CHAPTER 1
INTRODUCTION

1.1   Background of Study

Millions of dollars are lost each year due to corrosion. The costs of lost time, the replacement of materials of construction, and the constant personnel involvement in corrosion control are substantial and if not controlled, can be catastrophic. The control of the corrosion through the use of coatings, metallurgy, and non metallic materials of construction, cathodic protection, inhibitors and other methods has evolved into a science in its own right and has created industries devoted solely to corrosion controlled in specific areas [1]. Without these corrosion control methods, which effectively reduce environmental corrosion, the lost would be even higher.

Corrosion inhibitors are an effective means of corrosion control, and they are required in highly corrosive environments in which carbon steel is used. Corrosion inhibitors can be group into inorganic and organic inhibitors. Organic inhibitors usually designated as 'film-forming', protect the metal by forming a hydrophobic film on the metal surface. The effectiveness of these inhibitors depends on the chemical composition, their molecular structure, and their affinities for the metal surface. Because film formation is an adsorption process, the temperature and pressure in the system are important factors.

Jatropha oil is vegetable oil produced from the seeds of the *Jatropha* curcas. This plant can grow in marginal lands and common lands. The oil consists of two saturated fatty acids (palmitic and stearic acid) and unsaturated fatty acids (oleic acid and linoleic acid). These fatty acids are used in organic corrosion inhibitors. Throughout this research, the student would like to study on feasibility of Jatropha Oil as a corrosion inhibitor and the factors that affect the corrosivity when the metal protected by this Jatropha oil derived corrosion inhibitor.

1.2 Problem Statement

In recent years, the need for new anti-corrosive technologies in the coatings area has greatly increased due to the regulatory pressures on heavy metal pigment and volatile organic compounds-mostly solvents (VOC), [2]. Heavy metals such as chromium, lead, barium and even zinc have limited permission in certain usage. Corrosion inhibitors based on lead and chromate are the most environmentally unacceptable. Unfortunately, most of non-lead and non-chromate corrosion inhibitors in market nowadays, including semi-organic ones, also contain heavy metals listed by Superfund Amendments and Reauthorization Act (SARA) 313 which is one of the important federal heavy metal regulations [2]

1.3 Objectives

The objectives of the project are as follows:

- To identify the availability of Jatropha Oil as an organic corrosion inhibitors.
- To determine the effect of temperature and pH value on corrosivity.
- To determine the effect of Jatropha oil concentration on corrosivity.

1.4 Scope of Work

The scope of work for this project is to investigate the corrosion inhibitor by using Jatropha Oil. This project is begun with literature review on Jatropha Oil and its properties. Then, the student will relate the availability of Jatropha Oil with corrosion inhibitor. The experiment work shall be done in order to determine factors that affect the level of protection given by Jatropha oil. Two testing methods will be used in this experiments which are weight-loss and electrochemical methods.

CHAPTER 2
THEORY AND LITERATURE REVIEW

2.1   Corrosion Theory and Mechanism

A general definition of corrosion is the natural degradation through electrochemical or chemical reaction. Corrosion in an aqueous environment and in an atmospheric environment (which also involves thin aqueous layers) is an electrochemical process because corrosion involves the transfer of electrons between a metal surface and an aqueous electrolyte solution. This is due to tendency of metals to react electrochemically with oxygen, water, and other substances in the aqueous environment.

2.1.1   Classification of Corrosion

Corrosion has been classified in many different ways. One of the methods divides corrosion into low-temperature and high-temperature corrosion. Another separates corrosion into direct combination (or oxidation) and electrochemical corrosion. The preferred classification is wet corrosion and dry corrosion.

Wet corrosion occurs when a liquid is present. This usually involves aqueous solutions or electrolytes and accounts for the greatest amount of corrosion so far. A common example is corrosion of steel by water. Dry corrosion occurs in the absence of a liquid phase or above dew point of the environment. Vapors and gases are usually the corrodents. Dry corrosion is most associated with high temperatures. An example is attack on steel by the furnace gases.

The presence of even small amount of moisture could change the corrosion picture completely. For example, dry chlorine is practically noncorrosive to ordinary metal, but moist chlorine is extremely corrosive and attacks most of the common metals and alloys. The reverse is true for titanium-dry chlorine gas is more corrosive than wet chlorine.

2.1.2 Corrosion of Drill Pipe

The possible effect of drilling fluid on drilling string and casing corrosion became a matter of concern. Waters were usually salty; up to saturation when bedded salt was drilled and frequently were acidic. Also, contamination by acid gases (such as carbon dioxide and hydrogen sulphide) and by formation brines can cause severe corrosion.

In 1936, the American Petroleum Institute, Division of Production, Topical Committee on Materials, set up a subcommittee to investigate drill pipe corrosion fatigue. A significant conclusion was that corrosion fatigue endurance could not be reliably predicted from loss in weight tests without stress. Also, less corrosion was observed in mud of higher pH. Degradation of clay minerals by the hydroxyl ion starts at temperature above $200^{o}F$ ($149^{o}C$) when the pH of the mud is above 10. The wisest policy is to maintain the pH between 9 and 10, which in many wells will keep corrosion within acceptable limits.

Carbon dioxide dissolves in water and lowers the pH by forming carbonic acid. Corrosion is best controlled by maintaining the pH between 9 to 10 with sodium hydroxide, but when the inflow of the gases is large, the formation of excessive amounts of soluble of carbonates cause high viscosities. In such cases, calcium carbonate precipitate tends to form scale, thus setting up corrosion cells. This tendency may be offset by the use of scale inhibitors, and by cleaning the pipe during round trips.

2.1.3 Electrochemical Reactions

'Corrosion cell' is made up when the same metallic surface exposed to an aqueous electrolyte usually possesses sites for an oxidation (or anodic chemical reaction) that produces electrons in the metal, and a reduction (or cathodic reaction) that consumes the electrons produced by the anodic reaction. Dissolution of the metal in anodic reaction can form either soluble ionic products or an insoluble compound of the metal, usually an oxide. Several cathodic reactions are possible depending on what reducible species are present in the solution. Because these anodic and cathodic reactions occur simultaneously on a metal surface, they create an electrochemical cell of the type shown in Figure 1.
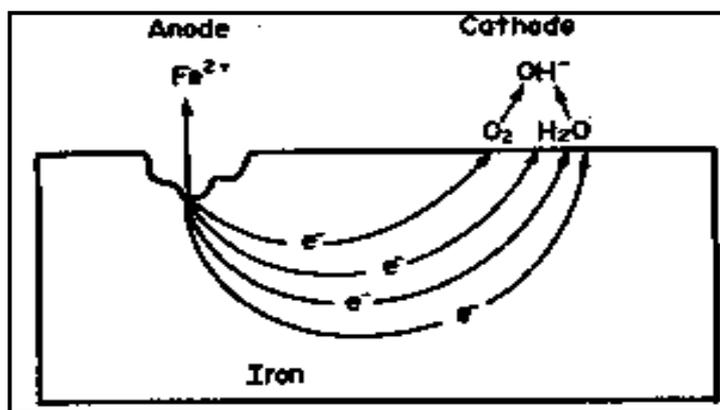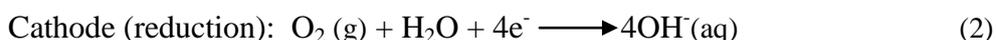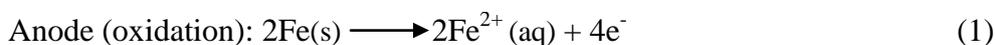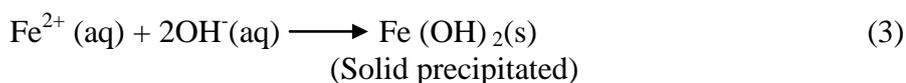

Figure 1: The electrochemical cell set up between anodic and cathodic sites on an iron surface undergoing corrosion. [6]
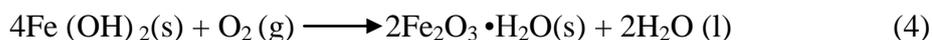
The complete corrosion reaction of iron can be shown as follow:

$$\text{Anode (oxidation): } 2Fe(s) \longrightarrow 2Fe^{2+} (aq) + 4e^- \tag{1}$$

$$\text{Cathode (reduction): } O_2 (g) + H_2O + 4e^- \longrightarrow 4OH^- (aq) \tag{2}$$

Within the surface, the hydroxide ions can move inward to react with the iron (II) ions moving from the oxidation region. Iron (II) hydroxide is precipitated.

$$Fe^{2+} (aq) + 2OH^- (aq) \longrightarrow Fe(OH)_2 (s) \tag{3}$$
$$\text{(Solid precipitated)}$$

Rust is then quickly produced by the oxidation of the precipitate.

$$4Fe(OH)_2 (s) + O_2 (g) \longrightarrow 2Fe_2O_3 \cdot H_2O(s) + 2H_2O (l) \tag{4}$$

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2.1.4 Corrosion Rate

Corrosion rate have been expressed in many ways such as percent weight loss, grams per square inch per hour and mils per year. The expression of mils per year readily calculated from weight loss of metal coupon during the corrosion test as below:

$$mpy = \frac{534 \, W}{DAT}$$

(5)

Where W = weight loss, mg

D = density of coupon, g/cm^3

A = area of coupon, sq. in

T = exposure time, hr

Environment always give the big impact to corrosion rate. Like most other chemical reactions, corrosion rates increase as temperature increases. For a constant humidity, an increase in temperature would therefore lead to a higher corrosion rate then fall to lower value. This is because the decrease of oxygen solubility when molecules more rapid evaporate as the increase of temperature. In a close system, the corrosion rate will increase with temperature until all oxygen has been consumed.

