EFFECTIVENESS OF *Mimosa pudica* EXTRACTS AS A BENIGN ANTI-CORROSION ADDITIVE.

BY

ONAH, JOHNPAUL CHIDIEBERE (B. Tech FUTO)

REG. NO: 20134872108

A THESIS SUBMITTED TO THE POST GRADUATE SCHOOL,

FEDERAL UNIVERSITY OF TECHNOLOGY, OWERRI.

IN PARTAIL FULFILLMENT OF THE REQUIREMENTS FOR THE AWARD OF THE DEGREE OF MASTER OF SCIENCE (M.Sc) IN ENVIRONMENTAL TECHNOLOGY.

OCTOBER, 2018.

CERTIFICATION

This is to certify that this work titled **"Effectiveness on *Mimosa pudica* Extracts as a Benign Anti-corrosion Additive"** was carried out by **Onah Johnpaul Chidiebere** with registration number **20134872108** in partial fulfillment for the award of the degree of Master of Science (M.Sc) in the Department of Environmental Technology, School of Environmental Sciences, Federal University of Technology, Owerri.

…………………………… **Prof. E.E Oguzie** (Principal Supervisor)	05/03/17 …………………… Date
…………………………… **Dr. P.N Okeke** (Head of Department, EVT)	05/03/19 …………………… Date
…………………………… **Prof E.E Oguzie** (Dean, SOES)	05/03/19 …………………… Date
…………………………… **Prof (Mrs) Nnenna N.Oti** (Dean of Post Graduate School)	…………………… Date
…………………………… **Prof. I.A Madu.** (External Examiner)	18/06/19 …………………… Date

DEDICATION

This project is dedicated to Almighty God, my family and friends for their immense contributions to my education.

ACKNOWLEDGEMENT

In the course of doing this research work, I have incurred indebtedness to many people too numerous to mention.

First and foremost, I wish to express my profound gratitude to my supervisor, Prof. E.E Oguzie whose experience coupled with wide counseling keenly supervised this project. I am also grateful to Dr.Chigbo for all his expert guidance and valuable contributions to the accomplishment of this work.

I wish to express my unalloyed gratitude to Head of department (HOD), Dr C.O. Nwoko and all my lecturers in the department for their moral and academic support towards the success of this work.

I immensely appreciate the efforts of my entire family Mr. and Mrs. L.E Onah who patiently endured various deprivations and resources of the family in pursuit of the Pearl of great Value to which we are committed simultaneously at different levels.

To the entire university community whose support and encouragement far exceeded my expectations, I say may the good lord bless you all.

Finally, I give thanks to God Almighty for his divine guidance.

TABLE OF CONTENT

CHAPTER TWO: LITERATURE REVIEW

CHAPTER THREE: MATERIALS AND METHODS

CHAPTER FOUR: RESULTS AND DISCUSSION

CHAPTER FIVE: CONCLUSION AND RECOMMENDATION

LIST OF TABLES page

LIST OF FIGURES page

ABSTRACT

This research investigated the suitability of *Mimosa pudica* leaf (MPL) and *Mimosa pudica* stem (MPS) extracts as an inexpensive and nontoxic corrosion inhibitor for mild steel in acidic environment. The experiments carried out to investigate the corrosion inhibition of mild steel in 1.0 M HCl and 0.5 M H_2SO_4 by *Mimosa pudica* leaf and stem extract were weight loss and potentiodynamic polarization methods to determine the extract adsorption potential, kind of protection and its corrosion inhibition efficiency. These parameters were evaluated and interpolated to map out the variations with time and extract concentration using Origin Pro8.0 and Power suite software. The data obtained were analyzed using Design Expert software to determine the optimum combination of extract concentration and exposure time to achieve maximum inhibition efficiency. The protection efficiency of the inhibitors were found to increase with increase in concentration of the inhibitor showing an optimum inhibition efficiency of 99% at 1000mg/L concentration of MPS inhibitor in 1.0 M HCl solution and 96% at the same concentration of MPL in the same acidic solution and in 0.5M H_2SO_4, the optimum inhibition efficiency was found to be 99% at 1000mg/L concentration of MPS extract and 97% at the same concentration of MPL in the same acidic solution. The inhibitor efficiencies determined by weight loss and polarization methods are in reasonable agreement. Based on the polarization results, the investigated MP extract can be classified as mixed-type inhibitor. The adsorption model obeys to the Langmuir adsorption isotherm. The result also revealed that MPS performs better than MPL in both acidic medium and that the efficiency of both extracts decreases with time. This suggests that MP extract is a good alternative corrosion inhibitor. It is therefore concluded that, to maximize the efficiency of MP extract, there is need to stabilize it to ensure a lasting efficiency with time.

