CORROSION OF MILD STEEL TOWARDS 1-BUTYL-3-METHYLIMIDAZOLIUM TETRACHLOROFERRATE

By

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

(Chemical Engineering)

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the Chemical Engineering Programme
Universiti Teknologi PETRONAS
In Partial Fulfillment of the requirement for the
Bachelor of Engineering (Hons) Chemical Engineering

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UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
May 2012

CERTIFICATION OF ORIGINALITY

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

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ABSTRACT

The ionic liquid 1-butyl-3-methylimidazolium tetrachloroferrate (Bmim [$FeCl_4$] have received vast attention recently, due to its versatility and broad usage in petrochemical industry. Several researches have been carried out to study the importance of Bmim [$FeCl_4$] in the industrial application and it plays important roles in many sector of the industry as in desulfurization of crude oil and synthesizing polymer (C.X. Wang, 2010). However, there is no paper report on the hazard of Bmim [$FeCl_4$]. Thus, this project is designed to develop the corrosion rate of Bmim [$FeCl_4$] towards A36 mild steel. The characteristic of the steel is studied to determine the composition of the steel which is crucial in the study of corrosion process. The A36 mild steel is widely used in the petrochemical industry as the raw material in piping and storage tank (L. Brockenbrough, 1994). Therefore, the study of corrosion of ionic liquid Bmim [$FeCl_4$] towards mild steel is very crucial. The rate of corrosion, mm/year is examined in this project. The main methods used to determine the corrosion rate is immersion test and Electrochemical Corrosion Measurement Techniques. The parameters of water content and temperature effect are studied in this project. The polarization technique is applied on both parameters and Tafel plot, Potentiodynamic and Linear Polarization Resistance (LPR) is designed. The technique and procedure highlighted in this project is based on several journals and reference books.

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CHAPTER 1: INTRODUCTION

1.1 Background Study

An ionic liquid (IL) is a salt that are liquid state at low temperature, which the ions are poorly coordinated and resulting to exist in liquid form at below 100°C or even at room temperature.. The advantages of using ionic liquid are it is thermally and hydrolytically stable, has wide liquid range and relatively low viscosity and wide electrochemical window. The awareness in ionic liquid stems from their prospective as 'green solvents' because of their non-volatile nature and thermal stability, which is the exceptional properties for application as solvents to substitute high toxic and volatile organic solvents (Wasercheid & Keim, 2004).

1-butyl-3-methylimidazolium tetrachloroferrate (Bmim [$FeCl_4$]) is one of the room temperature ionic liquids (RTILs) which it are salts that are liquid at or below room temperature. The invention of the magnetic behavior of Bmim[$FeCl_4$] leads to the entirely new field for ionic liquids and their application (D. Kogelnig, 2010). The research shown that Bmim [$FeCl_4$] can be used for the magnetic manipulation and transport of nitrogen bubbles, in catalytic and polymerization reactions. It also utilized in photo controllable paramagnetic supramolecular system (K. Bica, 2006).

Meanwhile, in the oil and gas industry, Bmim [$FeCl_4$] is used as the ionic liquid in desulfurization of crude oil. Bmim [$FeCl_4$] solid support ionic liquid is utilized to replace conventional method for sulfur removal in refining process which is catalytic hydrodesulfurization (HDS). The cost of current process of HDS is expensive as it is operates at high temperature and pressure. Furthermore, the process is inefficient to reduce aromatic sulfur compounds and obliges high hydrogen utilization under rigorous condition (Fa-tang Li, 2008).

Thus, ionic liquid was pioneered as alternative to remove sulfur from the natural raw crude oil. It is chosen as the best method because Bmim [$FeCl_4$] have an exceptional removal behavior for organic sulfur and nitrogen compounds which characterize a new rank of solvents and extracting agent. In addition, the ionic liquid has a low melting salt with no quantifiable vapor pressure. Bmim [$FeCl_4$] solid support ionic liquid is capable to extract organic sulfur species from the natural raw crude oil by using solid-liquid extraction technique.

In all of these processes, mild steel is involved as in pipes and tanks. Apart from all of the usage of Bmim [$FeCl_4$], there is no scientific study or research conducted on the behavior of Bmim [$FeCl_4$] towards corrosion. Therefore, the objective of this project is to study the corrosion of mild steel towards iron chloride and the ionic liquid of Bmim [$FeCl_4$] is utilized in this experiment.

1.1.1 Corrosion

Corrosion can be described as the degradation of a material because of reaction with its environment, and corrosion of most metal is inevitable which implies deterioration of physical properties of the material that can be a weakening of the material due to a loss of cross-sectional area (Barbara A.Shaw, 2006). Most metals utilized in the production of facilities are subject to corrosion because of elevated energy content of the elements in metallic composition. The phenomena of corrosion affect all industrial sectors including chemical and petrochemical industry. Corrosion of metallic structure has a vast impact on the economy, including transportation, utilities, manufacturing, production and infrastructure. Major companies lose millions and billions of dollars to replace and repair materials and machineries deteriorated by corrosion. The cost of corrosion is estimated at more than 4.2% of the nation's gross national product (GNP) in a number of countries (G. Abdellah, 2008).

There are many other effects of corrosion in the plant. One of them is unscheduled shut down. Consider the example of a large polymer plant which was forced to shut down due to excessive leakage of condenser tube. The plant is committed to supply products on a continuous basis at a fixed price to large cross-section of industrial customer. Losses of the production of the polymer will the company to purchase polymer from other polymer producing company to supply the customers. Furthermore, the unscheduled shutdown of a chemical plant or refinery possibly will involve interference of activities not only at the plant where failure take places, but also may suspend operations of several other plants which depend on the first plant for their supply of raw materials.

1.1.2 A36 Mild Steel

The steel tested in this project is A36 mild steel. The mainly frequent steel used and the slightest expensive of all steel is mild steel. There are many metal elements along with carbon operate as hardening agents which thwart dislocations from occurring inside the iron crystals and foil the lattice layers from sliding past each other. This is what makes steel harder than iron.

Table 1: Mild steel's chemical composition

Chemical composition	C	Mn	P	S	Si	Fe
Mass %	20	51	1.3	3.9	17	6.8

http://www.astm.org

In chemical industry, most of the pipeline in the world is created using a mild version of steel. This mild makeup allows the pipe to not only be easily welded into place, but also lets the pipeline flex and avoid cracking and breaking under pressure. The corrosive properties of the steel pipeline dictate that proper sealing of the pipe be accomplished through painting or a process often used on pipelines that involves wrapping the pipe with a corrosive-resistant material. Mild steel is available in a variety of structural shapes that are easily welded into pipe, tube and tubing. Mild steel pipes and tubes are easy to fabricate, readily available, and cost less than most other metals.

Mild steel also is broadly used in the fabricated of storage tank. There two tank that usually manufactured from low carbon mild steel which are vertical storage tank and cone bottom tanks. Vertical storage tanks are made-up of low carbon mild steel. They are welded externally for maximum resistance to corrosion and weather and internally welded to prevent crevices and pockets. Precision's cone bottom tanks are fabricated of low carbon mild steel. They are welded externally for maximum resistance to corrosion and weather, and welded internally to prevent crevices and pockets.

1.2 Problem Statement

For their versatility and properties, ionic liquid have a vast array of prospective functions for chemical industry and especially for petroleum industry, which the number of study and papers on the evaluation of ILs for industrial applications has been increase significantly over the years. However, there are no research has been done on the corrosion behavior of ionic liquid Bmim [$FeCl_4$] towards mild steel. The research on the corrosion rate needs to be done as it is vital to define the corrosion behavior of the material (G. Abdellah, 2008). Corrosion rate are determine from the loss in weight and exposure time of the mild steel which are inserted in appropriate locations in the steel. The corrosion rates in a chemical plant are dominated by several major factors which are solution chemistry, metallurgical and mechanical effect such as stress and strain.

Moreover, the corrosion mechanism of Bmim [$FeCl_4$] is not scientifically proven. Corrosion mechanism is important as modern corrosion science has its roots in electrochemistry and metallurgy. Electrochemistry contributes an understanding of the mechanism that is basic to the corrosion of all metallic objects. Metallurgy provides knowledge of the characteristics of metals and their alloys as well as the methods of combining the various metals (P. Marcus, 2002).

From the corrosion control standpoint, the best approach is to identify the factors that cause the corrosion in order to reduce it. There are some factor effecting corrosion like environmental factor and temperature. Temperature considerations are imperative as speed of electrochemical attack is increased in a high temperature. H_2O content also is one of the major factors contributing to the corrosion and polarization technique will be used to study these factors.

1.3 Objectives

Objectives of this project are:

1. To measure the corrosion rate of pure Bmim [$FeCl_4$].
2. To study the effect of temperature to the corrosion of pure Bmim [$FeCl_4$].
3. To study the effect of H_2O content to the Bmim [$FeCl_4$] rate of corrosion.

1.4 Scope of Work

This project essentially focuses on the corrosion behavior of ionic liquid 1-butyl-3-methylimidazolium tetrachloroferrate towards mild steel. Several stages are divided in order to achieve all the objectives of the project precisely. This project will be done according to time frame and planned scheduled. The corrosion behavior due to Bmim [$FeCl_4$] affected by the following process parameters which are open and control environment, H_2O content and temperature.

Table 2: Parameters that will be tested in the experiment

Parameter	Testing condition
Open and control environment	• Immersion test • Control environment: Sample placed in the glove box • Open environment: Sample leave to the room temperature and pressure
Temperature	• Temperature of 30, 40, 50, and 60 °C is studied
H_2O content	• H_2O volume of 0.2 ml, 1ml, 5ml and 10ml is mixed with 10 ml Bmim [$FeCl_4$] and examined

1.5 Relevancy of the Project

This project is relevant to the industry as ionic liquids have shown great potential to be explored in the future. Bmim [$FeCl_4$] has been used widely in the industrial where mild steel is involved as the material in pipeline and reactor. However, there is no research or study has been conducted so far to examine the corrosion rate of ionic liquid Bmim [$FeCl_4$] towards mild steel.

1.6 Feasibility of the Project

This project is practical within the time framework since it engages mainly experimental based activities which the university fully possess the equipment. Additionally, all the resources required for this project are generally accessible in the university. Thus it is not necessary to consume time for travelling to other locations in order to get the necessary resources or equipment usage.

CHAPTER 2: LITERATURE REVIEW

2.1 Basic Corrosion Theory

Corrosion is principally an electrochemical progression which there is two reactions occur in equilibrium except there is an exterior mechanism. Electrochemical reaction composed of two half-cell reactions which are anodic reaction and a cathodic reaction, where the reaction releases electrons and consumes electrons respectively. There are three common cathodic reactions, oxygen reduction which is fast, hydrogen evolution from neutral water, slow, and hydrogen evolution from acid, fast.