Besides, the corrosion rate is influented by pH value of the solution. The potential of hydrogen, (pH) is defined as the negative logarithm of the hydrogen concentration. Its efffect is dependent on concentration of dissolved oxygen and the type of acid that control the pH. The corrosion rate is high in acid range and high alkaline range because the corrosion in under anodic control. For mild alkaline and and neutral solutions, some corrosion protection is provided since the corrosion rate is under cathodic control.

2.2 Jatropha Curcas Oil

There is a growing interest in *Jatropha curcas* as a biodiesel "miracle tree" to help alleviate the energy crisis and generate income in rural areas of developing countries. Some common names include Jatropha, physic nut, Barbados nut, purging nut, pig nut, fig nut, and it is sometimes referred to as the biodiesel or diesel tree [10]. *Jatropha curcas L* belongs to the euphorbia family, is native to tropical America and naturalised throughout tropical and subtropical parts of Asia and Africa. Jatropha curcas is a drought-resistant bush or small tree with spreading branches and stubby twigs that grows to 20 feet high under favorable conditions.

The oil fraction of J. curcas contained saturated fatty acids mainly palmitic acid (16:0) with 14.1% and stearic acid (18:0) with 6.7%. Unsaturated fatty acids consisted of oleic acid (18:1) with 47.0%, and linoleic acid (18:2) with 31.6% [3]. The oil contained a high percentage of monounsaturated oleic and polyunsaturated linoleic acid indicating it has a semi-drying property [4]; hence, it may be potentially useful for the surface coating industry.

In addition, the oil also contained a higher concentration of unsaturated fatty acids than the saturated ones, and when combined with oxygen and exposed to the air forms a film, characteristic of 'drying oils' [5]. The physico-chemical parameters of *J. curcas* oil are presented in Table 1. The high iodine value (116 mg.l2.g-1) indicates a preponderance of unsaturated fatty acid. The fatty acids, acid value and peroxide values are relatively high compared with most seed oils.

Table 1: Physic-Chemical parameter of Jatropha Curcas oil [5]

Composition	Percentage
Color	Golden yellow
Specific gravity	0.8601
Refractive index	1.4735
Free fatty acids (%)	38
Acid value (mg. KOH. g^{-1})	4.24
Saponification value (mg. KOH. g^{-1})	169.9
Iodine value (mg. I_2. g^{-1})	111.6
Peroxide value (mg reac. $O_2 g^{-1}$)	3.5

2.2.1 Jatropha Trees and Leaves

The tree itself has been used for erosion control, fire wood, as hedge plant and for plant protection. Leaves have been used for rearing of silkworm, in dyeing, medicines, and as an anti-inflammatory substance. Latex has medicinal and pesticidal control properties. It has been used for wound healing as protease has medicinal use.

Figure 2(a): Jatropha leaves [18]

2.2.2 Flowers

The flowers in the form of a number of clusters and the colors yellow green. There are more than 15 flowers for each of the bunch. Jatropha is part of the monoecious plant and the flower is unisexual. The flowers attract bees and thus the plant has a potential use in honey production.

Figure 2(b): Jatropha Flowers [18]

2.2.3 Seeds

Seeds have been used as insecticide, food/fodder (when detoxified), seed oil for soap production, fuel, lubricant, insecticide, medicine and when mixed with iron oxide, they can be used in varnish. Seed cake is useful as fertilizer or in biogas production. Briquettes can be used as fuel, nutraceuticals, or, after further processing, as fodder (when detoxified), and seed shells are combustible. Fruit hulls are combustible, contain tannin and can be used as green manure and in biogas production. Lastly, the roots contain oil with strong antihelminthic properties [11].

Figure 3(a): Jatropha fruits [18]   Figure 3(b): Mature Jatropha seeds [18]

2.3 Jatropha Curcas Extraction

Analysis of the Jatropha Curcas seed shows the composition of 6.20% moisture, 18.00% of proyein, 38.00% of fat, 17.00% carbohydrate, 15.50 % of fibre and 5.30% of ash.The chemical anaIysis of Jatropha curcas Oil:

Table 2: Chemical analysis of Jatropha Curcas [11]

Parameter	Value
Acid Value:	38.2
Saponification:	169.9
Iodine Value:	101.7
Viscosity (31°C) cp:	40.4

There are many processes to extract oil from the seeds and commonly used namely mechanical extraction and chemical extraction. Since there is about 6.2% of moisture content in the seed, the Jatropha seed need to be dried in the oven at 70^oC to reduce its moisture content. Using mechanical method to extract the oil, either whole seeds or kernels or both of it can be fed into the process. Meanwhile, only kernels can be applied into the process if chemical extraction is used.

2.3.1 Mechanical Extraction

Mechanical extraction processes are suitable for both small- and large-capacity operations. The three basic steps in these processes are (a) kernel pre-treatment, (b) screw-pressing, and (c) oil clarification.

(a) Kernel Pre-Treatment

Proper kernel pre-treatment is necessary to efficiently extract the oil from the kernels. The feed kernels must first be cleaned of foreign materials that may cause damage to the screw-presses. Magnetic separators commonly are installed to remove metal debris, while vibrating screens are used to sieve sand, stones or other undesirable materials. A swinging hammer grinder and breaker rolls then breaks the kernels into small fragments. The kernel fragments subsequently are subjected to

flaking in a roller mill. The thickness of kernel cakes is progressively reduced as it travels from the top roller to the bottom.

(b) Screw Pressing

The kernel flake is then fed to the screw-press, which consists of an interrupted helical thread which revolves within a stationary perforated cylinder called the cage or barrel. The meal is forced through the barrel by the action of the revolving worms. The kernel flake is compressed as it passes through the barrel. The expelled oil drains through the perforation of the lining bars of the barrel, while the de-oiled cake is discharged through an annular orifice.

(c) Oil clarification

The expelled oil invariably contains a certain quantity of 'fines and foots' that need to be removed. The oil from the presses is drained to a vessel or tank. It is then either pumped to a decanter or revolving coarse screen to remove a large part of the solid impurities. The oil is then pumped to a filter press to remove the remaining solids and fines in order to produce clear oil prior to storage. The cakes discharged from the presses are conveyed for bagging or bulk storage. Figure 4 shos the process involves in oil extraction.
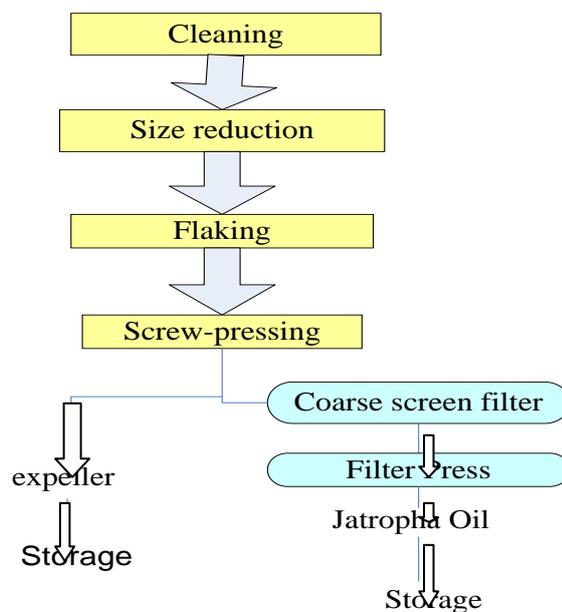
Figure 4: Mechanical extraction of kernel.

2.3.2   Solvent Extraction (Chemical)

Mechanical extraction, had its limits in terms of oil recovery. Because pressing generates heat and high temperatures damage both the oil and meal, an oil content of the press cake below 5-6% was difficult to achieve. Solvent extraction was developed because it allows a more complete extraction at lower temperatures. It begins to be economically attractive where large quantities of seed can be processed.

Many solvents, both petroleum and non-petroleum types have been used in the laboratory to extract oil from oilseeds such as [7]:

- Hydrocarbon solvents (hexane, heptane, pentane): Usually hexane is used. Extraction-grade hexane has an an n-hexane content between $48 - 98\%$ and a narrow distillation range. It is free of nitrogen and sulphur and unsaturated compounds and sufficiently stable to be used indefinitely.
- Halogenated solvents (trichloroethylene, dichloromethane)
- Water (with and without enzymes): Water only finds application as a solvent in the extraction of oil from palm, olive and coconuts. Its drawbacks are more than 10% residual oil in cake and energyintensive separation and drying.