Keywords

Mimosa pudica extract, HCl, H_2SO_4 ,Corrosion, Weight Loss, Potentiodynamic Polarization, Inhibition Efficiency, Concentration and Time.

CHAPTER ONE

INTRODUCTION

1.1 BACKGROUND INFORMATION

Metals are the pre-eminent important materials used in structural and decorative applications and with few exceptions; all metals are subject to degradation by chemical reaction of the metal with its environment. The deterioration and destruction of these metals is an un- avoidable but controllable process and it has a significant impact on the development of a country which can be compare to any natural disaster in term of damage cost (Koch and Brongermph, 2002).

Corrosion may be referred as an interaction (electrochemical reaction) of a metal with the surrounding environment, causing a slow, steady and irreversible deterioration in the metal in both physical and chemical properties (Charlton, 2002).This process can be controlled by employing some techniques such as the use of protective coating on metals using organic and inorganic molecules, plastics and polymers; and or cathodic and/or anodic protection using organic and inorganic inhibitors.

ISO 8044 Standard(ISO 8044:1999) defines it as physicochemical interaction between a metal and its environment that results in changes in the properties of the metal, and which may lead to significant impairment of the function of the metal, the environment, or the technical system, of which these form a part". The petroleum industry is one of the most affected by corrosion due to the presence of many corrosive substances in the crude oil, which affect equipments and pipelines from the extraction of crude oil to the transportation of final products.

The factors that can cause corrosion can be identified as physical, chemical, electrochemical or microbiological (Bastidas *et al*, 2005)

1.1.1 CHEMISTRY OF CORROSION

Common structural metals are obtained from their ores or naturally occurring compounds by the expenditure of large amounts of energy. These metals can therefore be regarded as being in a metastable state and will tend to lose their energy by reverting to compounds more or less similar to their original states. Since most metallic compounds, and especially corrosion products, have little mechanical strength, a severely corroded piece of metal is quite useless for its original purpose.

Virtually all corrosion reactions are electrochemical in nature, at anodic sites on the surface the iron goes into solution as ferrous ions, this constituting the anodic reaction. As iron atoms undergo oxidation to ions they release electrons whose negative charge would quickly build up in the metal and prevent further anodic reaction, or corrosion. Thus this dissolution will only continue if the electrons released can pass to a site on the metal surface where a cathodic reaction is possible. At a cathodic site the electrons react with some reducible component of the electrolyte and are themselves removed from the metal. The rates of the anodic and cathodic reactions must be equivalent according to Faraday's Laws, being determined by the total flow of electrons from anodes to cathodes which is called the "corrosion current", Icor. Since the corrosion current must also flow through the electrolyte by ionic conduction the conductivity of the electrolyte will influence the way in which corrosion cells operate. The corroding piece of metal is described as a "mixed electrode" since simultaneous anodic and cathodic reactions are proceeding on its surface (Oguzie *et al*, 2012). The mixed electrode is a complete electrochemical cell on one metal surface.