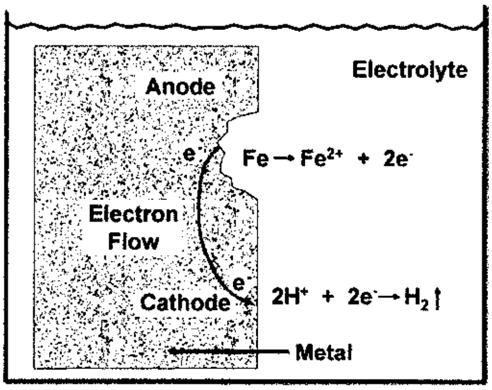

Figure 1: Corrosion Cell

The electron transfer from anode to cathode for iron and steel:

Anodic reaction:

$Fe \rightarrow Fe^{2+} + 2e^-$

Cathodic reactions:

- $O_2 + 4 H^+ + 4e^- \rightarrow 2H_2O$ (oxygen decline in acidic solution)
- $1/2\ O_2 + H_2O + 2e^- \rightarrow 2\ OH^-$ (oxygen lessening in neutral or basic solution)

- $2\ H^+ + 2e^- \rightarrow H_2$ (hydrogen formation from acidic solution)
- $2\ H_2O + 2e^- \rightarrow H_2 + 2\ OH^-$ (hydrogen growth from neutral water)

2.2 Formation of Corrosion Cell

As a result of differential in energy between the metal and the electrolyte, corrosion cells are form on metal surfaces which contact with the electrolyte. Dissimilar area on the metal surface could also have diverse potentials relating to the electrolyte. These dissimilarities could be affected by metallurgical factors, where there are differences in the composition, microstructure, fabrication, and field installations, or else environmental factors. Mild steel is the mainly utilized material in the chemical plant. Stainless steels (Fe-Cr-Ni) and nickel base corrosion resistant alloys (CRA), such as Incoloys (Ni-Fe-Cr), Inconels (NiCr) and Hastelloys (Ni-Cr-Mo-Fe-Co) are also applied in extremely corrosive environments.

i) Metallurgical factors

Steel is an alloy of iron (Fe) and carbon (C) which is quite soluble in liquid iron at steel manufacturing temperatures. Small quantity of carbon and manganese are inserted in the soft and malleable pure iron to increase its strength and toughness. During steelmaking, the majority of the carbon is oxidized. The remaining carbon and post fabrication heat treatment resolves the microstructure of steels which determines it strength and hardness. In highly deformed metals, the grains are deformed and the grain arrangement is utterly disordered (M. I. Masoud, 2005). Generally, the material is more reactive in electrochemical environments. In addition, the influence of impurities, inclusions, grain boundaries and differences in granule orientation also may results in drastically diverse electrochemical reactivates in many metals and alloys. This will cause high corrosion possibility.

ii) Environmental factors

Due to difference in the concentration of forceful ions in the environment, part of the same steel may corrode in a different way such as, a pipeline could go by several formations or soils with diverse water composition, consequently, the different rates of corrosion might experience by different section of the pipe. Likewise, a pipeline across a dry land will exposed to lower concentration of salt as compared to a pipeline crossing a river where sections of pipe exposed to higher salt concentrations turn out to be anodic and corrode. Variation in the oxygen concentration on the metal surface will results in particularly insidious forms of corrosion.

2.3 Overview of Ionic Liquid

Ionic liquids are salts that have low enough melting points to be liquid at room temperature. Applied research has focused on exploring the practicalities of using different ionic liquids in chemical processes. Ionic liquids are non-volatile, highly thermally stable, and have tunable miscibility, viscosity and solubility (Wasserscheid, 2003).

Because ionic liquids are composed of only ions, they show very high ionic conductivity, no volatility, and no flammability (H. Ohno, 2005). In many cases, the use of ionic liquids has been considered as the key element in the design of more environmentally benign methods for separations. These solvents (ionic liquids) display several properties that make them feasible as a potential basis for “greener“ separation processes, such as negligible vapor pressure, a wide liquid range, and good thermal stability (D. Rodgers, 2005). Currently, most of the attention in ionic liquids is centered on the design of new solvents. While the development of “new solvents” has led the direction of possible applications of ionic liquids, there is more potential for development in electrochemical applications as well.

2.3.1 Room Temperature Ionic Liquid (RTIL)

Room temperature ionic liquids (RTILs) are basically organic salts which exist in liquid form at ambient temperature. It normally composed of relatively large asymmetric organic cations and inorganic or organic anions. Due to the impressive characteristics of RTILs such as insignificant vapor pressure and solubility of a wide range of organic and inorganic compounds, they have generated a lot of interest in the industry as green solvents. Ionic liquids come in two main categories, namely simple salts where single anion and cation as well as binary ionic liquids where salts with equilibrium are involved. Figure 2 shows simple room temperature ionic liquids.

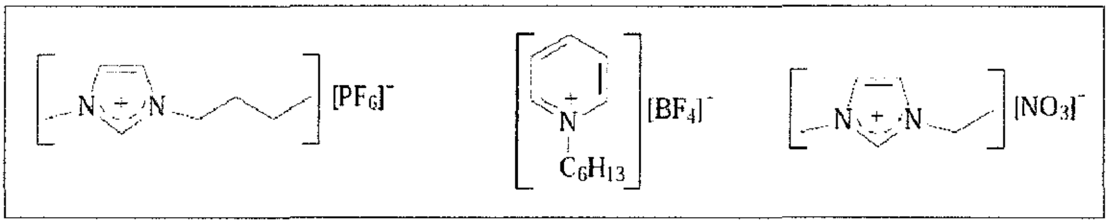

Figure 2: Room Temperature Ionic Liquid

2.3.2 Imidazolium Based Ionic Liquid

Imidazole is defined as organic compounds of the heterocyclic series which consists of two nitrogen atoms and three carbon atoms at nonadjacent locations such as $C_3H_4N_2$. Salts of imidazole is where the imidazole ring is in the cation are known as imidazolium salts such as imidazolium chloride. These salts are produced from the protonation or exchange at nitrogen of imidazole where it is applied as ionic liquids and precursors to stable carbons. Salts where a deprotonated imidazole is an anion are also achievable where these salts are known as imidazolide salts, for instance, sodium imidazolide.

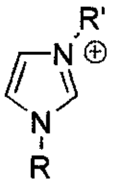

Figure 3: Imidazolium Structure

2.4 1-butyl-3-methylimidazolium tetrachloroferrate Bmim [$FeCl_4$]

Recently, the interest of ionic liquid 1-butyl-3-methylimidazolium tetrachloroferrate has increasing due to its properties and versatility. There are several technologies have been explored to maximize the utilization of Bmim [$FeCl_4$] in the industrial application. The function of Bmim[$FeCl_4$] in the polymer synthesizing is proved in the research which it can be used as a easy and efficient ionic liquid (IL)-assisted mechanochemical path to form a set of nanostructured graphene nanosheet/polypyrrole (GNS/PPy) compounds with diverse PPy loading (Xiangjun Lu, 2011). 1-butyl-3-methylimidazolium tetrachloroferrate (Bmim[$FeCl_4$]) can be applied as the dispersant of GNS as well as the catalyst in the synthesis of PPy. The research conducted on composites and pure PPy has conclude to main outcome, which is first, GNS/PPy composites show enhanced conductivity and thermal stability as compared with chaste PPy and secondly, the microstructure of the GNS/PPy composites is reliant on the loading of PPy.

In addition, application of ionic liquids has increased significantly in the chemical research area as a catalyst in petrochemical. Recent study shows that the catalytic activity of 12-tungstophosphoric acid immobilized on the Bmim [$FeCl_4$] ionic liquid as a highly efficient and eco-friendly catalytic system for rapid and chemoselective direct conversion of methoxymethyl ethers (MOM-ethers) or ethoxymethyl ethers (EOM-ethers) into their corresponding bromides, iodides and nitriles under microwave irradiation (M. Moghadam, 2010). High yields of products are attained in this reaction, which remarkable reactivity are shown by the catalyst and was reused several times.

Furthermore, Bmim [$FeCl_4$] also applied in the sulfur removal of crude oil (A. Doroodian, 2010). The cost of hydrodesulfurization (HDS) which is widely used is very high. Therefore, Bmim [$FeCl_4$] properties and capability in removing sulfur is examined to overcome this problem.

Table 3: Sulfur Removal by Ionic Liquids

Ionic Liquid	**Final Sulfur Content in Hydrcarbon (ppm)**	**% of Total Removed Sulfur in Hydrocarbon**
[BMIM][$FeCl_4$]	>10	99
[BDMIM][$FeCl_4$]	>10	99
[BDIM][$FeCl_4$]	14	97
[OP][$FeCl_4$]	152	70
[HEMIM][$FeCl_4$]	352	35

From Table 3, the ionic liquids with the tetrachloroferrate anion can roughly quantitatively eliminate the sulfur content of the sample original model; particularly the ionic liquids contain imidazolium cation. As a result, the ionic liquids can be utilized for the deep desulfurization of hydrocarbon mixtures, such as gasoline, kerosene, jet fuel and light cyclical oil. Therefore, the use of ionic liquids containing halogens of Fe (III) can be concluded as an anion for these functions, where these compounds presented very high competence for removing sulfur containing compounds from gasoline, diesel and other petroleum fractions.

The other research that proves the versatility of Bmim [$FeCl_4$] is that it is applied as a catalyst in the depolymerization of poly (ethylene terephthalate) (PET) in ethylene glycol (C.X. Wang, 2010). As compared to other magnetic liquid such as $FeCl_3$ or 1-butyl-3-methylimidazolium chloride ([Bmim]Cl), Bmim [$FeCl_4$] demonstrates superior catalytic activity for the glycolysis of PET. The proposed mechanism shows that the high catalytic activity of Bmim [$FeCl_4$] is accredited to the synergic outcome of its cation and anion. However, there is no study of corrosion for Bmim [$FeCl_4$].

2.5 The Study of Corrosion Rate

The study of corrosion inhibition of 3% de-aerated NaCl acidic solution towards carbon steel is done with amine-fatty acid corrosion inhibitor, which different temperatures are tested using potentiodynamic polarization technique (J Buchweishaija, 2002). The corrosion rate was calculated both in the presence and absence of inhibitor where the inhibition decreased with the decreasing in inhibitor concentration and also decreased as the temperature rises. The inhibitor operates as a mixed category at lesser temperature and predominantly anodic at high temperature. In this study, the interrelationship between the surface coverage and inhibitor's concentration was also examined. The electrochemical experiments were performed, where the cell was placed in a water bath and the temperature controlled in the range of 30 °C to 60 °C. The Electrochemical Measurement Techniques in a range of -250 mV to 250 mV is also analyzed in the experiment.

