

SWEET CORROSION INHIBITION OF CARBON STEEL USING A BLEND OF PALM-JATROPHA LEAF EXTRACT IN 3.5% NaCl SOLUTION

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MASTER of Materials Science and Engineering
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Abuja, Nigeria
December, 2017

CERTIFICATION

This is to certify that the thesis titled **“SWEET CORROSION INHIBITION OF CARBON STEEL USING A BLEND OF PALM-JATROPHA LEAF EXTRACT IN 3.5% NACL SOLUTION”** submitted to the school of postgraduate studies, African University of Science and Technology (AUST), Abuja, Nigeria for the award of the Master's degree is a record of original research carried out by Chinwego Chinenye Annexcel in the Department of Materials Science and Engineering.

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JATROPHA LEAF EXTRACT IN 3.5% NaCl SOLUTION**

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ABSTRACT

This research focused on studying the suitability of a blend of plant extracts for sweet corrosion inhibition on carbon steel in 3.5% NaCl solution. A comparative study was carried out to investigate the efficacy of sweet corrosion inhibition using palm, jatropa and palm-jatropa extracts at concentrations of 100 ppm, 200 ppm, and 400 ppm to show the effect of blending on the inhibitive performance of plant extracts. An analysis of the extracts was carried out and weight losses as well as electrochemical measurements of the steel samples were obtained. The extracts were characterised using FTIR spectroscopy, indicating the presence of phenols, amines, esters and alkaloids, which are active ingredients for corrosion inhibition. The spectrum of the palm-jatropa blend indicated a strong presence of phenols, alkaloids, imines and oximes. The best inhibition efficiency was obtained at PJE 200 (60.9%). The weight loss and electrochemical studies showed that palm extract is suspected to be an anodic inhibitor and a blend with jatropa extract would give better inhibition efficiency at a critical concentration.

Keywords: corrosion inhibition, carbon steel, electrochemical studies, inhibition efficiency, CO₂ corrosion

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LIST OF ABBREVIATION

FTIR – Fourier-Transform Infrared Spectroscopy

IE – Inhibition Efficiency

JE – Jatropha Extract

PE – Palm Extract

PJE – Palm-Jatropha Extract

MPY - Millimeter per Year

XPS – X-ray Photoelectron Spectroscopy

CHAPTER 1:INTRODUCTION

1.1 Background

Corrosion is a phenomenon that confirms the second law of thermodynamics, namely, the universal law of entropy that describes the degree of disorderliness or uncertainty in a system. Flakes of rust observed on a bar of iron as well as green patina observed on a bronze structure provide evidence of the effects of corrosion. This phenomenon can be explained in various ways but, in general, it is considered to be nature's way of reversing an unnatural process back to a lower energy state by the deterioration of the metal or its properties as a result of its exposure to different aggressive environments (Asrar et al., 2016). It can be either general or localised. The corrosive environment may be acidic, basic or neutral in some circumstances (Suah & Soboyejo, 2014). In the oil and gas industry, the pipelines, bolts and other structural materials are made of metals which were once stable iron carbonates or iron sulphide ores but, after refining, become unstable with a tremendous quantity of energy. Thus corrosion, by releasing the high energy, would tend to return the metal to a stable state (Gbarnjah & Soboyejo, 2014).

One of the most important impacts of corrosion is safety. Safety should be uppermost in the minds of industrial personnel because accidents do occur, in spite of taking all necessary precautions. So, not only is corrosion expensive but it also poses numerous risks to human life and safety. Corrosion can also have an impact on the environment. Corrosion-related failure of oil or gas pipelines or oil tanks can have severe detrimental effects on the environment in the form of water and air pollution, leading to the demise of aquatic life (Sastri, 2011). Corrosion costs affect capital and operational expenditure as well as health, safety and the environment. According to the National Association of Corrosion Engineers (NACE), in a study carried out on corrosion costs and preventive strategies in the United States, results show that the total annual estimated direct cost of corrosion in the U.S. is a staggering \$276 billion—approximately 3.1% of the nation's gross domestic product (GDP). This reveals that, although corrosion management has improved over the past several decades, more and better ways to encourage, support and implement optimal corrosion control practices must be found (International, 2002).

In addition, a report by Corrosion Control Solutions on a number of countries including the United Kingdom, Sweden, Germany and Finland showed that the annual cost of corrosion was as high as 5% of GNP (CORR-EX, 2017). Furthermore, Nigeria, which is a major user of ferrous and non-ferrous metal, is also experiencing its own share of corrosion problems in different industrial sectors, one of which is the oil and gas industry. Research work conducted on the causes of oil pipeline failures in oil and gas industries in the Niger delta area of Nigeria for the period between 1999 and 2010 showed that the major causes of failure included: ageing, corrosion, mechanical failures, welding defects, pressure surge problems, stress and wall thickness. From the results obtained, recommendations were given on measures to minimize these failures. According to the Gas and Oil Pipeline Standards (GOST) of Nigeria, the standard lifetime of a pipeline is 33 years but these research findings revealed that 42% of failures were mechanically induced, 18% were caused by corrosion, third party activity contributed 24%, 10% resulted from operational error and 6% were caused by natural hazards(Achebe, Nneke & Anisiji, 2012).

According to (Kermani, Harrop & Intl, 1996), sweet corrosion causes about 70% of oilfield failures and this has posed a serious problem in the oil and gas industry for about 60 years. This can be attributed to the poor resistance of carbon and low alloy steels to such an attack, causing both general and localised corrosion. Carbon steel is the most widely used material in the oil and gas industry because of its low cost and attractive combination of mechanical properties. Due to its susceptibility to corrosion, it is usually coated and chemicals are applied to mitigate/reduce corrosion (Aribo, Sanumi, Ogunbadejo & Ige, 2016). As environmental concerns are extremely important in the field of engineering, the use of eco-friendly inhibitors has been on the rise. The toxic effect that most synthetic corrosion inhibitors have is the motivation behind the use of natural products, such as plant extracts, as corrosion inhibitors. They are environmentally acceptable, inexpensive, readily available and renewable (Olawale, Oyawale, Adediran & Obafemi, 2016).

1.2 Problem Statement

The deterioration of metals in the presence of water is an industrial challenge especially in the oil and gas industry where water is a by-product of production thus, making corrosion a pervasive issue. Industrial corrosion can lead to process breakdowns that culminate in explosions or the release of chemicals dangerous to health.

Gases, such as carbon dioxide and hydrogen sulphide, aggravate the problem and must be considered when proffering solutions. The various factors that contribute towards the risk of corrosion include, but are not limited to, the type of reservoir, acids used in stimulation, oil well equipment, like the tubing and casing, as well as the operating conditions of the equipment (Rajeev, Surendranathan & Murthy, 2012).

The high level of toxicity that most synthetic inhibitors possess is the reason for the ongoing research work on natural inhibitors for corrosion control. The inorganic methods contain toxic phosphate; chromate and arsenic compounds that pose a high level of environmental risk, especially to human health, such as lung cancer, which is known to have arisen as a result of long-term exposure to inorganic inhibitors such as chromates and phosphates. Hence, laws have been imposed to reduce their use as corrosion mitigation substances.

Although various industries have sought out means to prevent or reduce corrosion by using organic coatings and plastic linings, it has been discovered that the former undergo delamination as a result of their thinness (usually in millimetres). In addition, the significant loss of mechanical toughness can interfere with the process for which the equipment was designed. This is because delamination might change the heat transfer properties, and any changes to the structural detail can lead to premature failure in the equipment.

1.3 Aim and Objectives

This work is aimed at understanding the synergistic effect of palm-jatropha extracts for corrosion inhibition of X65 steel in 3.5% NaCl. The specific objectives were to:

- i. formulate inhibitors from extracts of palm leaves, jatropha leaves and a blend of both,
- ii. determine the phyto-chemical properties of the plant extracts,
- iii. test the corrosion of the carbon steel treated with various concentrations of inhibitors, by using a weight-loss technique,
- iv. compare the inhibition properties of extracts from palm leaves, jatropha leaves and a blend of both and,
- v. determine the effect of the increase in the concentration of the plant extracts on their inhibitive efficiency.

1.4 Significance of the Study

- I. Due to the increasing ecological awareness, as well as strict environmental regulations limiting the use of toxic substances as corrosion inhibitors, the attention of researchers is currently focused on developing green alternatives when mitigating corrosion (Adesanmi, 2015).
- II. Most of the synthetic inhibitors are not only toxic but also expensive. Thus, it is of utmost importance to source inhibitors that are cheap as well as being environmentally friendly.
- III. As a result of the availability of plants and the simplicity of processing them into inhibitors, they are less expensive than inhibitors produced using synthetic chemicals (Ameh, Magaji & Salihu, 2012).
- IV. Major industries such as food, petroleum and power production, as well as chemical and electrochemical industries use carbon steel because of its versatile mechanical properties and low cost. The major problem with carbon steel is its dissolution in an aggressive medium. Corrosion of iron and steel in seawater in the presence of carbon dioxide gas is one of the major areas of concern in oil and gas industries. Carbon dioxide is a corrosive compound found in natural gas, crude oil, and in condensate and produced water. It is one of the most common environments in the oil field industry (Oilfield Wiki, 2016).

1.5 Justification of Research

The increasing ecological awareness as well as strict environmental regulations and policies, resulting in the need to develop environmentally friendly processes, has initiated the development of green alternatives, into which category oil palm and jatropha leaves fall, to mitigate corrosion. Jatropha trees are found predominantly in the northern parts of Nigeria while oil palm trees are more abundant in the southern parts of the country. The fruit of the oil palm tree is a food commodity while those of jatropha can be used for biofuel.

However, the leaves of both are of little or no use to those that cultivate them. It therefore makes sense to utilize the leaves in the production of corrosion inhibitors, in place of the harmful chemicals currently being used (Okafor, Ebenso, El-Etre & Quraishi, 2012).

1.6 Scope of Study

This study is limited to experimental work performed in the multifunctional laboratory of the African University of Science and Technology, Abuja which focussed on the corrosion inhibition of palm-jatropha extract on X65 carbon steel in a deaerated 3.5% NaCl solution, using weight-loss and electrochemical studies.

CHAPTER 2: LITERATURE REVIEW

2.1 Introduction

This chapter provides a review of the literature and data that exist concerning corrosion and corrosion control, the various approaches taken to mitigate corrosion and how they have been applied in the oil and gas sector. Accordingly, a discussion of the meaning of corrosion, the necessary factors for it to occur, the various types of corrosion and the environments that aggravate it, will be presented. This will be followed by a description of the thermodynamics and kinetics of corrosion processes, including adsorption isotherms. This chapter will also discuss various corrosion mitigation techniques including coatings, cathodic protection and inhibitors, with a focus on green inhibitors, as well as corrosion testing and inspection methods. Finally, a critical analysis of the benefits and challenges of implementing this novel inhibitor in the oil and gas industry will be discussed.

2.2 Overview on Corrosion

Corrosion is the deterioration of a substance or its properties as a result of an undesirable reaction with the environment. It is irreversible, degenerative and related to the second law of thermodynamics, that is, “energy spontaneously tends to flow only from being concentrated in one place to becoming diffused or dispersed and spread out.”(Bell, n.d.)

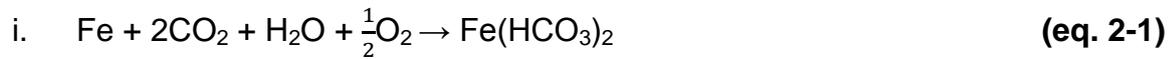
The process can result in a loss of function of the component or system. In addition, according to IUPAC, “corrosion is an irreversible interfacial reaction of a material (metal, ceramic, and polymer) with its environment which results in its consumption or dissolution into the material of a component of the environment” (IUPAC, 2012). Often corrosion results in effects detrimental to the usage of the material. It should be noted that physical or mechanical processes such as melting and evaporation, abrasion or mechanical fracture are not included in the term corrosion (Nataraja, 2017).

2.2.1 Theories of Corrosion

Generally, there are three theories of corrosion. They are:

Acid Theory: This theory suggests that a metal corrodes because of the acidic environment to which it is exposed.

Thus, corrosion is by atmospheric carbon dioxide, moisture and oxygen. Fe(OH)_3 , $\text{Fe(HCO}_3)_2$ and Fe(OH)CO_3 are usually the products of this corrosion as shown in the equations below:



Dry or Chemical (Direct Combination) Corrosion: This type of corrosion results from the direct chemical action of atmospheric gases such as O_2 , halogens, H_2S , SO_2 , N_2 or anhydrous inorganic liquids on the surface of a metal that is in contact with them. The resulting effect of corrosion is more uniform than localised corrosion. The types of chemical corrosion include:

a) Corrosion by oxygen

The degradation of metals due to the presence of a high oxygen concentration occurs as a result of direct reaction of metals with oxygen in the absence of moisture. Alkali and alkaline earth metals react with oxygen at room temperature and form corresponding oxides, while some other metals react at elevated temperatures. Metals such as Ag, Au and Pt are not oxidised because they are noble metals. Oxidation occurs at the surface of the metal and forms an oxide film, which tends to act as a barrier against further oxidation. The oxide film formed can be stable, unstable, volatile or porous and this determines if corrosion will continue or will be halted.

b) Corrosion by other gases, for example, H_2S , SO_2 , N_2 and CO_2

These gases react with metals in a dry atmosphere to form a corrosion product, which may be protective or non-protective. For example, in petroleum industries, H_2S attacks steel, forming FeS scale, which is porous and interferes with normal operations.

c) Wet or Electrochemical Corrosion: This is a common form of corrosion that occurs when the metal comes into contact with a conducting liquid or when two dissimilar metals are immersed partly or fully in a solution. A galvanic cell is formed on the surface of the metal because some parts act as the anode and the others as the cathode.

The anode is the metal that corrodes and undergoes oxidation thereby losing electrons while the cathode is any other conducting material that undergoes reduction and therefore gains electrons. The chemical in the environment, together with the humidity, acts as an electrolyte thus enabling the migration of ions between the cathode and the anode. Metals exposed to water are prone to corrosion if they are unprotected but this can be delayed or even prevented by natural or artificially contrived conditions in the environment.

For electrochemical corrosion to occur, four elements must be present:

- i. **Anode or Anodic Area:** This is the site where corrosion would take place. It is the electrode of an electrochemical cell that oxidises. Thus, electrons leave the surface of the anode into the electrolyte while the metal becomes ionised. The chemical equation (using steel as an example) for this is:



- ii. **Cathode or Cathodic Area:** This is a different conductive material with fewer tendencies to corrode. It is the part where reduction takes place. Thus, electrons that have left the anode flow towards the cathode. The chemical equation for this is:



It should be noted that this oxygen-reduction reaction, otherwise known as redox reaction, is for an aerated medium, that is, where oxygen exists as one of the corrosion reactants.

