.70 \text{ g/cm}^3$$

$$\text{Corrosion Rate} = \frac{(8.76 \times 10^4) \times 0.371 \text{ g}}{19.144 \text{ cm}^2 \times 672 \text{ hr} \times 2.07 \text{ g/cm}^3}$$

$$\text{Corrosion Rate} = 935.648 \times 10^{-3} \text{ mmpy}$$

According to these results, the corrosion rates were decrease with addition concentration of honey as inhibitor compared to the sample tested without inhibitor as presented in Figure 4.2. The test without inhibitor addition had the highest corrosion rate is about 935.648×10^{-3} mmpy compared to the others. The addition of natural honey as inhibitor shows the decreasing corrosion rates due to their mixture of various compounds consists of carbon (C), nitrogen (N), sulfur (S) and oxygen (O) by which all can be absorbed on the corroded metal (Radojcic, I, et al 2008). The thin layer was performed on the Aluminum Alloy based on adsorption by inhibitor onto the surface to reduce the corrosion rates. The results also show the mass loss continues decrease every hour and occur at every metal surface.

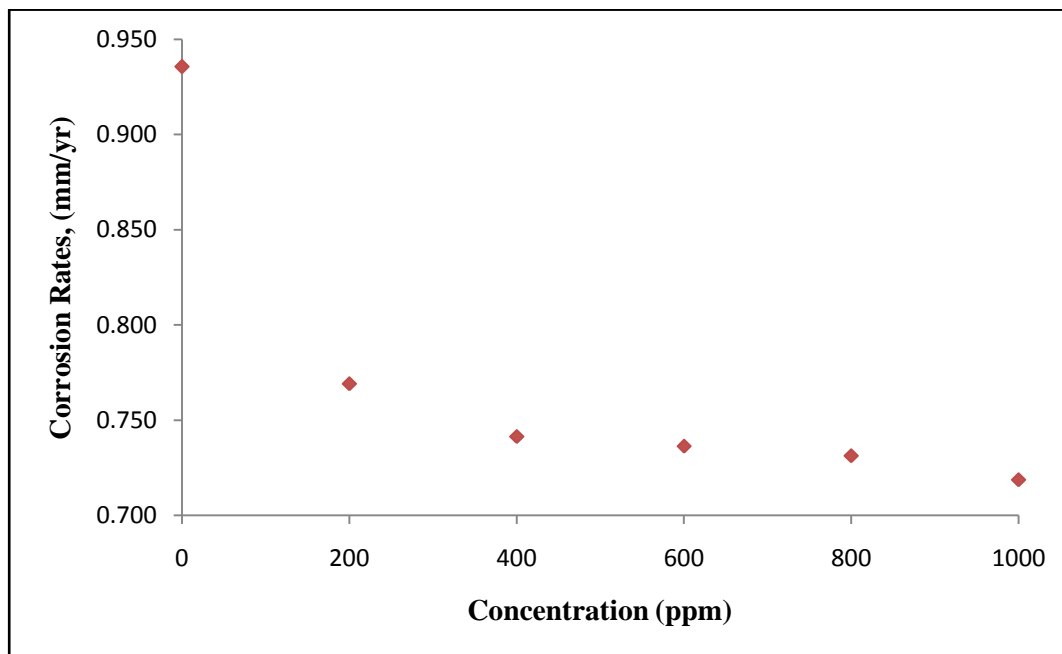


Figure 4.2: Corrosion rates versus concentration in 5% H₂SO₄ solution

The corrosive behavior of 5% sulfuric acid was breaking the protective layer of aluminum oxide and slowly increases the corrosion rate. According to Rosliza, R et al. 2008, the weight loss of 6061 Aluminum Alloy in sulfuric acid solution increased with immersion time but the corrosion rates are vice versa. In the beginning of immersion time, the corrosion rate was reported higher due to their formation of oxide layer and then slowly decrease depends to immersion time. This transition can be assumed as the formation of aluminum oxide layer (Al₂O₃) on the metal surface formed slowly and at the same time, the corrosion rate increase rapidly.

The Table 4.6 below shows the pH value for 5% H₂SO₄ solution with and without inhibitor concentration. The average pH value range from 0.34-0.35 shows that the addition small amounts of natural honey as inhibitor was unaffected the pH value of 5% H₂SO₄ solution as well as the acidic region of the solution. Based on Figure 2.1 in chapter 2, the passivity of aluminum alloy is uncontrolled and unstable when the pH value below than 4. In acids, aluminum dissolves to Al³⁺ ions according to the following reaction:



The oxide layer dissolves and Al^{3+} ions are formed.

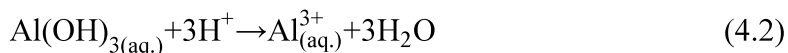


Table 4.6: pH value for 5% H_2SO_4 solution with and without inhibitor presence

Measuring no.	pH samples					
	No inh.	200 ppm	400 ppm	600 ppm	800 ppm	1000 ppm
1	0.35	0.33	0.34	0.36	0.34	0.35
2	0.35	0.34	0.36	0.35	0.33	0.34
3	0.36	0.34	0.35	0.35	0.35	0.34
Average	0.35	0.34	0.35	0.35	0.34	0.34

The investigation about corrosion types of 6061 Aluminum Alloy in 5% H_2SO_4 solution was conducted by using metallurgical microscope and the result was shown in Figure 4.3 below and the others were shown in Appendix D. This type of corrosion is prone to uniform corrosion because this type of corrosion develops very small diameter pits in order measure in micrometer and continuous decrease in thickness over the entire surface area of the metal (Vargel, C. 2004). As a result, the addition of inhibitor decreases the corrosion rates of 6061 Aluminum Alloy by performing the thin layers such as Figure 4.4.

With aluminum, this type of corrosion is observed especially in highly acidic or alkaline media, in which the solubility of the natural oxide film is high. The dissolution rate of the film is greater than its rate of formation. However, the ratio of both rates can change over time (Vargel, C. 2004). Immersion aluminum in acidic solution was released the hydrogen corresponds to the anodic dissolution reaction of aluminum such as:



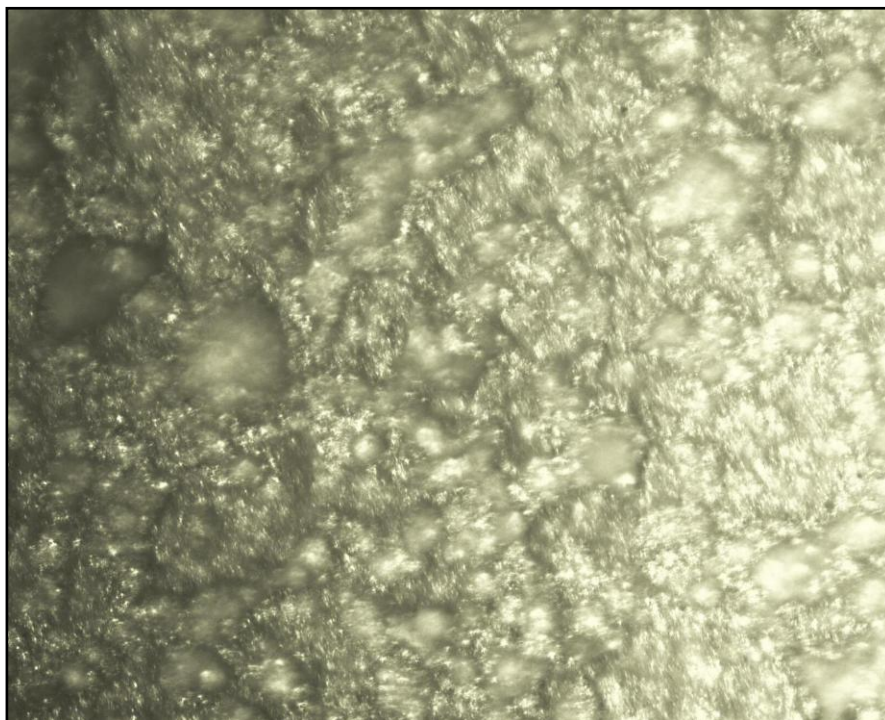


Figure 4.3: Micro surface under magnification (20 X) of 6061 Aluminum Alloy in 5% H_2SO_4 solution without inhibitor presence

The schematic diagram below illustrated the function of inhibitor to protected 6061 Aluminum Alloy from corrosive ability by 5% H_2SO_4 solution. The adsorption molecule into surface metal act as thin layer to delay the corrosion attack the base metal as shown in Figure 4.4 (b). According to Rosliza, R et al. 2009, the carbon and these molecules contain in honey cover the surface of the sample by developed thin layer of the corrosion resistant molecules and giving a protection against corrosion.