Another corrosion rate study of mild steel is done on the thiophene azodyes (1-3) in 2 M HCl where there are two techniques used namely EIS and LPR (A.S. Fouda, 2010). In this study, weight loss method is also examined. The effect of corrosion is analyzed at different temperature ranging from 30 °C to 60 °C. The mild steel's weight loss is also studied, where the initial and final weight after immersed in the thiophene azodyes (1-3) is recorded. The morphology of the steel at the initial and final experiment is examined by using Scanning Electron Microscope (SEM).

However, there are no study has been done on the corrosion rate and corrosion behavior of Bmim [$FeCl_4$] towards mild steel.

2.5.1 Electrochemical Impedance Spectroscopy (EIS)

Recently, EIS technique has establish prevalent applications in the field of materials' characterization. One of the method's advantages is the utilization of very small amplitude signals without significantly disturbing the properties being measured as compared to laboratory techniques. It is routinely used in the characterization of batteries, coatings, fuel cells and corrosion phenomena.

It has also been used broadly as a tool for investigating mechanisms in electro deposition, electro dissolution, corrosion studies and passivity. An electrochemical process may often be modeled by linear circuit elements such as resistors, capacitors, and inductors. For instance, the corrosion reaction itself can often be modeled by one or more resistors. The capability of EIS to model a corrosion process such as the corrosion rate in this manner gives rise to one practical attribute of the electrochemical impedance technique. Simple corrosion cell can be used to model the electrochemical corrosion process. Such modeling can facilitate understanding and lead to better prediction of corrosion rates and overall corrosion behavior.

2.5.2 Linear Polarization Resistance (LPR)

Linear Polarization Resistance is the electrochemical technique where it is the only corrosion monitoring technique that able to calculate the corrosion rate directly, in real time. The polarizing voltage of 10 mV is selected as being well within the limits for which the linear connection between I_{corr} and $\Delta E/\Delta I$ holds. In addition, the value is suitably small as to cause no significant or enduring distraction of the corrosion progression, so that consequent capacity stay relevant. Anodic and cathodic sites frequently alter position, and they present within a continuously conductive surface, making direct measurement of I_{corr} impossible.

Small, externally-imposed and potential shifts (ΔE) will construct quantifiable current flow (ΔI) at the corroding electrode. The manners of the externally imposed current are managed, as is that of I_{corr} by the measure of complexity with which the anodic and cathodic corrosion processes occur. Therefore, the superior intricacy will provide lesser value of I_{corr} and ΔI for a particular potential shift. At small value of ΔE, ΔI is directly proportional to I_{corr} and hence to the corrosion rate. The Stern-Geary equation is as followed:

$$\frac{\Delta E}{\Delta I} = \frac{\beta_a \beta_c}{2.3\,(I_{corr}\,(\beta_a \beta_c)}$$

Where, β_a and β_c are the Tafel slopes of the anodic and cathodic reactions respectively.

2.5.3 Scanning Electron Microscope (SEM)

SEM has expanded new areas of study in the medical and physical science communities since its development in the early 1950's. Nowadays, SEM is broadly used in the engineering application, where it is a type of microscope that applies electrons to form an image instead of using light. Its ability allows researchers and scientist to examine a much bigger array of specimens. The scanning electron microscope capability produces many advantages as compared to the traditional microscopes where it not just permitted more number of specimens to be in focus at one time, it also has much higher resolution that makes specimens capable to be magnified at much higher levels. Since SEM utilizes electromagnets rather than lenses, the researchers and scientists are more in control of the extent of magnification.

As a start-up process, the specimens must first be made electrically conductive where the sample is coated with conductive materials such as thin layer of gold, platinum, palladium, and alloy. The coating process is done by sputter coater where electric field and argon gas are utilized. The sample is first positioned in a vacuum condition of a small chamber. Argon gas and an electric field will then get rid of the electrons from the argon, which makes the atoms positively charged. The argon ions will be attracted to a negatively charged gold foil. These argon ions attack the gold atoms at the surface of the gold foil, making these gold atoms fall and resolve on the surface of the sample, consequently producing a thin gold coating.

2.5.4 Weight Loss Method

The weight loss technique measurement is based on geometric weight loss measurement. This measurement needs long exposure time to the ionic liquid in certain condition accomplish quantifiable corrosion rate. The weight loss method is applicable for corrosion rate measurements. The initial weight before the sample immersed is measured and recorded. After several time, the sample is take out and weigh to attain the weight difference. The corrosion rate in mm yr^{-1} is determined by the following equation (B. Si Ali, 2012).

$$\text{Corrosion rate (mm yr}^{-1}\text{)} = \frac{8.76\ x\ 10^4\ x\ W}{D\ x\ A\ x\ t}$$

Where; the coefficient, K is 8.76×10^4, W is the weight loss (g), D is the density (g/cm^3), A is the exposed area (cm^2), t is exposure time in hour.

CHAPTER 3: METHODOLOGY

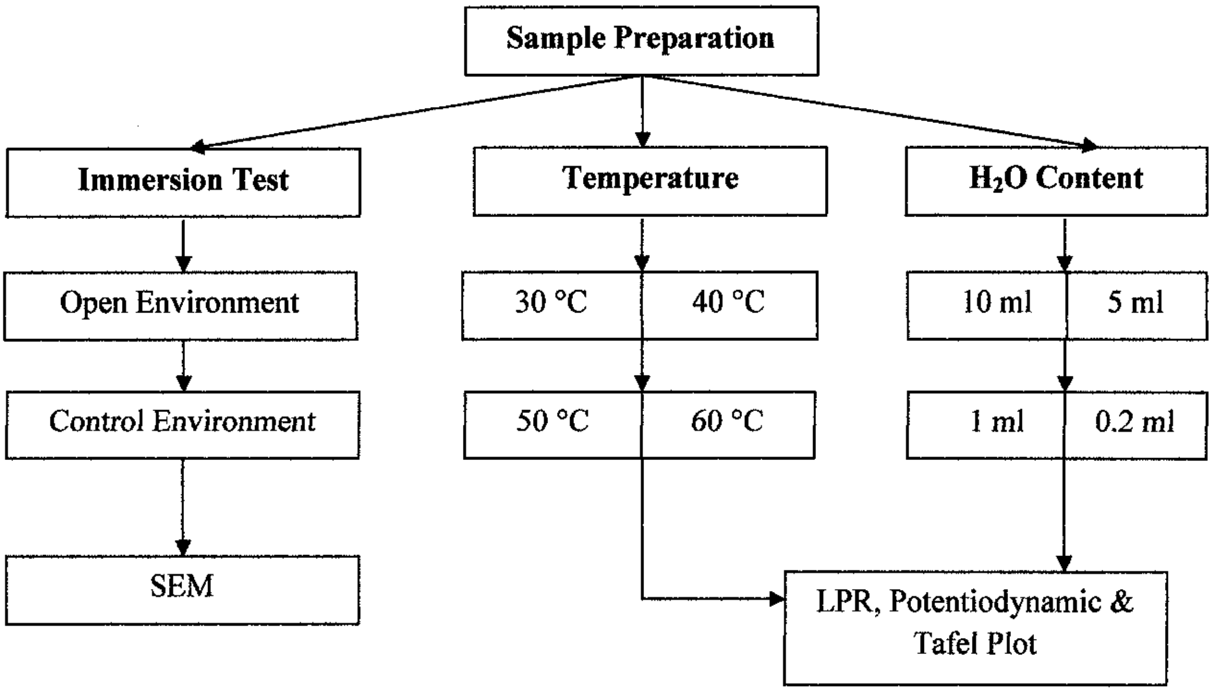

Figure 4: Flowchart of Methodology Involves

3.1 Sample Preparation

1. A piece of A36 mild steel plate is cut into smaller pieces on the dimension of 525 mm^3 by using abrasive cutter machine.

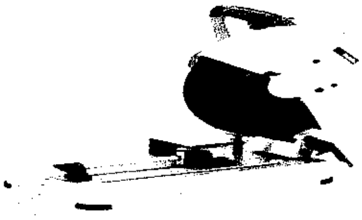

Figure 5: Abrasive Cutter

3.2 Immersion Test

1. The sample is grinded using Metkon FORCIPOL 2V Grinder & Polisher through increasingly fine polishing paper sequentially from 240, 320, 400,600,800 and 1200 grit.

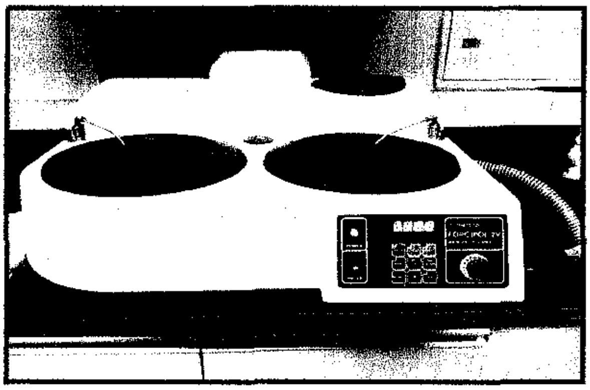

Figure 6: Grinder Machine

2. The sample is then polished using 6um DIAMAT Polycrystalline Diamond from 6um to 3um and finally 1um.
3. The samples degreased with acetone, rinsed in distilled water and then dried in the oven.
4. The dimension of two samples is measured the weight recorded.
5. Immersed the samples into two separate bottles which first specimens placed in the glove box for control environment and the other one in the room for open environment

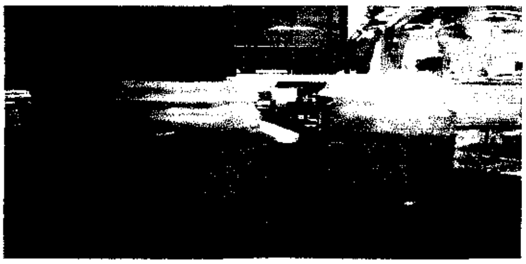

Figure 7: Specimens for Immersion Test

6. After 60 days, the samples' dimension and weight difference is recorded as well as examined using SEM.

3.2.1 Scanning Electron Microscopy (SEM) Testing

1. The samples are coated with palladium alloy using sputter coater.

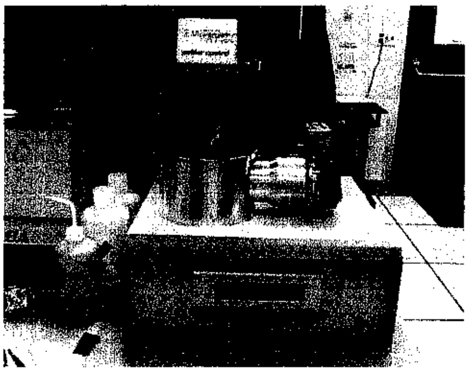

Figure 8: Sputter Coater

2. Firmly mounted the samples in the small chamber and placed in the SEM
3. The samples' surfaces are examined.

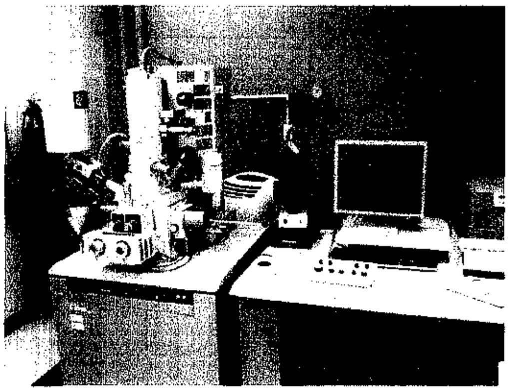

Figure 9: Scanning Electron Microscope

3.3 Electrochemical Measurement Techniques

1. New steels will be spot welded with copper wire before submerged into the epoxy resin and leave to harden for about 24 hours.