- iii. **Electrolyte:** This is usually a conductive liquid referred to as the chemical environment. It contains ions that migrate in an electric field.
- iv. **Electrical Contact between the Anode and Cathode:** This is necessary for the transfer of electrons from the anode to the cathode. It could be a metal-to-metal contact or a fastener, depending on the electrochemical corrosion process. For example, bulk steel can serve as a conductive metallic pathway that links the cathode to the anode while a salt bridge is used when considering a galvanic cell (Romer, Bell, Duranceau, Foreman & AWWA Research Foundation, 2004).

The absence of any one of these would stop corrosion. For example, coatings on the surface of a metal and the formation of an invisible oxide film (passive film) on metals, like stainless steel and titanium, act to prevent corrosion. The corrosion process involves two chemical changes: oxidation and reduction. The metal that is attacked undergoes an anodic change and is referred to as the anode while the corrosive agent, being reduced, undergoes a cathodic change. It should however be noted that, for corrosion to occur, there is no need for discrete (physically independent) anodes and cathodes. Innumerable micro-level anodic and cathodic areas can be generated at the same (single) surface on which anodic (corrosion) and cathodic (reduction) reactions occur (Nataraja, 2017).

Types of Corrosion

There are various types of corrosion that occur in nature. They can be categorised based on the environment in which they occur, for example, metallurgically influenced, mechanically assisted and environmentally induced cracking and the mode of occurrence, for example, general or localised. This is summarised in Table 2.1 below:

Table 2.1: Classification of corrosion types

General	Localised	Metallurgical Influenced	Mechanically Assisted	Environmentally Induced Cracking
Atmospheric	Crevice	Intergranular	Erosion-Corrosion	Stress Corrosion Cracking (SCC)
Galvanic Stray Current	Fillform Pitting	Dealloying	Fretting Corrosion Fatigue	Hydrogen Damage Liquid Metal Embrittlement
General Biological Corrosion Molten Salt High Temperature	Localised Biological Corrosion		Cavitation and Water Deep Impingement	Solid Metal Induced Embrittlement

2.2.2 Corrosion in the Oil and Gas Industry

The oil and gas industry is one of the industries where corrosion raises major economic and safety concerns as a result of the inherent corrosiveness of the impurities in crude oil and natural gas. Corrosion problems occur within this industry in at least three general areas: production, transport/storage and refining.

It has been established by corrosion experts that carbon dioxide (CO₂), hydrogen sulphide (H₂S) and free water are the highly corrosive media in the oil/gas wells and pipelines (Paul, Pattanayak & Guchhait, 2014). In addition, studies show that, in the petroleum industry, sweet or sour crude (containing CO₂ and H₂S respectively) is being used in operating units which handle both saturated and unsaturated hydrocarbons at elevated or low temperatures and pressures. The environment of these operations could either be sea or ordinary water systems (Schweitzer, 2013).

Understanding corrosion within the oil and gas industry necessitates a quick review of the construction materials typically employed. Steels are the predominant materials used for constructing onshore and offshore structures, including desalination plants, which are subjected to general or localized corrosion of varying degree (Malik, Ahmad, Andijani & Al-Fouzan, 1999). Carbon steel is an alloy of iron with up to 2 percent carbon that increases the strength of the material and its corrosion resistance. The steel may also contain trace quantities of other metals such as nickel or chromium. Carbon and low-alloy steels are primarily affected by general or uniform corrosion. In nature, iron occurs as various forms of oxides, which are then refined as ores to produce steel. Therefore, they have a tendency to return to their oxidized state. Variables, such as water (a common solvent for salt and acid solutions), corrosive gases, flow rate, temperature, as well as some organic systems, determine the rate of corrosion (Schweitzer, 2007). Most of the equipment used in the upstream sector of the oil and gas industry is made of steel and they handle strong acids and bases, thus requiring special corrosion protection. Corrosive vapour and acid condensates containing carbon dioxide, sulphur dioxide and hydrogen sulphide are usually encountered in this sector. Thus, methods of treatment, as suggested by Schweitzer (2013), would include inhibitors, appropriate materials and finally, the use of economics as a criterion in making the final decision.

In the petroleum industries, corrosion may be divided into two types: wet corrosion and dry corrosion. Wet corrosion occurs at low temperatures, that is, below the dew or boiling point of water. Thus, corrosion occurs because of the presence of an aqueous phase. Meanwhile, at higher temperatures (above the boiling point of water), corrosion occurs in the absence of an aqueous phase and is termed dry corrosion.

Temperature, pressure and composition of the aqueous, gaseous and oil phases influence wet corrosion and although the amount of water in refineries and petrochemical plants is usually small, the corrosiveness is high and localised where water is in contact with the metal. If dissolved hydrogen sulphide (H₂S), carbon dioxide (CO₂) and chloride ions (Cl⁻) are contained in the water, corrosion may occur even for produced water as low as 0.1% or may not begin until after several years of production.

Besides refineries in the oil and gas industry, pipelines operating between the oil and gas wells and processing plants have corrosion problems similar to those in the refineries. Flow rate is one of the factors that influences corrosion rate. Low flow rates may lead to pitting corrosion whereas, at high flow rates, flow-induced corrosion and erosion may occur (Papavinasam, 2011). Corrosion rates of several metals and alloys induced by slowly flowing seawater are shown in Table 2.2 below:

Table 2.2: Corrosion rate in mm/year for metals and alloys in stagnant or slowly flowing seawater

Materials	Corrosion rate
Haste alloy	≈0
Titanium	≈0
AISI 316 Stainless Steel	Insignificant, except for deep pits
AISI 304 Stainless Steel	Insignificant, except for deep pits
Nickel-Chromium alloys	Insignificant, except for deep pits
Aluminium	0.001-0.005, except for pits
Nickel-Copper alloys	Usually <0.025, except for pits
Nickel	Usually <25, except for deep pits
70-30 Copper-Nickel 0.5 Fe	0.003-0.012
90-10	0.003-0.012
Copper	0.013-0.090
Admiralty Brass	0.013-0.050
Aluminium Brass	0.013-0.030
Bronze	0.025-0.050
Nickel-aluminium Bronze	0.025-0.050
Nickel-aluminium-manganese Bronze	0.025-0.050
Manganese	0.025-0.075

Zinc	0.020-0.085
Austenitic nickel-alloyed cast iron	0.050-0.070
Carbon Steel	0.100-0.160

2.2.3 Sweet Corrosion

Corrosion that occurs in oilfields exists in different forms, of which sweet (CO₂) corrosion and sour (H₂S) corrosion in the produced fluids, and corrosion by oxygen dissolved in water injection, are by far the most prevalent forms of attack found in oil and gas production (Villamizar, Casales, Gonzalez-Rodriguez & Martinez, 2007). It has been established that the majority of the oil and gas pipeline failures are as a result of sweet corrosion of carbon and low alloy steels (López, Schreiner, De Sánchez & Simison, 2003).

Sweet corrosion was reported as early as 1940. It occurs generally in oxygen-free environments and varies according to the precise environmental conditions. Steel pipes in contact with production fluids are prone to this kind of damage, which occurs as a result of the dissolution of CO₂ in the source water forming carbonic acid. In production tubing and pipe systems there is usually CO₂ in the gas phase and this contact with carbon dioxide causes corrosion. In addition, high rates of water condensation in wet gas pipelines leads to corrosion. The water phase dissolves CO₂, leading to the occurrence of cathodic reactions. The water phase also contains considerable amounts of dissolved salts, and thus its conductivity is high (Fontana, 1987). The reactions involved in sweet corrosion of carbon steel in typical anaerobic aqueous solution are given below (Abd El-Lateef, Abbasov, Aliyeva & Ismayilov, 2012):



FeCO_3 and $\text{Fe}(\text{HCO}_3)_2$ are the corrosion products that build up over time during the process of sweet corrosion, partially passivating the corroding steel surface because of their limited solubility thereby, altering the chemical nature of the steel surface and ultimately affecting the corrosion rate of the carbon steel as well as the corrosion control practice involved. Thus, it is of utmost importance to understand precisely the microstructure and the characteristics of the surface film resulting from sweet corrosion in order to achieve better protection of the steel.

Sweet corrosion occurs at all stages of the production process from downhole to surface equipment and processing facilities. Factors such as temperature, partial pressure of CO_2 and pH influence the mechanism of sweet corrosion (Kermani & Morshed, 2003). Although significant progress has been achieved in understanding the mechanism of CO_2 corrosion in the oil and gas industry, an understanding of its inhibition mechanism and the kinetics of the inhibition process necessary to quantify formulations that will provide a desirable level of protection, remains incomplete (Abd El-Lateef et al., 2012).

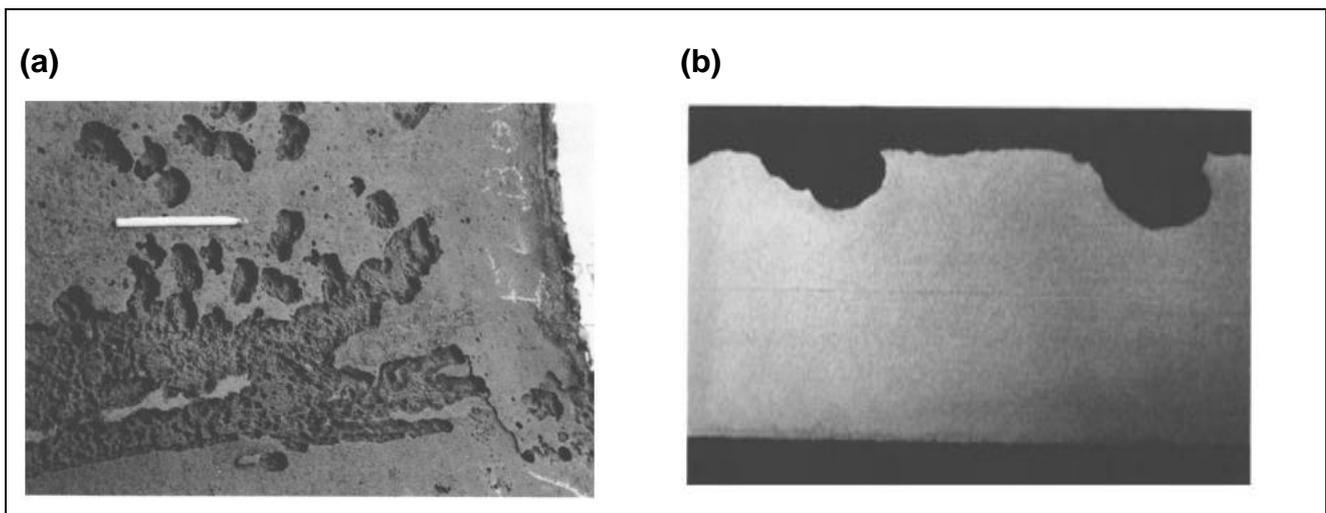


Figure 2.1: Flow-affected corrosion in a flow line for oil and gas in a field in the North Sea (a) A view of the pipe bottom (b) Cross-section of typical pits (2.5x) (Photo: J.M. Drugli, SINTEF Corrosion Centre)

Studies have shown that sweet corrosion rates in the presence of crude oil seen in the field are much lower than those obtained under laboratory conditions are. This is as a result of the inhibitive effect that the crude oil has on the steel.

This can either be due to the wettability effect, where crude oil entrains the water and prevents it from wetting the steel surface, either continuously or intermittently, or by inhibition due to certain components in the crude oil which reach the steel surface either by direct contact or by first partitioning into the water phase.

Furthermore, corrosion inhibitors are added to the produced fluids in order to reduce corrosion failures when the environment becomes highly aggressive or the scales formed on the steel are non-protective. Most of these inhibitors are organic compounds, containing nitrogen or sulphur functionalities. They are surface-active agents that preferentially adsorb onto any surface or interface of a system and alter the surface and interfacial energies, even at low concentrations. This surface-active property comes from their amphipathic, lipid-like, molecular structure, which contains a polar head group, having strong attraction to water, referred to as a hydrophilic head and a non-polar hydrocarbon chain (hydrophobic tail).

2.3 Various Mitigation/Prevention Methods

Various methods have been put forward as being effective in controlling the corrosion of materials. They include, amongst others:

2.3.1 Material Selection

In selecting materials to control corrosion, standard corrosion references are important. In addition, cost plays an important role. As it is not always economically feasible to use the material that provides the optimum corrosion resistance, either another alloy and/or some other measure must be used (Callister & Wiley, 2007). This process takes into account all statutory and regulatory requirements: the project design criteria, such as design lifetime, inspection and maintenance philosophy; type and frequency of interventions; and safety and environmental profiles. Materials for tubing and downhole equipment exposed to produced or injected fluids are selected based on their corrosiveness and the operational conditions throughout the lifetime of the wells. Carbon steel, in combination with corrosion control measures, is often used as the basis for the material selection for most injection wells. Requirements for corrosion monitoring on topside facilities are defined for the fluid to be injected.

2.3.2 Coatings

Paints and coatings on metals are both decorative and protective. They are made from naturally occurring compounds, synthetic materials or a mixture of both. The mechanism of protection for organic coatings occurs by forming a barrier action from the layer or from active corrosion inhibition provided by pigments in the coating (Schweitzer, 2013).

2.3.3 Cathodic Protection

This involves the application of an external current to reduce the corrosion rate to practically zero so that, despite the corrosive environment, the metal can remain indefinitely with a very low corrosion rate. Metals such as steel, copper, brass, lead and aluminium are protected using this method when they are soils and almost all aqueous media (Schweitzer, 2013). The process employs a galvanic couple whereby a more reactive metal is connected to the metal that needs to be protected, resulting in the former undergoing oxidation. Upon oxidation, it gives up electrons and protects the metal. The oxidised metal is often referred to as the sacrificial anode (Callister & Wiley, 2007). For cases where the electrolyte resistivity is high and where the structures are large, impressed current cathodic protection is used. This does not involve the use of galvanic anodes, as they cannot economically deliver enough current to provide protection. Instead, the anodes are connected to a DC power source which works by delivering controlled amounts of DC current to the surfaces submerged in water with the aid of ultra-reliable zinc electrodes and combined anodes of metal oxide.

2.3.4 Corrosion Inhibition

Studies have shown that internal corrosion of product pipelines in the oil and gas industry can be controlled by coatings and inhibitors (a few parts per million). These inhibitors are substances which, when added in small concentrations to an environment, reduce the corrosion rate of a metal exposed to that environment. Inhibitors often play an important role in the oil extraction and processing industries where they have always been considered to be the first line of defence against corrosion. The specific inhibitor would depend on both the alloy and the corrosive environment (Callister & Wiley, 2007). In practice, most inhibitors are developed by empirical experimentation and are proprietary in nature.

Therefore, their composition is not disclosed, making it difficult to understand inhibition completely. But it is possible to classify inhibitors according to their mechanism and composition (Fontana, 1987).