Most pickling inhibitors function by forming an adsorbed layer on the metal surface, probably no more than monolayer in thickness, which essentially block discharge of H^+ and dissolution of metal ions. Some inhibitors block the cathodic reaction (raise hydrogen overpotential) more than the anodic reaction or vice versa but adsorption appears to be general over all rather than at the specific at specific anodic and cathodic sites and both reaction tend to be retarded (Revie, R.W. and Uhlig, H.H. 2008).

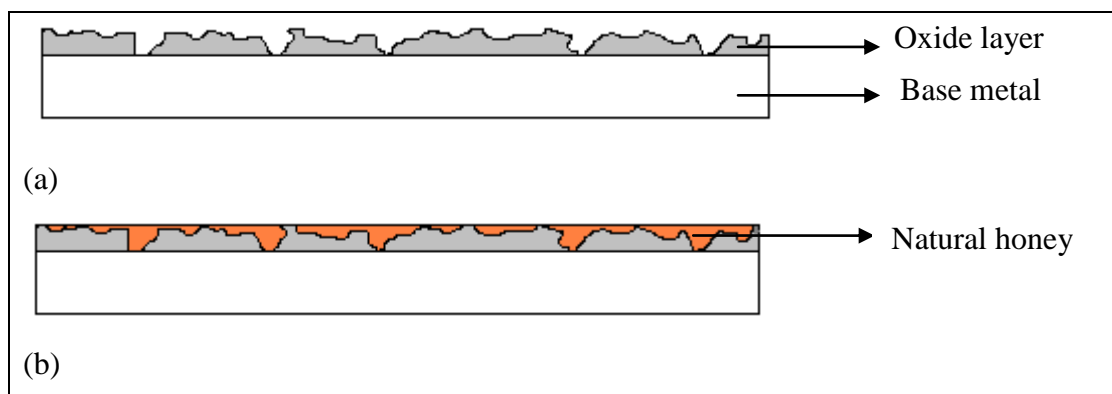


Figure 4.4: Schematic diagram of surface film (a) without inhibitor (b) with inhibitor presence

4.3 POTENTIODYNAMIC POLARIZATION MEASUREMENT

Potentiodynamic polarizations were measured in the absence and presence of inhibitor in a potential range between -1.5V to 3.5V. Figure 4.5 below explain the changed anodic and cathodic polarization curve of 6061 Aluminum Alloy in 5% H₂SO₄ solution with and without inhibitor presence. The black line curve represents sample tested without inhibitor and to be as references. The colors curve much polarized in both anodic and cathodic site. This variation shows that natural honey acts as mixed-type corrosion resistance. The addition of natural honey also increased the potential of 6061 Aluminum Alloy more to the cathodic site.

The trend curve show the anodic current density increases rapidly when the potential moves to a more positive direction. This behavior is likely due to the rapid dissolution attack by H₂SO₄ solution on 6061 Aluminum Alloy. The oxidation of aluminum to Al³⁺ ion at the anodic region is rapidly increase can be categorized to uniform corrosion due to the small pit occur at the surface metal. The anodic current density also experiences little fluctuations before it increases. However, the flat region indicates that it is passivated. In the cathodic region, only the reduction of H⁺ ions can be seen.

The addition of natural honey at all studied concentration decreased the anodic and cathodic current density. This behavior can be assume as the adoption molecules

into the surface metal cause in slowly reduction of H^+ ion as well as oxidation of aluminum into Al^{3+} ion.

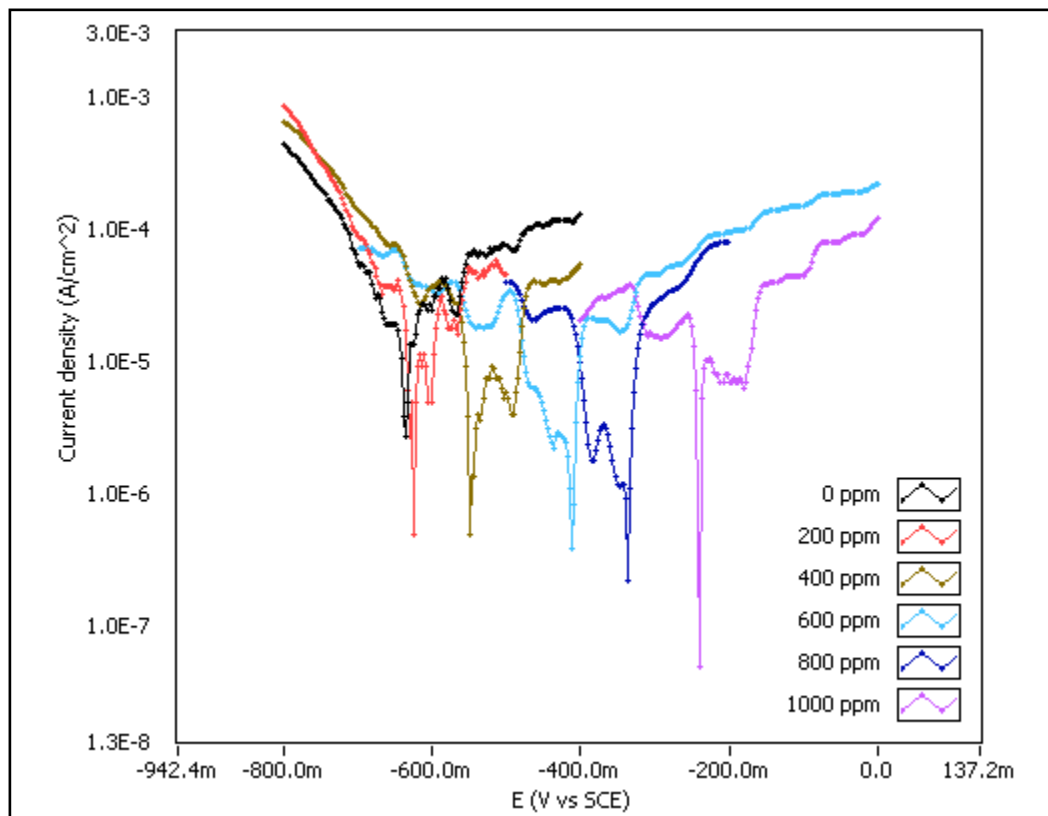


Figure 4.5: Potentiodynamic polarization curve of 6061 Aluminum Alloy in 5% H_2SO_4 with various concentrations of honey

Inspection in Table 4.7 below reveals that corrosion rates decrease with increasing the concentration of honey ranging from 0 ppm to 1000 ppm. The electrochemical parameters such as corrosion potential (E_{corr}), corrosion current density (I_{corr}), anodic tafel slopes (b_a), and cathodic tafel slope (b_c) also listed in Table 4.7 below. The change in E_{corr} is assumed to be related to the growth of passive layer at the surface of metal (Rosliza, R et al. 2008). The value of corrosion current density, I_{corr} is significant decline when the concentration of honey was increased. The example of calculation corrosion rate was shown in example below by using Equation 3.7.

