THE EFFECT OF TEMPERATURE ON NATURAL CORROSION INHIBITION OF CARBON STEEL BY USING PINEAPPLE PEELS *(ANANASCOMOSUS L.)*EXTRACT IN ACID SOLUTION

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ABSTRACT

Corrosion inhibitor is one of the suitable methods to control metals from corrosion. Pineapple is chosen as a corrosion inhibitor for this research because pineapple is one of the abundant resource in Malaysia and reported that pineapple peels have potential to be extract due to large amount of waste after processing. This corrosion inhibitor is biodegradable and do not harm environment which is not contain any toxic chemical. The purpose of this research is to study the inhibition efficiency of the pineapple peels from carbon steel coupon by considering effect of temperature, molarity of the acid and concentration of the inhibitor. The coupon was leaved for one week in acidic solution for 7 days and weight loss method is used to calculate the efficiency of inhibitor and corrosion rate in each parameters. From the results, it shows that corrosion rate decrease when inhibitor concentration and temperature increase while molarity of acid decrease. From this research, the highest value of inhibition efficiency which is 80.34% is obtained at 0.0340g/mL of inhibitor concentration. Hence, inhibitor concentration plays and important role to prevent metal from rusting and give more effect towards the metal besides other parameters. The corrosion inhibition action is afforded by the adsorption of the components of the inhibitor onto the carbon steel surface. Therefore, it is shows that the adsorption of the compound in the pineapple peels towards the coupon is responsible for corrosion inhibition effect.

ABSTRAK

Perencat karat adalah salah satu cara terbaik untuk mengawal pengaratan terhadap logam. Nenas dipilih sebagai perencat karat untuk kajian ini adalah kerana Malaysia merupakan pengeluar nenas terbesar dan kulit nenas juga berpotensi untuk diekstrak dan selalunya akan dibuang selepas pemprosesan nenas. Perencat karat ini sangat mesra alam dan tidak memberi kesan terhadap persekitaran kerana tidak mengandungi bahan kimia bertoksik. Tujuan kajian ini dilakukan adalah untuk mengkaji kemampuan perencat karat daripada nenas dengan mengkaji kesan terhadap kepekatan asid, kepekatan perencat karat dan suhu persekitaran. Metal karbon ditinggalkan di dalam larutan asid selama seminggu dan kaedah kehilangan berat digunakan untuk mengetahui kebolehan perencat karat pada sesetengah keadaan. Hasil daripada keputusan eksperimen yang telah dijalankan, kepekatan perencat karat yang tinggi di dalam kepekatan larutan acid yang rendah pada suhu yang tinggi akan mengurangkan karat pada kadar yang lebih cepat. Daripada kajian yang telah dilakukan, ini menunjukkan bahawa semakin tinggi kepekatan perencat karat dan suhu, semakin berkurang kepekatan asid dan dapat meningkatkan kebolehan perencat karat di samping mengurangkan pengaratan terhadap metal karbon. Daripada eksperimen yang telah dijalankan, kebolehan perencat karat yang paling tinggi adalah pada 80.34% pada 0.0340g/mL kepekatan perencat karat. Ini membuktikan bahawa, kepekatan perencat karat memainkan peranan yang penting untuk mengelakkan metal daripada pengaratan. Perencat karat dibantu oleh beberapa komponen yang dapat membantu menjaga metal daripada berkarat. Kompenan yang terkandung di dalam perencat karat dapat memberi kesan terhadap kebolehan perencat karat dalam mengawal pengaratan terhadap metal.