2.3.4.1 Classification of Inhibitors

The selection of inhibitors is based on the metal and the environment. Inhibitors can be classified into environmental conditioners and interface inhibitors (as shown in Figure 2.2). Environmental conditioners act by scavenging the aggressive substances in the medium. For example, in near-neutral and alkaline solutions, reduction of oxygen is a common reaction. Thus, in such situations, corrosion can be controlled by decreasing the oxygen content using scavengers such as hydrazine.

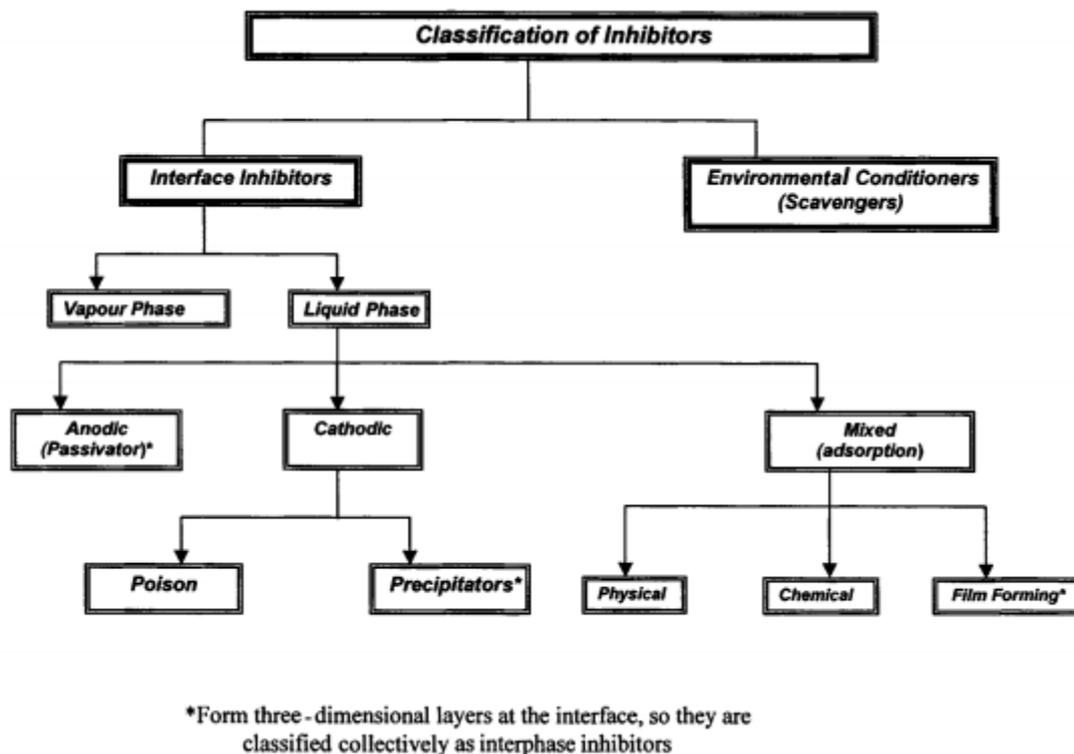


Figure 2.2: Classification of inhibitors
(Papavinasam, 2011)

In contrast, interface inhibitors control corrosion by forming a film at the metal/environment interface and can be classified into liquid and vapour phase inhibitors.

i Anodic Inhibitors: These are usually used in near-neutral solutions where sparingly soluble corrosion products, such as oxides, hydroxides or salts, are formed. They act by forming a protective oxide film on the metal's surface, thus causing a large anodic shift of the corrosion potential. This shift forces the metallic surface into the passivation region. Chromates, nitrates, tungstates and molybdates are some examples of anodic inhibitors. The metal surface to be inhibited is usually oxide free allowing the inhibitor easy access to retard the anodic electrochemical processes of corrosion on the metal surface. Anodic inhibitors cause the formation or maintenance of a passive film on the material surface and this film essentially prevents the anodic dissolution of the metal. The effectiveness of anodic inhibitors depends on the metal.

When the concentration of an anodic inhibitor is not sufficient, corrosion may be accelerated, rather than inhibited. Therefore, it is necessary to know the critical concentration above which inhibitors are effective and this depends on the nature and concentration of aggressive ions (Papavinasam, 2011).

Mechanistic Aspect of Anodic Inhibitors

This can be explained by using the polarization diagram of an active-passive metal as shown in Figure 2.3. In the absence of inhibitors, corrosion would occur (point A). As the concentration is increased, the rate of corrosion also increases to a critical concentration and corrosion rate (point B). At this critical concentration, a rapid transition of the metal to the passive state occurs and thus, corrosion rate is decreased (point C).

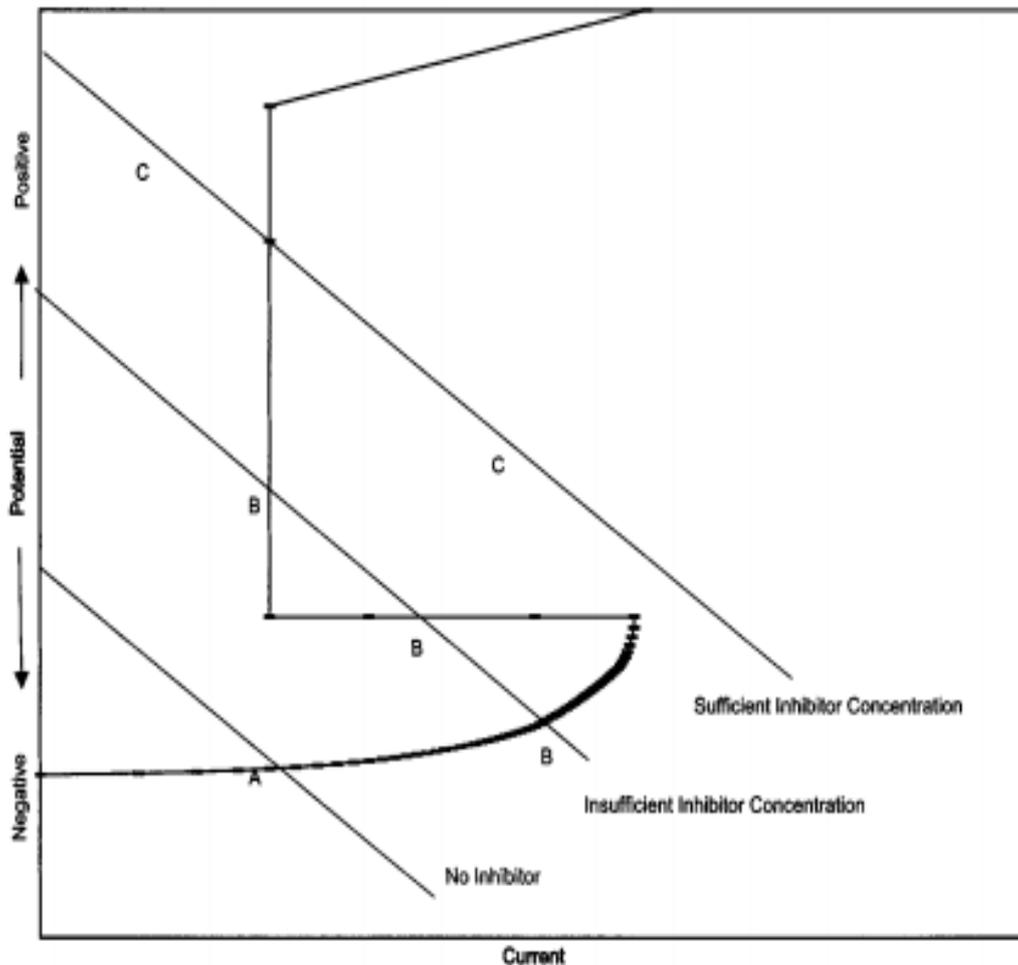


Figure 2.3: Polarization diagram of an active-passive metal showing the dependence of the current on concentration of passivation-type inhibitor (Papavinasam, 2011)

ii Cathodic Inhibitors: These act by either slowing the reaction on the cathode itself (cathodic poisons) or selectively precipitating on cathodic areas in order to limit the diffusion of the reducing species to the surface (cathodic precipitators). The corrosion rates can also be reduced by the use of oxygen scavengers that react with dissolved oxygen. Sulphite and bisulphite ions are examples of oxygen scavengers that can combine with oxygen to form sulphate. The rate of oxygen reduction controls the rate of corrosion in cathodic inhibitors, that is, cathodic inhibitors act by forming thick (compared to passive) films on the metal surface. These films reduce oxygen transport to the surface.

iii Mixed Inhibitors: These work by reducing both the cathodic and anodic reactions by forming precipitates on the surface thus indirectly blocking both anodic and cathodic sites. About 80% of inhibitors are organic compounds that cannot be specifically classified as anodic or cathodic. Thus, they are referred to as mixed inhibitors. Hard water that has a high concentration of calcium and magnesium is less corrosive than soft water because of the tendency of the salts in the hard water to precipitate onto the surface of the metal, thus forming a protective film. The most common inhibitors of this category are the silicates and the phosphates. Sodium silicate, for example, is used to prevent corrosion in many domestic water softeners. In aerated hot water systems, sodium silicate protects steel, copper and brass. However, protection is not always reliable and depends heavily on the pH level. Phosphates also require oxygen for effective inhibition. Silicates and phosphates do not afford the degree of protection provided by chromates and nitrites; however, they are very useful in situations where non-toxic additives are required (Schweitzer, 2007).

Mixed inhibitors function in three ways: physical adsorption, chemisorption and film formation. Physical adsorption, also known as electrostatic adsorption, occurs when the metal is positively charged and adsorption of negatively charged (anionic) inhibitors is facilitated. Although, physically adsorbed inhibitors interact rapidly, they are easily removed from the surface. Increase in temperature facilitates desorption. On the other hand, chemisorption takes place at a slower rate and, as temperature increases, adsorption and inhibition also increase. This process is not completely reversible.

Studies show that the most effective inhibitors are those that are chemically adsorbed. This process involves charge transfer between the inhibitor molecules and the metal surface.

iv Volatile Corrosion Inhibitors: These are compounds transported in a closed environment to corrosion sites by volatilization from a source. For example, morpholine and hydrazine are volatile compounds transported with steam to prevent corrosion in the condenser tubes. They do this by neutralizing acidic carbon dioxide or by shifting surface pH towards less acidic and corrosive values. In closed vapour spaces, such as shipping containers, volatile solids such as salts of dicyclo-hexylamine, cyclohexylamine and hexamethyleneamine are used (Maarten, 2011).

2.3.4.2 Synergism and Antagonism of Inhibitors

It has been shown that the lateral interaction between inhibitor molecules in the presence of two or more adsorbed species can significantly affect the performance of an inhibitor. A synergistic effect arises if the interaction is attractive. This means that the degree of inhibition in the presence of both inhibitors is higher than the sum of the individual effects. For example, a study on synergised plant leaf extracts (*Jatropha curcas* and *Sida acuta*) as substitutes for toxic additives in alkyd resin primer for corrosion inhibition of steel in 1.0 M HCl showed that the extract effectively inhibited corrosion more than the synthetic chemical (Folorunso, Anthony & Abidemi, 2016). The inhibition efficiency of a mixture of formaldehyde and furfurylimine is higher than the inhibition efficiency when these inhibitors are used separately. In some cases, there could be an antagonistic effect which would lead to a decrease in the inhibition efficiency. This was shown when nicotine and thiourea were used as mixed inhibitors and a decrease in the inhibition efficiency was experienced compared to when they were used separately.

2.3.4.3 Green Inhibitors

Green approaches to corrosion mitigation involve using substances, techniques and methodologies that reduce feedstock, products, by-products, solvents, reagents, and so forth that, while combating corrosion, can harm human health or the environment. Such approaches include:

- using plant extracts, bio-chemicals, biodegradable organic substances as well as green inorganic inhibitors;
- developing corrosion inhibitors based on waste products;
- using ion exchange pigments, coatings and corrosion by-products to control corrosion.

The use of green corrosion inhibitors for the protection of metals can be traced to the last half of the nineteenth century where extracts of glue, gelatine and bran were used to inhibit the corrosion of iron in acids. This and subsequent discoveries of effective inhibitors for metals were the result of empirical studies (Sastri, 2011). The increased awareness of environmental and human health has resulted in increased research into corrosion inhibitors made from plant extracts and other non-toxic substances.

Table 2.3: Different plants that have been used as green inhibitors

Plant Extracted	Material Solution	Effect of Temperature on %Inhibition Efficiency	Effect of Concentration on % Inhibition Efficiency	Methods	Adsorption Isotherm	Reference
<i>Pterolobium hexapetalum</i> and <i>Celosia argentea</i>	Industrial water	Decrease	Increase	Weight loss and electrochemical impedance spectroscopy	Langmuir	(Kumar & Mohana, 2014)
<i>Anibaros aeodora</i>	HCl	Done at constant temperature	Increase	Potentiodynamic polarization and electrochemical impedance spectroscopy	Langmuir	(Chevalier et al., 2014)
<i>Neem</i>	Salt water	Done at constant temperature	Increase	Weight loss		(Chigondo & Chigondo, 2016)
<i>Citrus aurantifolia</i>	H ₂ SO ₄	Decrease	Increase	Weight loss	Langmuir and Freundlich	(Caroline, Kobe & Ganiyu, 2015)
<i>Hibiscus sabdariffa</i>	H ₂ SO ₄ and HCl	Done at constant temperature	Increase	Weight loss	Langmuir	(Murthy & Vijayaragavan, 2017)
<i>Elaeis guineensis</i>	HCl	Decrease	Increase	Weight loss, potentiodynamic polarization, and electrochemical impedance spectroscopy	Langmuir	(Hussin, Rahim, Ibrahim & Brosse, 2016)

2.4 *Jatropha curcas* and *Elaeis guineensis* plants

Jatropha curcas, whose common name is Barbados or physic nut, is a tall bush/shrub or small tree that can grow up to 6 meters tall on marginal and poor soil. It can grow in arid, semi-arid and wasteland climates. It belongs to the Euphorbiaceae family and its genus name is derived from the Greek word *jatr'os* (doctor) and *troph'e* (food), which implies medicinal uses. It has a lifespan of around 50 years. The tree is deciduous with leaves falling off under conditions of stress. It can be found predominantly in the wild or semi-cultivated areas in Central and South America, Africa, India and South East Asia.



Figure 2.4: *Jatropha curcas* plant

(“*Jatropha* plants – DESERTIFICATION,” 2017)

J. curcas offers a wide range of benefits, such as, low-cost cultivation; drought resistance and land reclamation. It can be grown almost anywhere, even in sandy, saline or otherwise infertile soil, and can be propagated easily. It is capable of stabilizing sand dunes, acting as a windbreak and combating desertification. It naturally repels both animals and insects because it contains pesticide and fungicidal properties, which make the use of pesticides unnecessary. The tree does not exhaust the nutrients in the soil and does not require expensive crop rotation and fertilizers.