Figure 10: Samples Mounting

2. The samples are then grinded by wet silicon carbide paper leaving a working area of steel of 0.40 cm^2 and polished.
3. The samples degreased with acetone, rinsed in distilled water and leave dried in the oven.
4. The experiment conducted in a conventional three-electrode glass cell assembly with grafite as counter electrode (auxiliary), AgCl as reference electrode and A36 mild steel as working electrode.
5. The experimental setup is as below:

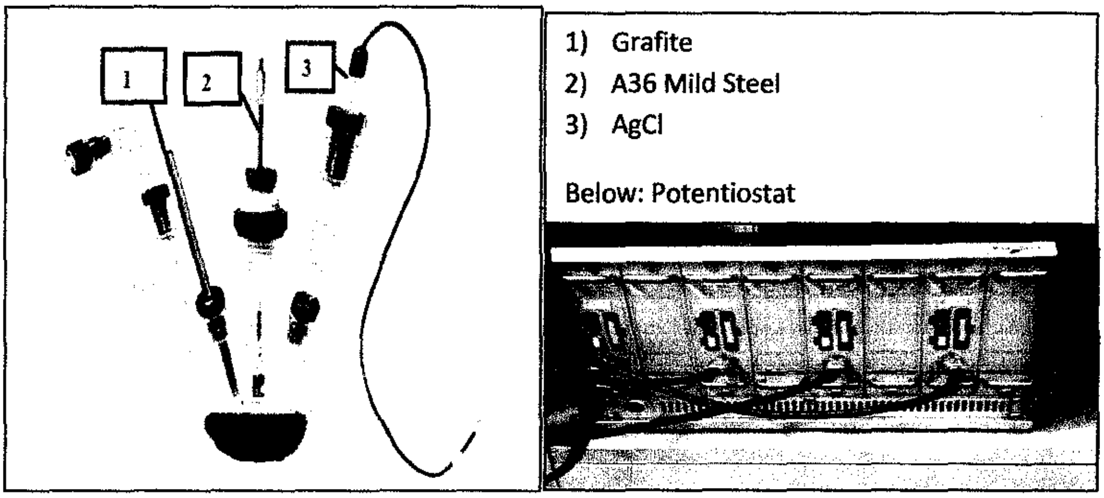

Figure 11: Experimental Setup

6. The corrosion cell is connected to potentiostat which connected to the computer.
7. The corrosion rate will be measured using IVMan and WEIS software where Potentiodynamic, LPR and Tafel plot can be obtained.

3.3.1 Temperature Effect

1. The temperatures of 30 °C, 40 °C, 50°C and 60 °C is tested in the experiment.
2. The corrosion cell is immersed in the water where water bath will control the desired temperature.
3. The experimental setup is as figure below:

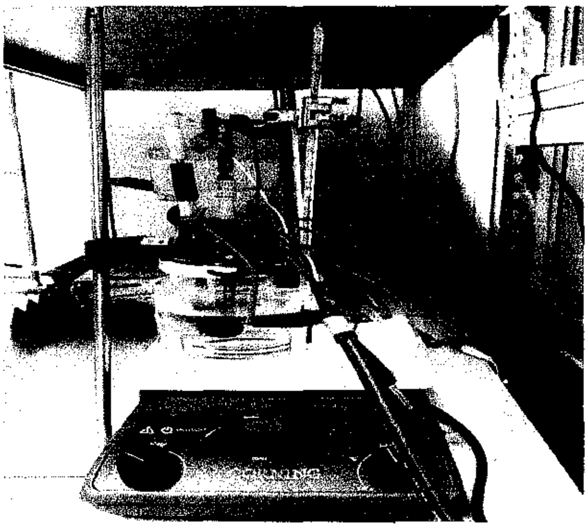

Figure 12: Temperature Effect

3.3.2 H_2O Content

1. The ionic liquid Bmim [$FeCl_4$] is mixed with water content of 10 ml, 5 ml, 1 ml and 0.2 ml.
2. The mixture is then placed in the corrosion cell and examined.

3.4 Gantt Chart

No.	Detail/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14		1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Briefing on Final Year Research Project Background	■						MID-SMESTER BREAK								SEM-BREAK														
2	Preliminary Research Work/Literature survey		■	■	■	■	■																							
3	FYP seminar "Research Methodology"			■																										
4	Preparation of Extended Proposal Defense				■	■																								
5	Sample Preparation				■	■	■		■	■	■																			
6	Immersion Test					■	■		■	■	■	■	■	■																
7	Study on temperature content																■	■	■	■	■		MID-SMESTER BREAK							
8	Study on water content																			■	■	■		■	■					
9	EDX																									■	■			
10	FYP Final Presentation																											■	■	
11	Dissertation submission																											■	■	

CHAPTER 4: RESULTS & DISCUSSIONS

4.1 Immersion Test

Sample A, Glove box:

Height: 2.82 mm

Width: 13.03 mm

Length: 14.3 mm

Duration: 10/2/2012 - 26/4/2012, 76 days

Weight:

1. Initial: 4076.18 mg
2. Final: 4062.58 mg

Weight difference: 13.60 mg

Corrosion rate (mm yr^{-1}) = $\frac{8.76\ x\ 10^4\ x\ W}{D\ x\ A\ x\ t}$

$$= \frac{8.76\ x\ 10^4\ x\ 0.0136}{7.85\ x\ 1.8633\ x\ 1824} = \mathbf{0.04465\ mm\ yr^{-1}}$$

Sample B, Open Environment:

Height: 2.80 mm

Width: 13.78 mm

Length: 12.8 mm

Duration: 10/2/2012 - 26/4/2012, 76 days

Weight:

1. Initial: 3792.48 mg
2. Final: 3566.29 mg

Weight difference: 226.19 mg

Corrosion rate (mm yr^{-1}) = $\frac{8.76\ x\ 10^4\ x\ W}{D\ x\ A\ x\ t}$

$= \frac{8.76\ x\ 10^4\ x\ 0.22619}{7.85\ x\ 9.4438\ x\ 1824}$ = **0.14653 mm yr^{-1}**

Weight loss method was carried out to obtain the corrosion rate of Bmim [$FeCl_4$] where two parameters were investigated which are control environment and open environment. For control environment, at oxygen and water concentration below than 0.1 ppm, the corrosion rate obtain is 0.04465 mm yr^{-1}. For sample B, at room environment, the corrosion rate of the ionic liquid obtains is 0.14653 mm yr^{-}1. Corrosion rate at room environment is higher than the control environment, which proves that the concentration level of oxygen and water favor high corrosion rate.

4.1.1 Scanning Electron Microscopy (SEM)

SEM is a method of getting the images of the surface structure of the sample. The electrons beam will be attracted to the atoms and creates the patterns and images of the structure. Three samples were tested. The first sample is control sample which is a36 mild steel before immersed in the Bmim [$FeCl_4$]. The second sample is the mild steel after being immersed in the ionic liquid for 76 days and set at control environment. Meanwhile, the third sample is placed at room environment.

a) Control Sample

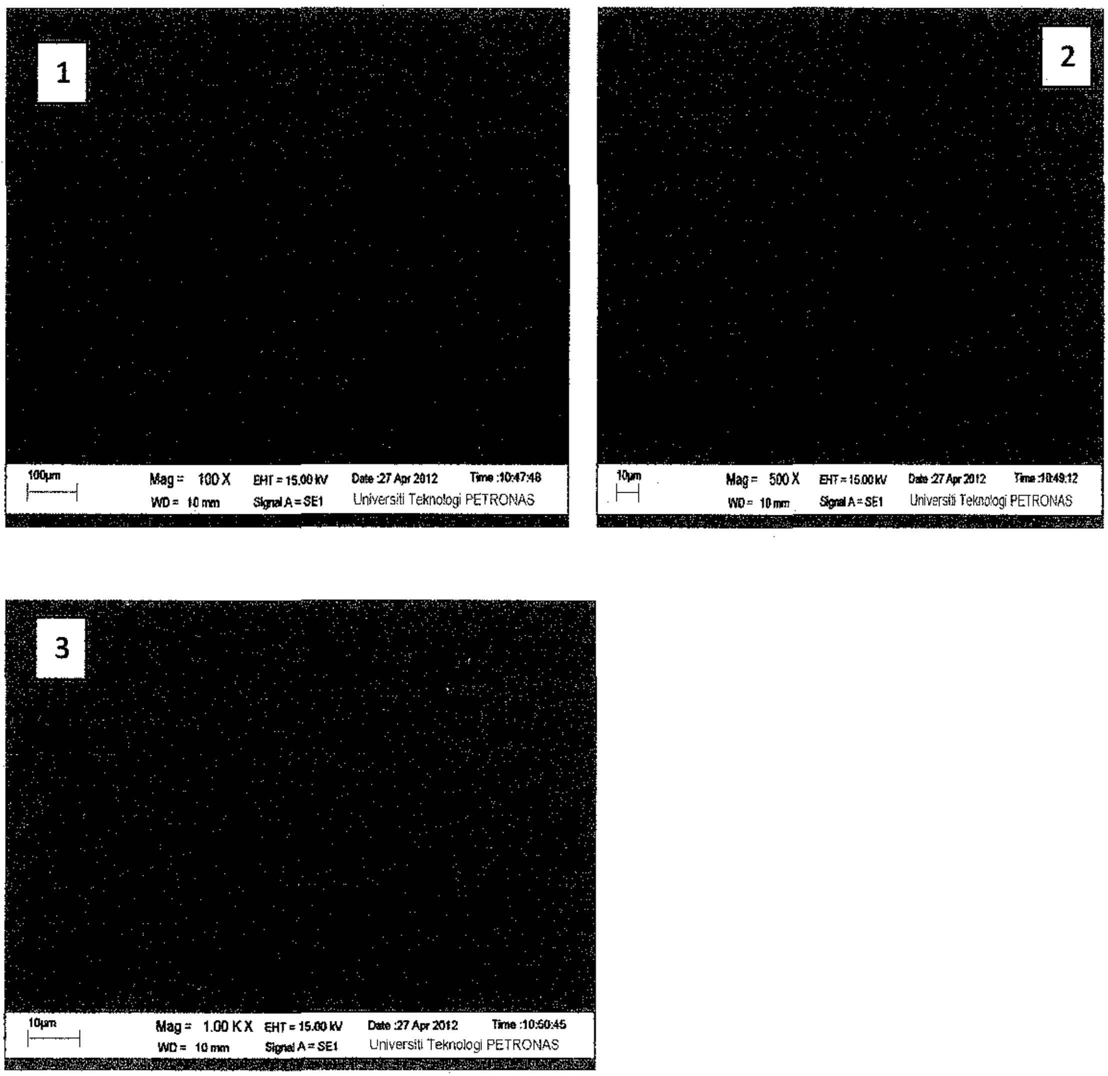

Figure 13: SEM images, for the control sample which is not corroded (1) 100x (2) 500x (3) 1000x Magnification