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LIST OF ABBREVIATIONS

A	surface area exposed
D	density of carbon steel
K	constant in corrosion rate Equation (4.11)
T	time of exposure in hours
W_a	initial weight before corrosion
W_o	weight after corrosion
V_o	corrosion rate without inhibitor
V_i	corrosion rate with inhibitor

LIST OF ABBREVIATIONS

AFM	Atomic Force Microscope
CRS	Cold Rolled Steel
DSCLE	Sun leaf extract
EIS	Electrochemical Impedance Spectroscopy
GC	Gas Chromatography
MPIB	Malaysian Pineapple Industry Board
SCC	Stress corrosion cracking

1 INTRODUCTION

1.1 *Motivation and statement of problem*

Pipeline affect our daily lives of most people in this world and most of the chemical industry used carbon steel for the pipelines materials because of the high availability of carbon. Pipeline usually transport gasses and liquid substances through millions of miles of underground pipelines that contain crude oil, natural gas liquids, gasoline, diesel fuel, jet fuel, carbon dioxide, anhydrous ammonia, and other commodities. Pipeline allows continuous, stable and high capacity to supply of hydrocarbon compared to the other forms of transport. Therefore, pipelines are cost effective, efficient and readily expandable. Pipeline also supported by pumping and compression stations that carry billions of cubic meters of our energy needs. For example in oil and gas transportation, it is currently provide 54% of the world's primary energy needs and there are over 3500 000 km of high pressure oil and gas pipeline around the world (P. Hopkins, 2014). Most of the pipelines are operated for a number of years and without inspection through this pipelines, either buried in the ground, exposed to the atmosphere or submerged in water there are still have probabilities for the pipelines to corrode and having an external damage. Buried pipeline transportation in the ground is believed as the most efficient method of transporting oil, gas or chemicals. It has a lower rate and is relatively reliable, but corrosion is still a major problem for its safe operation (Wenhe *et al.,* 2014).

Corrosion is one of the leading causes of pipelines failures (both gas and hazardous liquids) in the United States. As shown in Figure 1-1, corrosion has been responsible for 18 percent of the significant incidents (both onshore and offshore) in the 20 year period through 2008 in United States (Michael 2008). Almost any environment can cause corrosion, which occurs under numerous complex conditions in pipeline system. It also can occur when the acidic gases such as H_2S and CO_2 are dissolved in water, it create an acidic environment which in the vicinity of the steel will cause severe corrosion.