When using any solvent extraction,the most important decision is the selection of the solvent used. The properties which should be considered when choosing the appropiate solvent are selectivity, insolubility, density, chemical reactivity and viscosity. One of the apparatus used in solvent extraction is Soxhlet extractor. Soxhlet extraction is ideal for solid samples and the solutes required are extracted via the process of percolation.

2.3.3   Soxhlet Extractor

A Soxhlet extractor is a piece of laboratory apparatus invented in 1879 by Franz von Soxhlet [15]. It was originally designed for the extraction of a lipid from a solid material. Normally a solid material containing some of the desired compound is placed inside a thimble which is loaded into the main chamber of the Soxhlet extractor. The Soxhlet extractor is placed onto a flask containing the extraction

solvent. The Soxhlet is then equipped with a condenser. The solvent is heated to reflux. The solvent vapour travels up a distillation and floods into the chamber housing the thimble of solid. The condenser ensures that any solvent vapour cools, and drips back down into the chamber housing the solid material.

The chamber containing the solid material slowly fills with warm solvent. Some of the desired compound will then dissolve in the warm solvent. When the Soxhlet chamber is almost full, the chamber is automatically emptied by a siphon side arm, with the solvent running back down to the distillation flask. This cycle may be allowed to repeat many times. During each cycle, a portion of the non-volatile compound dissolves in the solvent. After many cycles the desired compound is concentrated in the distillation flask. After extraction the solvent is removed, typically by a rotary evaporator, yielding the extracted compound. The non-soluble portion of the extracted solid remains in the thimble, and discarded. Below is a schematic representation of a Soxhlet extractor.

1: Stirrer bar

2: Still pot (the still pot should not be overfilled and the volume of solvent in the still pot should be 3 to 4 times the volume of the soxhlet chamber)

3: Distillation path

4: Thimble

5: Solid

6: Siphon top

7: Siphon exit

8: Expansion adapter

9: Condensor

10: Cooling water

Figure 5: Soxhlet Extractor [17]   11: Cooling water out
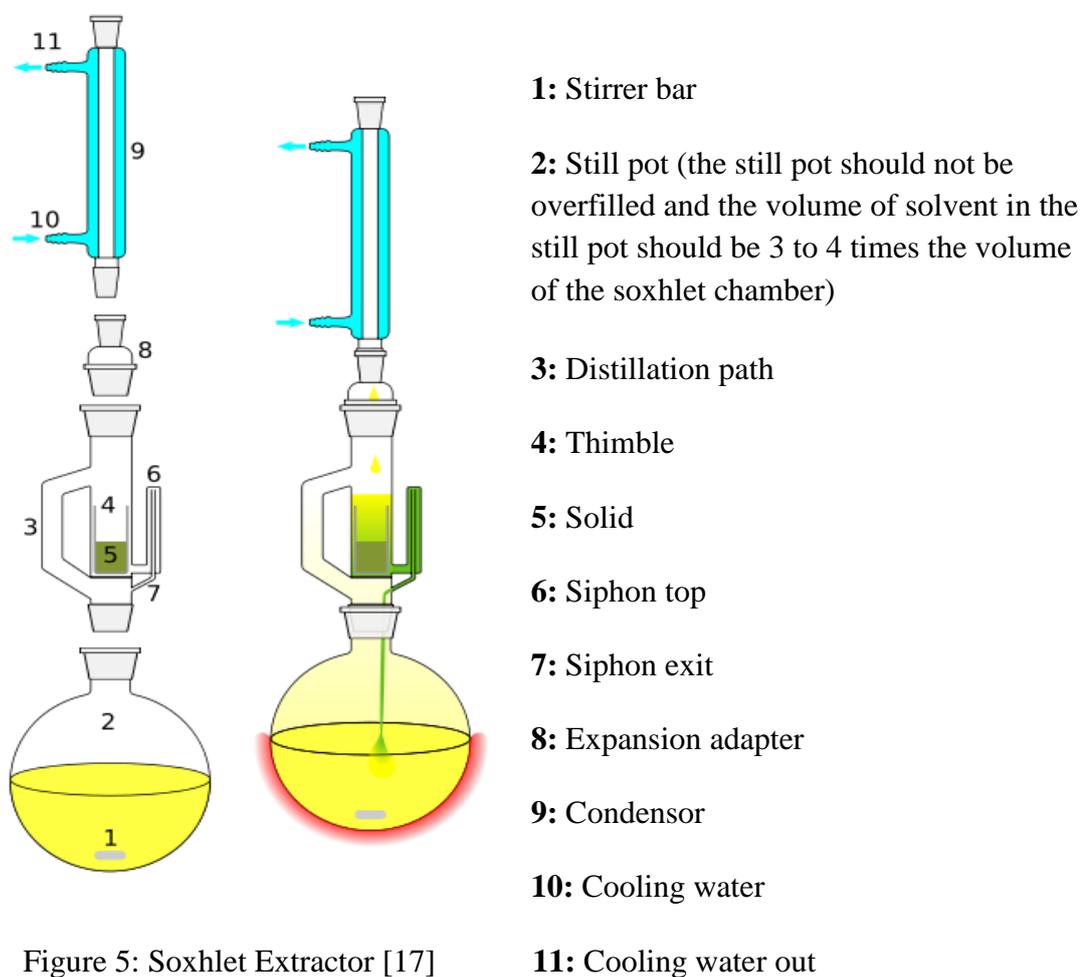
2.4   Theory of Inhibitors

Corrosion inhibitors are defined as materials that when present in a system in relatively small quantities will produce a reduction in metal loss due to corrosion attack. These inhibitors can interfere with the anodic or cathodic reaction and can form a protective barrier on the metal surface against corrosive agents. The inhibitors usually contain three elements:

- One or more active inhibitor components
- A solvent base
- Certain additives, such as surfactants, dispersants, demulsifiers, and defoamers

Solvents are used to dilute inhibitors to control physical characteristics (such as viscosity and pour point) and to assist inhibition.

2.4.1   Physical Characteristics

Physical characteristics of inhibitors must be considered when evaluating a potential application. These include:

- Physical form
- Solubility
- Emulsion forming tendencies
- Thermal stability
- Compatibility with other chemicals
- Thermal stability

2.4.1.1   Physical Form

Inhibitors may be in the form of solid or liquid. Solid inhibitors have been made in the shape of stick that will sink in the system and then slowly dissolve and be produced back. These sticks are rarely used. Most corrosion inhibitors are in liquid form and have densities that range from 840g/L to 1440g/L.

2.4.1.2 Solubility

The formation of an inhibitor film and its life are primarily governed by the solubility of that product in the system. There are soluble, insoluble and dispersible types of inhibitors.

A product is soluble in fluid if it forms a clear mixture that does not separated. A product is insoluble in a fluid when it will separate after mixing to form an identifiable layer. Materials are dispersible in a fluid if they form a mixture that is not clear and separate slowly.

Different solubility is required for different applications of corrosion inhibitors. An inhibitor to be added continuously to a water flood should be water soluble or highly dispersible. On the other hand, in the application of periodic treatment; continuing protection requires some degree of insolubility of the inhibitors in the fluid to which it is exposed.

2.4.1.2 Emulsion-Forming Tendencies

Because of the chemical nature of most corrosion inhibitors, there is a positive tendency in water oil-systems to form emulsions. Some of these emulsions will break down quite readily, while other is extremely stable and practically impossible to break. The inclusion of an emulsifier in a corrosion inhibitor can be one of the alternatives of this problem.

2.4.1.3 Thermal Stability

Corrosion inhibitors generally have temperature limits above which they will lose their effectiveness and change their chemical composition. This temperature may be variable for any one inhibitor, depending on such conditions as pressure and presence of water.

2.4.2 Selection of Inhibitor

Many factor are involved in the selection of inhibitors, includes:

- Identification of the problem to be solved
- Corrosives present
- Type of system
- Pressure and temperature
- Velocity
- System composition

Although problems such as rod break or pipe line leaks may initially be seen as purely corrosion failures, the actual cause of the problem could be oxygen, scale or bacteria. If attack is due to oxygen entry, installation of gas blanket could greatly reduce corrosion.

The presence of corrosives such as H_2S and CO_2 greatly influences the choice of an inhibitor. Some inhibitors perform best in sweet fluids, while other inhibitors work best in sour fluids. Even the concentration of NaCl has a bearing on the choice of inhibitors.

Both pressure and temperature have on influence on inhibitor selection. Inhibitors may get polymerized and formed sludge at high temperature and pressure condition. Pressure influences the corrosivity of H_2S and CO_2.

Velocity is another factor need to be considered. For example, in the pipeline system, low velocity might be insufficient to displace water from low areas in the line. In the case of dry gas pipelines with low velocity, a water soluble inhibitor should be selected and should be injected continuously. If the velocity is high enough to prevent the accumulations of water in low areas of a dry gas line, an oil-soluble inhibitor should be batch treated.