b) Mild Steel in Control Environment

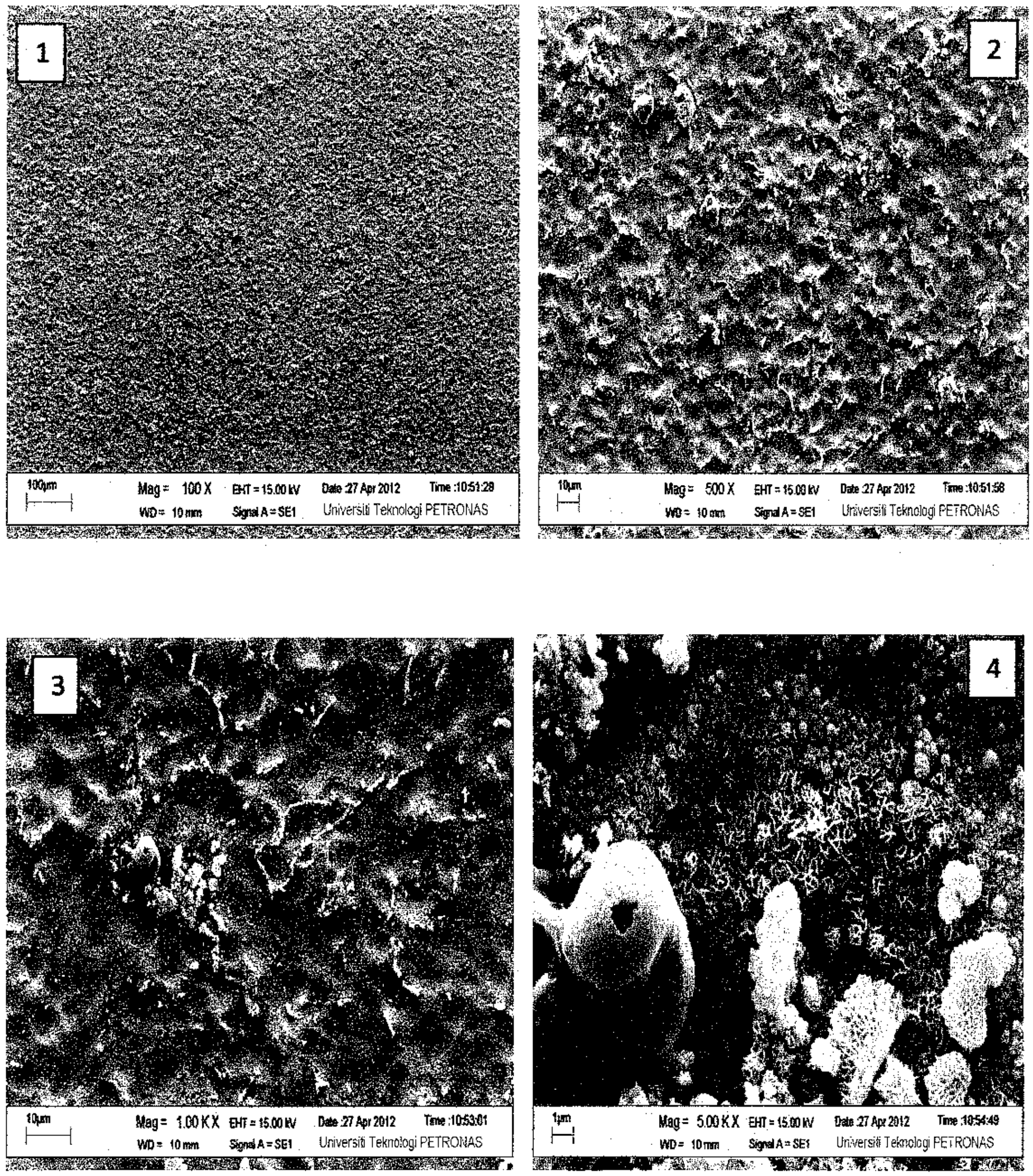

Figure 14: SEM images, for the mild steel which immersed in the BMIM [$FeCl_4$] at control environment (1) 100x (2) 500x (3) 1000x (4) 5000x Magnification

c) Mild Steel in Open Environment

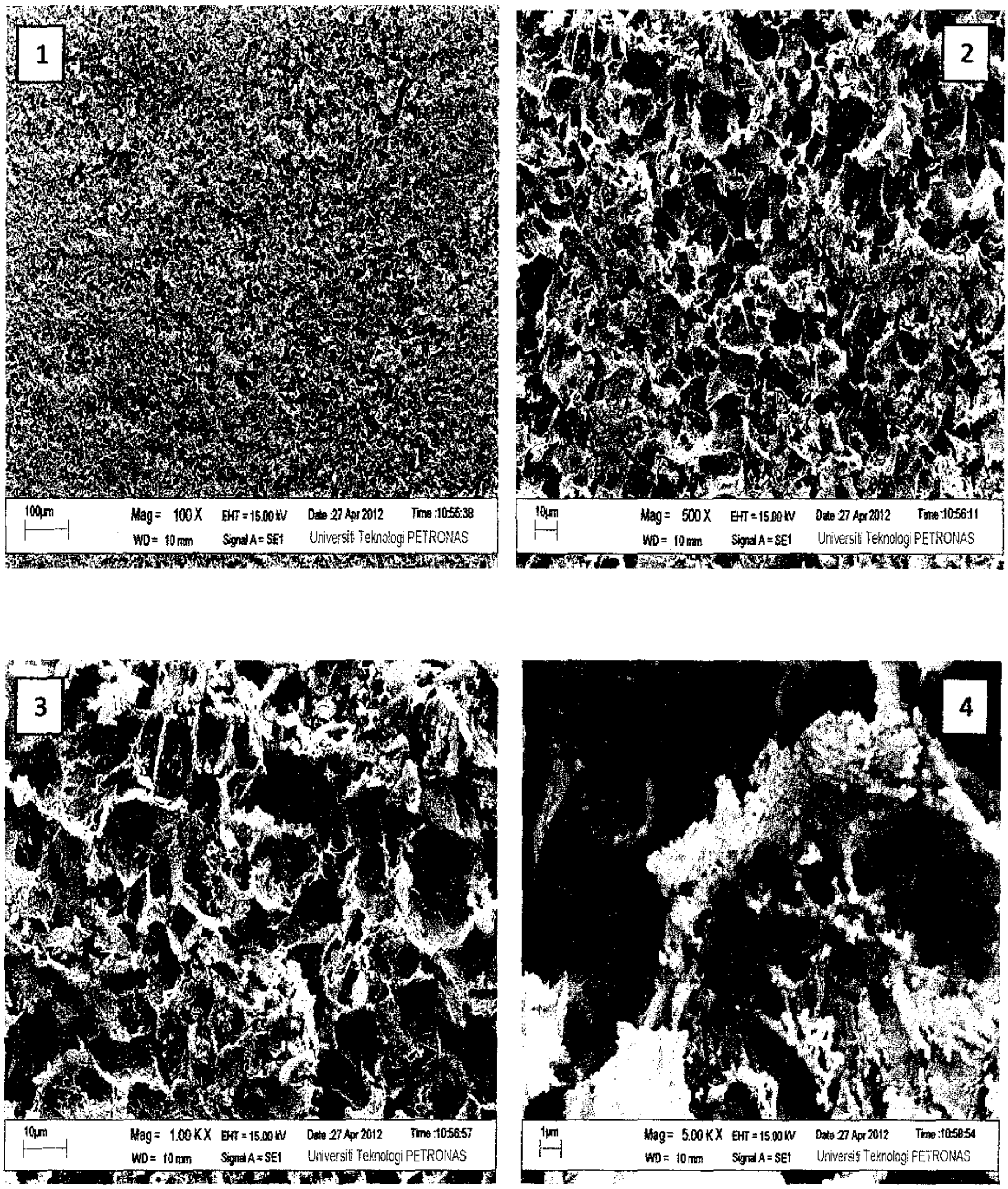

Figure 15: SEM images, for the mild steel which immersed in the BMIM [$FeCl_4$] at open environment (1) 100x (2) 500x (3) 1000x (4) 5000x Magnification

For the control sample, it can be seen from the SEM images shown above that the mild steel film layer is free from corrosion. Even at 1000x Magnification there are no corrosive layer detected.

For the second sample which is mild steel immersed in the Bmim [$FeCl_4$] at control environment, the pearlite/ferrite microstructure are formed at the sample's surface. At the control environment, the oxygen and water concentration is below 0.1 ppm.

The third sample is placed at the open environment which is room environment. The SEM images show that rough surface morphology after being immersed in the Bmim [$FeCl_4$] at room environment which indicate higher corrosion attacks on the mild steel.