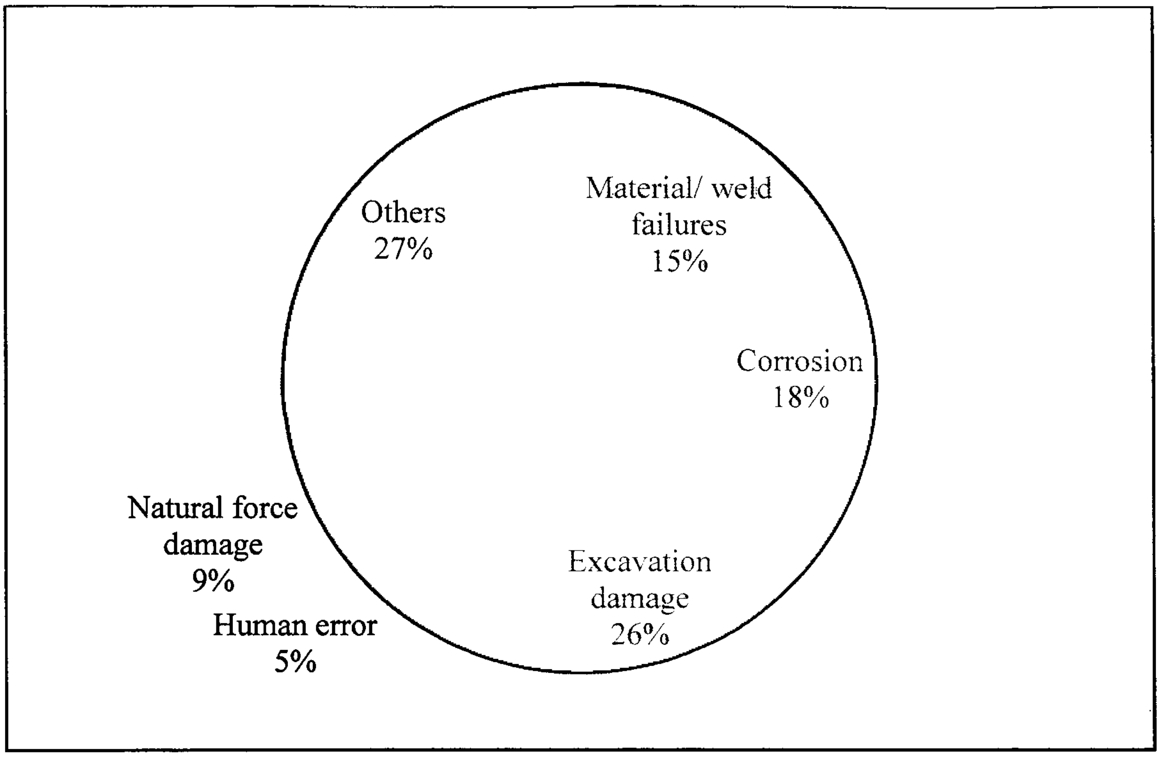

Figure 1.1: Causes of pipeline failures in United States

As shown in Figure 1-1, corrosion has been responsible for 18 percent of the significant incidents (both onshore and offshore) in the 20 year period through 2008 in United States (Michael 2008). Almost any environment can cause corrosion, which occurs under numerous complex conditions in pipeline system. It also can occur when the acidic gases such as H_2S and CO_2 are dissolved in water, it create an acidic environment which in the vicinity of the steel will cause severe corrosion. Sometimes, in oil wells oxygen is one of the corrosive gases too. In the oil and gas industries, corrosion may be localized or uniform. Localized corrosion, can be create under the insulators, sediment and bacteria, was 10 to 100 times faster than uniform corrosion lead to destructiôn and there are many costs and risks associated with it. Corrosion or external damage can weaken the structure of the pipeline. Corrosion may cause a leakage and make it as a unsafe for transporting fluids. However, technologist nowadays can control this entire problem on pipelines especially to the main problem of the pipeline which is corrosion. Based on human equipment, production management or environmental factor, the corrosion may cause an accident such as leakage and this may lead to the explosion (Jiang, 1999).

Some of the ways to overcome these problems are linings and protective coatings, cathodic protection, material selection and inhibitors (Sankara Papavinasam, 2014). Coating is the first defence line in front of a corrosive environment when pipe has been buried. Good function of coating depends on its adhesiveness rate to the metal surface. Initial adhesion and durability in contact situations are factors that are cause to high efficiency in the long term. The quantity of initial adhesion has the relationship with flow of coating and wetting of the surface by applying a coating and depends on clean and ready of the pipe surface (Amir Samimi 2012). However, the corrosion of pipelines' coatings is one of the main problems in oil and gas industries for which a large amount of money is spent each year.

Besides coating, corrosion inhibitor also can help to reduce the corrosion rate in pipeline. Corrosion inhibitor is a chemical substance organic or inorganic which when added (Andrade *et al.,* 2001) is a required amount to corrosive environment decreases the rate of corrosion. Corrosion inhibitors frequently work as anodic, cathodic or mixed inhibitors (Pasheco *et al.,*2011, Xu *et al.,*2008) by adsorbing themselves on the metallic surface (physical adsorption) by forming a film layer on the surface. Inhibitors reduce corrosion rate activities by increasing the cathodic and anodic polarization behaviour, decreasing the mobility of ions to the surface of the metal, raising the electrical resistance of the metallic surface and creating a barrier film on the surface of the metal. For industrial and large scale applications, cost, avaibility and environmental friendliness are essential considerations (Ji *et al.,*2011). Environmental concern must be evaluated first before choosing the best method to control the corrosion on pipelines. Cost of organic inhibitors is relatively low, but many of the effective inhibitors such as chromate, mercride, arsenate are very toxic and harmful to both humans and environment whereas plant extracts corrosion inhibitors are cheap, non toxic and also environmental friendly.