It also grows quickly and establishes itself easily. *Jatropha curcas* has played a major role in the treatment of various diseases, including bacterial and fungal infections (Segura-Campos & Betancur-Ancona, 2017). Different parts of the *J. curcas* plant, including the stem, bark, twigs, leaves and seeds, can be used to treat various diseases. It is also used for the production of aviation fuel (Prasad & Prasad, 2017). Compounds that have been isolated from *J. curcas* leaves include the flavonoid apigenin and its glycosides vitexin and isovitexin. The leaves have also been reported to contain steroid sapogenins and alkaloids (Aliyu, Balogun, Funsho & Arogundade, 2012; Staubmann, Schubert-Zsilavec, Hiermann & Kartnig, 1998). The major reason for the cultivation of *J. curcas* is for the oil from the seeds, which is used to manufacture biodiesel and jet fuel but its use as a corrosion inhibitor is on the rise. Metals such as copper alloys and mild steel have been used to study its corrosion inhibition efficiency in different environments and it has been shown to be a good corrosion inhibitor in the researched environments and temperatures (Aribo et al., 2016).

Elaeis guineensis is a palm species whose common name is the African oil palm. It originated in the tropical rainforest region of West Africa, particularly in Guinea, and can also be found on the eastern coast of Nigeria. The southern coast of Nigeria was originally called the palm oil coast by the first Europeans who arrived there and traded in the commodity. Different parts of this plant are used in African medicine for the treatment of various diseases.



Figure 2.5: Oil palm

Mature palms can grow as tall as 20 m and are single-stemmed. The leaves are pinnated and have a length of approximately 3-5 m. During the harvest of the fruit, the fronds, which consist mainly of cellulose, lignins and hemicelluloses, are discarded. Lignins are considered a valuable source of antioxidant phenolic compounds and are potent free radical scavengers.

2.5 Principles of Corrosion Inhibition

In order to protect metals and alloys from corrosion when exposed to air, most metals have their surfaces covered with oxide films. When such a metal is immersed in an aqueous solution, dissolution of the oxide film occurs.

The extent of dissolution tends to be greater in an acidic solution than a near-neutral solution. If the near-neutral solution contains inhibiting anions, the oxide film is prevented from dissolving and is made stable by passivation which can effectively prevent the corrosion of the metal (Thomas & Gareth, 2017). Transition of the metal/solution interface in acidic solutions is attributed to the adsorption of the inhibitor molecules at the metal/solution interface to form a protective film. The adsorption rate occurs rapidly, thus shielding the metal surface from the aggressive solution (Chao, Lin & Macdonald, 1981). A very important step in inhibition by organic-based film forming in corrosion inhibitors is adsorption, which results from the polar or charged nature of the organic molecule. First, a physisorption surface film is established through Van der Waal's forces and further stabilizes through chemisorption to form a donor type bond (Pearson, 1968).

The use of organic molecules as a corrosion inhibitor is one of the most practical methods for the protection of metals against corrosion and it is becoming increasingly popular. The existing data from various research works show that organic inhibitors act by adsorption and protect the metal by film formation. Organic compounds bearing heteroatoms with high electron density, such as phosphor, sulphur, nitrogen, oxygen or those containing multiple bonds which are considered as adsorption centres, are effective as corrosion inhibitors (Döner, Solmaz, Özcan & Kardaş, 2011). An inhibitor is adsorbed on a metal surface as a result of its chemical structure, molecular size, the nature and charged surface of the metal, and distribution of charge over the whole inhibitor molecule. The adsorption process occurs by replacing solvent molecules from the metal surface with ions and molecules accumulated near the metal/solution interface. The ion accumulation at the metal/solution interface usually occurs in excess of those required to balance the charge on the metal at the operating potential. These ions replace solvent molecules from the metal surface, and their centres reside at the inner Helmholtz plane. This phenomenon is termed specific adsorption, contact adsorption. The anions are adsorbed when the metal surface has an excess positive charge in an amount greater than that required to balance the charge corresponding to the applied potential. The exact nature of the interactions between a metal surface and an aromatic molecule depends on the relative coordinating strength towards the given metal of the particular groups present (Ritchiea, Baileyb & Woodsa, 1999).

In principle, two modes of adsorption can be considered. One in which the neutral molecules of the leaves can be adsorbed on the steel's surface through a chemisorption mechanism. This would involve displacing the water molecules from the steel surface and sharing electrons between the heteroatoms of the inhibitor and the iron. The inhibitor molecules can also adsorb on the steel surface based on donor-acceptor interactions between π -electrons of the aromatic/heterocyclic ring and vacant d-orbitals of surface iron. Since, in principle, the steel surface bears the positive charge in acidic solutions (Mu, Zhao, Liu & Gu, 1996), it becomes difficult for the protonated leaf extract to approach the positively charged mild steel surface (H_3O^+ /metal interface). This results from the electrostatic repulsion. Thus, the chloride ions, having a smaller degree of hydration, would bring excess negative charges in the vicinity of the interface and favour more adsorption of the positively charged inhibitor molecules. The protonated leaf extract would thus be absorbed through electrostatic interactions between the positively charged molecules and the negatively charged metal surface. Therefore, the heteroatoms in the different parts of the plant extract which contain active constituents bring about synergism between the molecules, thus accounting for the good inhibition efficiencies (Singh, Ebenso & Quraishi, 2012).

2.5.1 Inhibition of Sweet Corrosion

CO_2 , which is the major corrosive agent in sweet corrosion, acts in two ways- it increases the amount of hydrogen formed on the cathode and forms the carbonate-oxide films on the surface of the metal. Thus, in order to prevent this type of corrosion, the first approach is to use any available means to make the cathodic process efficiently inhibitive, while the second approach would involve the formation of protective carbonate oxide films or deposits, which can be achieved by pH regulation of the medium and temperature.

Three types of inhibitors can be used to slow down the cathodic process effectively:

- inhibitors that can displace depolarisers (HCO_3^- ions, H_2CO_3 and $\text{CO}_2\cdot\text{H}_2\text{O}$ molecules) from the surface of the metal;
- film-forming inhibitors that are impermeable to depolarisers;
- bond-forming inhibitors with depolarisers.

To an extent, these inhibitors can also prevent the occurrence of the anodic process. For the first case, organic substances, which have close chemical structures to carbonate ions, that is, carboxyl or carbonyl groups will be the most promising reagents. Thus, carbonic acids, ketones, and esters should be effective inhibitors of CO₂ (at optimal dimensions of the molecules and their concentrations in the solution). However, substances with these compounds are almost never used as inhibitors. Most compounds used by the domestic oil and gas industry are organic high-molecular weight compounds, which contain nitrogen: amines, amides, and imidazoles, nitrogen-bearing heterocyclic compounds, quaternary ammonium bases and their salts. All of these compounds efficiently inhibit mainly the anodic process and this is based on their adsorption properties which depend on the nature and state of the metal surface, the type of corrosive environment and the chemical structure of the inhibitor (Abd El-Lateef et al., 2012).

2.5.2 Adsorption Isotherms

The principle of adsorption occurs by chemical (irreversible) and physical (reversible) bonding and the adsorbed material is called the adsorbate while the adsorbing face is the adsorbent. The numbers of molecules of the fluid phase which will adsorb to a given solid surface at equilibrium, depend on several factors including:

- i. pH which increases as adsorption reduces;
- ii. relative concentration of the solute in the solvent;
- iii. adsorption forces;
- iv. degree of ionization;
- v. the available surface area;
- vi. the prevailing temperature (Yawas, 2005).

2.6 Corrosion Tests on Inhibition

Testing to determine the efficacy of inhibitors in mitigating corrosion can be done using electrochemical as well as weight loss methods.

2.6.1 Weight Loss Method

Weight loss is one of the most effective and simplest methods for monitoring corrosion. It is a quantitative method used in measuring and monitoring the internal and external corrosion in metallic structures.

In order to measure the weight loss, metal coupons are connected to the holder and immersed in the electrolyte or corrosive medium. The principle is to measure the weight loss of the coupons after being exposed to a specific environment. In addition, the weight loss method can record several types of corrosion by probing the finite details of the corrosion events. This cannot be done by other methods.

2.6.2 Electrochemical Studies

This involves carrying out electrochemical measurements using a conventional three-electrode cylindrical glass cell and a potentiostat. A platinum or graphite electrode usually serves as the counter electrode. Saturated calomel or silver chloride serves as the reference while the working electrode is the workpiece being tested. This method can be used to measure the open circuit delay, linear polarisation, potentiodynamic polarisation and the electrode impedance spectroscopy.

2.7 Characterization and Microstructural Analysis

Microstructural analysis is carried out on the plant extracts as well as the corroded metal surfaces. The plant extracts are characterised in order to ascertain the functional groups present in them and, by this, the ability to inhibit corrosion can be predicted.

After corrosion has taken place on the steel coupons, they are characterised using various equipment such as the scanning electron microscope, energy dispersive X-ray spectroscopy and X-ray photoelectron spectroscopy. This equipment enables one to view the surface of the corroded samples and to determine the elements present.

2.7.1 Fourier-Transform Infra-Red Spectroscopy (FTIR)

FTIR has proven to be a useful tool for characterizing and identifying compounds or functional groups (chemical bonds) in an unknown mixture of plant extracts (Hazra, Roy, Sen & Laskar, 2007; Sasidharan, Chen, Saravanan, Sundram & Yoga Latha, 2011). It has been shown to be powerful in identifying the types of chemical bonds present in compounds. The wavelength of light absorbed is characteristic of the chemical bond and, by interpreting the infrared absorption spectrum, the chemical bonds in a molecule can be determined (Ashokkumar & Ramaswamy, 2014).

2.7.2 Scanning Electron Microscopy

This produces images of a specimen by scanning it with a focused beam of electrons and data is collected over a selected area of the surface of the sample. A 2-D image which displays spatial variations in properties, including chemical characterisation, texture and orientation of materials, is generated. It is also capable of performing analyses of selected point locations on the sample.

An EDS component is usually applied in conjunction with the SEM analysis to determine the elements in or on the surface of the sample for qualitative information, measure elemental composition for semi-quantitative results and identify foreign substances that are not organic in nature and coatings on metal (Laboratory Testing Inc., 2017).

2.7.3 X-ray Photoelectron Spectroscopy

This measures the elemental composition in parts per thousand, empirical formula, chemical and electronic state that exist within a material. It is a surface-sensitive quantitative spectroscopic technique that analyses the chemical composition of the surface of a material in its original state or after some surface treatment. X-ray photoelectron spectroscopy (XPS) identifies chemical elements from the binding energy spectra of X-ray photoelectrons (Yang, 2008).

CHAPTER 3: MATERIALS AND EXPERIMENTAL METHODOLOGY

3.1 Introduction

In order to study the inhibitive performance of the plant extracts, the samples and the carbon steel samples have to be adequately prepared and exposed to the deaerated CO₂ environment. Experiments were therefore conducted to obtain the extracts and study the corrosion.

3.2 Materials and Equipment Used

The materials used in this research include:

- i. carbon steel from SCC in Abuja State, Nigeria;
- ii. 5 g of jatropha leaf extract;
- iii. 5 g of palm leaf extract;
- iv. analytical grade sodium chloride;
- v. 10 Erlenmeyer flasks and stoppers of 500 ml;
- vi. orifice connectors;
- vii. measuring cylinders of 1000 ml and pipettes of 10 ml for measuring the salt solution and extract respectively;
- viii. 120 grit emery papers;
- ix. ethanol for extraction;
- x. acetone for degreasing metal samples;
- xi. carbon dioxide gas;
- xii. a rotary evaporator for solvent evaporation;
- xiii. cloth for cleaning debris from specimens;
- xiv. distilled water for cleaning specimen and electrolyte preparation;
- xv. soxhlet apparatuses for extraction;
- xvi. a digital weighing balance.

3.3 Experimental Procedures

This section presents the steps taken to: obtain the extracts, simulate a sweet environment and characterize the extracts and the metal samples.

3.3.1 Sample Preparation

3.3.1.1 Preparation of Extracts

Palm fronds were obtained from new Kuchingoro, Games Village, Abuja while the jatropa leaves were obtained from Chaza, Suleja, Niger State. The leaves were washed with water and cut into small pieces, as shown in Figures 3.1 and 3.2. Then they were oven dried for three days at 50 °C (Figure 3.3) and finely ground using a commercial blender (Figure 3.4). The mass of the ground jatropa leaves was obtained.



Figure 3.1: Jatropha leaves



Figure 3.2: Palm fronds



Figure 3.3: Oven drying of the leaves



Figure 3.4: Blending leaves with a commercial blender

Extraction of the active ingredients was done using a 500 ml soxhlet apparatus as shown in Figure 3.5. Ethanol was used as the solvent because of its ability to extract the active ingredients of the plants efficiently. For each extraction, 12 g of the ground leaves was used with 200 ml of ethanol in a flat bottom flask fixed to the end of the apparatus and the condenser, which were tightly fixed at the bottom end of the extractor. An average of 8 cycles was used for the extraction process, which took approximately 30 minutes. A batch process was used; extraction was done simultaneously using six soxhlet extractors. The extractors were heated on heating mantles at 70⁰C. The solvent remained in contact with the powdery leaf material while the extraction was taking place.

After three days, the soxhlet extraction was completed and a rotary evaporator was used to obtain the extract (Figure 3.6).



Figure 3.6: Rotary evaporation



Figure 3.5: Soxhlet extraction

3.3.1.2 Preparation of the Steel Samples

The X65 steel samples used for the experiment were obtained from SCC pipes, Ushafa, Abuja FCT where they were prepared adequately. The steel sample was analysed using Solaris charge-coupled device (CCD) at SCC Steel Company, Abuja, Nigeria with the elemental composition shown in Table 3.1. They were obtained specifically from a section of steel pipes used in oil and gas industries (Figure 3.7). The dimensions of the steel used were 40 mm x 20 mm x 17.4 mm (Figure 3.8).



Figure 3.7: Oil pipeline pools

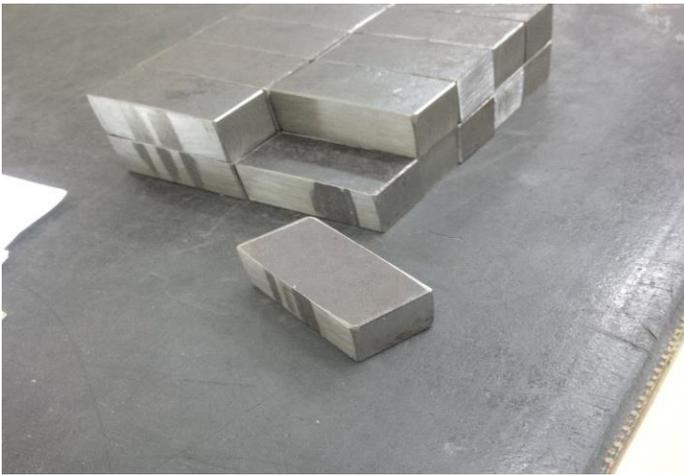


Figure 3.8: Metal coupons sectioned from the pipelines

Prior to the experiment, the prepared steel samples were polished with 120 grit sand paper, degreased with acetone and lastly air-dried.