The most common and important electrochemical reactions in the corrosion of iron are thus anodic reaction (corrosion)

$Fe \rightarrow Fe^{2+} + 2e$ 1.1

Cathodic reaction (simplified)

$2H^+ + 2e \rightarrow H_2$ 1.2

$H_2O + 1/2O_2 + 2e \rightarrow 2OH$ 1.3

1.1.2 THE CONSEQUENCES OF CORROSION

The consequences of corrosion are many and varied and the effects of these on the safe, reliable and efficient operation of equipment or structures are often more serious than the simple loss of a mass of metal. When the structural integrity of some metallic facilities such as storage tanks, pipelines, ships, railcars, tanker trucks, and nuclear waste facilities—all of which store and/or transport potentially hazardous materials is weakened by corrosion, the environment and public safety are threatened. These consequences can be categorized as economic/social effect and environmental effects.

ECONOMIC/SOCIAL EFFECTS

The need for partial/total replacement and plant repairing shutdown after a corrosion incident incur economic loss in every establishment. Some of the major economic/social effects of corrosion can be summarised as follows:

1. Hazards or injuries to people arising from structural failure or breakdown (e.g. bridges, cars, aircraft).

2. Loss of time in availability of profile-making industrial equipment.

4. Loss of technically important surface properties of a metallic component. These could include frictional and bearing properties, ease of fluid flow over a pipe surface, electrical conductivity of contacts, surface reflectivity or heat transfer across a surface.

5. Added complexity and expense of equipment which needs to be designed to withstand a certain amount of corrosion, and to allow corroded components to be conveniently replaced.

ENVIRONMENTAL EFFECTS

Corrosion can cause numerous environmental problems. For instance, water systems can become contaminated and unsuitable for transporting drinking water as a result of corrosion. Also, structures such as storage tanks, pipelines, ships, railcars, tanker trucks, and nuclear waste facilities—all of which store and/or transport potentially hazardous materials—can be highly subject to the harmful effects of corrosion. When the structural integrity of these facilities is weakened by corrosion, the environment and public safety are threatened.

In Nigeria, corrosion of pipelines and tankers accounts for 50% of all oil spills (Nwilo and Badejo, 2007). It leads to rupturing or leaking of production infrastructures. A reason that corrosion accounts for such a high percentage of all spills is that as a result of the small size of the oilfields in the Niger Delta, there is an extensive network of pipelines between the fields, as well as numerous small networks of flow lines—the narrow diameter pipes that carry oil from wellheads to flow stations—allowing many opportunities for leaks. The transport of huge quantities of oil creates opportunities for major oil spills through a combination of human and natural hazards. In onshore areas, most pipelines and flow lines are laid above ground. Pipelines, which have an estimate life span of about fifteen years, are old and susceptible to corrosion. Many of the pipelines are as old as twenty to twenty-five years (Human Rights Watch, 1999).According to Uchegbu, 2002, the problem inherent in oil spills is widespread in their effects and long- lasting in duration. The effects of oil spills throughout the world may be the same since the vegetation, water bodies, soil and ground water are affected. Oil is toxic to massive life, it causes water birds to drown when it soaks its feathers, may decimate fish and shell fish populations, and can severely damage the economics of beach resort areas; but as long as demand for petroleum remains high, some accidents are perhaps, inevitable, (Carla, 2000). The dark film of crude oil when present on the surface of water may hinder the aeration of water body and elevate its temperature by absorbing solar energy and killing aquatic lives. Renner, *et al* (2008) noted that diesel oil reduced the population of periwinkle for more than six months when present on the surface of water.