For this research, green corrosion inhibitor from extraction of pineapple peels is chosen as a raw material for corrosion inhibitor. Pineapple is a type of tropical plant believed to originate from East Area South America and introduced to Malaysia in the 16th century by Portuguese. It has long been recognized as one of the most popular subtropical fruit and grown extensively in Hawaii, Philippines, Caribbean area, Malaysia, Taiwan, Thailand, Australia, Mexico, Kenya, South Africa and Hainan province of China (Xie W *et al.,*2006). In Malaysia, Malaysian Pineapple Industry Board (MPIB) reported that for year 2008, Johor produced the highest yield of pineapple with 142,963.00 metric tons followed by Kelantan and Kedah (Nadzirah *et al.,* 2013). Therefore, the post harvest wastage of pineapple at the retail market is substantially high (Fernando and de Silva, 2000). Hence, the alternative to its efficient utilization are necessary (Correia *et al.,* 2007). Previous study proved that pineapple peels have potential to be extracted due to large amount of waste after processing (Ketnawa *et al.,* 2009). More than 70% of pineapple is consumed as fresh fruit in producing countries (Robert *et al.,* 2011). Pineapple waste is a by product of the pineapple processing industry and it consists of residual pulp, peels and skin. About 30% of the pineapples are turned into waste during the canning operation (Jamal *et al.,* 2009). These wastes can cause environmental pollution a problem if not utilized because it is still contains high content of carbohydrates as well as high fiber and low protein contents (Bhargava 2008). To overcome those problems, there is an alternative process for using these wastes. One of the ways is by using pineapple peels as an organic corrosion inhibitor.

1.2 Objectives

The following have been set for objectives of this research:

- To identify the effect of temperature, molarity of the acid and concentration of the inhibitor towards the efficiency of corrosion inhibitor.
- To study the relationship between corrosion rate and inhibition efficiency of the pineapple from carbon steel coupon.

1.3 Scope of this research

The following are the scope of this research:

i) For this research, there only some of the factor that influence the corrosion rate which are temperature, molarity of acid and concentration of the corrosion inhibitor.

ii) Corrosion rate can be determined from weight loss method. From the weight loss method, the efficiency of the inhibitor can be determined.

iii) The corrosion inhibitor was tested by using different temperature which is at 27-70 $^{\circ}C$, and different concentration of corrosion inhibitor is prepared by 0.002-0.034 g / mL. The corrosion inhibitor is then dissolved in 1.0-8.0 M of HCl.

1.4 Organisation of this thesis

The structure of the reminder of the thesis is outlined as follow:

Chapter 2 provides a description of the pipeline and the cause of the pipeline corrosion. The definition of the corrosion inhibitor also have stated in this chapter. Besides that, the types of the corrosion inhibitor from the previous research are explained well in this topic as well as the effect that can affect the corrosion inhibitor. A summary of the experiment from the different corrosion inhibitor also been stated in chapter 2.

Chapter 3 gives a review on how the process of the extraction of pineapple peels and the effect of the corrosion inhibitor towards the parameters. The parameters used are concentration of the acid, molarity of the corrosion inhibitor, and the temperature.

Chapter 4 is about how the corrosion rate is calculated from the weight loss method. It also shows the relationship between the corrosion rate and the inhibition efficiency at every parameter.

Chapter 5 draws together a summary of the thesis and outlines the future work which might be derived from the model developed in this work.