Table 4.7: The electrochemical parameters in 5% H₂SO₄ solution

c(ppm)	Potentiodynamic Polarization				
	E_{corr} (mV)	I_{corr} ($\mu\text{A}/\text{cm}^2$)	b_a (V/div)	b_c (V/div)	CR (10^{-3} mm/yr)
0	-600.700	90.943	1.344	0.168	992.987
200	-562.482	77.879	1.034	0.222	850.344
400	-608.466	71.839	0.834	0.166	784.398
600	-641.425	65.103	0.784	0.168	710.848
800	-465.792	63.225	0.907	0.273	690.338
1000	-471.682	62.379	0.906	0.235	681.099

Example calculation of corrosion rates for without inhibitor

$$\text{CR} = K_1 \frac{i_{\text{corr}}}{\rho} EW$$

Where,

$$i_{\text{corr}} = 90.943 \mu\text{A}/\text{cm}^2$$

$$K_1 = 3.27 \times 10^{-3} \text{ mm g}/\mu\text{A cm y}$$

$$\rho = 2.70 \text{ g}/\text{cm}^3$$

$$EW = 9.01$$

$$\text{CR} = (3.27 \times 10^{-3}) \times \frac{90.943}{2.70} \times 9.01$$

$$\text{CR} = 992.380 \times 10^{-3} \text{ mm}/\text{yr}$$

4.4 INHIBITOR EFFICIENCY AND ADSORPTION ISOTHERM

The performances various concentration of inhibitor can be evaluated by using Equation 3.5. Table 4.8 and 4.9 shows the values of inhibition efficiencies obtained by weight loss measurements and potentiodynamic polarization for 6061 Aluminum Alloys in a 5% H₂SO₄ solution in the absence and presence of different concentration of natural honey. Inhibitor efficiency, I_{eff} and surface covered by inhibitor molecules, θ were calculated and shown in example below. The value of I_{eff} and θ for the various

concentrations are mentioned in Table 4.8 and 4.9 for weight loss measurement and potentiodynamic polarization respectively.

The data reveals that the inhibition efficiency increases with increasing in honey concentration. Thus, the surface coverage, θ due to the adsorption of honey at metal surface increasing with increased inhibitor concentration. This phenomenon happen due to the surface coverage absorbed onto the surface metal increase with increasing the concentration of honey. The higher surface coverage on the metal surface was found at the higher performance inhibition of honey. According to Et-Etre, A.Y. 1998, the carbohydrates that which the major component of honey exhibits and ideal medium for fungi growth. The fungi colony grows with time at the expense of the honey concentration. Consequently the inhibition efficiency is greatly reduced. Fungi produce highly corrosive organic acids that corroded the aluminum (Revie, R.W. et al. 2008).

This behavior can be conclude as the presence of inhibitor became a reason to reduce the corrosion by blocking the corroded surface with their organic compound and the absence of inhibitor, the solution is in contact to the surface metal and corrode the passive film.

Table 4.8: Inhibitor efficiency for weight loss measurement with different concentration of honey for 6061 Aluminum Alloy corrosion in 5% H₂SO₄ solution

c (ppm)	R_0 (10^{-3} mmpy)	R_i (10^{-3} mmpy)	I_{eff} (%)	θ
200		769.198	17.790	0.178
400		741.457	20.755	0.208
600	935.648	736.413	21.294	0.213
800		731.369	21.833	0.218
1000		718.759	23.181	0.232

Example calculation for weight loss measurement

a) Inhibitor efficiency for 200 ppm

$$I_{\text{eff}} = \frac{R_0 - R_i}{R_0} \times 100\%$$

where:

I_{eff} = efficiency of inhibitor, %

$R_0 = 935.648 \times 10^{-3}$ mmpy

$R_i = 769.198 \times 10^{-3}$ mmpy

$$I_{\text{eff}} = \frac{935.648 \times 10^{-3} - 769.198 \times 10^{-3}}{935.648 \times 10^{-3}} \times 100\%$$

$$I_{\text{eff}} = 17.790\%$$

b) Surface coverage, θ for 200 ppm

$$\theta = 1 - \frac{R_i}{R_0}$$

where,

$R_0 = 935.648 \times 10^{-3}$ mmpy

$R_i = 769.198 \times 10^{-3}$ mmpy

$$\theta = 1 - \frac{769.198 \times 10^{-3}}{935.648 \times 10^{-3}}$$

$$\theta = 0.178$$

Table 4.9: Inhibitor efficiency for potentiodynamic polarization with different concentration of honey for 6061 Aluminum Alloy corrosion in 5% H₂SO₄ solution

c (ppm)	I_{corr} ($\mu\text{A}/\text{cm}^2$)	I'_{corr} ($\mu\text{A}/\text{cm}^2$)	I_{eff} (%)	θ
200		77.879	14.365	0.144
400		71.839	21.007	0.210
600	90.943	65.103	28.413	0.284
800		63.225	30.478	0.305
1000		62.379	31.409	0.314

Example calculation for potentiodynamic polarization

a) Inhibitor efficiency for 200 ppm

$$I_{\text{eff}} = 100 \times \left(1 - \frac{I'_{\text{corr}}}{I_{\text{corr}}} \right)$$

where:

I_{eff} = efficiency of inhibitor, %

$$I'_{\text{corr}} = 77.879 \mu\text{A}/\text{cm}^2$$

$$I_{\text{corr}} = 90.943 \mu\text{A}/\text{cm}^2$$

$$I_{\text{eff}} = 100 \times \left(1 - \frac{77.879}{90.943} \right)$$

$$I_{\text{eff}} = 14.365\%$$

b) Surface coverage, θ for 200 ppm

$$\theta = 1 - \frac{I'_{\text{corr}}}{I_{\text{corr}}}$$

where,

$$I'_{\text{corr}} = 77.879 \mu\text{A}/\text{cm}^2$$

$$I_{\text{corr}} = 90.943 \mu\text{A}/\text{cm}^2$$

$$\theta = 1 - \frac{77.879}{90.943}$$

$$\theta = 0.144$$

The Figure 4.6 below shows the relationship between c/θ and c inhibitor concentration. The linear dependence of c/θ versus c inhibitor concentration in 5% H_2SO_4 solution suggesting that the adsorption natural honey onto 6061 Aluminum Alloy surface obeys Langmuir's adsorption isotherm. The value of the adsorption equilibrium constant, K was determine as intercept on ordinate when $c = 0$. This action of an inhibitor in aggressive acid media is assumed to be due adsorption at the metal interface.

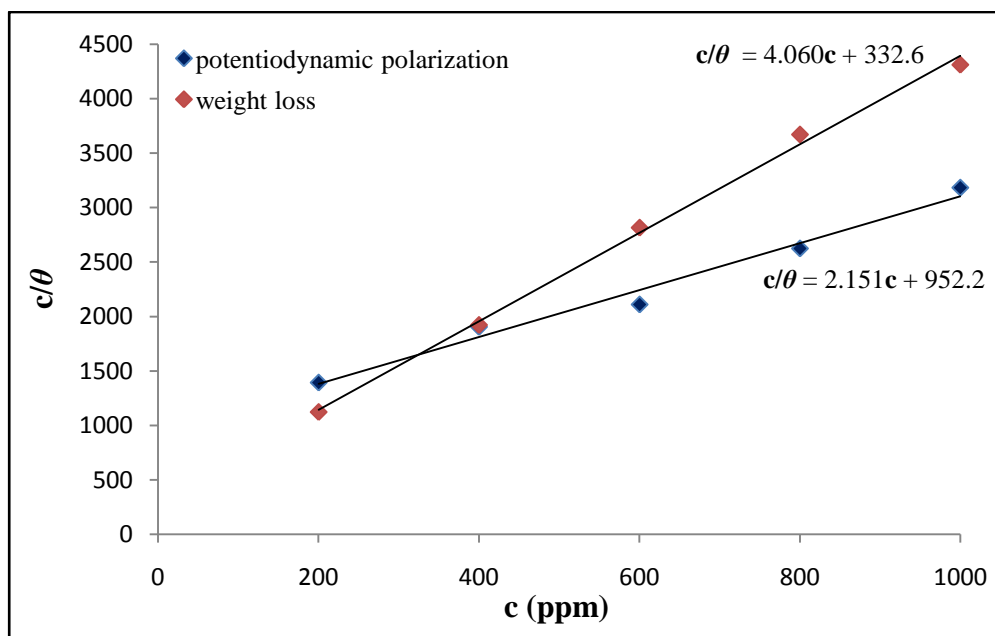


Figure 4.6: The relationship between inhibitor concentration c (ppm) and c/θ

Example calculation for thermodynamic relations

$$k = \frac{1}{55.5} \exp \left[-\frac{\Delta G}{RT} \right]$$

where:

$$R = 8.314 \text{ J/K.mol}$$

$$T = 298.16 \text{ K}$$

a) Weight loss measurement

$$c/\theta = 4.060c + 332.6$$

When $c = 0$

$$c/\theta = 332.6$$

Then,

$$\ln 332.6 = 1/k$$

$$k = 0.172 \text{ m}^3\text{mol}^{-1}$$

$$k = 1.722 \times 10^3 \text{ dm}^3\text{mol}^{-1}$$

$$k = \frac{1}{55.5} \exp \left[-\frac{\Delta G}{RT} \right]$$

$$\Delta G = \ln (k \times 55.5) \times RT$$

$$\Delta G = \ln (1.722 \times 10^3 \times 55.5) \times 8.134 \times (25 + 273.16)$$

$$\Delta G = -27.812 \text{ kJ/mol}$$

b) Potentiodynamic polarization

$$c/\theta = 2.151c + 952.2$$

When $c = 0$

$$c/\theta = 952.2$$

Then,

$$\ln 952.2 = 1/k$$

$$k = 0.145 \text{ m}^3\text{mol}^{-1}$$

$$k = 1.458 \times 10^3 \text{ dm}^3\text{mol}^{-1}$$

$$k = \frac{1}{55.5} \exp \left[-\frac{\Delta G}{RT} \right]$$

$$\Delta G = \ln (k \times 55.5) \times RT$$

$$\Delta G = \ln (1.458 \times 10^3 \times 55.5) \times 8.134 \times (25 + 273.16)$$

$$\Delta G = -27.408 \text{ kJ/mol}$$

The negative value of ΔG showed that the adsorption were spontaneous and might have physical mechanism. The heat of adsorption can be assumed to physical adsorption where the behavior of this adsorption usually less than 40 kJ/mol and it is an instantaneous process as a function of surface coverage. Comparing with chemisorptions, the heat of adsorption are about 40-400 kJ/mol and the reaction can be rapid or slow (Goel, A. 2006). The obtained value of ΔG may suggest as the indication of physisorptions mechanism.

4.5 SUMMARY

The overall results were shown in Table 4.10 below.

Table 4.10: Overall result for weight loss measurement and potentiodynamic polarization

Results	Sample with honey concentration, ppm					
	No inh.	200	400	600	800	1000
Weight loss measurement						
CR (10^{-3} mmpy)	935.648	769.198	741.457	736.413	731.369	718.759
I_{eff} (%)	-	17.790	20.755	21.294	21.833	23.181
θ	-	0.178	0.208	0.213	0.218	0.232
$k \times 10^3$ (dm^3/mol)				1.722		
ΔG (kJ/mol)				-27.812		
Potentiodynamic Polarization						
I_{corr} ($\mu\text{A}/\text{cm}^2$)	90.943	77.879	71.839	65.103	63.225	62.379
CR (10^{-3} mm/yr)	992.987	850.344	784.398	710.848	690.338	681.099
I_{eff} (%)	-	17.790	20.755	21.294	21.833	23.181
θ	-	0.178	0.208	0.213	0.218	0.232
$k \times 10^3$ (dm^3/mol)				1.458		
ΔG (kJ/mol)				-27.408		

The analysis of corrosion inhibition can be summaries as:

- (i) The corrosion rates were decreased when increasing the concentration of honey,
- (ii) The performances of honey were increased when increasing the inhibitors concentration,
- (iii) Natural honey acts as mixed-types corrosion resistance,
- (iv) The corrosion type of 5% H₂SO₄ onto 6061 Aluminum Alloy was uniform corrosion,
- (v) The surface coverage increased when increasing inhibitor addition,
- (vi) The linear dependence of c/θ versus c inhibitor concentration obeys Langmuir's adsorption isotherm
- (vii) The negative value free energy adsorption can be assumed as physisorptions mechanism.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 INTRODUCTION

The corrosion inhibition of 6061 Aluminum Alloy in 5% H₂SO₄ solution was succeeded evaluated by using weight loss method and potentiodynamic polarization. The results were analyzed and discussed in previous chapter and the comparison between each concentration of honey will carry out some conclusions. Therefore, the conclusions have to be linked to the objective in earlier chapter including corrosion inhibition of honey, the effect of variation concentration and corrosion behavior.

In addition, the recommendations can give some new investigation related to this topic to strengthen the research and provide new idea in corrosion science. This includes the type of solution, material, and methods. This will help to accesses the effectiveness of honey as inhibitor in various conditions.

5.2 CONCLUSIONS

Inhibitor is the most practical method to protect metal against corrosion. Therefore, to protect 6061 Aluminum Alloy into aggressive media produce by 5% H₂SO₄ solution, the addition small amount natural honey into solution will extend the life spend of material. The weight loss method and potentiodynamic polarization technique were used to study this topic, thus several conclusion could be drawn from the study are:

- (i) Natural honey in 5% H_2SO_4 solution acts as inhibitors for 6061 Aluminum Alloy.
- (ii) The corrosion rates of 6061 Aluminum Alloy in H_2SO_4 are significantly reduced upon the addition of natural honey.
- (iii) Uniform corrosion occurs onto the surface metal due to exposure in acid environment.
- (iv) The inhibitor efficiency increased with the increasing of their concentration.
- (v) The studied of corrosion inhibitor shows that the honey act as mixed-type corrosion resistant.
- (vi) The adsorption of natural honey on the 6061 Aluminum Alloy surface in 5% H_2SO_4 solution obey Langmuir's adsorption isotherm.

5.3 RECOMMENDATIONS

The corrosion inhibition of 6061 Aluminum Alloy in acidic media introduced by honey was limited to several conditions. Thus, for further work to study detail about this topic, some suggestions could be made into consideration such as:

- (i) Operate many kinds of experiment by using various concentrations of H_2SO_4 solution into 10%, 5% and 1% concentration.
- (ii) Comparison in various acidic solutions such as nitric acid, hydrochloric acid, acetic acid and others.
- (iii) Made an immersion testing by every 5, 10, 15, 20, 25 and 30 days to evaluate the variation of corrosion rates and corrosion behavior for each day.
- (iv) Use SEM (scanning electron microscope) after exposure to determine the reaction and nature of honey onto surface 6061 Aluminum Alloy with comparing without inhibitor presence.

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APPENDIX A
PROJECT PLANNING (GANTT CHART): FINAL YEAR PROJECT 1

Work Progress	Week														
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Get the project title and arrange discussion time with supervisor.	■														
Find the problem statement and project objectives.	■	■	■												
Find scope of the project, hypothesis. Verify problem statement, project objectives, scope and hypothesis.			■	■											
Do research and collect the information		■	■	■	■	■	■	■	■	■	■	■	■		
Study and Learning the theory					■	■	■	■	■	■					
Do the design of the experiment and state the experimental procedure								■	■	■	■	■	■	■	■
Report Writing (Chapter 1, 2, 3) (Introduction, Literature review, Methodology)			■	■	■	■	■	■	■	■	■	■	■		
Submit draft thesis and prepare slide presentation														■	
Final year project 1 presentation															■

Remark:

- Planning progress
- Actual progress

APPENDIX B
MASS LOSS DUE TO REPETITIVE CLEANING CYCLE

Table 6.1: Mass sample after cleaning (1st cycle)

Weighing no.	Concentration of honey					
	No inh.	200 ppm	400 ppm	600 ppm	800 ppm	1000 ppm
1	8.797	8.856	8.863	8.885	8.880	8.894
2	8.798	8.855	8.863	8.886	8.879	8.892
3	8.797	8.855	8.862	8.886	8.880	8.894
Average	8.797	8.855	8.863	8.886	8.880	8.893