Table 3.1: Chemical analysis of steel samples

C%	Si%	P%	Mn%	S%	Cr%	Mo%	Ni%	Al%	Cu%	Nb%
0.061	0.137	0.013	1.604	0.003	0.019	<0.002	0.335	0.022	0.267	0.039
B%	Ti%	V%	W%	Mg%	Ca%	Ce%	La%	As%	Pb%	W%
0.0005	0.012	<0.001	<0.005	0.0012	0.0066	<0.002	0.006	<0.005	0.010	<0.005
Ni%	N%	Zn%	Zr%	Co%	Sb%	Fe%				
0.335	<0.003	0.001	0.003	<0.002	0.014	97.444				

3.3.1.3 Preparation of Corrosion Medium

The medium used for the corrosion experiments was 3.5% NaCl which was prepared by dissolving 35 g of analytical grade sodium chloride in one litre of distilled water.

The volume of the electrolyte used for the experiment was 250 ml.

Thus, the volume control was 250 ml of 3.5% NaCl while the volume of the different inhibitors, palm, jatropha and palm-jatropha, was prepared using the neutralization formula at 100 ppm, 200 ppm and 400 ppm: $C_aV_a=C_bV_b$

Ten different corrosion media were obtained for the experiment.

3.3.2 Experimental Set-up for Weight Loss Measurement

250 ml of each medium was poured into ten different Erlenmeyer flasks with stoppers which were connected with orifice connections. The system was deaerated by passing carbon dioxide gas through the flasks at about 16 psi for about 8 hours. The flasks were then sealed and the experiment began.

Weight loss measurements were taken every 24 hours for 6 days using a digital weighing balance.



Figure 3.9: Weight loss experimental set-up



Figure 3.10: Weighing metal samples with a digital weighing balance

3.3.3 Experimental Set-up for Electrochemical Studies

The electrochemical study was carried out using a Gamry Ref 650 Potentiostat in the ADB laboratory of African University of Science and Technology, Abuja. A standard Ag/AgCl cell was used as the reference electrode while graphite and X65 steel coupons served as the counter and working electrode respectively.



Figure 3.11: Electrochemical studies set-up

The experiment was carried out on 10 samples each in varying media – control, 100 ppm, 200 ppm and 400 ppm for PE, JE and PJE in an airtight beaker (150 ml) and CO₂ was pumped in for about an hour before the open circuit potential as well as the linear polarisation resistance were obtained for each experiment. The scan rate was set to 0.125 mV/s, and initial to final electrode potential was -0.01 V to +0.01 V.

3.4 Sample Analysis

3.4.1 Fourier Transform Infrared Spectroscopy

A Fourier transform infrared spectroscopy machine was used to obtain the composition of the plant extracts (Figure 3.12).

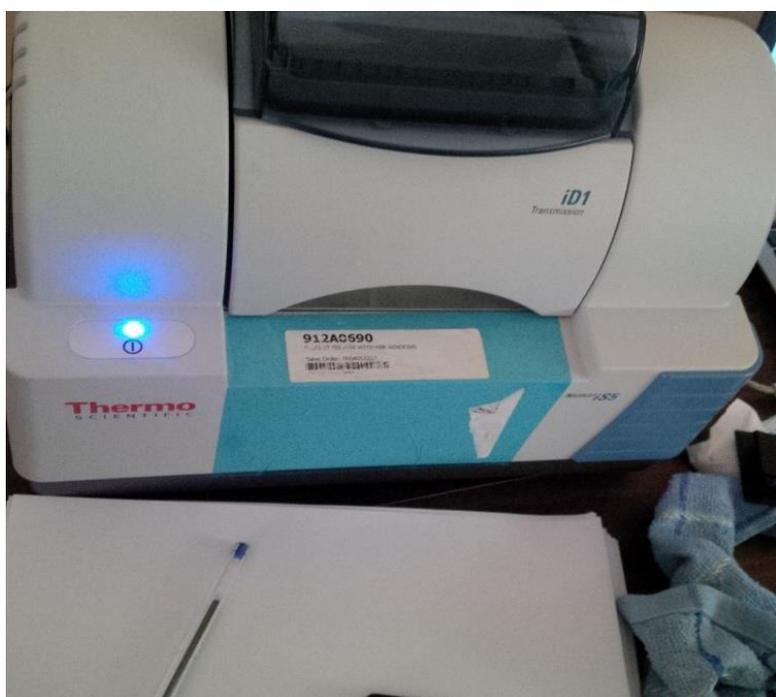


Figure 3.12: FTIR analysis of plant extracts

3.5 Computation of Results

i. Determination of weight loss

The weight loss of the coupon was determined by finding the difference between the initial weight and the final weight after immersion in the solution.

$$W_l = W_i - W_f$$

Where W_l = Weight loss after immersion of coupon

W_i = Initial weight of coupon

W_f = Final weight of coupon after immersion

ii. Determination of surface coverage

The surface coverage of the inhibitor was calculated using the equation;

$$\theta = \frac{W_i - W_f}{W_i}$$

iii. Determination of corrosion rate

From the standard expression for corrosion rate measurement, corrosion rate in mm per year (*mm/yr*) was calculated.

$$MPY = \frac{8.76 \times 10^4 W}{DAT}$$

Where W = weight loss in grams

D = density of metal

A = area in cm^2

T = time in hours

iv. Determination of inhibition efficiency

The inhibition efficiency was computed using the relationship:

$$IE = \frac{R_0 - R}{R_0} \times 100$$

Where R and R_0 are the corrosion rates with and without inhibitor, respectively.

CHAPTER 4: RESULTS AND DISCUSSION

4.1 Characterization of Plant Extracts

The *Jatropha curcas* and *Elaeis guineensis* leaf extracts used for this research was prepared and characterised at the Chemistry Department in Shetsco, Abuja, Nigeria using an iD5 Fourier transform infra-red spectroscopy. Figures 4.1, 4.2 and 4.3 give the FTIR spectrum of the extracts.