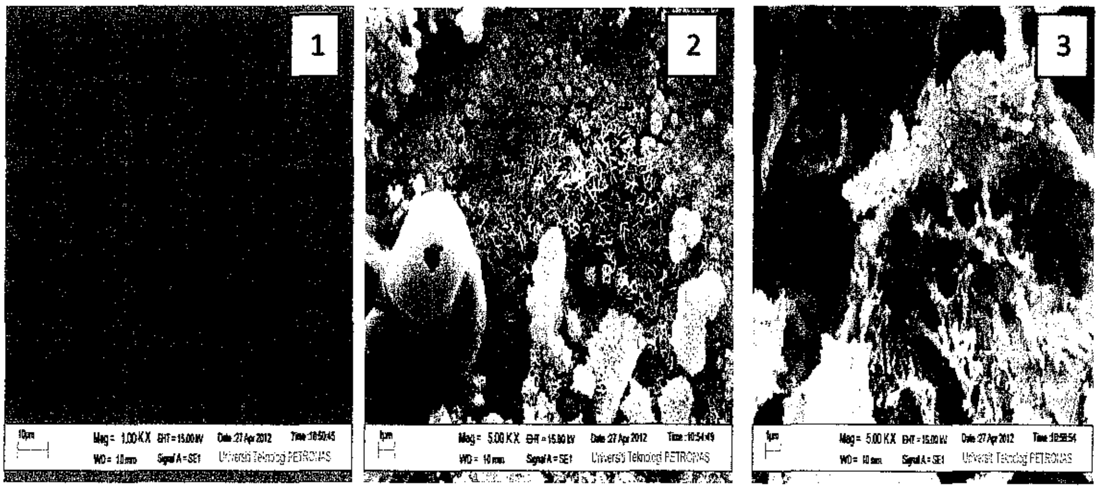

Figure 16: Comparison between (1) control environment and (2) room environment at 5000x Magnification.

The comparison between two samples proves that at room environment, the corrosion attack is the highest where the pearlite/ferrite microstructure formed at sample surface is the uppermost. At room environment, the oxygen concentration is 209,460 parts per million by volume or 20.946% of overall composition and the water vapor is ~0.40% over full atmosphere. The higher the oxygen and water concentration will speed up the corrosion rate of the mild steel.

4.2 Effect of Temperature

4.2.1 Tafel Plots

The Tafel Plots for the mild steel in Bmim [$FeCl_4$] was measured in various temperatures of 30 °C, 40 °C, 50 °C and 60 °C. The Tafel Plots were obtained at the potential range from -250 to +250 mV at a scan rate of 10 mV/minute and I Range (A) of 5 A. The kinetic parameters including corrosion current density, I_{corr} and corrosion rate are given in the table below.

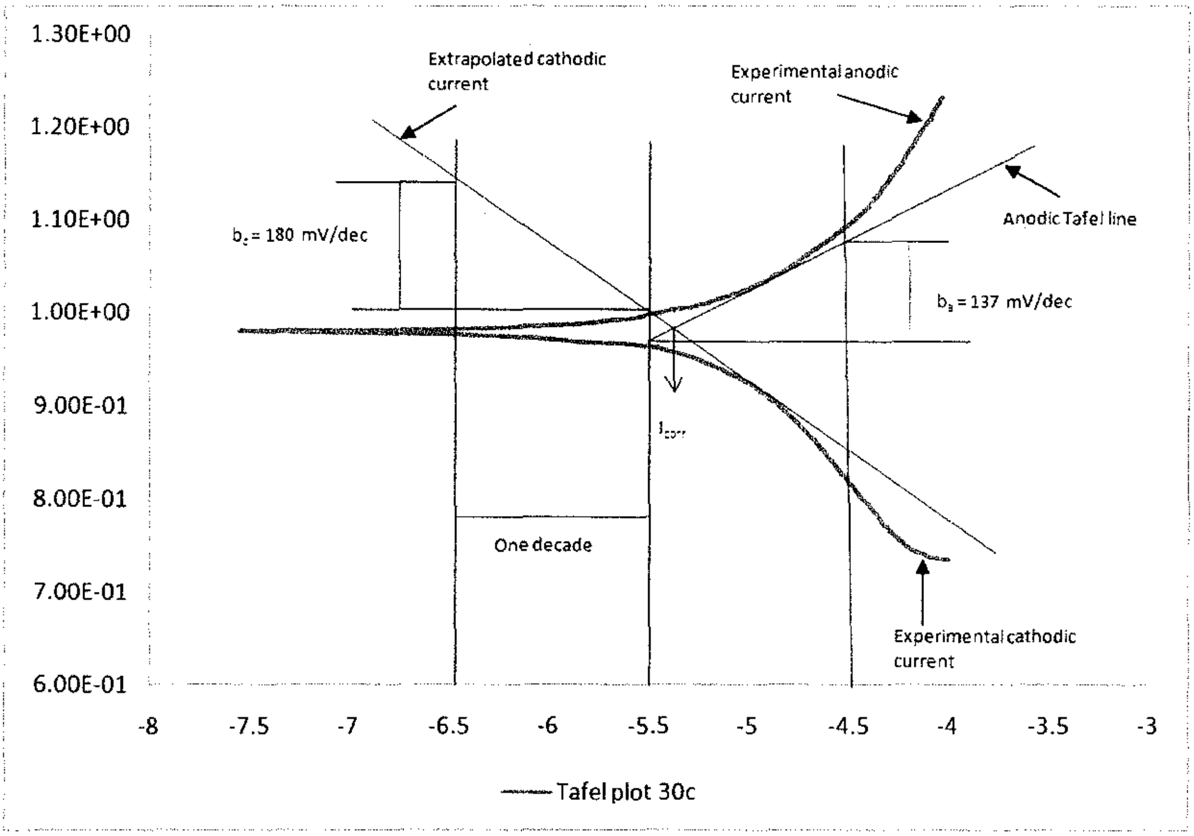

Figure 17: Tafel extrapolation method for corrosion current measurement of Bmim [$FeCl_4$] at 30 °C of Log I vs V

The polarization parameters corrosion current density, I_{corr}, anodic slope, βa and cathodic slope βc were extracted from the anodic and cathodic plot obtain from the IVMan software by using McCafferty method (E. McCaffetry, 2005). I_{corr} is attained from the interception between extrapolated cathodic current and anodic Tafel line.

Table 4: Summary of the electrochemical parameters extracted from Tafel Plot of mild steel in Bmim [$FeCl_4$] at diverse temperatures

Temp.	E_{corr} [V]	I_{corr} [V]	b_a [V/div]	b_c [V/div]	Rp [ohm]	CR [MPY]	CR [mmPY]
30 °C	980.857m	6.008u	0.137	0.180	5.617k	6.881	0.174800
40 °C	1.085	17.262u	0.061	0.150	3.096k	18.828	0.478305
50 °C	1.002	31.298u	0.095	0.116	725.407	34.138	0.867220
60 °C	893.023m	35.612u	0.108	0.099	627.965	38.842	0.986742

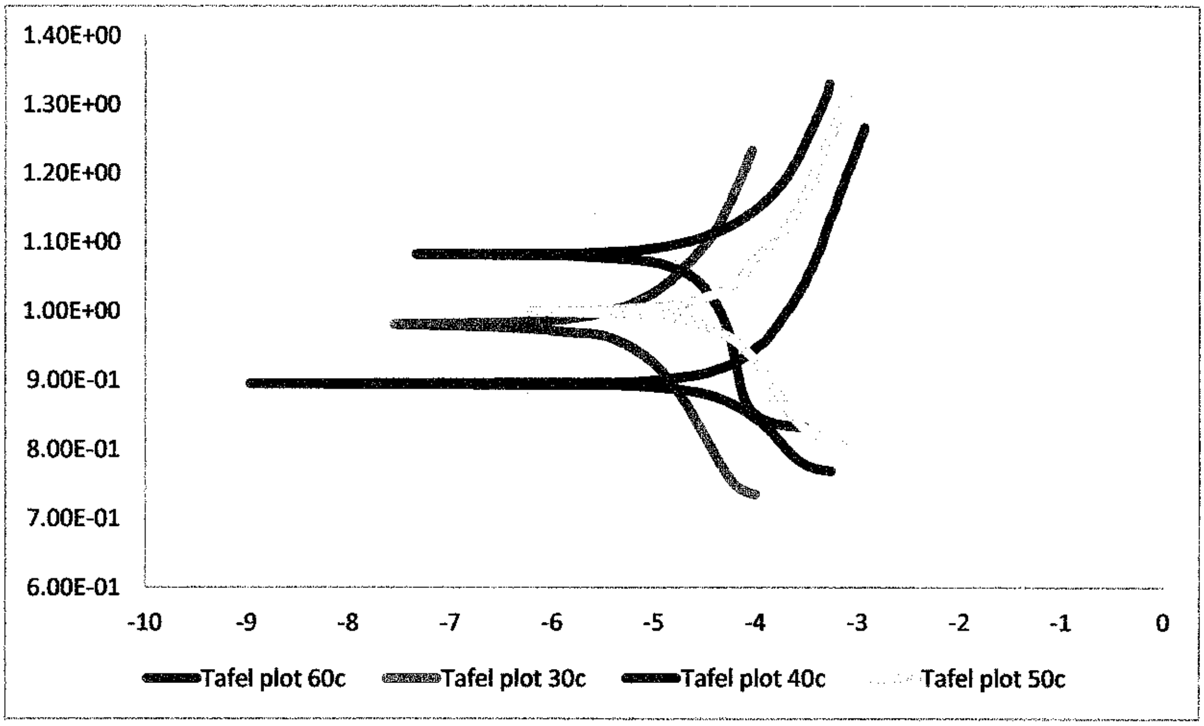

Figure 18: Tafel extrapolation method for corrosion current measurement of V vs Log I in ionic liquid Bmim [$FeCl_4$] at various temperatures

The data on Table 4 and Tafel plot shows that the increase in temperature will produce huge impacts on corrosion rate of Bmim [$FeCl_4$] towards mild steel. As the temperature increases from 30 °C to 60 °C, the corrosion potential altered towards active directions and constructed raise in corrosion rate. This happens due to absorb of Bmim [$FeCl_4$] towards the electrode surface. Therefore, the kinetic of cathodic reactions will be increase. Thus, the corrosion rate will be higher as the temperature rises.

4.2.2 Linear Polarization Resistance

The linear polarization resistance (LPR) for the mild steel in Bmim [$FeCl_4$] was measured in the temperatures of 30 °C, 40 °C, 50 °C and 60 °C. Therefore, the LPR plots were obtained at the potential range from -30 to +30 mV at a scan rate of 1 mV/minute and I Range (A) of 5 A. the kinetic parameters are recorded in the table below. LPR can be used to determine the corrosion rate using electrochemical means. The connection between the polarization resistance (*R p*) and corrosion rate (CR) is shown by the following equation

$$CR = \frac{\beta_a \beta_c M}{2.3\,(\beta_a + \beta_c(R_p FZD)}$$

Where β_a and β_c are the anodic and cathodic respectively, F is the Faraday constant, M is molecular weight, Z is the metal's valence and D is density of the mild steel (B. Si Ali, 2012).