Table 6.2: Mass sample after cleaning (2nd cycle)

Weighing no.	Concentration of honey					
	No inh.	200 ppm	400 ppm	600 ppm	800 ppm	1000 ppm
1	8.795	8.855	8.862	8.886	8.880	8.893
2	8.794	8.856	8.861	8.887	8.879	8.892
3	8.795	8.857	8.861	8.887	8.881	8.892
Average	8.795	8.856	8.861	8.887	8.880	8.892

Table 6.3: Mass sample after cleaning (3rd cycle)

Weighing no.	Concentration of honey					
	No inh.	200 ppm	400 ppm	600 ppm	800 ppm	1000 ppm
1	8.795	8.854	8.863	8.885	8.879	8.890
2	8.795	8.856	8.863	8.885	8.879	8.891
3	8.796	8.855	8.864	8.886	8.880	8.890
Average	8.795	8.855	8.863	8.885	8.879	8.890

Table 6.4: Mass sample after cleaning (4th cycle)

Weighing no.	Concentration of honey					
	No inh.	200 ppm	400 ppm	600 ppm	800 ppm	1000 ppm
1	8.794	8.854	8.861	8.886	8.880	8.891
2	8.795	8.854	8.862	8.885	8.879	8.892
3	8.795	8.852	8.860	8.885	8.880	8.891
Average	8.795	8.853	8.861	8.885	8.880	8.891

Table 6.5: Mass sample after cleaning (5th cycle)

Weighing no.	Concentration of honey					
	No inh.	200 ppm	400 ppm	600 ppm	800 ppm	1000 ppm
1	8.796	8.854	8.862	8.884	8.879	8.891
2	8.794	8.854	8.861	8.885	8.879	8.892
3	8.795	8.852	8.862	8.886	8.878	8.893
Average	8.795	8.853	8.862	8.885	8.879	8.892