Crude oil is associated with some toxic heavy metals most of which contaminate the oil through underground deposits, especially lead and chromium. Iron is in great abundance in tropical and subtropical aquifers and is associated with crude oil deposits. High iron concentrations in groundwater are widely reported from developing countries, where iron is often an important water quality issue (Enijiugha and Nwanna, 2004). Mangrove forests are included in a highly complex trophic system. If oil directly affects any organism within an ecosystem, it can indirectly affect a host of other organisms (MERCK, 2007). These floral communities rely on nutrient cycling, clean water, sunlight, and proper substrate. With ideal conditions they offer habitat structure, and input of energy via photosynthesis to the organisms they interact with. The effects of oil spills on mangroves are known to acidify the soils, halt cellular respiration, and starve roots of vital oxygen (MERCK, 2007).The loss of mangrove forests is not only degrading life for plants and animals, but for humans as well (Alexa, 2006). Mangroves forests have been a major source of wood for local individuals. They also are important to a variety of species vital to subsistence practices for local indigenous groups, which unfortunately see little to none of the economic benefits of oil. Mangroves also provide essential habitat for rare and endangered species like the manatee and pygmy hippopotamus. Inya, 1997, outlined the adverse effect of oil exploration and exploitation and revealed that this activity is massively threatening the subsistent peasant economy, the environment and basic survival of the people of the Delta. Cutler and Saundry, 2006 listed economic impacts of oil exploitation to include, recreational sports and fishing losses, tourism losses, (existence value and replacement cost of birds) and mammal's acute mortality, positive impact was strong spill related business such as hotels, taxis, car rentals and boat charters.

1.1.3 CORROSION PREVENTION

By retarding either the anodic or cathodic reactions the rate of corrosion can be reduced. This can be achieved in several ways:

1. Conditioning the Metal

This can be sub-divided into two main groups:

(a) Coating the metal, in order to interpose a corrosion resistant coating between metal and environment. The coating may consist of:

(i) another metal, e.g. zinc or tin coatings on steel, (ii) a protective coating derived from the metal itself, e.g. aluminium oxide on “anodised” aluminium, (iii) organic coatings, such as resins, plastics, paints, enamel, oils and greases. The action of protective coatings is often more complex than simply providing a barrier between metal and environment. Paints may contain a corrosion inhibitor, zinc coating in iron or steel confers cathodic protection. (Koch,*et al*,2016)

(b) Alloying the metal to produce a more corrosion resistant alloy, e.g. stainless steel, in which ordinary steel is alloyed with chromium and nickel. Stainless steel is protected by an invisibly thin, naturally formed film of chromium $Cr_2 O_3$.

2. Conditioning the Corrosive Environment

(a) Removal of Oxygen: By the removal of oxygen from water systems in the pH range 6.5-8.5, one of the components required for corrosion would be absent. The removal of oxygen could be achieved by the use of strong reducing agents e.g. sulphite.

However, for open evaporative cooling systems this approach to corrosion prevention is not practical since fresh oxygen from the atmosphere will have continual access.

3. Electrochemical Control: Since corrosion is an electrochemical process, its progress may be studied by measuring the changes, which occur in metal potential with time or with applied electrical currents. (Oguzie,*et al*,2004). Conversely, the rate of corrosion reactions may be controlled by passing anodic or cathodic currents into the metal. If, for example, electrons are passed into the metal and reach the metal/electrolyte interface (a cathodic current) the anodic reaction will be stifled while the cathodic reaction rate increases. This process is called cathodic protection and can only be applied if there is a suitable conducting medium such as earth or water through which a current can flow to the metal to be protected. Cathodic protection may be achieved by using a DC power supply (impressed current) or by obtaining electrons from the anodic dissolution of a metal low in the galvanic series such as aluminium, zinc or magnesium (sacrificial anodes). Similar protection is obtained when steel is coated with a layer of zinc.

In certain chemical environments, it is sometimes possible to achieve anodic protection, passing a current, which takes electrons out of the metal and raises its potential. Initially this stimulates anodic corrosion, but in favourable circumstances, this will be followed by the formation of a protective oxidised passive surface film.

1.4.1 ENVIRONMENTAL/SAFETY ASPECTS OF CORROSION CONTROLTECHNIQUES

There have been growing concern about certain compounds like chromate, arsenic, phosphate compounds and many others as inhibitors for corrosion control, mainly due to the issue of toxicity, and to that, there have been calls for development of less toxic corrosion inhibiting additives.