Table 5: Summary of the electrochemical parameters extracted from Liner Polarization Resistance of mild steel in Bmim [$FeCl_4$] at diverse temperatures

Temp.	Rp [ohm]	b_a [V/div]	b_c [V/div]	CR [MPY]	CR [mmPY]
30 °C	2.932k	0.120	0.120	969.309m	0.024624
40 °C	1.008k	0.120	0.120	28.289	0.716094
50 °C	0.944k	0.120	0.120	29.654	0.945235
60 °C	870.957	0.120	0.120	32.267	0.828839

Table 5 shows that the corrosion rate is increasing as the temperature rises from 30 °C to 60 °C which indicates the rising temperature will effect on the corrosion of Bmim [$FeCl_4$] towards mild steel. From linear polarization resistance value, it shows that addition of the temperature resulted into a decrease in the polarization resistance, Rp of the mild steel from 2.932k ohm at 30 °C to 870.957 ohm at 40 °C. Reduce in Rp generally suggested a decline in the corrosion resistance of the steel in the presence of higher temperature. This shows that the metal is exposed towards higher corrosion. The behavior of polarization resistance can be clarified in terms of the stability of the external formed corrosion products layer. The Rp rate remains more or less constant if this layer is very stable and it is not separated from the metal surface. The Rp value will be fluctuating as if this layer formed and detached again.

4.2.3 Potentiodynamic

The Potentiodynamic polarization curves for the temperature of 30 °C, 40 °C, 50 °C and 60 °C is displayed in the Figure 19 while the summary of electrochemical parameters are recorded in the Table 6. The polarization curves were obtained at the potential range from -500 mV to 2 V at a scan rate of 10 mV/minute and I Range (A) of 5 A. The kinetic parameters including corrosion current density, I_{corr} and corrosion rate are given in the Table 6 below.

Table 6: Summary of the electrochemical parameters extracted from Potentiodynamic extrapolation.

Temp.	E_{corr} [V]	I_{corr} [V]	b_a [V/div]	b_c [V/div]	Rp [ohm]	CR [MPY]	CR [mmPY]
30 °C	264.729m	19.768u	0.379	0.221	5.710k	20.099	0.178628
40 °C	892.709m	21.021u	0.427	0.236	3.136k	22.928	0.582468
50 °C	702.711m	22.737u	0.497	0.561	1.031k	24.800	0.630003
60 °C	500.922m	98.582u	0.564	0.618	0.299k	107.525	0.974352

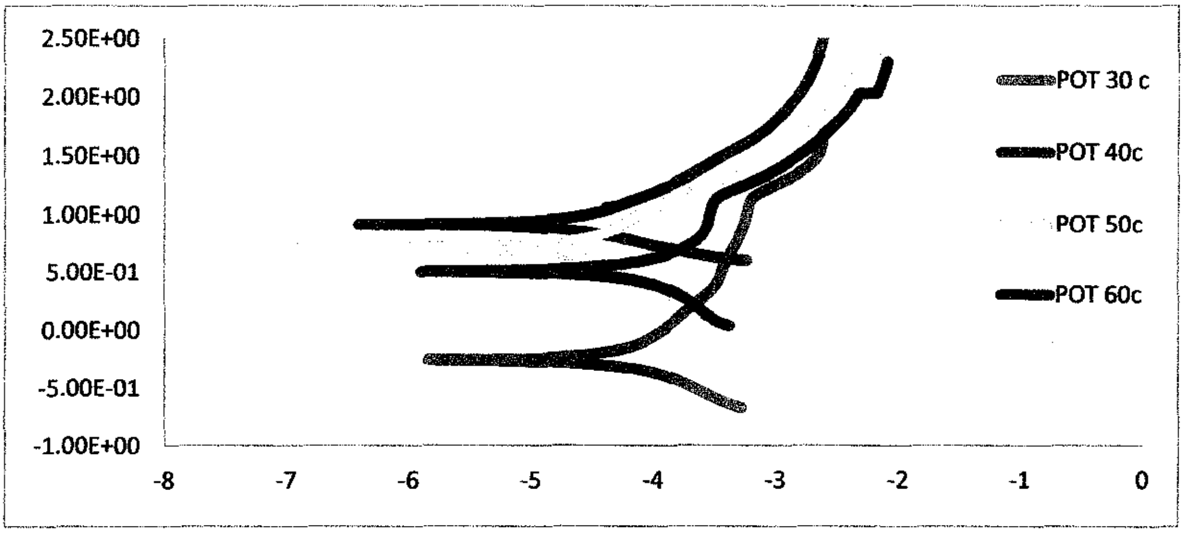

Figure 19: Potentiodynamic extrapolation method for corrosion current measurement of V vs Log I in ionic liquid Bmim [$FeCl_4$] at various temperatures

The four polarizations exhibit almost similar behavior, suggesting that the corrosion mechanisms are the same for all temperatures. The corrosion potential shifted towards only positive direction which indicates there are no passivation has occurred. This figure indicates at 30 °C, active dissolution of the mild steel between -0.3 V until it reaches 2.0 V. The same trend showed by polarization at 40 °C and 50 °C where corrosion takes place between 1.0 V to 2.0 V and 0.8 V to 2.0 V respectively. For extrapolation at 60 °C, it can be seen that the region of active dissolution ranges from 0.4 V to 0.7 V while between 0.7 V to 1.0 V current density is independent of potential. At higher potential values, 1.0 V to 2.0 V, the corrosion reoccurs.

4.2.4 Corrosion Rate Summary of Bmim $[FeCl_4]$ with the Effect of Temperature

Table 7: Summary of corrosion rate obtains at different extrapolation.

Temperature	Tafel [mmPY]	LPR [mmPY]	Potentiodynamic [mmPY]
30 °C	0.174800	0.024624	0.178628
40 °C	0.478305	0.716094	0.582468
50 °C	0.867220	0.845235	0.630003
60 °C	0.986742	0.928839	0.974352

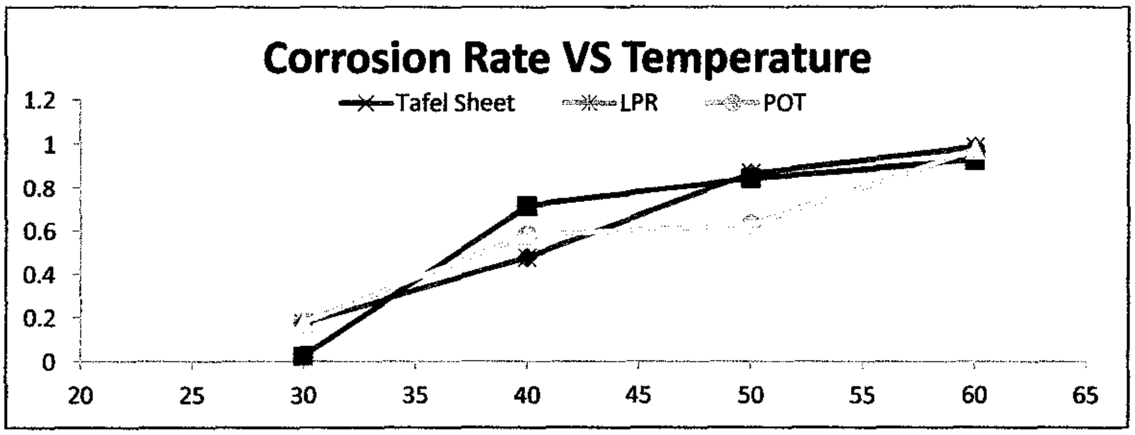

Table 7 summarizes all the corrosion rates at respective temperature by different types of extrapolation. It is observed that every extrapolation produce the same trend where the increase in temperature will produce the rises of corrosion rate.

4.3 Effect of Water Content

4.3.1 Tafel Plots

The Tafel Plots for the mild steel in Bmim [$FeCl_4$] was measured in a mixture of water content of 0.2 ml, 1 ml, 5 ml and 10 ml with Bmim [$FeCl_4$]. The Tafel Plots were obtained at the potential range from -250 to +250 mV at a scan rate of 10 mV/minute and I Range (A) of 5 A. The kinetic parameters including corrosion current density, I_{corr} and corrosion rate are given in the table below.

Table 8: Summary of the electrochemical parameters extracted from Tafel Plot of mild steel in Bmim [$FeCl_4$] at various water content

H_2O	E_{corr} [V]	I_{corr} [V]	b_a [V/div]	b_c [V/div]	Rp [ohm]	CR [MPY]	CR [mmPY]
0.2 ml	235.182m	6.228u	0.191	0.157	6.003k	6.793	0.172570
1 ml	-2889.889m	34.2784u	0.178	0.102	820.680	37.388	0.949798
5 ml	-261.203m	342.137u	0.202	0.297	152.291	373.175	9.480
10 ml	-80.212m	24.706u	0.309	0.195	2.104k	26.947	0.684552

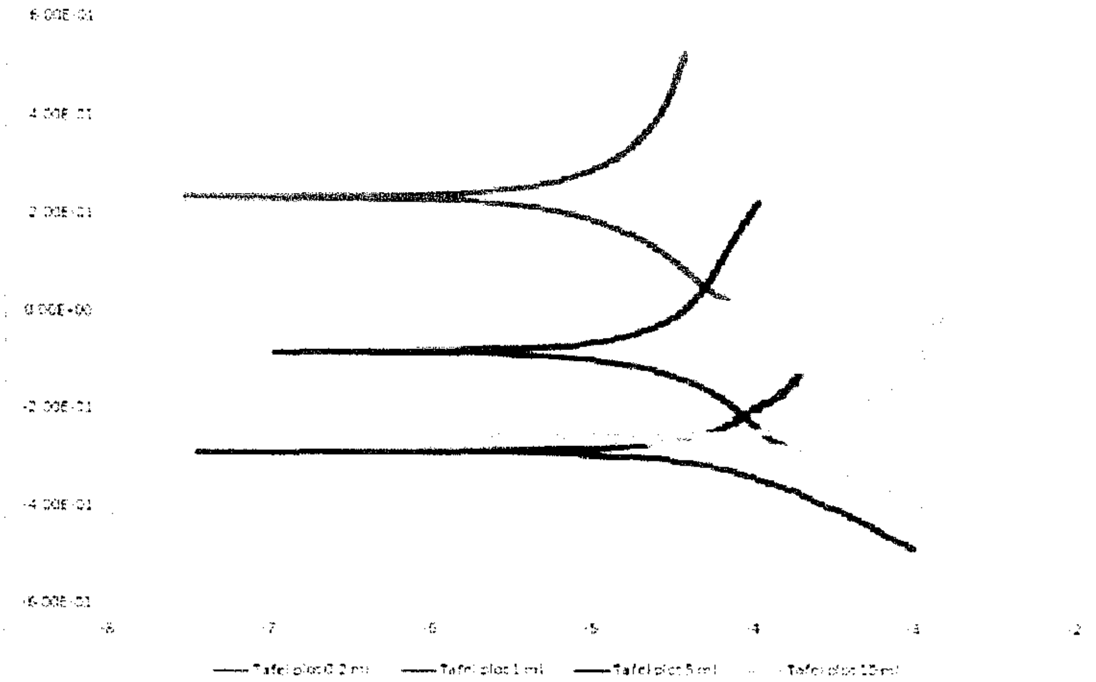

Figure 20: Tafel extrapolation method for corrosion current measurement of V vs Log I in ionic liquid Bmim [$FeCl_4$] at assorted water content