APPENDIX C
GRAPH REPETITIVE CLEANING CYCLE

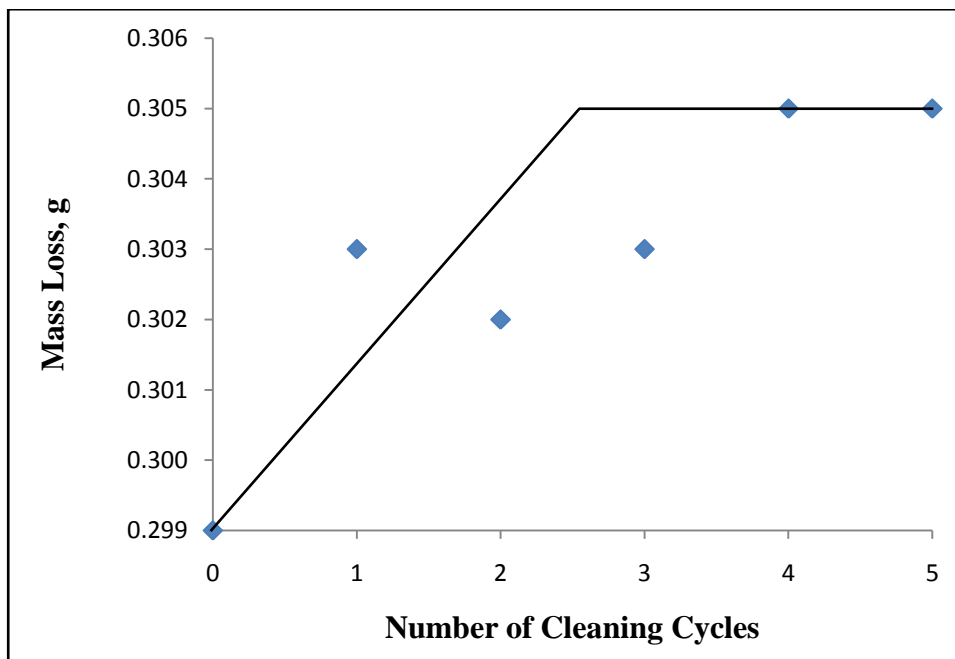


Figure 6.1: Mass loss for 200 ppm resulting from repetitive cleaning cycle

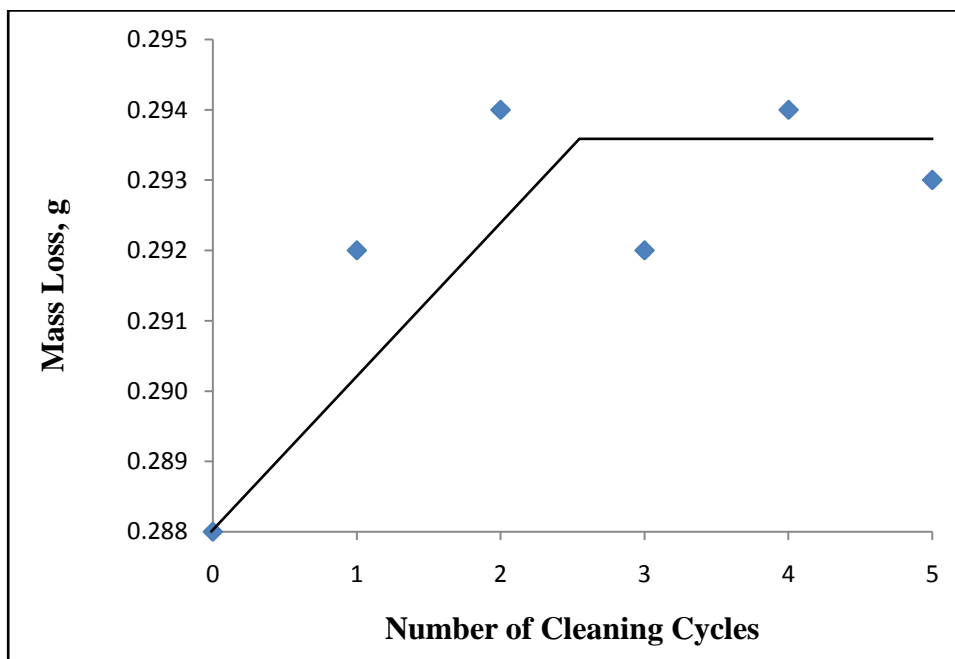


Figure 6.2: Mass loss for 400 ppm resulting from repetitive cleaning cycle

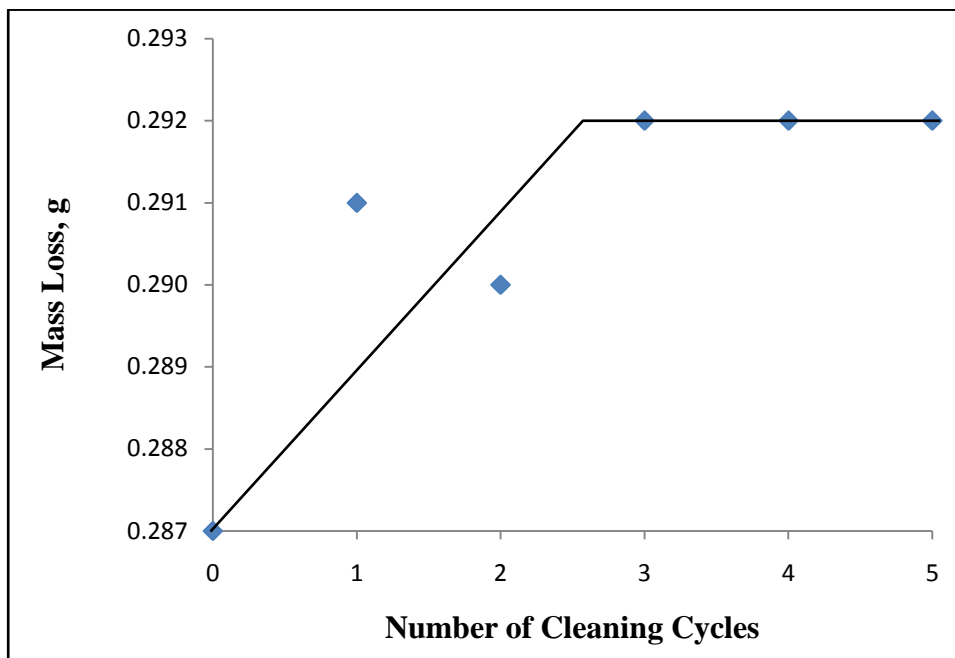


Figure 6.3: Mass loss for 600 ppm resulting from repetitive cleaning cycle

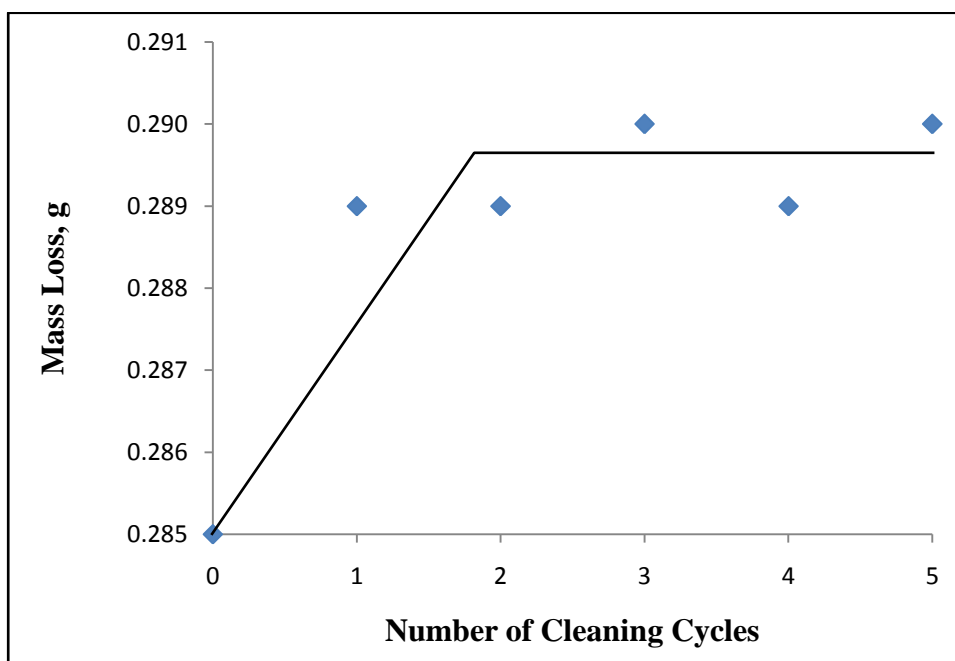


Figure 6.4: Mass loss for 800 ppm resulting from repetitive cleaning cycle

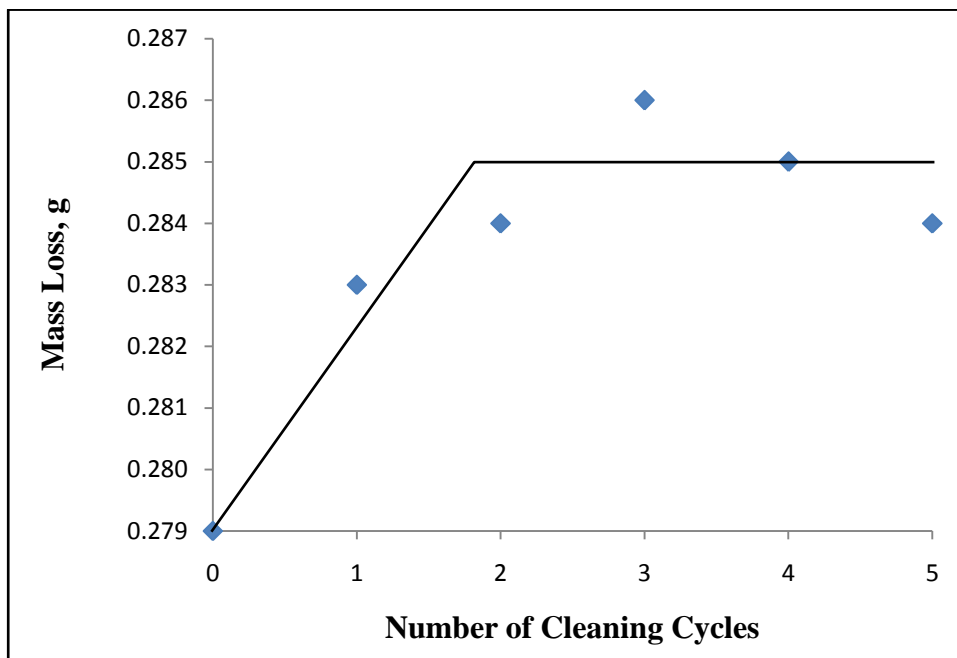


Figure 6.5: Mass loss for 1000 ppm resulting from repetitive cleaning cycle

APPENDIX D
MICRO SURFACE OF 6061 ALUMINUM ALLOY

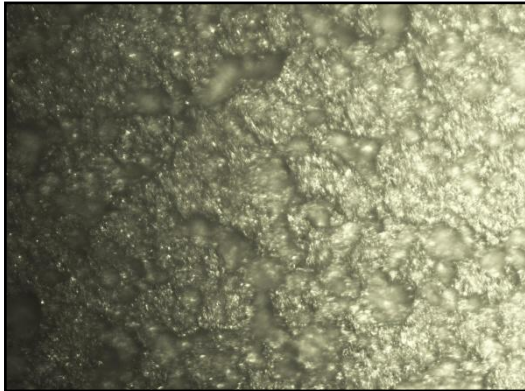


Figure 6.6: Sample for 200 ppm

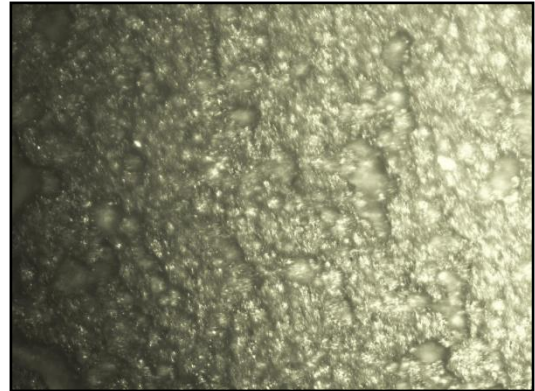


Figure 6.9: Sample for 800 ppm

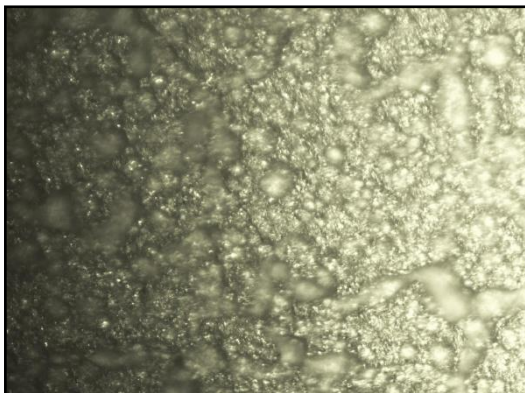


Figure 6.7: Sample for 400 ppm

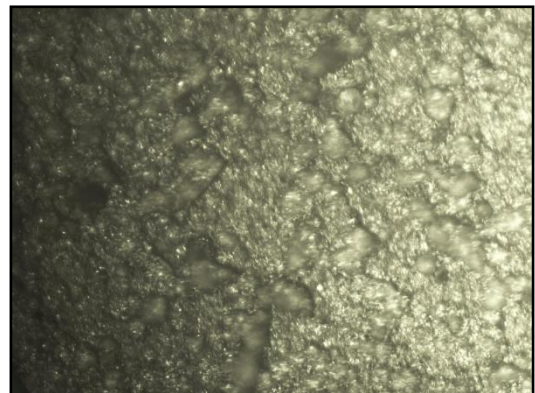


Figure 6.10: Sample for 1000 ppm

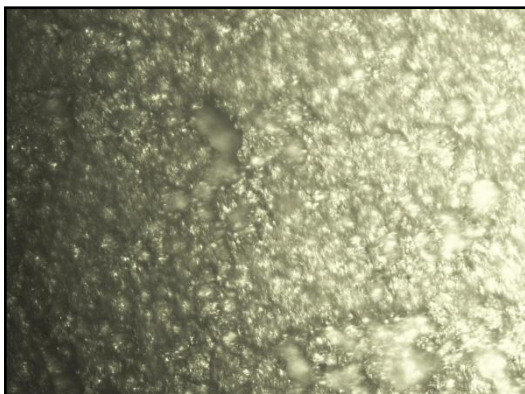


Figure 6.8: Sample for 600 ppm

APPENDIX E
CORROSION RATES FOR WEIGHT LOSS METHOD

Calculation of corrosion rates for 200 ppm

$$\text{Corrosion Rate} = \frac{K \times W}{A \times T \times D}$$

where,

$$K = 8.76 \times 10^4 \text{ is constant value}$$

$$W = 0.305 \text{ g}$$

$$A = 19.144 \text{ cm}^2$$

$$T = 672 \text{ hr}$$

$$D = 2.70 \text{ g/cm}^3$$

$$\text{Corrosion Rate} = \frac{(8.76 \times 10^4) \times 0.305 \text{ g}}{19.144 \text{ cm}^2 \times 672 \text{ hr} \times 2.07 \text{ g/cm}^3}$$

$$\text{Corrosion Rate} = 769.198 \times 10^{-3} \text{ mmpy}$$

Calculation of corrosion rates for 400 ppm

$$\text{Corrosion Rate} = \frac{K \times W}{A \times T \times D}$$

where,

$$K = 8.76 \times 10^4 \text{ is constant value}$$

$$W = 0.294 \text{ g}$$

$$A = 19.144 \text{ cm}^2$$

$$T = 672 \text{ hr}$$

$$D = 2.70 \text{ g/cm}^3$$

$$\text{Corrosion Rate} = \frac{(8.76 \times 10^4) \times 0.294 \text{ g}}{19.144 \text{ cm}^2 \times 672 \text{ hr} \times 2.07 \text{ g/cm}^3}$$

$$\text{Corrosion Rate} = 741.457 \times 10^{-3} \text{ mmpy}$$

Calculation of corrosion rates for 600 ppm

$$\text{Corrosion Rate} = \frac{K \times W}{A \times T \times D}$$

where,

$$K = 8.76 \times 10^4 \text{ is constant value}$$

$$W = 0.292 \text{ g}$$

$$A = 19.144 \text{ cm}^2$$

$$T = 672 \text{ hr}$$

$$D = 2.70 \text{ g/cm}^3$$

$$\text{Corrosion Rate} = \frac{(8.76 \times 10^4) \times 0.292 \text{ g}}{19.144 \text{ cm}^2 \times 672 \text{ hr} \times 2.07 \text{ g/cm}^3}$$

$$\text{Corrosion Rate} = 736.413 \times 10^{-3} \text{ mmpy}$$

Calculation of corrosion rates for 800 ppm

$$\text{Corrosion Rate} = \frac{K \times W}{A \times T \times D}$$

where,

$$K = 8.76 \times 10^4 \text{ is constant value}$$

$$W = 0.290 \text{ g}$$

$$A = 19.144 \text{ cm}^2$$

$$T = 672 \text{ hr}$$

$$D = 2.70 \text{ g/cm}^3$$