2 LITERATURE REVIEW

2.1 Overview

Corrosion is influence by environment and the metal type. The presence of carbon dioxide, hydrogen sulphide and free water in the production fluid can cause severe damage and corrosion problems especially in oil and gas pipeline (Yong Bai *et al.*, 2014) One of the way to prevent and minimize the corrosion rate in pipeline is by using natural corrosion inhibitor. There are many types of natural corrosion inhibitor that had been found by the previous research. Most of the natural corrosion inhibitor are made up from the plant leaves or peels such are *Bambusa Arundinacea, Aloe Vera, Chenopodium Ambrosioides, Nauclea latifolia, Ginko* and *Gossipium Hirsutum.* Through this research, the inhibition from these extract are contain some of complex organic species which are amino acids, alkaloids and nitrogen bases, carbohydrates and proteins. This complex organic contain polar function which are the major part of adsorption. The adsorption may affect the efficiency of the corrosion inhibitor. From the previous research of the corrosion inhibitor from pineapple, it was studied using weight loss and hydrogen evolution methods at 30-60 °C. It was found that the pineapple leaves extract inhibited the acid induced corrosion of mild steel

2.2 Types of corrosion

Corrosion is deterioration of essential properties in a material due to reactions with its environment. Pipeline corrosion limits pipeline flow, increases pig runs and wear and deteriorates product delivery. Pipeline life is shortened because of pipeline corrosion. There are some types of corrosion which are uniform or general corrosion, pitting, crevice corrosion, intergranular corrosion and environmental induced cracking.

2.2.1 General corrosion

Uniform corrosion or general corrosion is known as a type of corrosion attack (deterioration) that is more or less uniformly distributed over the entire exposed surface of a metal. Figure 2.1 shows the surface effect produced by most direct chemical attacks by an acid. Uniform corrosion also refers to the corrosion that proceeds at approximately the same rate over the exposed metal surface. Cast irons and steels corrode uniformly when exposed to open atmospheres, soils and natural waters, leading to the rusty appearance. On a polished surface, this type of corrosion is first seen as a general dulling of the surface and, if allowed to continue, the surface becomes rough and possibly frosted in appearance. The discoloration or general dulling of metal created by its exposure to elevated temperatures is not to be considered as uniform etch corrosion. The use of chemical-resistant protective coatings or more resistant materials will control these problems. While this is the most common form of corrosion, it is generally of little engineering significance, because structures will normally become unsightly and attract maintenance long before they become structurally affected.

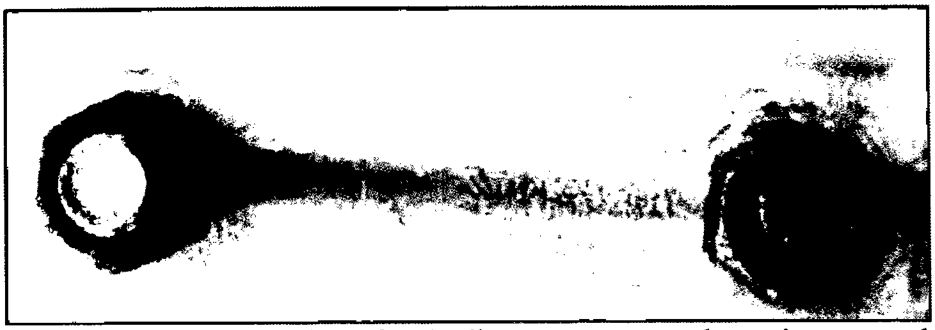

Figure 2.1: Uniform distribution of cathodic reactants over the entire exposed metal surface.