4.1.1 Extract Analysis

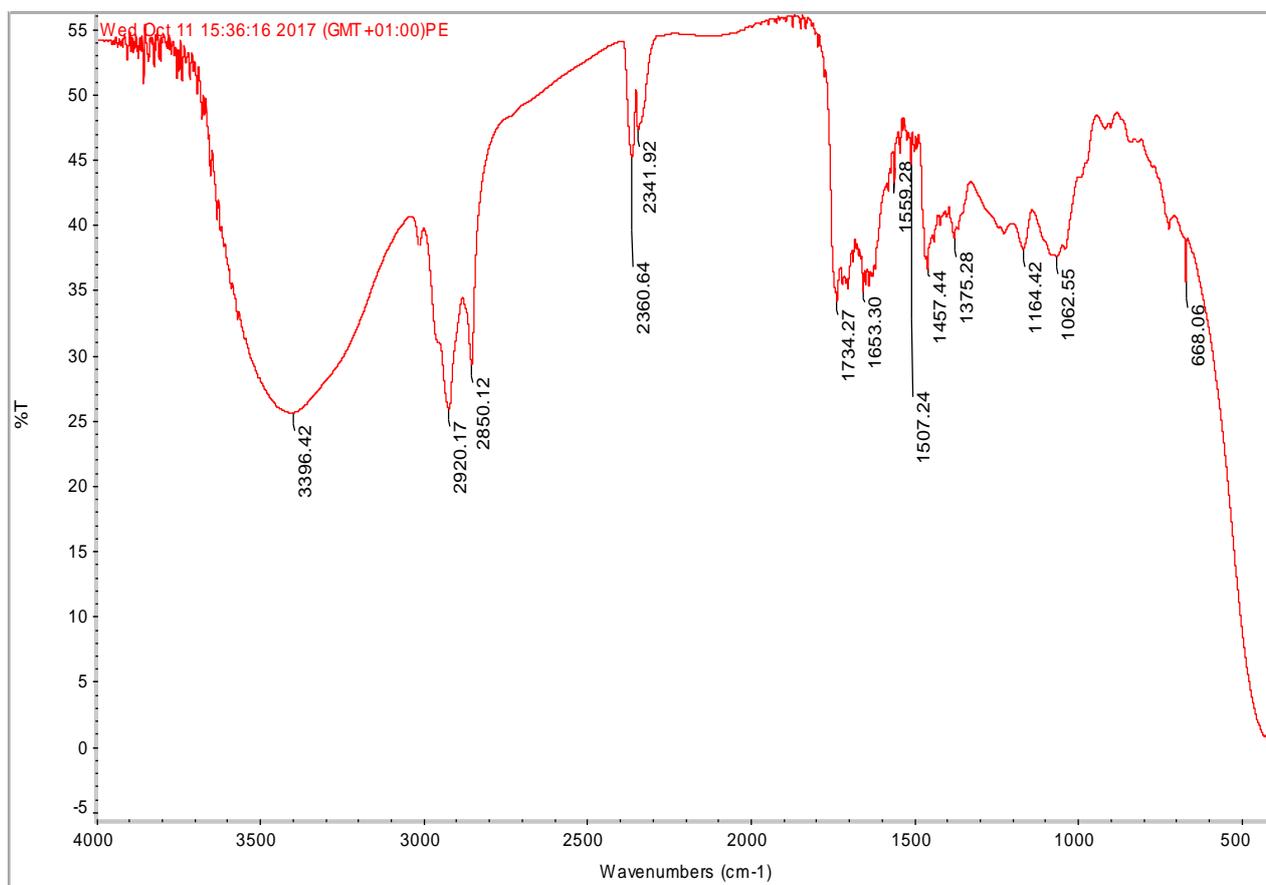


Figure 4.1: FTIR analysis of palm extract

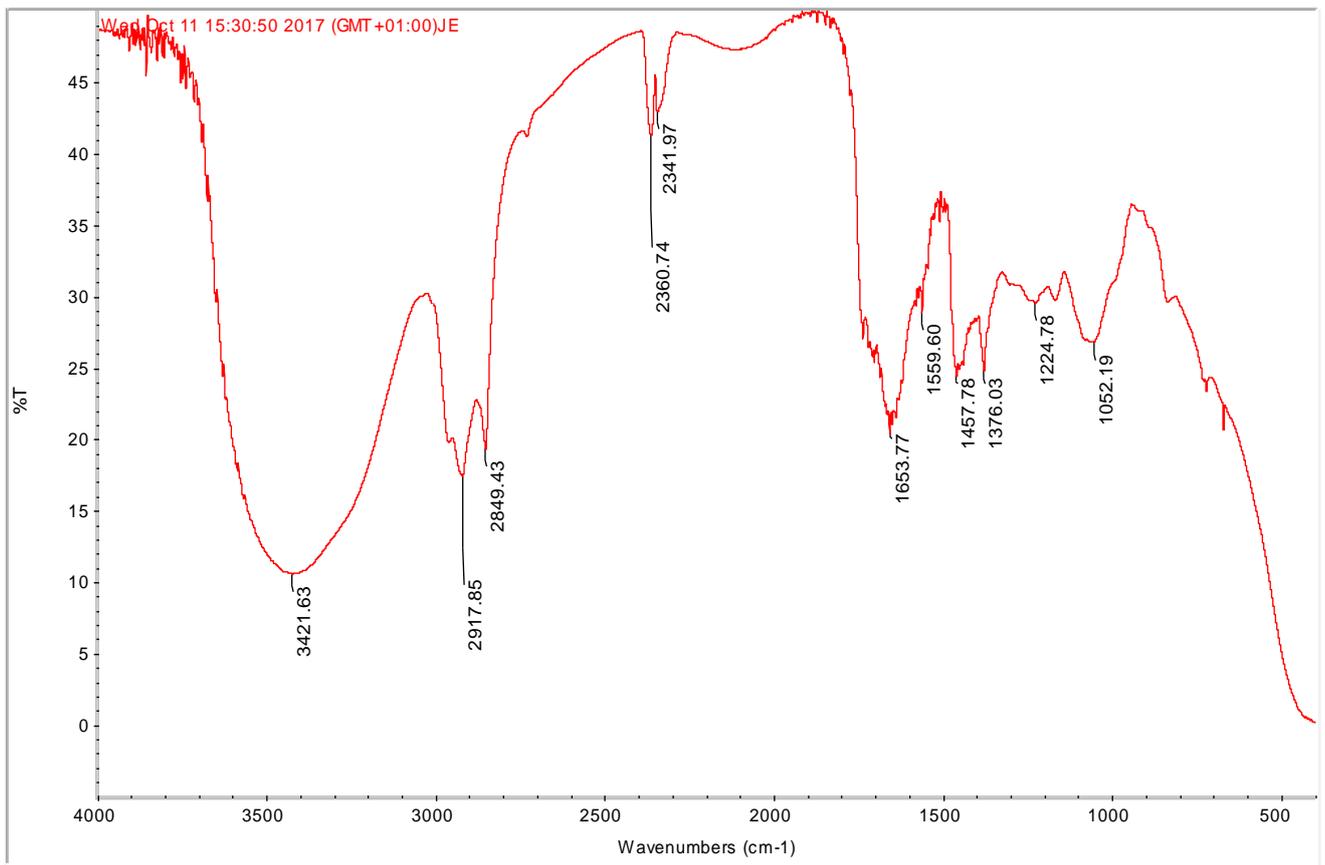


Figure 4.2: FTIR analysis of jatropha extract

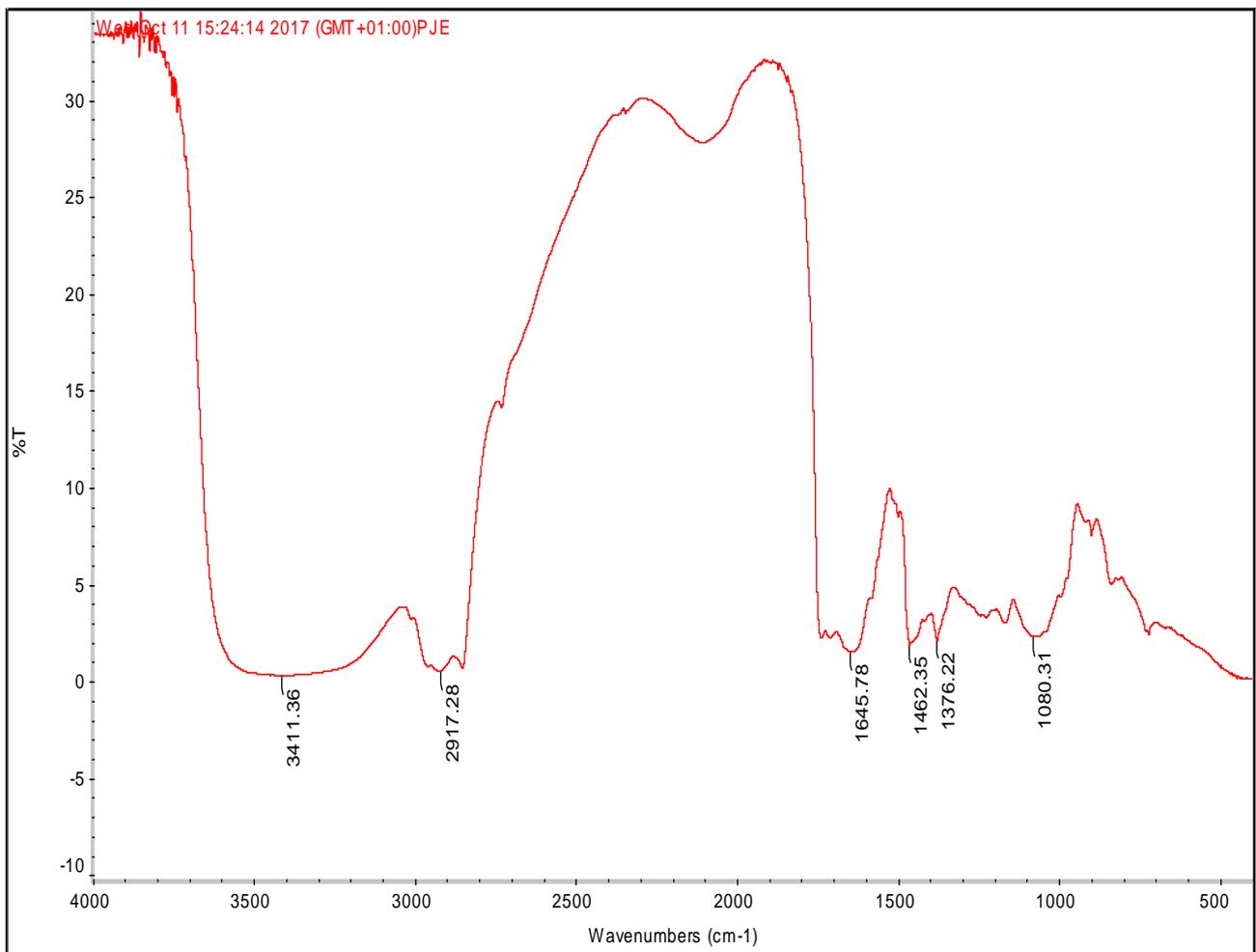


Figure 4.3: FTIR analysis of palm-jatropha extract

4.2 Weight Loss Measurements

Table 4.1: Corrosion lab analysis after 24 hours for palm extract

		(No inhibition)	PE ₁₀₀	PE ₂₀₀	PE ₄₀₀
Determination of weight loss	w_i	112.2499	111.222	109.91	112.021
	w_f	112.2015	111.165	109.858	111.96
	Δw	0.0484	0.0568	0.0519	0.0613

Determination of surface coverage	θ	0.000431181	0.00051	0.00047	0.00055
Determination of corrosion rate					
	D (g/cm ³)	7.86	7.86	7.86	7.86
	A (cm ²)	36.4	36.4	36.4	36.4
	T ₁ (hours)	24	24	24	24
	R_{corr}	0.617467774	0.72463	0.66212	0.78204
Determination of inhibition efficiency	IE%		-17.355	-7.2314	-26.653

Table 4.2: Corrosion lab analysis after 24 hours for jatropha extract

		(No Inhibition)	JE ₁₀₀	JE ₂₀₀	JE ₄₀₀
Determination of weight loss	w_i	112.2499	110.253	111.36	111.726
	w_f	112.2015	110.194	111.301	111.683
	Δw	0.0484	0.0587	0.0589	0.0428
Determination of surface coverage	θ	0.000431181	0.00053	0.00053	0.00038
Determination of corrosion rate	D (g/cm ³)	7.86	7.86	7.86	7.86
	A (cm ²)	36.4	36.4	36.4	36.4
	T ₁ (hours)	24	24	24	24
	R_{corr}	0.617467774	0.74887	0.75142	0.54603
Determination of inhibition efficiency	IE%		-21.281	-21.694	11.5702

Table 4.3: Corrosion lab analysis after 24 hours for palm-jatropha extract

		(No inhibition)	PJE ₁₀₀	PJE ₂₀₀	PJE ₄₀₀
Determination of weight loss	w_i	112.2499	111.321	112.553	111.951
	w_f	112.2015	111.285	112.503	111.921
	Δw	0.0484	0.0353	0.0502	0.0296
Determination of surface coverage	Θ	0.000431181	0.00032	0.00045	0.00026
Determination of corrosion rate	D (g/cm ³)	7.86	7.86	7.86	7.86
	A (cm ²)	36.4	36.4	36.4	36.4
	T ₁ (hours)	24	24	24	24
	R_{corr}	0.617467774	0.45034	0.64043	0.37762
Determination of inhibition efficiency	IE%		27.0661	-3.719	38.843

Table 4.4: Corrosion lab analysis after 48 hours for palm extract

		(No inhibition)	PE ₁₀₀	PE ₂₀₀	PE ₄₀₀
Determination of weight loss	W_i	112.2015	111.165	109.858	111.96
	W_f	112.1779	111.138	109.838	111.932
	ΔW	0.0236	0.0268	0.0194	0.0276
Determination of surface coverage	Θ	0.000210336	0.00024	0.00018	0.00025
Determination of corrosion rate	D (g/cm ³)	7.86	7.86	7.86	7.86
	A (cm ²)	36.4	36.4	36.4	36.4
	T ₂ (hours)	48	48	48	48
	R_{corr}	0.150539664	0.17095	0.12375	0.17605
Determination of inhibition efficiency	IE%		-13.559	17.7966	-16.949

Table 4.5: Corrosion lab analysis after 48 hours for jatropa extract

		(No inhibition)	JE ₁₀₀	JE ₂₀₀	JE ₄₀₀
Determination of weight loss	W_i	112.2015	110.194	111.301	111.683
	W_f	112.1779	110.171	111.275	111.667
	Δw	0.0236	0.0234	0.0265	0.0166
Determination of surface coverage	Θ	0.000210336	0.00021	0.00024	0.00015
Determination of corrosion rate	D (g/cm ³)	7.86	7.86	7.86	7.86
	A (cm ²)	36.4	36.4	36.4	36.4
	T ₂ (hours)	48	48	48	48
	R_{corr}	0.150539664	0.14926	0.16904	0.10589
Determination of inhibition efficiency	IE%		0.84746	-12.288	29.661

Table 4.6: Corrosion lab analysis after 48 hours for palm-jatropha extract

		(No inhibition)	PJE ₁₀₀	PJE ₂₀₀	PJE ₄₀₀
Determination of weight loss	w_i	112.2015	111.285	112.503	111.921
	w_f	112.1779	111.267	112.482	111.901
	Δw	0.0236	0.0186	0.0214	0.0199
Determination of surface coverage	Θ	0.000210336	0.00017	0.00019	0.00018
Determination of corrosion rate	$D(\text{g/cm}^3)$	7.86	7.86	7.86	7.86
	$A (\text{cm}^2)$	36.4	36.4	36.4	36.4
	T_2 (hours)	48	48	48	48
	R_{corr}	0.150539664	0.11865	0.13651	0.12694
Determination of inhibition efficiency	IE%		21.1864	9.32203	15.678

Table 4.7: Corrosion lab analysis after 72 hours for palm extract

		(No inhibition)	PE ₁₀₀	PE ₂₀₀	PE ₄₀₀
Determination of weight loss	w_i	112.1779	111.138	109.838	111.932
	w_f	112.1662	111.117	109.818	111.912
	Δw	0.0117	0.0219	0.0199	0.0202
Determination of surface coverage	θ	0.000104299	0.0002	0.00018	0.00018
Determination of corrosion rate	D (g/cm ³)	7.86	7.86	7.86	7.86
	A (cm ²)	36.4	36.4	36.4	36.4
	T ₃ (hours)	72	72	72	72
	R_{corr}	0.049754635	0.09313	0.08463	0.0859
Determination of inhibition efficiency	IE%		-87.179	-70.085	-72.65

Table 4.8: Corrosion lab analysis after 72 hours for jatropa extract

		(No inhibition)	JE ₁₀₀	JE ₂₀₀	JE ₄₀₀
Determination of weight loss	w_i	112.1779	110.171	111.275	111.667
	w_f	112.1662	110.153	111.258	111.654
	Δw	0.0117	0.0178	0.0172	0.0128
Determination of surface coverage	Θ	0.000104299	0.00016	0.00015	0.00011
Determination of corrosion rate	D (g/cm ³)	7.86	7.86	7.86	7.86
	A (cm ²)	36.4	36.4	36.4	36.4
	T ₃ (hours)	72	72	72	72
	R_{corr}	0.049754635	0.0757	0.07314	0.05443
Determination of inhibition efficiency	IE%		-52.137	-47.009	-9.4017

Table 4.9: Corrosion lab analysis after 72 hours for palm-jatropha extract

		(No inhibition)	PJE ₁₀₀	PJE ₂₀₀	PJE ₄₀₀
Determination of weight loss					
	W_i	112.1779	111.267	112.482	111.901
	W_f	112.1662	111.24	112.463	111.887
	ΔW	0.0117	0.0269	0.0185	0.0147
Determination of surface coverage					
	θ	0.000104299	0.00024	0.00016	0.00013
Determination of corrosion rate					
	D (g/cm ³)	7.86	7.86	7.86	7.86
	A (cm ²)	36.4	36.4	36.4	36.4
	T ₃ (hours)	72	72	72	72
	R_{corr}	0.049754635	0.11439	0.07867	0.06251
Determination of inhibition efficiency					
	IE%		-129.91	-58.12	-25.641

Table 4.10: Corrosion lab analysis after 96 hours for palm extract

		(No inhibition)	PE ₁₀₀	PE ₂₀₀	PE ₄₀₀
Determination of weight loss	W_i	112.1662	111.1165	109.8184	111.912
	W_f	112.154	111.0954	109.8043	111.8987
	Δw	0.0122	0.0211	0.0141	0.0133
Determination of surface coverage	θ	0.000108767	0.00019	0.000128	0.000119
Determination of corrosion rate	D (g/cm ³)	7.86	7.86	7.86	7.86
	A (cm ²)	36.4	36.4	36.4	36.4
	T ₄ (hours)	96	96	96	96
	R_{corr}	0.038910676	0.067296	0.044971	0.042419
Determination of inhibition efficiency	IE%		-72.9508	-15.5738	-9.01639

Table 4.11: Corrosion lab analysis after 96 hours for jatropha extract

		(No inhibition)	JE ₁₀₀	JE ₂₀₀	JE ₄₀₀
Determination of weight loss	w_i	112.1662	110.154	111.258	111.654
	w_f	112.154	110.135	111.225	111.639
	Δw	0.0122	0.0182	0.0328	0.0151
Determination of surface coverage	θ	0.000108767	0.00017	0.00029	0.00014
Determination of corrosion rate	D (g/cm ³)	7.86	7.86	7.86	7.86
	A (cm ²)	36.4	36.4	36.4	36.4
	T ₄ (hours)	96	96	96	96
	R_{corr}	0.038910676	0.05805	0.10461	0.04816
Determination of inhibition efficiency	IE%		-49.18	-168.85	-23.77

Table 4.12: Corrosion lab analysis after 96 hours for palm-jatropha extract

		(No inhibition)	PJE ₁₀₀	PJE ₂₀₀	PJE ₄₀₀
Determination of weight loss	w_i	112.1662	111.24	112.463	111.887
	w_f	112.154	111.221	112.45	111.868
	Δw	0.0122	0.0188	0.0133	0.0181
Determination of surface coverage	θ	0.000108767	0.00017	0.00012	0.00016
Determination of corrosion rate	D (g/cm ³)	7.86	7.86	7.86	7.86
	A (cm ²)	36.4	36.4	36.4	36.4
	T ₄ (hours)	96	96	96	96
	R_{corr}	0.038910676	0.05996	0.04242	0.05773
Determination of inhibition efficiency	IE%		-54.098	-9.0164	-48.361

Table 4.13: Corrosion lab analysis after 120 hours for palm extract

		(No inhibition)	PE ₁₀₀	PE ₂₀₀	PE ₄₀₀
Determination of weight loss	w_i	112.154	111.0954	109.8043	111.8987
	w_f	112.1366	111.0814	109.7881	111.8758
	Δw	0.0174	0.014	0.0162	0.0229
Determination of surface coverage	θ	0.000155144	0.000126	0.000148	0.000205
Determination of corrosion rate	D (g/cm ³)	7.86	7.86	7.86	7.86
	A (cm ²)	36.4	36.4	36.4	36.4
	T ₅ (hours)	120	120	120	120
	R_{corr}	0.044396443	0.035721	0.041335	0.05843
Determination of inhibition efficiency	IE%		-49.18	-168.85	-23.77

Table 4.14: Corrosion lab analysis after 120 hours for jatropha extract

		(No inhibition)	JE ₁₀₀	JE ₂₀₀	JE ₄₀₀
Determination of weight loss	w_i	112.154	110.1354	111.2247	111.6386
	w_f	112.1366	110.111	111.2071	111.6303
	Δw	0.0174	0.0244	0.0176	0.0083
Determination of surface coverage	Θ	0.000155144	0.000222	0.000158	7.43E-05
Determination of corrosion rate	D (g/cm ³)	7.86	7.86	7.86	7.86
	A (cm ²)	36.4	36.4	36.4	36.4
	T ₅ (hours)	120	120	120	120
	R_{corr}	0.044396443	0.062257	0.044907	0.021178
Determination of inhibition efficiency	IE%		-40.2299	-1.14943	52.29885

Table 4.15: Corrosion lab analysis after 120 hours for palm-jatropha extract

		(No inhibition)	PJE ₁₀₀	PJE ₂₀₀	PJE ₄₀₀
Determination of weight loss	w_i	112.154	111.221	112.4497	111.8684
	w_f	112.1366	111.2079	112.4429	111.854
	Δw	0.0174	0.0131	0.0068	0.0144
Determination of surface coverage	θ	0.000155144	0.000118	6.05E-05	0.000129
Determination of corrosion rate	D (g/cm ³)	7.86	7.86	7.86	7.86
	A (cm ²)	36.4	36.4	36.4	36.4
	T ₅ (hours)	120	120	120	120
	R_{corr}	0.044396443	0.033425	0.01735	0.036742
Determination of inhibition efficiency	IE%		24.71264	60.91954	17.24138