CHAPTER 3
METHODOLOGY

3.1   Introduction

Corrosion testing is divided into four types of classification which are laboratory tests, pilot plant or semi-works tests, plant and field tests. Laboratory tests are characterized by small specimens and small volumes of solutions, and actual conditions are simulated insofar as conveniently possible. In this laboratory tests, the variables that take into account are exposure time to the environment, concentration of Jatropha oil inhibitor, pH values and temperature.

There are two methods of corrosion testing applied in this research; weight-loss method and electrochemical corrosion cell. The corrosion rate is easily to be calculated base on the formula in these two methods. Besides, these methods are environmental friendly. Below is the flow chart of methodology in the experiment.

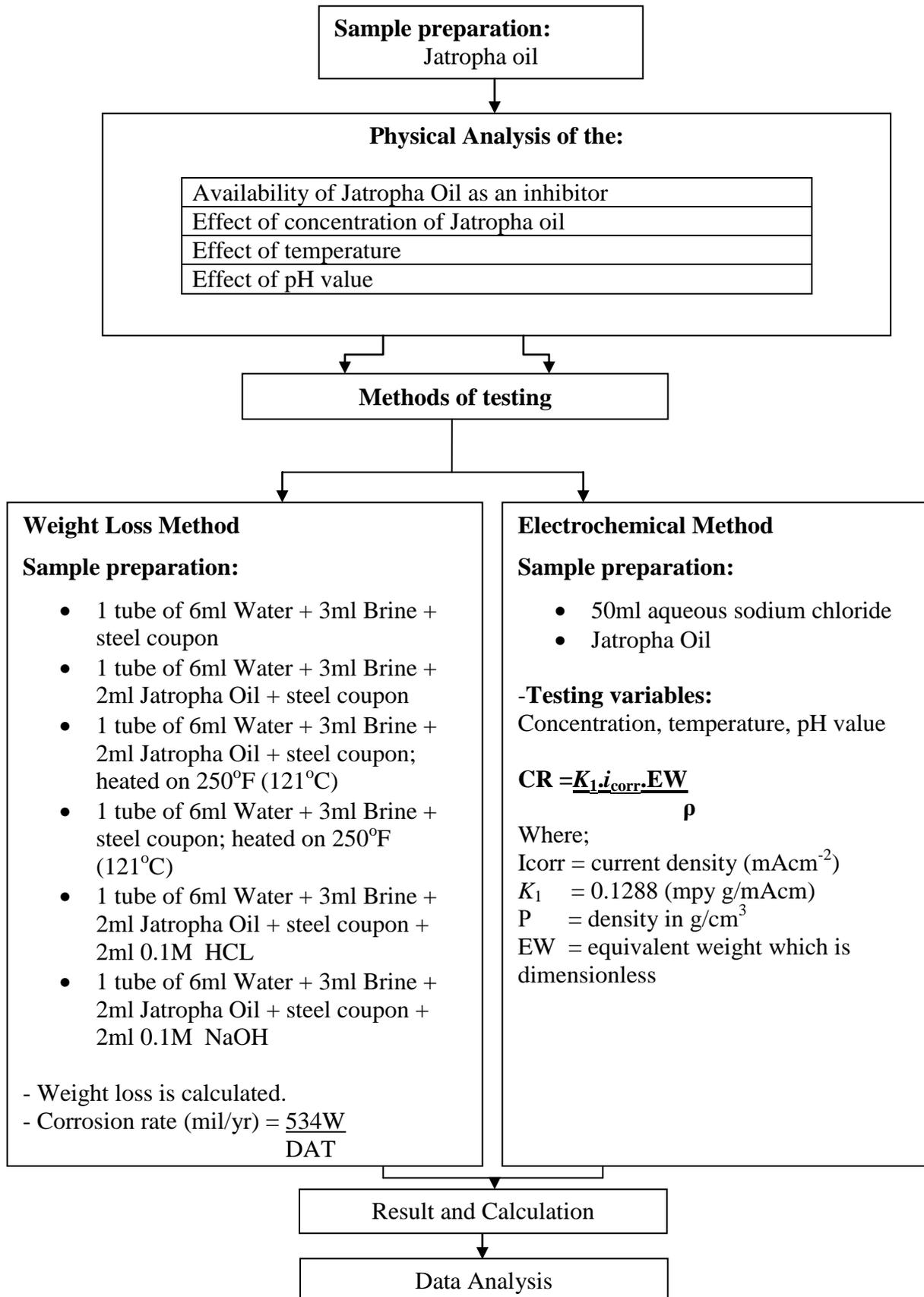Please refer to Appendix I for Gantt chart.

Sample preparation:
Jatropha oil

Physical Analysis of the:

| Availability of Jatropha Oil as an inhibitor |
| Effect of concentration of Jatropha oil |
| Effect of temperature |
| Effect of pH value |

Methods of testing

Weight Loss Method

Sample preparation:

- 1 tube of 6ml Water + 3ml Brine + steel coupon
- 1 tube of 6ml Water + 3ml Brine + 2ml Jatropha Oil + steel coupon
- 1 tube of 6ml Water + 3ml Brine + 2ml Jatropha Oil + steel coupon; heated on 250°F (121°C)
- 1 tube of 6ml Water + 3ml Brine + steel coupon; heated on 250°F (121°C)
- 1 tube of 6ml Water + 3ml Brine + 2ml Jatropha Oil + steel coupon + 2ml 0.1M HCL
- 1 tube of 6ml Water + 3ml Brine + 2ml Jatropha Oil + steel coupon + 2ml 0.1M NaOH

- Weight loss is calculated.
- Corrosion rate (mil/yr) = $\dfrac{534W}{DAT}$

Electrochemical Method

Sample preparation:

- 50ml aqueous sodium chloride
- Jatropha Oil

-**Testing variables:**
Concentration, temperature, pH value

$$CR = \dfrac{K_1 . i_{corr} . EW}{\rho}$$

Where;
I_{corr} = current density (mAcm^{-2})
K_1 = 0.1288 (mpy g/mAcm)
P = density in g/cm^3
EW = equivalent weight which is dimensionless

Result and Calculation

Data Analysis

Figure 6: Flow diagram of methodology

3.2 Sample Preparation

3.2.1 Chemically Oil Extraction

Jatropha Curcas seeds must be dehulled and ground in order to increase efficiency of the extraction. By using blender, Jatropha Curcas seeds were ground to small particle size. It was then placed in the oven to release the moisture content. The oven was set at 70°C in 48 hours. The moisture content of the seeds was measured using Mettler Toledo moisture analyzer. The seed were placed in the sample pan until its entire surface homogeneously covered by the seeds. Prior to start calculating the sample's moisture content, ramp time which was 3 minutes must be set then followed by setting the desired temperature which 100°C. The reading of the moisture content started when the temperature reached 100°C and it was hold for 30 minutes.

The oil will be extracted chemically by using hexane as the solvent since it can extract the oil up to 99% of the total amount of the oil available in the seed and also environmental friendly. By using Soxhlet extractor 30 grams of Jatropha seeds were accurately weighed and placed in the thimble. Meanwhile, 140 ml of hexane with purity 99.9% was measured and put inside of the round bottom flask. Then, extraction time started after the equipment had been set up and reached at desired temperature, boiling point of hexane (68°C). To get more accurate data, this process was varied from 2 hours to 6 hours with increment 2. This equipment can be seen as figure 7.

Figure 7: Soxhlet extractor available in laboratory [17]

3.3 Weight- Loss Corrosion Test using Jatropha Oil in varies Condition

In the test, coupons are exposed in fluids with and without Jatropha oil and evaluated on a weight-loss basis.

3.3.1 Preparation of specimen (carbon steel coupon)

1. All rough edges on the surface of coupons were smoothened.
2. The coupons were dipped into acetone to degrease their surfaces.
3. They were etched in a 5% HCl solution.
4. Then, neutralized the coupons with sodium bi-carbonate solution.
5. Clean under tap water by scrubbing with nylon brush, then rinsed with acetone.
6. The coupons were dried and then weight.

3.3.2 Procedure for weight loss test

1. Six pieces of ASTM 102.6 carbon mild steel of 5mm thick were immersed in six different tubes.
2. Each tube contains:
   - 1 tube of 6ml Water + 3ml Brine + steel coupon
   - 1 tube of 6ml Water + 3ml Brine + 2ml Jatropha Oil + steel coupon
   - 1 tube of 6ml Water + 3ml Brine + 2ml Jatropha Oil + steel coupon; heated on $120^{o}C$ ($250^{o}F$)
   - 1 tube of 6ml Water + 3ml Brine + steel coupon; heated on $120^{o}C$ ($250^{o}F$)
   -
   - 1 tube of 6ml Water + 3ml Brine + 2ml Jatropha Oil + steel coupon + 1ml 0.1M HCL
   - 1 tube of 6ml Water + 3ml Brine + 2ml Jatropha Oil + steel coupon + 1ml 0.1M NaOH

3. The test tubes with the above static condition will be evaluated after 24 hours.

3.3.3   Procedure after test

1. The coupons were removed from the test sample, the immersed in sodium-bi-carbonate solution.
2. The coupons were cleaned by scrubbing with nylon brush under the tap water.
3. The clean coupons were dried and then weighed.