$$\text{Corrosion Rate} = \frac{(8.76 \times 10^4) \times 0.290 \text{ g}}{19.144 \text{ cm}^2 \times 672 \text{ hr} \times 2.07 \text{ g/cm}^3}$$

$$\text{Corrosion Rate} = 731.369 \times 10^{-3} \text{ mmpy}$$

Calculation of corrosion rates for 1000 ppm

$$\text{Corrosion Rate} = \frac{K \times W}{A \times T \times D}$$

where,

$$K = 8.76 \times 10^4 \text{ is constant value}$$

$$W = 0.285 \text{ g}$$

$$A = 19.144 \text{ cm}^2$$

$$T = 672 \text{ hr}$$

$$D = 2.70 \text{ g/cm}^3$$

$$\text{Corrosion Rate} = \frac{(8.76 \times 10^4) \times 0.285 \text{ g}}{19.144 \text{ cm}^2 \times 672 \text{ hr} \times 2.07 \text{ g/cm}^3}$$

$$\text{Corrosion Rate} = 718.759 \times 10^{-3} \text{ mmpy}$$

APPENDIX F
CORROSION RATES FOR POTENTIODYNAMIC POLARIZATION

Calculation of corrosion rates for 200 ppm

$$CR = K_1 \frac{i_{\text{corr}}}{\rho} EW$$

Where,

$$i_{\text{corr}} = 77.879 \mu\text{A}/\text{cm}^2$$

$$K_1 = 3.27 \times 10^{-3} \text{ mm g}/\mu\text{A cm y}$$

$$\rho = 2.70 \text{ g}/\text{cm}^3$$

$$EW = 9.01$$

$$CR = (3.27 \times 10^{-3}) \times \frac{77.879}{2.70} \times 9.01$$

$$CR = 850.344 \times 10^{-3} \text{ mm}/\text{yr}$$

Calculation of corrosion rates for 400 ppm

$$CR = K_1 \frac{i_{\text{corr}}}{\rho} EW$$

Where,

$$i_{\text{corr}} = 71.839 \mu\text{A}/\text{cm}^2$$

$$K_1 = 3.27 \times 10^{-3} \text{ mm g}/\mu\text{A cm y}$$

$$\rho = 2.70 \text{ g}/\text{cm}^3$$

$$EW = 9.01$$

$$CR = (3.27 \times 10^{-3}) \times \frac{71.839}{2.70} \times 9.01$$

$$CR = 784.398 \times 10^{-3} \text{ mm}/\text{yr}$$

Calculation of corrosion rates for 600 ppm

$$CR = K_1 \frac{i_{\text{corr}}}{\rho} EW$$

Where,

$$i_{\text{corr}} = 65.103 \mu\text{A}/\text{cm}^2$$

$$K_1 = 3.27 \times 10^{-3} \text{ mm g}/\mu\text{A cm y}$$

$$\rho = 2.70 \text{ g}/\text{cm}^3$$

$$EW = 9.01$$

$$CR = (3.27 \times 10^{-3}) \times \frac{65.103}{2.70} \times 9.01$$

$$CR = 710.848 \times 10^{-3} \text{ mm}/\text{yr}$$

Calculation of corrosion rates for 800 ppm

$$CR = K_1 \frac{i_{\text{corr}}}{\rho} EW$$

Where,

$$i_{\text{corr}} = 63.225 \mu\text{A}/\text{cm}^2$$

$$K_1 = 3.27 \times 10^{-3} \text{ mm g}/\mu\text{A cm y}$$

$$\rho = 2.70 \text{ g}/\text{cm}^3$$

$$EW = 9.01$$

$$CR = (3.27 \times 10^{-3}) \times \frac{63.225}{2.70} \times 9.01$$

$$CR = 690.338 \times 10^{-3} \text{ mm}/\text{yr}$$

Calculation of corrosion rates for 1000 ppm

$$CR = K_1 \frac{i_{\text{corr}}}{\rho} EW$$

Where,

$$i_{\text{corr}} = 62.379 \mu\text{A}/\text{cm}^2$$

$$K_1 = 3.27 \times 10^{-3} \text{ mm g}/\mu\text{A cm y}$$

$$\rho = 2.70 \text{ g}/\text{cm}^3$$

$$EW = 9.01$$

$$CR = (3.27 \times 10^{-3}) \times \frac{62.379}{2.70} \times 9.01$$

$$CR = 681.099 \times 10^{-3} \text{ mm}/\text{yr}$$

APPENDIX G
INHIBITOR EFFICIENCY FOR WEIGHT LOSS MEASUREMENT

Calculation for 400 ppm

$$I_{\text{eff}} = \frac{R_0 - R_i}{R_0} \times 100\%$$

where:

I_{eff} = efficiency of inhibitor, %

$R_0 = 935.648 \times 10^{-3}$ mmpy

$R_i = 741.457 \times 10^{-3}$ mmpy

$$I_{\text{eff}} = \frac{935.648 \times 10^{-3} - 741.457 \times 10^{-3}}{935.648 \times 10^{-3}} \times 100\%$$

$$I_{\text{eff}} = 20.755\%$$

Calculation for 600 ppm

$$I_{\text{eff}} = \frac{R_0 - R_i}{R_0} \times 100\%$$

where:

I_{eff} = efficiency of inhibitor, %

$R_0 = 935.648 \times 10^{-3}$ mmpy

$R_i = 736.413 \times 10^{-3}$ mmpy

$$I_{\text{eff}} = \frac{935.648 \times 10^{-3} - 736.413 \times 10^{-3}}{935.648 \times 10^{-3}} \times 100\%$$

$$I_{\text{eff}} = 21.294\%$$

Calculation for 800 ppm

$$I_{\text{eff}} = \frac{R_0 - R_i}{R_0} \times 100\%$$

where:

I_{eff} = efficiency of inhibitor, %

$R_0 = 935.648 \times 10^{-3}$ mmpy

$R_i = 731.369 \times 10^{-3}$ mmpy

$$I_{\text{eff}} = \frac{935.648 \times 10^{-3} - 731.369 \times 10^{-3}}{935.648 \times 10^{-3}} \times 100\%$$

$I_{\text{eff}} = 21.833\%$

Calculation for 1000 ppm

$$I_{\text{eff}} = \frac{R_0 - R_i}{R_0} \times 100\%$$

where:

I_{eff} = efficiency of inhibitor, %

$R_0 = 935.648 \times 10^{-3}$ mmpy

$R_i = 718.759 \times 10^{-3}$ mmpy

$$I_{\text{eff}} = \frac{935.648 \times 10^{-3} - 718.759 \times 10^{-3}}{935.648 \times 10^{-3}} \times 100\%$$

$I_{\text{eff}} = 23.181\%$

APPENDIX H
INHIBITOR EFFICIENCY FOR POTENTIODYNAMIC POLARIZATION

Calculation for 400 ppm

$$I_{\text{eff}} = 100 \times \left(1 - \frac{I'_{\text{corr}}}{I_{\text{corr}}} \right)$$

where:

I_{eff} = efficiency of inhibitor, %

$$I'_{\text{corr}} = 71.839 \mu\text{A}/\text{cm}^2$$

$$I_{\text{corr}} = 90.943 \mu\text{A}/\text{cm}^2$$

$$I_{\text{eff}} = 100 \times \left(1 - \frac{71.839}{90.943} \right)$$

$$I_{\text{eff}} = 21.007\%$$

Calculation for 600 ppm

$$I_{\text{eff}} = 100 \times \left(1 - \frac{I'_{\text{corr}}}{I_{\text{corr}}} \right)$$

where:

I_{eff} = efficiency of inhibitor, %

$$I'_{\text{corr}} = 65.103 \mu\text{A}/\text{cm}^2$$

$$I_{\text{corr}} = 90.943 \mu\text{A}/\text{cm}^2$$

$$I_{\text{eff}} = 100 \times \left(1 - \frac{65.103}{90.943} \right)$$

$$I_{\text{eff}} = 28.413\%$$

Calculation for 800 ppm

$$I_{\text{eff}} = 100 \times \left(1 - \frac{I'_{\text{corr}}}{I_{\text{corr}}} \right)$$

where:

I_{eff} = efficiency of inhibitor, %

$$I'_{\text{corr}} = 63.225 \mu\text{A}/\text{cm}^2$$

$$I_{\text{corr}} = 90.943 \mu\text{A}/\text{cm}^2$$

$$I_{\text{eff}} = 100 \times \left(1 - \frac{63.225}{90.943} \right)$$

$$I_{\text{eff}} = 30.478\%$$

Calculation for 1000 ppm

$$I_{\text{eff}} = 100 \times \left(1 - \frac{I'_{\text{corr}}}{I_{\text{corr}}} \right)$$

where:

I_{eff} = efficiency of inhibitor, %

$$I'_{\text{corr}} = 62.379 \mu\text{A}/\text{cm}^2$$

$$I_{\text{corr}} = 90.943 \mu\text{A}/\text{cm}^2$$

$$I_{\text{eff}} = 100 \times \left(1 - \frac{62.379}{90.943} \right)$$

$$I_{\text{eff}} = 31.409\%$$

APPENDIX I
SURFACE COVERAGE, θ FOR WEIGHT LOSS MEASUREMENT

Calculation for 400 ppm

$$\theta = 1 - \frac{R_i}{R_0}$$

where,

$$R_0 = 935.648 \times 10^{-3} \text{ mmpy}$$

$$R_i = 741.457 \times 10^{-3} \text{ mmpy}$$

$$\theta = 1 - \frac{741.457 \times 10^{-3}}{935.648 \times 10^{-3}}$$

$$\theta = 0.208$$

Calculation for 600 ppm

$$\theta = 1 - \frac{R_i}{R_0}$$

where,

$$R_0 = 935.648 \times 10^{-3} \text{ mmpy}$$

$$R_i = 736.413 \times 10^{-3} \text{ mmpy}$$

$$\theta = 1 - \frac{736.413 \times 10^{-3}}{935.648 \times 10^{-3}}$$

$$\theta = 0.213$$

Calculation for 800 ppm

$$\theta = 1 - \frac{R_i}{R_0}$$

where,

$$R_0 = 935.648 \times 10^{-3} \text{ mmpy}$$

$$R_i = 731.369 \times 10^{-3} \text{ mmpy}$$

$$\theta = 1 - \frac{731.369 \times 10^{-3}}{935.648 \times 10^{-3}}$$

$$\theta = 0.218$$

Calculation for 1000 ppm

$$\theta = 1 - \frac{R_i}{R_0}$$

where,

$$R_0 = 935.648 \times 10^{-3} \text{ mmpy}$$

$$R_i = 718.759 \times 10^{-3} \text{ mmpy}$$

$$\theta = 1 - \frac{718.759 \times 10^{-3}}{935.648 \times 10^{-3}}$$

$$\theta = 0.232$$

APPENDIX J
SURFACE COVERAGE, θ FOR WEIGHT LOSS MEASUREMENT

Calculation for 400 ppm

$$\theta = 1 - \frac{i_{\text{corr}}'}{I_{\text{corr}}}$$

where,

$$i_{\text{corr}}' = 71.839 \mu\text{A}/\text{cm}^2$$

$$I_{\text{corr}} = 90.943 \mu\text{A}/\text{cm}^2$$

$$\theta = 1 - \frac{71.839}{90.943}$$

$$\theta = 0.210$$

Calculation for 600 ppm

$$\theta = 1 - \frac{i_{\text{corr}}'}{I_{\text{corr}}}$$

where,

$$i_{\text{corr}}' = 65.103 \mu\text{A}/\text{cm}^2$$

$$I_{\text{corr}} = 90.943 \mu\text{A}/\text{cm}^2$$

$$\theta = 1 - \frac{65.103}{90.943}$$

$$\theta = 0.284$$

Calculation for 800 ppm

$$\theta = 1 - \frac{I'_{\text{corr}}}{I_{\text{corr}}}$$

where,

$$I'_{\text{corr}} = 63.225 \mu\text{A}/\text{cm}^2$$

$$I_{\text{corr}} = 90.943 \mu\text{A}/\text{cm}^2$$

$$\theta = 1 - \frac{63.225}{90.943}$$

$$\theta = 0.305$$

Calculation for 1000 ppm

$$\theta = 1 - \frac{I'_{\text{corr}}}{I_{\text{corr}}}$$

where,

$$I'_{\text{corr}} = 62.379 \mu\text{A}/\text{cm}^2$$

$$I_{\text{corr}} = 90.943 \mu\text{A}/\text{cm}^2$$

$$\theta = 1 - \frac{62.379}{90.943}$$

$$\theta = 0.314$$