2.2.2 Pitting

Passive metals, such as stainless steel, resist corrosive media and can perform well over long periods of time. However, if corrosion does occur, it forms at random in pits. Pitting is most likely to occur in the presence of chloride ions, combined with such depolarizers as oxygen or oxidizing salts. Methods that can be used to control pitting include maintaining clean surfaces, application of a protective coating, and use of inhibitors or cathodic protection for immersion service. Molybdenum additions to stainless steel are intended to reduce pitting corrosion. The rust bubbles or tubercules on the cast iron above indicate that pitting is occurring. Researchers have found that the environment inside the rust bubbles is almost always higher in chlorides and lower in pH (more acidic) than the overall external environment. This leads to concentrated attack inside the pits. Pitting corrosion is an important design consideration for containment such as required for steel pipelines used in the offshore oil and gas industry. There is extensive reliance on cathodic protection and protective coatings but corrosion still appears as one of the main causes of failure in the marine offshore environment, perhaps because current anti-corrosion measures are not always sufficient. For weld zones, a generally accepted explanation of sensitization assumes that at sensitization temperature, chromium carbides precipitate at the grain boundaries, causing impoverishment of the adjacent matrix and therefore expected to be susceptible to both intergranular and pitting corrosion. This is a corrosion that occurs in materials that have protective films. It is an attack with localized holes on the metal's surface. The attack can penetrate the metal very rapidly, while some parts of the metal surface remain free from corrosion. Pitting is vigorous when the solution on the metal surface contains chloride, hypochlorite or bromide ions. Other harmful solutions are those that contain fluorides and iodides, while sulfides and water are known to enhance the pitting process. When metal is exposed, its available electrons are given up, and thus tiny pits begin to form on the metal surface. This then grows to become a rapid attack that results in massive damage of the metal. The oxidizing cation of iron, copper and mercury, among others, enables the formation of pitting even when there is no supply of oxygen in the metal surface. Stainless steel, chromium, passive iron, cobalt, aluminium, copper and associated alloys are all prone to pitting corrosion. A tubercular morphology can be seen where pits develop. Pitting is not always local in nature, as even when intrinsic defects in the solution-metal interface, the potential nuclei remains intact. Their development and stabilization show a random nature, and galvanic coupling established in the zones of discontinuity where metal dissolution occurs, lead to the formation of small anodes.

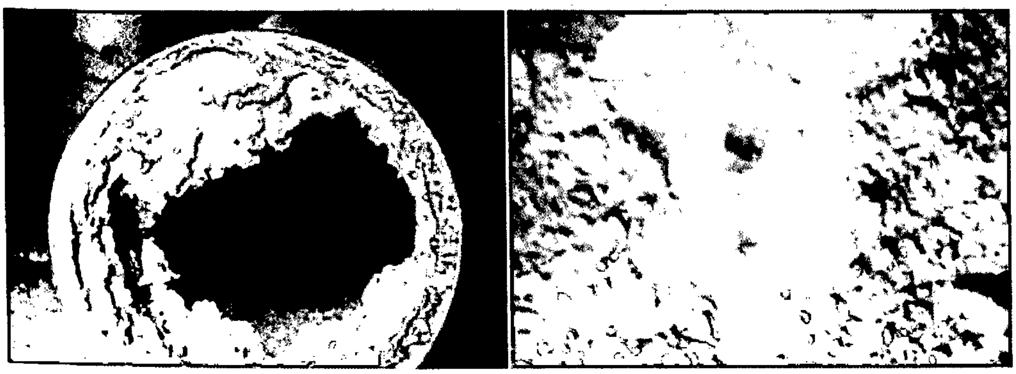

Figure 2.2: Example of pitting in pipeline

2.2.3 Stress corrosion cracking

It happens because of the conjoint action of stress and a corrosive environment which leads to the formation of a crack which would not have developed by the action of the stress or environment alone. Typically cracking failures are seen in pressure vessels, pipe work, highly stressed components and in systems when an excursion from normal operating conditions or the environment occurs. The stresses that cause cracking are either produced as a result of the use of the component in service or residual stresses introduced during manufacturing. The environment that can cause cracking is either the permanent service environment such as sea water or a temporary one caused by operations such as cleaning of the system which can leave a residue, or if the stress is applied during the operation initiate cracking.