Table 4.16: Corrosion lab analysis after 144 hours for palm extract

		(No inhibition)	PE ₁₀₀	PE ₂₀₀	PE ₄₀₀
Determination of weight loss	w_i	112.1366	111.0814	109.7881	111.8758
	w_f	112.1214	111.068	109.7691	111.864
	Δw	0.0152	0.0134	0.019	0.0118
Determination of surface coverage	θ	0.000135549	0.000121	0.000173	0.000105
Determination of corrosion rate	D (g/cm ³)	7.86	7.86	7.86	7.86
	A (cm ²)	36.4	36.4	36.4	36.4
	T ₆ (hours)	144	144	144	144
	R_{corr}	0.03231925	0.028492	0.040399	0.02509
Determination of inhibition efficiency	IE%		11.84211	-25	22.36842

Table 4.17: Corrosion lab analysis after 144 hours for jatropa extract

		(No inhibition)	JE ₁₀₀	JE ₂₀₀	JE ₄₀₀
Determination of weight loss	W_i	112.1366	110.111	111.2071	111.6303
	W_f	112.1214	110.0952	111.194	111.6203
	Δw	0.0152	0.0158	0.0131	0.01
Determination of surface coverage	θ	0.000135549	0.000143	0.000118	8.96E-05
Determination of corrosion rate	D (g/cm ³)	7.86	7.86	7.86	7.86
	A (cm ²)	36.4	36.4	36.4	36.4
	T ₆ (hours)	144	144	144	144
	R_{corr}	0.03231925	0.033595	0.027854	0.021263
Determination of inhibition efficiency	IE%		-3.94737	13.81579	34.21053

Table 4.18 Corrosion lab analysis after 144 hours for palm-jatropha extract

		(No inhibition)	PJE ₁₀₀	PJE ₂₀₀	PJE ₄₀₀
Determination of weight loss	W_i	112.1366	111.2079	112.4429	111.854
	W_f	112.1214	111.1923	112.4254	111.8379
	ΔW	0.0152	0.0156	0.0175	0.0161
Determination of surface coverage	θ	0.000135549	0.00014	0.000156	0.000144
Determination of corrosion rate	D (g/cm ³)	7.86	7.86	7.86	7.86
	A (cm ²)	36.4	36.4	36.4	36.4
	T ₆ (hours)	144	144	144	144
	R_{corr}	0.03231925	0.03317	0.03721	0.034233
Determination of inhibition efficiency	IE%		-2.63158	-15.1316	-5.92105

Table 4.19: Summary of the corrosion rates and inhibition efficiencies at different time intervals for weight loss measurements

Time (hrs)	Inhibitors (concentrations)	Corrosion rates (mm/yr)	Inhibition efficiency (%)
24	PE ₁₀₀	0.72463	-17.355
	PE ₂₀₀	0.66212	-7.2314
	PE ₄₀₀	0.78204	-26.653
	JE ₁₀₀	0.74887	-21.281
	JE ₂₀₀	0.75142	-21.694
	JE ₄₀₀	0.54603	11.5702
	PJE ₁₀₀	0.45034	27.0661
	PJE ₂₀₀	0.64043	-3.719
	PJE ₄₀₀	0.37762	38.843
48	PE ₁₀₀	0.17095	-13.559
	PE ₂₀₀	0.12375	17.7966
	PE ₄₀₀	0.17605	-16.949
	JE ₁₀₀	0.14926	0.84746
	JE ₂₀₀	0.16904	-12.288
	JE ₄₀₀	0.10589	29.661
	PJE ₁₀₀	0.11865	21.1864
	PJE ₂₀₀	0.13651	9.32203
	PJE ₄₀₀	0.12694	15.678
72	PE ₁₀₀	0.09313	-87.179
	PE ₂₀₀	0.08463	-70.085
	PE ₄₀₀	0.0859	-72.65
	JE ₁₀₀	0.0757	-52.137

Time (hrs)	Inhibitors (concentrations)	Corrosion rates (mm/yr)	Inhibition efficiency (%)
	JE ₂₀₀	0.07314	-47.009
	JE ₄₀₀	0.05443	-9.4017
	PJE ₁₀₀	0.11439	-129.91
	PJE ₂₀₀	0.07867	-58.12
	PJE ₄₀₀	0.06251	-25.641
96	PE ₁₀₀	0.0673	-72.951
	PE ₂₀₀	0.04497	-15.574
	PE ₄₀₀	0.04242	-9.0164
	JE ₁₀₀	0.05805	-49.18
	JE ₂₀₀	0.10461	-168.85
	JE ₄₀₀	0.04816	-23.77
	PJE ₁₀₀	0.05996	-54.098
	PJE ₂₀₀	0.04242	-9.0164
	PJE ₄₀₀	0.05773	-48.361
120	PE ₁₀₀	0.035721	19.54023
	PE ₂₀₀	0.041335	6.896552
	PE ₄₀₀	0.05843	-31.6092
	JE ₁₀₀	0.062257	-40.2299
	JE ₂₀₀	0.044907	-1.14943
	JE ₄₀₀	0.021178	52.29885
	PJE ₁₀₀	0.033425	24.71264
	PJE ₂₀₀	0.01735	60.91954
	PJE ₄₀₀	0.036742	17.24138

Time (hrs)	Inhibitors (concentrations)	Corrosion rates (mm/yr)	Inhibition efficiency (%)
144	PE ₁₀₀	0.028492	11.84211
	PE ₂₀₀	0.040399	-25
	PE ₄₀₀	0.02509	22.36842
	JE ₁₀₀	0.033595	-3.94737
	JE ₂₀₀	0.027854	13.81579
	JE ₄₀₀	0.021263	34.21053
	PJE ₁₀₀	0.03317	-2.63158
	PJE ₂₀₀	0.03721	-15.1316
	PJE ₄₀₀	0.034233	-5.92105

The figures below show the graphical representations of the inhibition efficiencies after weight loss measurements.