The coupons will be weighted before and after the experiment and will be recorded in the table as in the result. The rate of corrosion can be calculated by equation (5).

3.4   Electrochemical Corrosion Cell

The electrochemical cell is set up with two electrodes in order to illustrate the different electrical conductivities of liquids (aqueous sodium hydroxide and mixture of aqueous sodium with Jatropha oil). The apparatus needed are:

     i. Power supply(2-5 V)
    ii. 2 x250mL beakers
   iii. Two inert electrode; zinc and cuprum rods
   iv. Ammeter, switch and connections
    v. Liquids: Aqueous sodium chloride and Jatropha oil

3.4.1   Effect of Concentration of Jatropha Oil

The purpose of this experiment is to investigate the availability of the Jatropha Oil as a corrosion inhibitor and its effectiveness towards the different fluid concentration. The corrosion rate will be calculated and compared to the results in weight-loss corrosion test method. Procedures of the experiment are as below:
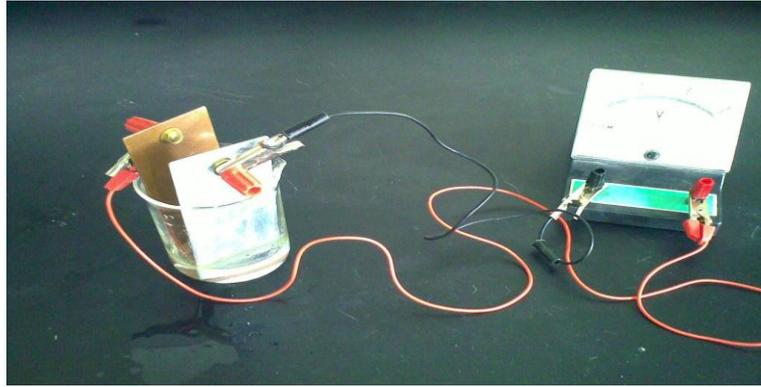
Figure 8: Electrochemical cell

1. After the apparatus is set up (figure 8), 50mL of aqueous sodium chloride is poured into a beaker.
2. The electrodes then immersed into the beaker and switch is closed.
3. The produced current is recorded and opens the switch.
4. The same electrodes are removed, cleaned and dried.
5. These steps are repeat using mixture of 50ml of aqueous sodium chloride with 5ml, 10ml, 15ml, 20ml,25ml of Jatropha Oil and the oil is added until the reading of ammeter is zero.

The corrosion rate from the electrochemical cell can be calculated by:

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

Where;

CR is given in mpy, icorr in mAcm^{-2}

K_1 is 0.1288 in unit of mpy g/mAcm

P is density in g/cm^3 (density of zinc is 7.14 g/cm^3)

EW is equivalent weight which is dimensionless

3.4.2   Effect of Temperature

In order to illustrate the effect of temperature upon energy levels by measurement of potential difference, the above experiment set up is applied. The beaker that contains mixture of 50ml of aqueous sodium chloride with suitable amount of Jatropha Oil is heated using the hotplate with temperature of 27°C. Then the apparatus is connected again and current is recorded. The same steps are repeated with 50°C, 70°C, 90°C, 115°C and 140°C. Then corrosion rate is calculated base on experiment data.

Figure 9: Electrochemical cell with temperature control

3.4.3   Effect of pH Value

5ml of 2M Hydrochloric acid (HCL) is added into the beaker that containing 35ml Jatropha oil derived inhibitor and 50ml aqueous sodium hydroxide. The electrodes are immersed into the beaker, and start the experiment and record the current that produced. After that, the apparatus and equipments are cleaned and dried. The experiment is repeated by using 10ml and 15ml 2M HCL. The same steps are repeated with using 0.1 M HCL acids, 0.0001M HCL acid, 2M sodium hydroxide (NaOH), 0.1M NaOH and 0.0001M NaOH. Then corrosion rate is calculated.

CHAPTER 4
RESULT AND DISCUSSION

4.1 RESULTS

In order to evaluate the availability of Jatropha oil as a corrosion inhibitor, the corrosion rate will be calculated in each experiment. In the weight loss test, the specimens have been exposed to specific condition for 24 hours.

4.1.1 Corrosion Rate in Weight- Loss Test

In the test, coupons in fluids with and without Jatropha oil are exposed in vary condition in order to observe the corrosivity. Weight loss is calculated in order to know the corrosion rate and determination of Jatropha Oil as anti-corrosion. The coupons will be weighted before and after the experiment and will be recorded in the table as below.

Table 3: Weight-loss corrosion test

Set	Mixture	Weight of coupon before test, g	Weight of coupon after test, g	Weight loss (g)	Corrosion rate, mpy
A	water+ brine + coupon steel	13.515	13.509	0.006	36.0
B	water+ brine + Jatropha oil + coupon steel	13.422	13.422	0.000	0.0
C	Water + Brine + Jatropha Oil + steel coupon; heated on 250^oF (121^oC)	13.506	13.499	0.003	5.7
D	Water + Brine + Jatropha Oil + steel coupon + 0.1M HCL	13.510	13.510	0.000	0
E	Water + Brine + Jatropha Oil + steel coupon + 0.1M NaOH	13.515	13.480	0.035	210

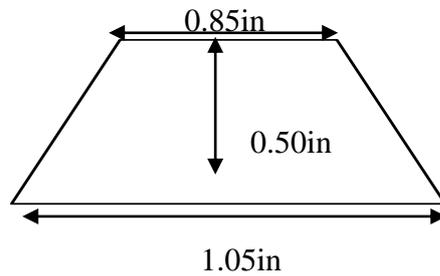The density of mild carbon steel is 7.85g/cm^3. The area of the coupon is calculated as figure 9:

Figure 10: Illustration of mild carbon steel coupon

Area= (half the sum of parallel sides) x (perpendicular distance between the parallel sides)

$\quad$ = [(0.85 + 1.05)/2] x 0.50

$\quad$ = 0.472in^2

Due to six metal surfaces have been exposed to the corrosion environment, with the 0.1986in thick of the coupon, the total area of the coupon is;

Total Area (A) = (0.472in^{2}x2) + (0.1968inx0.85inx2) + (0.1968inx 0.50inx2)

$\quad\quad\quad\quad$ = 1.48in^2

Corrosion rate will be expressed in mils per year. The expression of mils per year readily calculated from weight loss of metal coupon during the corrosion test as table 4:

$$\text{mpy} = \frac{534\ W}{DAT}$$

Where W = weight loss, mg

$\quad\quad$ D = density of coupon, g/cm^3

$\quad\quad$ A = area of coupon, sq. in

$\quad\quad$ T = exposure time, hr

Table 4: Corrosion rate calculation in mil/year (mpy)

Mixture	Corrosion Rate
water+ brine + coupon steel	$mpy = \dfrac{534W}{DAT}$ $= \dfrac{534 \times 6mg}{7.85g/cm^3 \times 1.48\ in^2 \times 24hr}$ $= \textbf{11.49 mil/year}$
water+ brine + Jatropha oil + coupon steel	$mpy = \dfrac{534W}{DAT}$ $= \dfrac{534 \times 0mg}{7.85g/cm^3 \times 1.48\ in^2 \times 24hr}$ $= \textbf{0 mil/year}$
Water + Brine + steel coupon; heated on 250°F (121°C)	$mpy = \dfrac{534W}{DAT}$ $= \dfrac{534 \times 60mg}{7.85g/cm^3 \times 1.48\ in^2 \times 24hr}$ $= \textbf{115 mil/year}$
Water + Brine + Jatropha Oil + steel coupon; heated on 250°F (121°C)	$mpy = \dfrac{534W}{DAT}$ $= \dfrac{534 \times 3mg}{7.85g/cm^3 \times 1.48\ in^2 \times 24hr}$ $= \textbf{5.7 mil/year}$
Water + Brine + Jatropha Oil + steel coupon + 0.1M HCL	$mpy = \dfrac{534W}{DAT}$ $= \dfrac{534 \times 0mg}{7.85g/cm^3 \times 1.48\ in^2 \times 24hr}$ $= \textbf{0 mil/year}$
Water + Brine + Jatropha Oil + steel coupon + 0.1M NaOH	$mpy = \dfrac{534W}{DAT}$ $= \dfrac{534 \times 35mg}{7.85g/cm^3 \times 1.48\ in^2 \times 24hr}$ $= \textbf{210 mil/year}$

4.1.2 Effect of Concentration of Jatropha Oil on it Inhibition

Electrochemical corrosion rate measurements often provide results in terms of electrical current. The conversion of these results to either mass loss or penetration rates requires additional electrochemical information. For this time of experiment, the digital ammeter has been used. Below is the result of the experiment for investigating the effect of concentration of Jatropha oil as inhibitor.