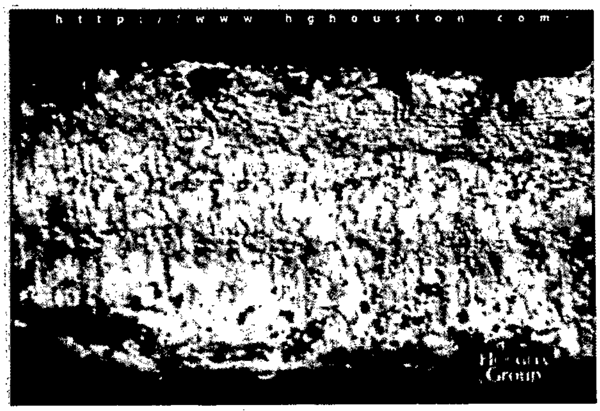

Figure 2.3 : Cracking of pipe fracture surface

Types of corrosion	Definition	Prevention
Uniform corrosion	Uniform corrosion or general corrosion is known as a type of corrosion attack (deterioration) that is more or less uniformly distributed over the entire exposed surface of a metal	• Cathodic protection • Inhibitor • Protective coatings
Pitting corrosion	Pitting is most likely to occur in the presence of chloride ions, combined with such depolarizers as oxygen or oxidizing salts.	• Proper selection of materials with known resistance to the service environment. • Control pH, chloride concentration and temperature. • Cathodic protection.
Stress corrosion cracking (SCC)	It happens because of the conjoint action of stress and a corrosive environment which leads to the formation of a crack which would not have developed by the action of the stress or environment alone. Typically, SCC failures are seen in pressure vessels, pipework, highly stressed components and in systems when an excursion from normal operating conditions or the environment occurs.	• Select the alloy that is not susceptible to the environment. • Avoid stress concentration

Figure 2.4 : Summarization of type of corrosion in pipeline

2.3 *Previous research on pineapple peels*

The corrosion inhibition of mild steel in HCl by pineapple leaves extract was studied using weight loss and hydrogen evolution methods at 30-60 °C. It was found that the pineapple leaves extract inhibited the acid induced corrosion of mild steel. The inhibition efficiency is increase with the increasing in the extract concentration and rise in temperature. Adsorption of the extract was found to obey Langmuir adsorption isotherm at all concentrations and temperature studied. By hydrogen evolution measurements, it is clearly seen that the hydrogen evolution rates were reduced in the presence of the extract compared to the blank acid solution. The volume of the hydrogen gas evolved varies linearly with reaction time. The results also indicate the decrease deflection of H_2 gas evolution on the introduction of the pineapple leaves extract showing that the extract actually afforded corrosion inhibition of mild steel in the acidic environment. The volume of hydrogen gas was observed to increase with increase in temperature in the absence and presence of the leaves extract. Also the volume of H_2 evolved was dependent on extract concentration, decreasing with increasing extract concentration.

2.4 *Previous research on bamboo leaf*

From the previous research, bamboo leaf extraction is also good as a corrosion inhibitor (Xianghong Li *et al.,* 2012). China is one of the richest countries in the world in terms of 500 bamboo species. Bamboo leaf extract is virtually non-poisonous and rich in flavonoids, amino acids and active sugar. Sun leaf extract (DSCLE) on the corrosion of cold rolled steel (CRS) in 1.0-5.0 M H_2SO_4 solution was determined by weight loss, potential dynamic polarization curves, electrochemical impedance spectroscopy (EIS) and atomic force microscope (AFM) methods. It shows that, DSCLE is chosen as a good inhibitor in 1.0M HCl and 0.5M H_2SO_4. Langmuir adsorption isotherm is obeyed by adsorption of DSCLE on CRS surface and acts as a mixed type inhibitor in both acids with efficiently protects or minimizes steel from corrosion. The corrosion rate increases with temperature both in unhibited and inhibited solutions, especially goes up more rapidly in the absence of inhibitor.

Theses result confirms that DSCLE acts as an effective inhibitor in the range of temperature studied. Besides that, organic compounds of bamboo leaf also show inhibitive effect via adsorption on metal surface and some adsorption isotherms.

2.5 Previous research on aloe vera

Aloe Vera also can be used and known as green corrosion inhibitor (Olesegun K. Abiola *et al.,* 2010). The effect of Aloe Vera leaves extract on the corrosion of zinc in 2 M HCl solution was determined by weight loss technique. Inhibition efficiency of the Aloe Vera in Zinc corrosion is 2 M HCl solution. Therefore, the inhibition efficiency is proportional to the concentration of the extract whereas inversely proportional with increasing temperature. The adsorption of the inhibitor on zinc surface was obeyed with Langmuir adsorption isotherm.