An inhibitor can be referred to a substance (or a combination of substances) added in a very low concentration to treat the surface that is exposed to a corrosive environment, that terminate or diminishes the corrosion of metal (Buchweishaija, 1997). They can be called site-blocking elements, blocking species or adsorption blockers due to their adsorption properties (Oguzie *et al*, 2010).

Arsenic is an element that raises much concern from the both environmental and human health standpoints. Arsenic is a protoplasmic poison due to its effect on sulphydryl group of cells interfering with cells enzymes, cell respiration and mitosis (Marchiset-Ferlay, *et al* 2012)

Soluble inorganic arsenic is acutely toxic. Intake of inorganic arsenic over a long period can lead to chronic arsenic poisoning (arsenicosis). Effects, which can take years to develop depending on the level of exposure, include skin lesions, peripheral neuropathy, gastrointestinal symptoms, diabetes, renal system effects, cardiovascular disease and cancer. Organic arsenic compounds are less harmful to health and are rapidly eliminated by the body (Cho et al, 2013).

1.1.5 *Mimosa pudica* OVERVIEW

Mimosa pudica is a creeping annual or perennial herb. It has has been found to have antiasthmatic, aphrodisiac, analgesic, and antidepressant properties. *M. pudica* is known to possess sedative, emetic, and tonic properties, and has been used traditionally in the treatment of various ailments including alopecia, diarrhea, dysentery, insomnia, tumor, and various urogenital infections. Phytochemical studies on *M. pudica* have revealed the presence of alkaloids, non-protein amino acid (mimosine), flavonoids C-glycosides, sterols, terpenoids, tannins, and fatty acids (Genest, *et al*. 2008).

Two well-known movements are observed in *M. pudica* L: one is the very rapid movement of the leaves when it is stimulated by touch, heating, etc., and the other is the very slow, periodical movement of the leaves called nyctinastic movement which is controlled by a biological clock (Ueda and Yamamura, 1999).The leaves of the sensitive plant M. pudica can adapt their closing response to electrical and mechanical stimulation so that they reopen to repeated stimulation. The more intense the stimuli and the longer the intertribal interval, the longer it takes to adapt. Leaves adapted to the effects of mechanical stimulation can still respond by closing to electrical stimulation and vice versa (Markin et al, 2008).

Many plants and herb species used traditionally have potential antimicrobial and antiviral properties (Shelef, *et al* 1983) and this has raised the optimistic thinking of scientists about the future of phyto-antimicrobial agents (Das *et al*, 1999). Several research works have been carried out to study about the phytochemical components of *Mimosa pudica* (Ahmad, *et al,* 2001; Arthur, 1954.) and also about the antimicrobial activity of the plant (Palacios, *et al*, 1991).The major chemical substances of interest in these surveys were the alkaloids and steroidal sapogenin (Lozoya and Lozaya, 1989). However, the methanolic extract of leaves of M. *pudica* showed the presence of bioactive components like terpenoids, flavonoids, glycosides, alkaloids, quinines, phenols, tannins, saponins and coumarin (Gandhiraja, *et al*, 2009). Roots of *Mimosa* contain tannin, ash, calcium oxalate crystals and alkaloid mimosine (Oudhia, *et al*, 2006).

1.2 PROBLEM STATEMENT

Basically, one problem associated with many industries especially those that widely use acid for pickling, de-scaling and as a cleaning agent for the removal of undesirable oxide film has been the deterioration of metals used in the fabrication of the reaction vessel and storage tanks. Material losses and corrosion consequences are priced so high that in some countries like the U.S. and England these factors have been estimated from 3 to 4% of the GDP. Though, over the years, there have been means of controlling these reactions but the means especially the use of synthesised inhibitors have proven to be detrimental to the environmental and also costly (Raja and Sethuraman, 2008). Recent studies estimate that the U.S. demand for corrosion inhibitors will rise by 4.1% per year to USD$ 2.5 billion in 2017. In 2012 they estimated that the market demand of inhibitors was divided on 26.6% to refining petroleum, 16.9% utilities, 16.7% gas and oil production, 15.3% chemical, 9.5% metals, 7.1% pulp and paper and 8.0% other (Argyropoulos,2007). These inhibitors may cause reversible (temporary) or irreversible (permanent) damage to organ system viz, kidney or liver, or to disturb a biochemical process or to disturb an enzyme system at some sites in the body (Ajayi *et al*, 2011; Okafor *et al*, 2012). In order to reduce the environmental and health impacts caused by these chemical substances, there is need to develop a new class of efficient corrosion inhibitors that are less toxic and inexpensive.