Table 5: Concentration of Jatropha oil with the current produced respectively

Concentration of Jatropha Oil (ml)	%-volume of Jatropha Oil	Current produced (mA)	i_{corr} (mAcm^{-2})	CR (mpy)
0	0	2.51	0.1673	0.0981
5	9.0	1.1	0.0733	0.0430
10	17	0.25	0.0167	0.0098
15	23	0.19	0.0127	0.0074
20	29	0.16	0.0107	0.0063
25	33	0.08	0.0053	0.0031
30	38	0.03	0.0020	0.0012
35	42	0	0.0000	0.0000
40	45	0	0.0000	0.0000

The graph below shows the relationship between varies in concentration of Jatropha Oil with respective current measured.
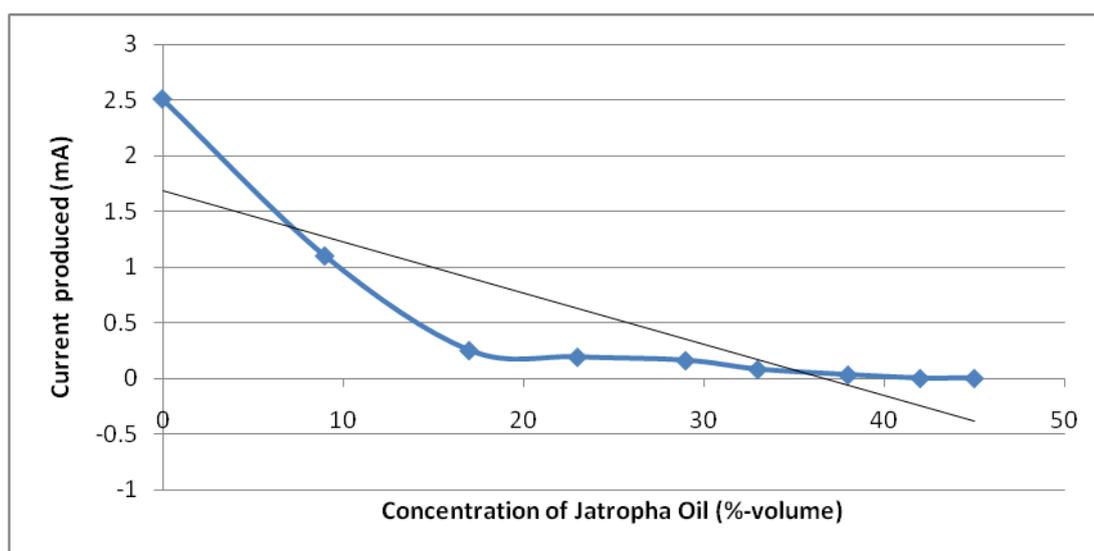

Figure 11: Concentration of Jatropha oil vs Current Produced

The first step is to convert the measured current value to current density. This is can be accomplished by dividing the total current to the geometric area of the electrode exposed to the solution. It is assumed that the current distributes uniformly across the area used in this calculation. For example, from galvanic cell, the measured current is 2.51mA and the respective current density can be calculated as below:

Current density, i_{corr} = <u>total anodic current, mA</u>

$$\qquad\qquad\text{Exposed specimen area, cm}^2$$

$$= \frac{2.51\text{mA}}{15\text{cm}^2}$$

$$= 0.1673\text{mAcm}^{-2}$$

Faraday's law can be used to calculate the corrosion rate (CR) from the corrosion current.

CR = <u>$K_1.i_{corr}.$EW</u>

$$\qquad\qquad\boldsymbol{\rho}$$

Where;

CR is given in mpy, icorr in mAcm^{-2}

K_1 is 0.1288 in unit of mpy g/mAcm

P is density in g/cm^3 (density of zinc is 7.14 g/cm^3)

EW is equivalent weight which is dimensionless.

EW (for zinc) = <u>Gram atomic weight</u>

$$\qquad\qquad\text{Number of electron transferred}$$

$$= 65/\ 2 = 32.5$$

CR = <u>0.1288 mpy g/mAcm x 0.1673mAcm^{-2} x 32.5</u>

$$\qquad\qquad 7.14\text{ g/cm}^3$$

$$= 0.0981\text{mpy}$$

4.1.3   Effect of Temperature on the Inhibition of Jatropha Oil

To investigate the effect of temperature to the inhibition efficiency by the Jatropha oil, the galvanic cell is set up. The beaker that contains mixture of 50ml of aqueous sodium chloride with 35ml of Jatropha Oil is heated using the magnetic stirred hotplate with temperature of 27°C (80°F). Then the apparatus is connected again and current is recorded. The same steps are repeated with 50°C, 70°C, 90°C, 115°C and 140°C. The measured current was recorded as below.

Table 6: Temperature of solution with the current produced respectively

Temperature (^{o}C)	Current Produced (mA)				i_{corr} (mAcm^{-2})	CR (mpy)
	1st run	2nd run	3rd run	Average		
27	0.330	0.300	0.300	0.310	0.0207	0.0121
50	0.200	0.250	0.280	0.243	0.0162	0.0095
70	0.160	0.200	0.070	0.143	0.0096	0.0056
90	0.110	0.160	0.090	0.120	0.0080	0.0047
115	0.001	0.000	0.000	0.000	0.0000	0.0000
140	0.000	0.000	0.000	0.000	0.0000	0.0000

The graph below shows the relationship between the temperatures with respective current measured.
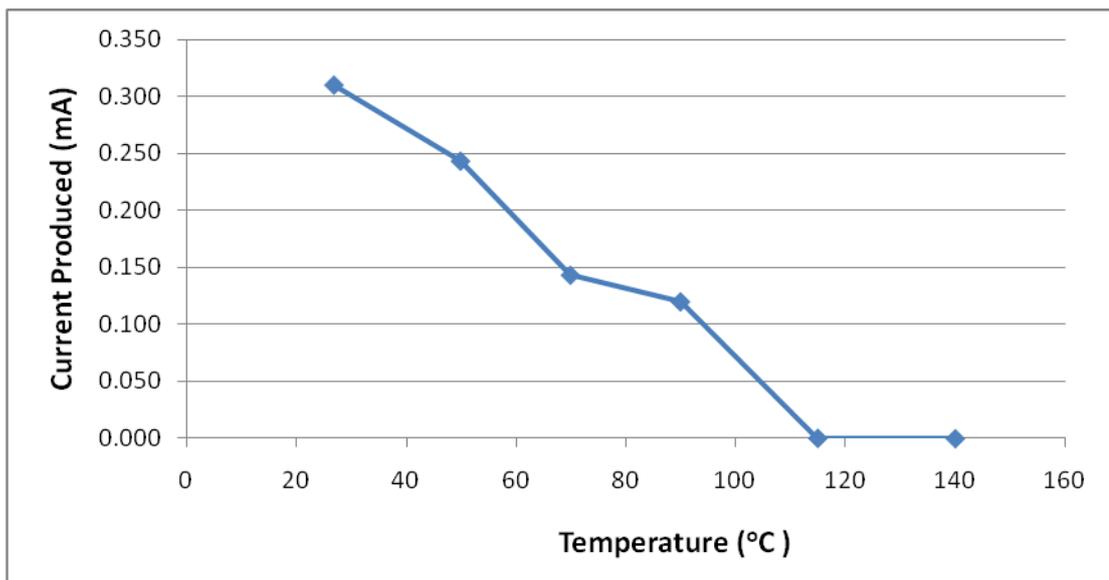

Figure 12: Graph of Temperatures vs Current Produced

4.1.4   Effect of pH value on the Inhibition of Jatropha Oil

To investigate the effect of temperature to the inhibition efficiency by the Jatropha oil, the galvanic cell is set up. Different concentration of acid and base are used gives different set of pH value. Table 6 and shows pH values with respective corrosion rates.

Table 7: Effect of different pH value to current produced and corrosion rate

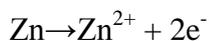| Molarity of NaOH (M)	Quantiy of NaOH (ml)	pH value	Current produced (mA)	i_{corr} (mAcm^{-2})	CR (mpy)
2	15	13.39	2.1	0.1400	0.0821
	10	13.28	1.56	0.1040	0.0610
	5	12.56	1.46	0.0973	0.0571
0.1	15	7.8	0.63	0.0420	0.0246
	10	7.6	0.54	0.0360	0.0211
	5	7.26	0.44	0.0293	0.0172
0.0001	15	6.07	0	0.0000	0.0000
	10	5.9	0	0.0000	0.0000
	5	5.8	0.23	0.0153	0.0090
Molarity of HCL (M)	Quantiy of HCL(ml)	pH value	Current produced (mA)	i_{corr} (mAcm^{-2})	CR (mpy)
0.0001	5	5.89	0.31	0.0207	0.0121
	10	5.27	0	0.0000	0.0000
	15	5.11	0	0.0000	0.0000
0.1	5	4.2	0.03	0.0020	0.0012
	10	2.59	0	0.0000	0.0000
	15	1.76	0	0.0000	0.0000
2	5	1.27	0	0.0000	0.0000
	10	1.21	0.1	0.0067	0.0039
	15	1.06	0.11	0.0073	0.0043

4.2   DISCUSSION

The corrosion test has been done by using weight loss method and electrochemical method. In weight loss method, the basic experiment has been done which is to test the availability of Jatropha oil to behave as a corrosion inhibitor. In order to investigate the effectiveness of the Jatropha oil as an organic inhibitor toward temperature, pH and concentration limits, the electrochemical method has been used for corrosion test.