Type of inhibitor	Explanation	Journal
Pineapple peels	The corrosion inhibiton of mild steel in HCl by pineapple leaves extract was studied using weight loss and hydrogen evolution methods at 30-60°C. It was found that the pineapple leaves extract inhibited the acid induced corrosion of mild steel. The inhibition efficiency increase with increase in the extract concentration and with rise in temperature.	U.F. Ekanem *et al.*, 2010
Bamboo leaf	Bamboo leaf extract is virtually non-poisonous and rich in flavonoids, amino acids and active sugar. Sun leaf extract (DSCLE) on the corrosion of cold rolled steel (CRS) in 1.0-5.0 M H_2SO_4 solution was determined by weight loss, potential dynamic polarization curves, electrochemical impedance spectroscopy (EIS) and atomic force microscope (AFM) methods. It shows that, DSCLE is chosen as a good inhibitor in 1.0M HCl and 0.5M H_2SO_4.	Xianghong Li *et al.*, 2012
Aloe Vera	The effect of Aloe Vera leaves extract on the corrosion of zinc in 2 M HCl solution was determined by weight loss technique. Inhibition efficiency of the Aloe Vera in Zinc corrosion is 2 M HCl solution. Therefore, the inhibition efficiency is proportional to the concentration of the extract whereas inversely proportional with increasing temperature.	Olesegun K. Abiola *et al.*,2010

Figure 2.5: Summarization of difference type of inhibitor.

2.6 *Effect of temperature towards the inhibition efficiency*

Usually, corrosion rate is increase when the temperature is increase. For the research of corrosion inhibition of Fig leaves extract (Taleb *et al.,* et al), the temperature range is tested at 25-50°C. The result shows when the temperature increases the corrosion rate increased and the inhibition efficiency decreased. This happens because of the physical adsorption in the inhibitor. Besides that, the same result was valso obtained for the esearch of corrosion inhibition from Nypa fruticans' wurmb (Orubite *et al.,* 2007). It was observed that the inhibition efficiency decreased with increase in temperature, a situation that is generally true of most chemical reactions. However addition of KI to the Nypa fruticans' wurmb extract showed a slightly different trend. For all the different concentrations of KI added to the highest concentration of Nypa fruticans' wurmb extract (0.36 g dm^{-3}) inhibition efficiency decreased as temperature increased from30-40OC but showed a dramatic increase at the highest temperature of $50^{O}C$ (This observation has been attributed to synergistic inhibition between Nypa fruticans' wurmb extract and KI for mild steel corrosion in the acid medium.

2.7 *Summary*

Pineapple is chosen as a corrosion inhibitor for this research rather than Aloe Vera or bamboo leaf because of pineapple is the most abundant resource in Malaysia. For the large production, pineapple peels is the best green corrosion inhibitor because it can easily be found in Malaysia and also have large amount of waste after processing (Ketnawa *et al.,* 2009).

3 MATERIALS AND METHODS

3.1 *Methods*

Inhibitor preparations

I. Pineapple peels was dried and grind into powder form.
II. Extract the solution by distillation process after stirred with methanol for 2 days.

↓

Carbon steel coupons

I. Polish machine and sand paper are used to scrub the steels coupon to expose clean, shining surface and washed with distilled water and ethanol.
II. The coupon is weighed before dipped in the HCL solution for 7 days.

↓

Weight loss method

I. Brushed the coupon after dipped in HCL by using Sb_2O_3 and $SnCl_2$.
II. The coupon is weighed again after dried in acetone hence the weight loss can be calculated

↓

Analysis

I. Effect of temperature, concentration of inhibitor and molarity of acid.
II. Components found in pineapple peels that can prevent corrosion.

Figure 3.1: Flowchart of the experimental method