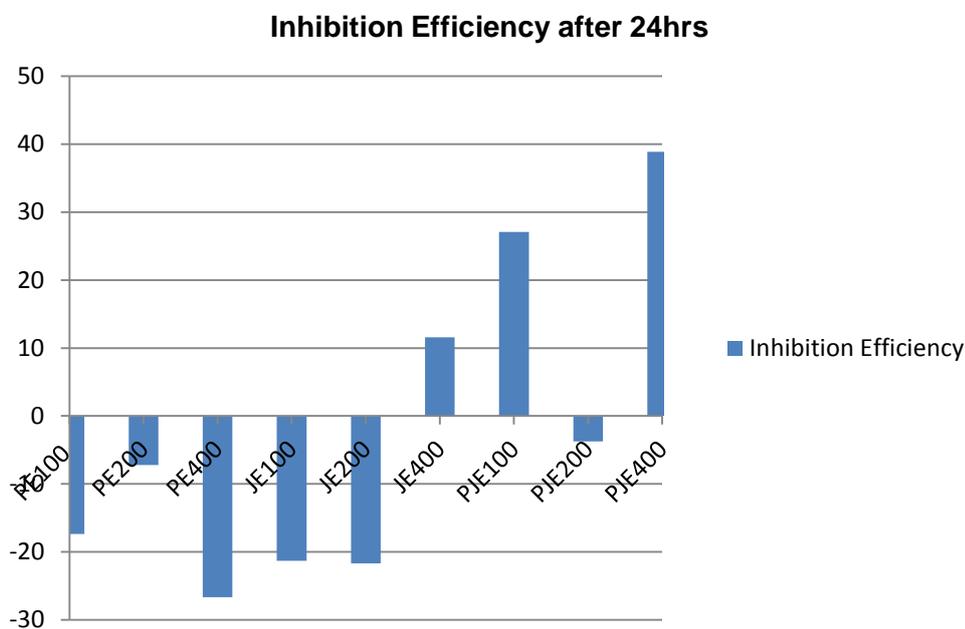


Figure 4.4: Inhibition efficiency after 24 hrs

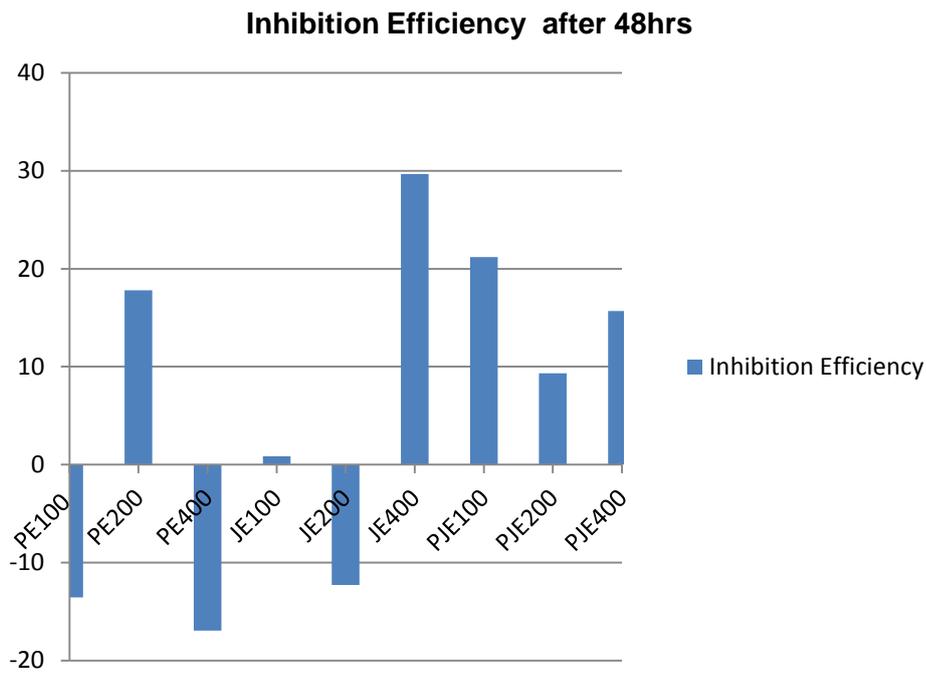


Figure 4.5: Inhibition efficiency after 48 hrs

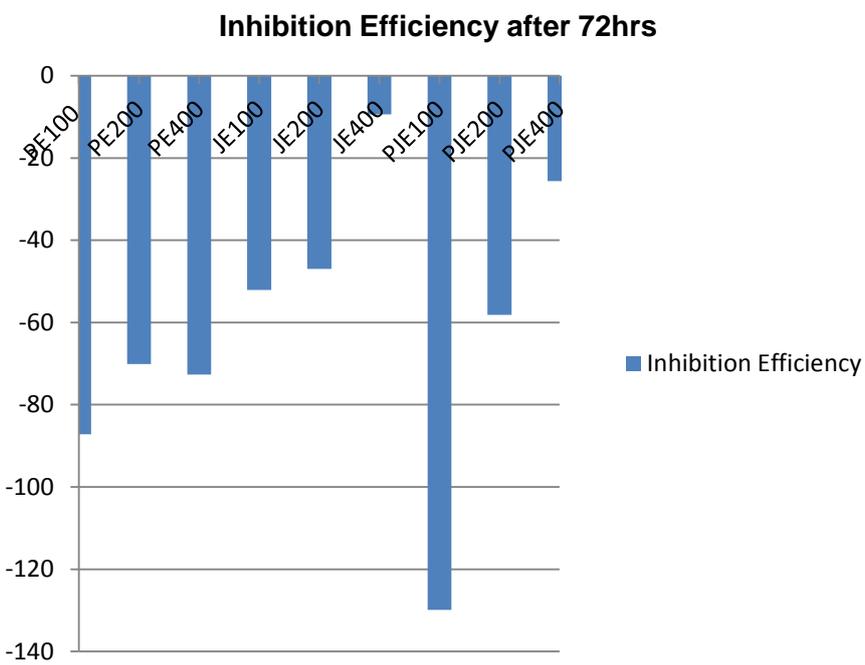


Figure 4.6: Inhibition efficiency after 72 hrs

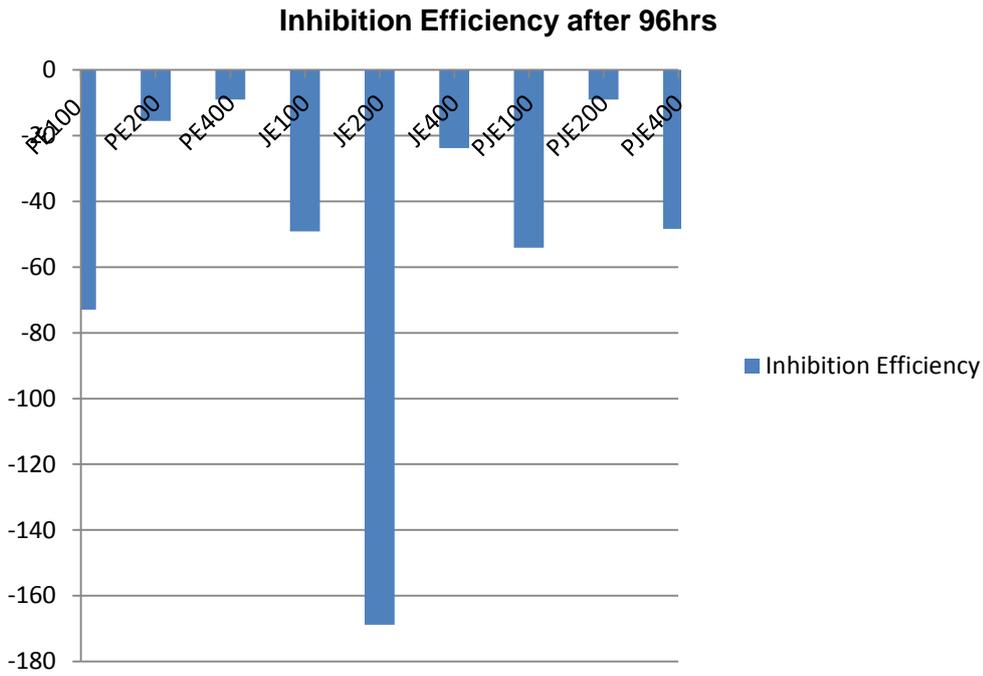


Figure 4.7: Inhibition efficiency after 96 hrs

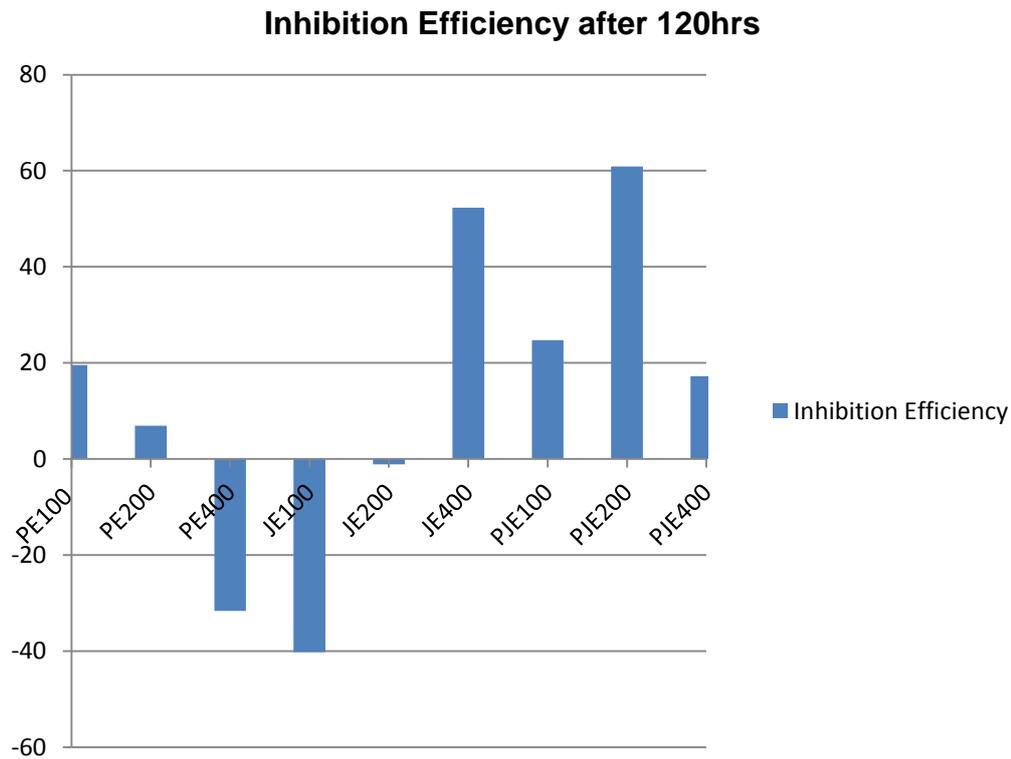


Figure 4.8: Inhibition efficiency after 120 hrs

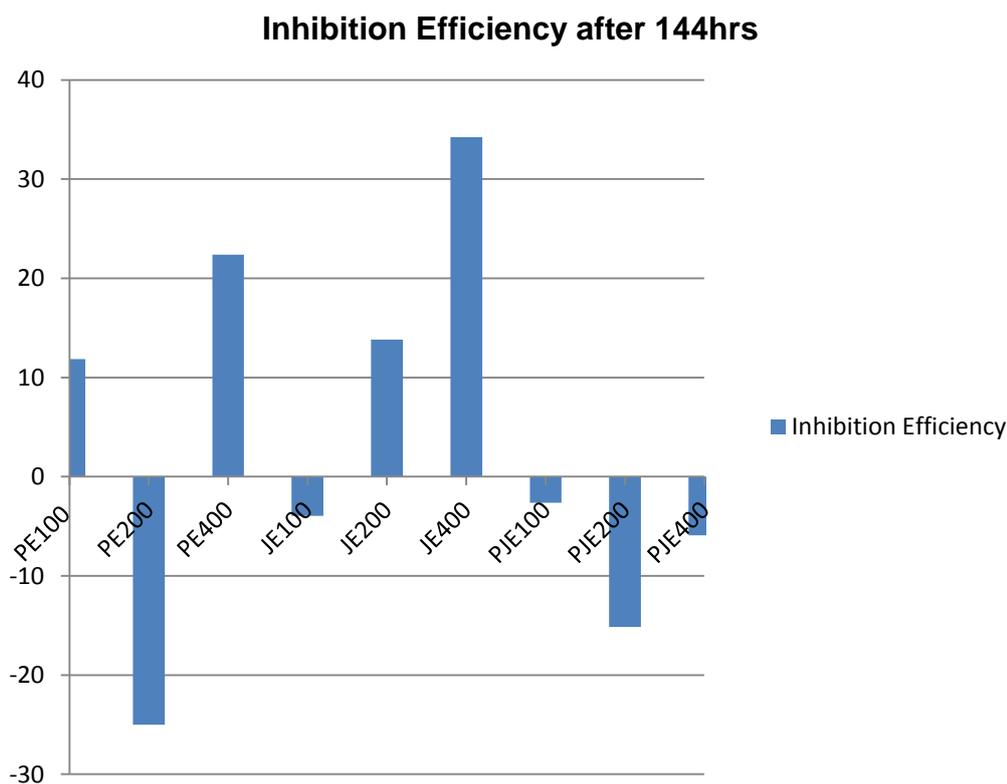


Figure 4.9: Inhibition efficiency after 144 hrs

4.3 Corrosion Rates from Electrochemical Studies

The Gamry Echem Analyst software was used to obtain the corrosion rates of each experiment from their linear polarisation resistance. This is represented in Table 4.20.

Table 4.20: Corrosion rates after electrochemical studies of the metal coupons at different inhibitor concentrations

Inhibitor (Concentrations)	Corrosion Rate (MPY) ($\times 10^{-6}$)	Corrosion Rate (mm/yr) ($\times 10^{-6}$)
Control	2.968	0.0754
PE ₁₀₀	597.3	15.171
PE ₂₀₀	1969	50.0126
PE ₄₀₀	142.7	3.625

JE ₁₀₀	8004	203.302
JE ₂₀₀	3.358	0.0853
JE ₄₀₀	2.816	0.0715
PJE ₁₀₀	1.688	0.0429
PJE ₂₀₀	12.08	0.307
PJE ₄₀₀	64.02	1.626

NB: 1 mpy = 0.0254 mm/y

4.3.1 Graphical Representation of Corrosion Rates after Electrochemical Studies

The graphs in Figures 4.10, 4.11 and 4.12 show the corrosion rates for the different inhibitors after electrochemical studies were carried out.

Corrosion Rate for Palm Extract

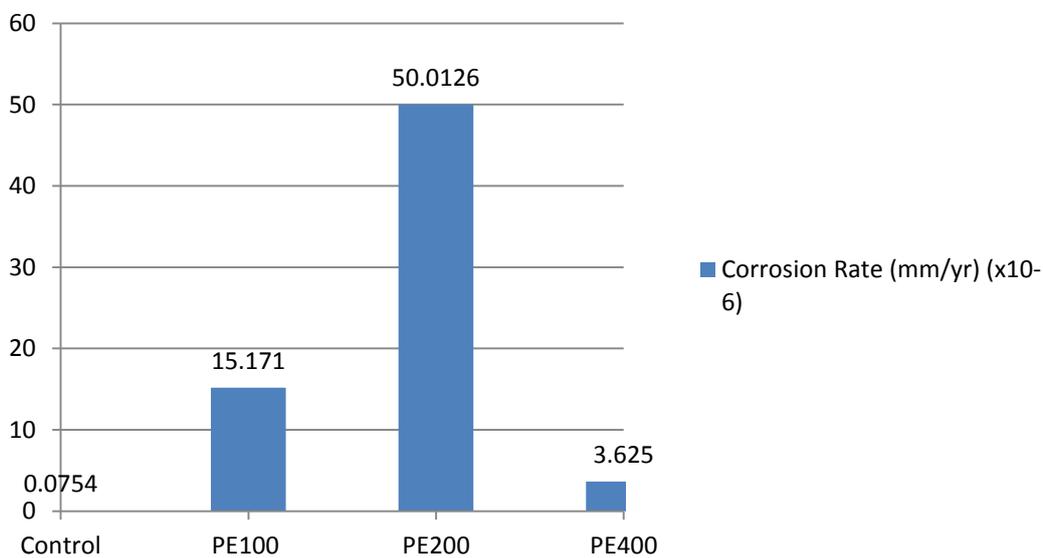


Figure 4.10: Corrosion rate for palm extract

Corrosion Rate for Jatropha Extract

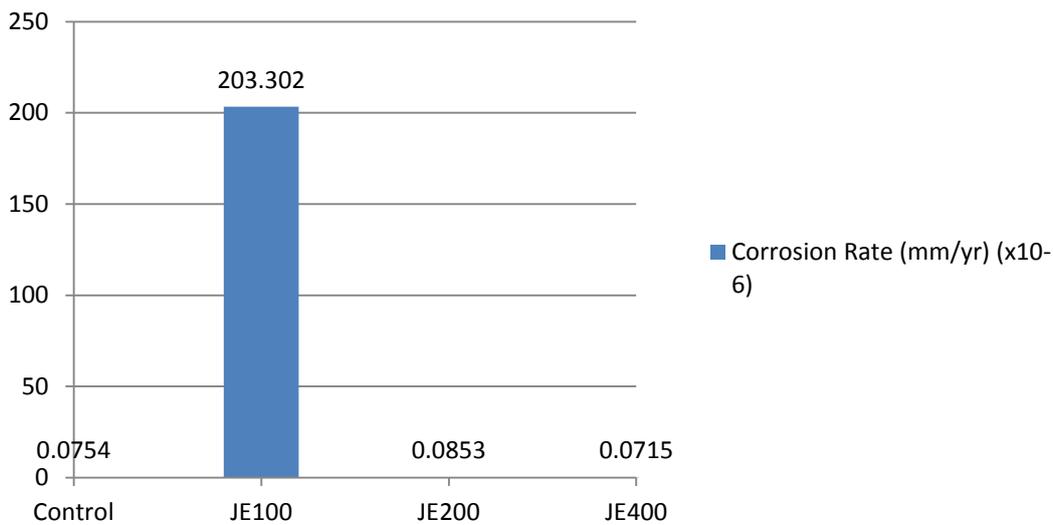


Figure 4.11: Corrosion rate for jatropha extract

Corrosion Rate for Palm-Jatropha Extract

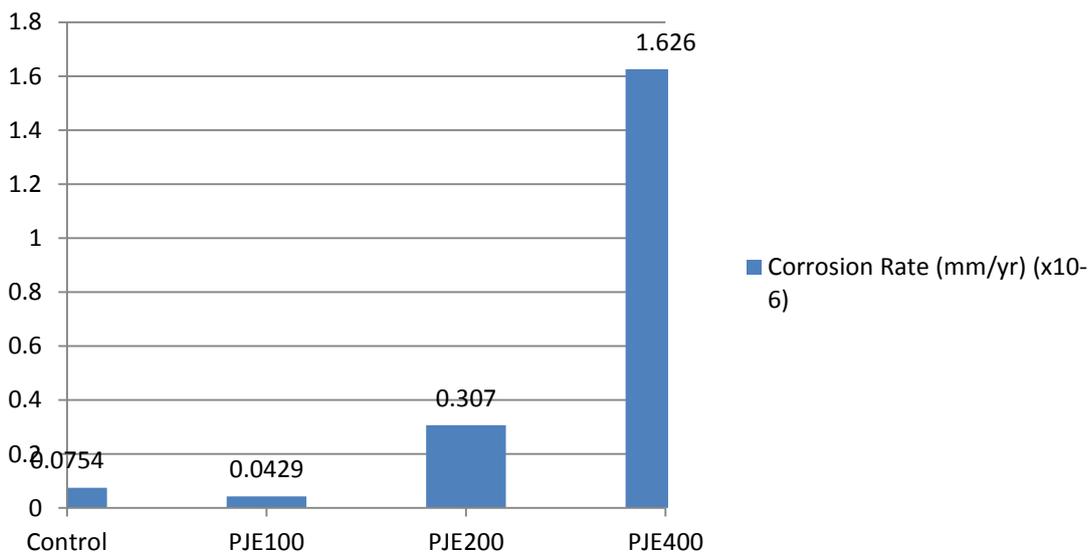


Figure 4.12: Corrosion rate for palm-jatropha extract

4.4 Implications of Results Obtained

4.4.1 FTIR Analysis

The IR spectrum of the palm extract is shown in Figure 4.10. The broad band at 3396 cm^{-1} depicts an O-H stretching vibration of bonded hydroxyl groups, alcohol or phenol. The peaks at 2929 cm^{-1} , 2860 cm^{-1} , and 2360 cm^{-1} are C-H stretching vibrations of saturated hydrocarbons.

This is due to the presence of aliphatic compounds. The strong, sharp band at 1734 cm^{-1} can be ascribed to the stretching vibrational frequency of C=O of esters and lactones. At 1559 and 1507 cm^{-1} , C=C aromatic rings can be predicted. Meanwhile, at 1457 cm^{-1} and 1375 cm^{-1} , the presence of N=O of nitro group exists. The weak bands at 1164 cm^{-1} and 1082 cm^{-1} are indications of C-O stretching vibration band of alcohols, ethers and esters. In addition, the peak at 668 cm^{-1} is an indication of the weak band assigned to the bending vibrational mode of amines.

The IR spectrum of the jatropha extract shown in Figure 4.11 is quite similar to the palm except for the absence of the bending vibrational mode of amines. The IR spectrum of the palm-jatropha leaves extract is shown in Figure 4.12. It is also similar to both palm and jatropha spectra except for the presence of imines and oxime moieties which is depicted by the weak band of the peak at 1645 cm^{-1} . In addition, the O-H stretching at 3411 cm^{-1} is stronger than both palm and jatropha extracts. Phenols, amines and alkaloids act as physical barriers to corrodents. They reduce the rate of corrosion by forming an adsorbed layer on the metal's surface.

4.4.2 Weight Loss/Electrochemical Measurements:

From the results of the weight loss as shown in Tables 4.1 to 4.19, it can be observed that initially, the corrosion rate was high at the onset of the reaction, however, as the exposure time increased, the corrosion rate decreased. High corrosion rates were observed at the initial stages of the exposure time because the substrate of the active sites had not been fully covered by the inhibitor molecules.

Table 4.20 gives a summary of the corrosion rates after the electrochemical studies and, for both weight loss and electrochemical experiments, it can be deduced that the inhibitors worked well at different concentrations. This can be attributed to the coverage the inhibitor films achieved. The palm extract appears to be an anodic inhibitor as it sharply increases the corrosion rate and a reduction is observed at 400 ppm of PE. This can also be observed when looking at the blend. The jatropha extract's inhibitive ability seemed to improve as the concentration increased. At low concentrations for palm-jatropha extract, the inhibitor performed well. The best inhibition efficiency was obtained at PJE 200 (60.9%).

CHAPTER 5: CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The following conclusions can be drawn from the results of this study.

- i. The plant extracts were successfully formulated as corrosion inhibitors from the leaves of palm and jatropa.
- ii. The extracts were characterised using FTIR spectroscopy, indicating the presence of phenols, amines and alkaloids, which are active ingredients for corrosion inhibition.
- iii. The spectrum of the palm-jatropa blend indicated a strong phenol compound and the presence of imines and oximes, which are active ingredients in corrosion inhibition.
- iv. The weight loss and electrochemical studies showed that palm extract is an anodic inhibitor and a blend with jatropa extract would give better inhibition efficiency at a critical temperature.

5.2 Recommendations and Future Work

- i. The above research was carried out at room temperature (25 0C). However, future investigations can be done at elevated temperatures of about 60-70 degrees Celsius.
- ii. An increase in the concentration of the extracts to 500, 700, 1000 ppm should be considered in order to determine the critical concentration above which inhibition would be favoured adequately.
- iii. SEM as well as XPS analysis should be carried out on corroded samples to view surface behaviours at the microscopic level and also to identify surface chemistry after corrosion has occurred.
- iv. The adsorption isotherms of the inhibited samples should be studied.

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