4.2.1   Calculation of Corrosion Rate by Faraday's Law

The mixed potential-theory consists of two simple hypothesis. First any electrochemical reaction can be divided into two or more partial oxidation and reduction reactions and there can be no net accumulation of electric charge during an electrochemical reaction. It can be experimentally demonstrated that electrochemical reactions are composed of two or more partial oxidation or reduction reactions. The second hypothesis is a restatement of the law of conservation of charge. It follows that during the corrosion of an electrically isolated metal sample, the total rate of oxidation must equal the total of reduction.

Corrosion involves the destructive attack of metal by chemical or electrochemical reaction with its environment. Usually corrosion consists of a set of redox reactions that are electrochemically in nature. The metal is oxidized to corrosion products at anodic sites:

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

Hydrogen is reduced at cathodic sites; $2H^+ + 2e^- \rightarrow H_2$

Faraday's Law states that 96486.7 Coulombs (equal to one faraday) of charge transfer will oxidize or reduce one Gram Equivalentweight of the material involved in the electrochemical reaction. Faraday's developed a number of Coulombs by dividing Avogadro's Number (6.02×10^{23} which equal the number of atoms of any specific atom whose weight equal its gram atomic weight) by the number of electrons in one coulomb.

A coulomb is that amout of electrical charge equal to the charge carried by 6.24×10^{18} electron;

$$\frac{6.02 \times 10^{23} \text{ Atoms/Grams Atomic Weight}}{6.24 \times 10^{18} \text{ electrons/coulomb}} = 96486.7 \text{ Coulombs/Faraday}$$

According to Faraday's Law;

$Q = \dfrac{nFW}{M}$

where   Q= charge passed (coulombs);

   n= number of electron transferred;

   W = weight of electoactive species (g)

   M = Molecular weight (g)

$W = \dfrac{QM}{nF}$

$= \dfrac{Q.EW}{F} = \dfrac{it \times EW}{F}$

W/t = Corrosion Rate(CR)

$= \dfrac{it \times EW}{\rho FA}$

$= \dfrac{K1 \times icorr \times EW}{\rho}$

EW (for zinc) = $\dfrac{\text{Gram atomic weight}}{\text{Number of electron transferred}}$

$= 65/2 = 32.5$

4.2.2   Weight Loss Corrosion Test

In the oilfield, the types of metal employed in equipment are numerous, but because of the cost versus performance properties of mild steel, it is most commonly used. Weight loss is probably the most common form of measurement of corrosion control effectiveness. In our experiment, mild carbon coupon steel are used as the specimen.

From the experiment, it was observed that the brine mixture without adding any of Jatropha oil cause the corrosion to the mild carbon steel coupon with the rates of 11.49 mpy. Meanwhile, the Jatropha oil in the second solution has form a film protection layer to the coupon and protect the mild carbon steel from corrode.

For an inhibitor to work effectively it must be dispersed to all wetted surfaces and under the system conditions it must be sufficiently effective to provide adequate protection. Inhibitor efficiency is defined from laboratory measurements, as the relative corrosion rate with and without inhibitor.

$$\text{Inhibitor efficiency} = \frac{CRo - CRinh}{CRo} \times 100\%$$

Where CRinh = corrosion rate in the presence of inhibitor while CRo = corrosion rate in the absence of inhibitor. From the weight loss corrosion test, the inhibitor efficiency is calculated as follow;

$$\text{Inhibitor efficiency} = \frac{11.49mpy - 0}{11.49mpy} \times 100\%$$
$$= 100\%$$

4.2.3   Effect of Jatropha Oil Concentration on Corrosion Behavior

From this experiment, the current reading indicates that there is oxidation and reduction reaction, hence the corrosion process occured. High value of current indicates the high value of corrosion. Generally with increasing inhibitor concentration, the corrosion current density and corrosion rate decrease as shown in Table 4 and Figure 12 respectively. Figure 12 below illustrate the relation between

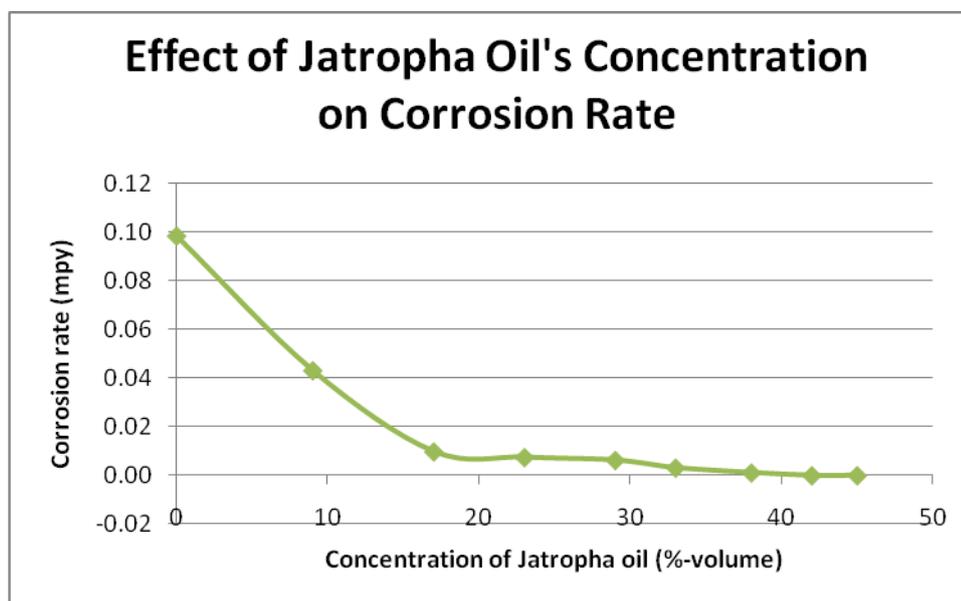the concentration of Jatropha oil as an organic corrosion inhibitor to the amount of corrosion rate.

Figure 13: The effect of concentration of Jatropha oil to corosion rate

The inhibitor efficiency is a function of inhibitor concentration, is typically above 90% for successful inhibitor. Inhibitor efficiency is defined from laboratory measurements, as the relative corrosion rate with and without inhibitor.

$$\text{Inhibitor efficiency} = \frac{CRo - CRinh}{CRo} \times 100\%$$

The rise of the inhibition efficiency is due to the inductive effect of the methyl groups. There has been a growing interest in the use of organic compounds as inhibitors for the aqueous corrosion of metals. A metal surface in an aquoeus environment will have a surface charge and the inhibitor will rapidly be adsorbed onto the metal surfaces. Adsorbed organic molecules prevent steel corrosion by blocking the active sites on the metal surfaces and the inhibition efficiency (E%) of an inhibitor depends strongly on the chemical structure or the functional groups on the inhibitor molecule.

4.2.4   Effect of Temperature on Inhibitor Effectiveness

Temperature is a crucial variable in any accelerated corrosion testing. Higher temperature means more energy available, thus faster rates for the chemical processes that cause both corrosion and degradation of cured film. Increasing the temperature within limits does not alter the corrosion reaction at the metal surface.

It is in interest to develop high performance corrosion inhibitors for high temperature and high shear flow application. A high performance corrosion inhibitor which improves performance and film life at high temperature and high shear will increase reliability of corrosion inhibition and less reliance on expensive corrosion resistant alloy thus reducing capex as well as opex.

From laboratory study, rate of corrosion uniformly decreases as shown in figure 13 this is attributed to the formation of more protective metal due to a decrease in the iron carbonate solubility and also to the competition between the mass transfer and corrosion rates.
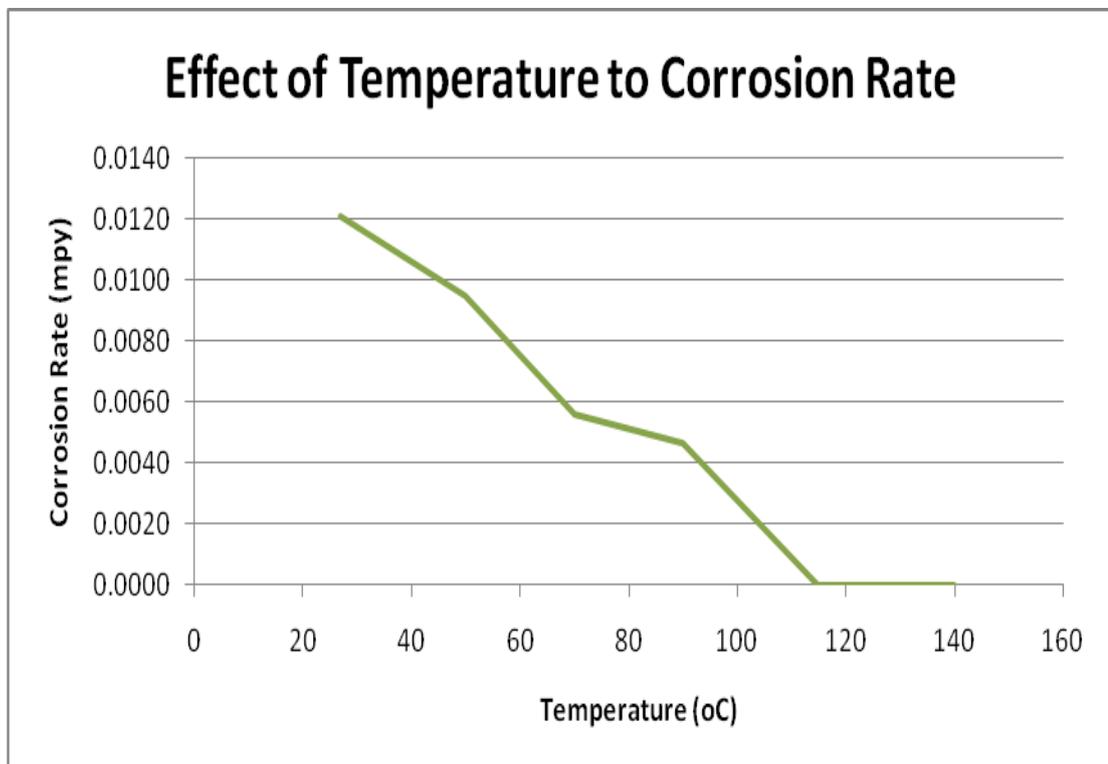