Figure 20 shows the Tafel plot of Bmim [$FeCl_4$] corrosion rate with the presence of water at 0.2 ml, 1 ml, 5 ml and 10 ml. the polarization parameters such as I_{corr}, βa and βc are shown in the Table 8. The polarization curves shows that in the present of increasing water level from 0.2 ml to 5 ml, the corrosion current density will increase which lead to linear rises of corrosion rate. However, the rate of corrosion declining as it reaches 10 ml, which is perchance due to the passivation or to a diverse structure and morphology of another new film formation, thus causing the mild steel in Bmim [$FeCl_4$] corrosion rate to be reduced. Another explanation towards this behavior is that the solution of 10 ml Bmim [$FeCl_4$] is already saturated with 10 ml of water. Therefore, there might be slight difference in the composition of the ionic liquid which resulting in different corrosion rate.

4.3.2 Linear Polarization Resistance

The linear polarization resistance (LPR) for the mild steel in Bmim [$FeCl_4$] was measured to gain the corrosion rate of the ionic liquid with the effect of water content of 0.2 ml, 1 ml, 5 ml and 10 ml. Therefore, the LPR plots were obtained at the potential range from -30 to +30 mV at a scan rate of 1 mV/minute and I Range (A) of 5 A. The kinetic parameters are recorded in the table below.

Table 9: Summary of the electrochemical parameters extracted from Liner Polarization Resistance of mild steel in Bmim [$FeCl_4$] at diverse water content

H_2O	Rp [ohm]	b_a [V/div]	b_c [V/div]	CR [MPY]	CR [mmPY]
0.2 ml	4.796k	0.120	0.120	5.925	0.150523
1 ml	1.706k	0.120	0.120	16.660	0.423233
5 ml	142.330	0.120	0.120	199.651	5.072
10 ml	2.151k	0.120	0.120	13.208	0.335532

In the polarization measurement, linear polarization resistance was used as criterion for the electrochemical corrosion evaluation for mild steel in Bmim [$FeCl_4$]. The deviation of the polarization resistance value with time, Rp, for the dissimilar water level is verified in Table 9. At the beginning of the experiment, the mixture between 10 ml Bmim [$FeCl_4$] with 0.2 ml water had the highest Rp value which is 4.796k ohm. However, the Rp value drop drastically as the water level increase to 1 ml and 5 ml, thus resulted in higher corrosion rate. On the other hand, the rise in Rp is observed as the water level reaches 10 ml which results in lower corrosion rate.

4.3.3 Potentiodynamic

The Potentiodynamic polarization curves for the water level of 0.2 ml, 1 ml, 5 ml and 10 ml is exhibit in the Figure 21 while the electrochemical parameters are recorded in the Table 10. The polarization curves were achieved at the potential range from -500 mV to 2 V at a scan rate of 10 mV/minute and I Range (A) of 5 A. The kinetic parameters including corrosion current density, I_{corr} and corrosion rate are given in the table below.

Table 10: Summary of the electrochemical parameters extracted from Potentiodynamic at different water content

H_2O	E_{corr} [V]	I_{corr} [V]	b_a [V/div]	b_c [V/div]	Rp [ohm]	CR [MPY]	CR [mmPY]
0.2 ml	167.158	20.241u	0.799	0.520	6.759k	22.077	0.560841
1 ml	-243.827m	73.351u	0.911	0.511	1.938k	80.006	2.032
5 ml	-282.920m	334.481u	0.353	0.642	295.481	364.824	9.268
10 ml	-155.063m	48.690u	0.729	0.470	2.55k	53.107	1.349

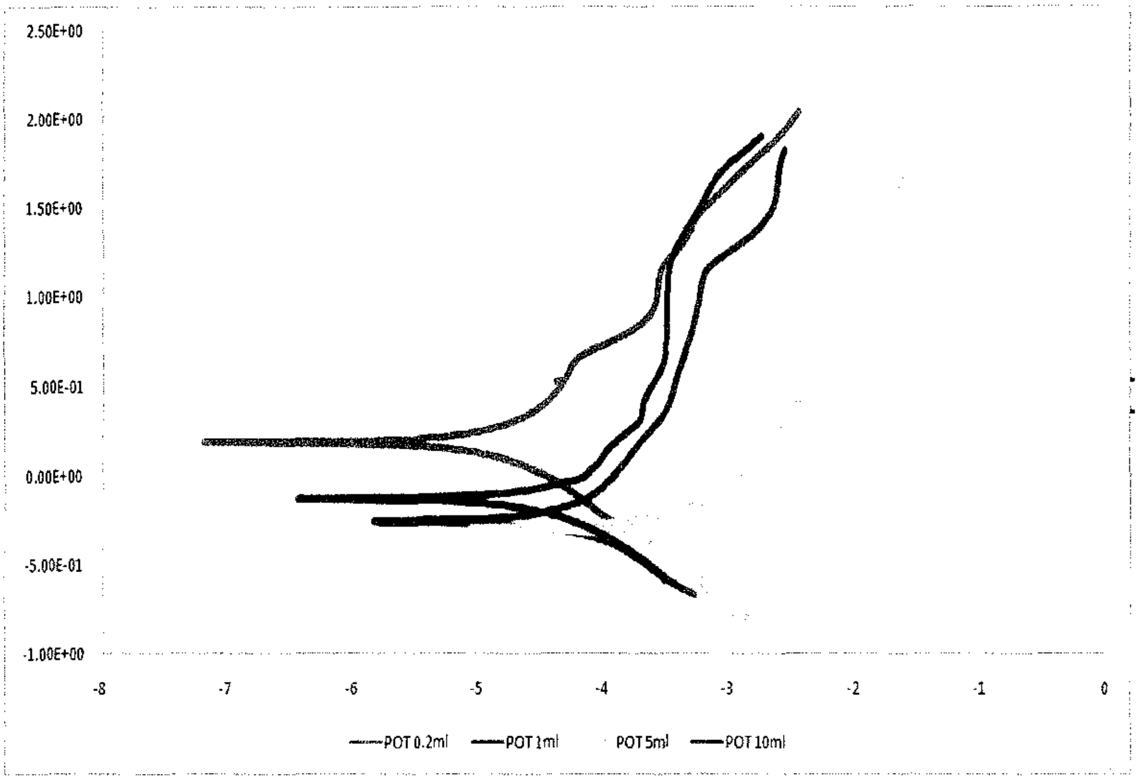

Figure 21: Potentiodynamic extrapolation at assorted water content of V vs Log I

The polarization curves obtain proposed the corrosion mechanism is the same at all water content as the demonstrated almost similar behavior. The corrosion potential shifted towards only positive direction which signifies there is only active metal dissolution and no passivation has occurred for water level of 0.2 ml, 1 ml and 5 ml. Therefore, the increase in corrosion rate is proportional with the increase in water level. However, for 10 ml water mixture, the active dissolution of mild steel occurred between -0.9 V to 0.7 V. At the passivation potential of 0.8 V to 1.1 V, the polarization curve displays reduce in anodic current and a minor decrease in the cathodic current. This behavior suggests that there is a slight change in the exterior of the specimen either by deposition of corrosion products or development of a porous thin film that produce lower corrosion rate.

4.3.4 Summary of Corrosion Rate of Bmim $[FeCl_4]$ with the Effect of Water Content

Table 11: Summary of corrosion rate obtains at three different extrapolations.

Water Content	Tafel [mmPY]	LPR [mmPY]	Potentiodynamic [mmPY]
0.2 ml	0.172570	0.150523	0.560841
1 ml	0.949798	0.423233	2.032
5 ml	9.480	5.072	9.268
10 ml	0.684552	0.335532	1.349

Table 11 sums up all the corrosion rates at different types of water level based on water level. It is observed that every extrapolation produce the same trend where the increase in water level from 0.2 ml to 10 ml will produce the rises of corrosion rate. However, the corrosion rate declining as the water level reaches 10 ml.

CHAPTER 5: CONCLUSION & RECOMENDATIONS

5.1 Conclusion

As the application of ionic liquid Bmim [$FeCl_4$] in the industrial application has increasing nowadays, the knowledge of corrosion behavior is very important. Therefore, the rate of corrosion of Bmim [$FeCl_4$] must be obtained. The methods of Tafel Plots, Potentiodynamic and Linear Polarization Resistance are proved to be vital in measuring the corrosion rate precisely. The SEM images of the sample for initial and final immersion testing also desired to study the rate of corrosion.

Based on the results obtain, the corrosion rate of pure Bmim [$FeCl_4$] at control environment is 0.04465 mm yr^{-1} while at room environment is 0.14653 mm yr^{-1}. Corrosion rate at control environment is lower which indicates that the lower concentration level of oxygen and water will results in lower corrosion rate. Furthermore, the comparison between SEM images also proves that the corrosion attack is the highest for room environment where the pearlite/ferrite microstructure formed at sample surface is the uppermost.

Meanwhile, the corrosion rate for effect of temperature is obtained through Electrochemical Measurement Techniques. From the extrapolations, it can be conclude that as temperature of Bmim [$FeCl_4$] rises, the corrosion rates will also increases. As for the water level, it demonstrates almost the same behavior. The corrosion rate increase as the water level rises from 0.2 ml to 5 ml. However, the corrosion rate decline as it reaches 10 ml. This phenomenon occurs due passivation or thin film formation on the surface of the steel. It also happen because the solution of 10 ml Bmim [$FeCl_4$] is already saturated with 10 ml of water. Therefore, the corrosion of Bmim [$FeCl_4$] decreases.

5.2 Recommendations

Throughout the project carried out, the following recommendations can be implemented to improve the result of the project:

1. Cyclic voltametric and Windows Stability of ionic liquid can be done to understand deeper on the corrosion behavior of ionic liquid Bmim [$FeCl_4$].
2. Energy Dispersive X-ray (EDX) analysis can be done in order to analyze the corrosion compound on the steel surface.

CHAPTER 6: REFERENCES

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