Figure 14: The effect of temperature of solution to corosion rate

In some system such as in drill pipe, elevated temperatures can influences the rheological properties of drilling fluids physically, chemically and electrochemically. An increase in temperature decreases the viscosity of the liquid phase. An increase in temperature increases the ionic activity of any electrolyte, and the solubility of any partially soluble salts that may be present in the mud. The consequence changes in the ionic and base-exchange equilibrium alter the balance between the interparticle attractive and repulsive forces, and hence the degree of dispersion.

From the experiment, Jatropha oil can be a good inhibitor in high temperature system, but further study because large number of variables involved in the elevated temperature fluid system.

4.2.5 Effect of pH value on Inhibitor Effectiveness

The pH value is an important parameter in corrosion of carbon steels and low alloy steels. The pH affects both the electrochemical reactions and the precipitation of corrosion products and other scales. A higher pH means there are fewer free hydrogen ions, and that a change of one pH unit reflects a tenfold change in the concentrations of the hydrogen ion. A pH less than 7 are considered to be acidic, while pH greater than 7 is considered to be basic or alkaline and a pH of 7 considered being neutral.

Corrosion of metals can occur in acid, neutral and alkaline solutions. Normally, low-pH acid waters accelerate corrosion by supplying hydrogen ions to the corrosion process. But, as shown in figure 14, the corrosion process is accelerated in high-pH alkaline solution. Jatropha oil gives the corrosion protection in acid solution. For 5ml (5.5%-volume) of acid or base is added, the corrosion rate is decreases from 0.0121mpy to 0mpy when the pH is in the range 5.89-1.27. When 10ml (10.5%-volume) of acid or base is added to the solution, the corrosion rate is decreases from 0.0211mpy to 0.0mpy when the pH is in range 7.6-5.9 and corrosion rate uniformly at 0mpy from pH of 5.9-2.59. For 15ml (15%-volume) of acid or base is added, corrosion rate uniformly at 0mpy from pH of 6.01-1.76.
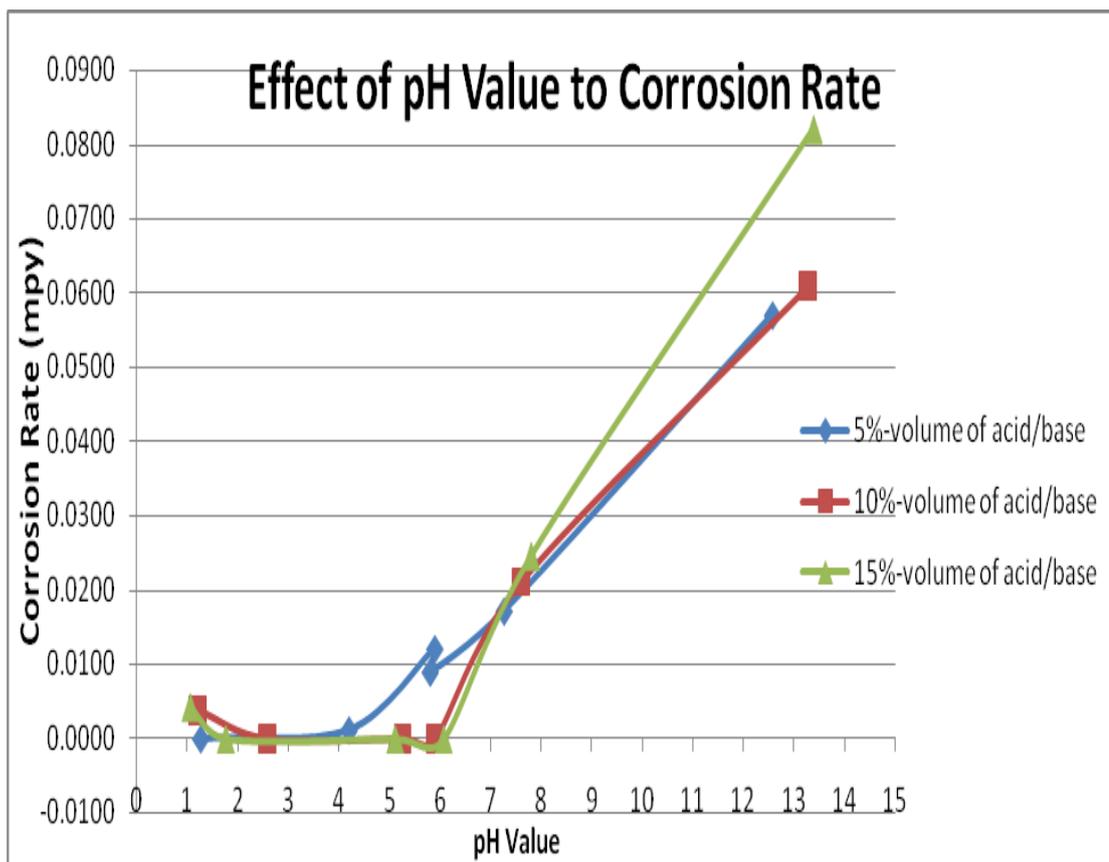
Figure 15: Effect of pH value on corrosion rate in the presence of inhibitor

The oil fraction of Jatropha oil contained saturated fatty acids mainly palmitic acid (16:0) with 14.1% and stearic acid (18:0) with 6.7%. Unsaturated fatty acids consisted of oleic acid (18:1) with 47.0%, and linoleic acid (18:2) with 31.6% [3]. Fatty acids and some form of basic nitrogen containing precursor are the principal active-ingredient sources for corrosion inhibitor. Historically, the first proprietary organic inhibitors were fatty imidazolines made from fatty acids and poly-ethylene amines. With this properties, Jatropha perform well as inhibitor in acidic system.

CHAPTER 5
CONCLUSION AND RECOMMENDATION

5.1   Conclusion

For the conclusion, the corrosion test has been done by using weight loss method and electrochemical method. From the weight loss corrosion test, we can conclude that Jatropha oil is designated as 'film-forming', protect the metal by forming a hydrophobic film on the metal surface. After the metal has been exposed to the corrosion environment for 24 hours, it's shown the inhibitor efficiency is 100%. The oil contained a high percentage of monounsaturated oleic and polyunsaturated linoleic acid indicating it has a semi-drying property; hence, it has high potential to use as corrosion protector.

From the study of effect of inhibitor's concentration, corrosion rate is decrease with the increase in concentration of Jatropha inhibitor. The concentration of 40-45% by volume of Jatropha oil will give the full protection to the metal surface. The effectiveness of Jatropha oil as inhibitor is due to the inductive effect of the methyl groups. Adsorbed organic molecules prevent steel corrosion by blocking the active sites on the metal surfaces and the inhibition efficiency (E%) of an inhibitor depends strongly on the chemical structure or the functional groups on the inhibitor molecule.

From laboratory study, rate of corrosion uniformly decreases with the increase of temperature. This is attributed to the formation of more protective metal due to a decrease in the iron carbonate solubility and also to the competition between the mass transfer and corrosion rates. Besides, Jatropha oil gives the corrosion protection in acid solution with the range of 1.5-6 pH value. As conclusion, Jatropha oil is a good organic corrosion inhibitor for acidic and high temperature system.

5.2   Recommendation

In order to get better results in the future, here are some recommendations that can be suggested.

- It is recommended to use the mild carbon steel that square or rectangle in shape. It is better to scrub the steel coupon with the sand paper first before degrease their surfaces with the acetone.
- It is recommended to extend the temperature range above 140°C (280°F) in order to facilitate further effect of high temperature to the effectiveness of Jatropha as an inhibitor.
- It is suggested to blend Jatropha oil with drilling mud fluid in order to test the corrosion protection in that system.
- For further evaluation, potential stat is the equipment that can be used to measure the corrosion rate.

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