1.3 AIM AND OBJECTIVES OF STUDY

1.3.1 AIM

To determine the effectiveness of *Mimosa pudica (Mp)* extract as an inexpensive and non-toxic corrosion inhibitor for mild steel in acidic environment.

1.3.2 OBJECTIVES

1. To evaluate the anti corrosion performance of *Mimosa pudica* extract
2. To determine the effect of *Mimosa pudica* extract concentration on inhibition efficiency.
3. To determine the effect of exposure time on *Mimosa pudica* extract inhibition efficiency.
4. To carry out a factorial design to determine the optimum combination of extract concentration and exposure time to achieve achieve maximum inhibition efficiency

1.4 JUSTIFICATION OF STUDY

Natural products have been studied extensively as corrosion inhibitors both in product mixtures extracted from natural sources such as plants or essentially pure products derived from animals or plants (i.e. vitamins and amino acids) (Mohamed, et al 2004).Successful developments of researches to obtain natural corrosion inhibitors are growing as quickly as the environmental consciousness is gaining ground (Otero and Bastidas, 1996).

Most plants extracts, because of their biocompatibility nature and high adsorption potentials owing to their biological origin tend to serve well as corrosion inhibitors and they are the flavanoid, alkaloids and other natural products obtained from natural sources (Raja and Sethuraman, 2008). In general, these compounds present conjugated aromatic structures, long aliphatic chains such as nitrogen, sulphur, and oxygen heteroatom with free electron pairs that are available to form bonds with the metal surface; in most cases, they act synergistically to exhibit good efficiency regarding the corrosion protection.

Phyto chemical studies on *Mimosa pudica* have revealed the presence of these conponents (alkaloids, non-protein (mimosine), flavonoid C-glycosine, sterole, terpenoids, tannins, and fatty acid) that provides the adsorption and biocompatibility potential. (Pande *et al*, 2010).

1.6 SCOPE OF STUDY

This research work encompasses the design of the experiment using a Design Expert Software 9.0 to determine the best *Mimosa pudica* extract concentration range and the numbers of experimental runs that will give the best result. The variables to be considered are

(I) Concentration of inhibitor (*Mimosa pudica* leaf and stem extract)

(II) Exposure time

(III) Corrosive environment (0.5M H_2SO_4 and 1M HCL)

EXPERIMENTAL TECHNIQUES

The experiment involved the analysis of *Mimosa pudica* leaf and stem extract separately in an electrochemical cell containing mild steel as the working electrode and acidic medium as the electrolyte to determine Potentiodynamic polarization caused by the extract.

This work also involved gravimetric measurement (weight loss measurement) to determine weight loss difference of mild steel in acidic medium in absence, present of different concentrations of mimosa pudica leaf, and stem extract at different exposure time. These parameters were chosen because they are the parameters commonly used to quantify the corrosion inhibition properties of a substance.

DATA ANALYSIS

The data obtained from the experiments were analysed and interpreted using these computational softwares;

(I) Origin Pro 8 (for gravimetric data)

(II) Power Suite software (for electrochemical data)

(III) Design Expert software to vary selected input parameters to theoretically optimize the corrosion inhibition process.

CHAPTER TWO

LITRATURE REVIEW

2.1 INTRODUCTION

With few exceptions, all metals are subject to degradation by chemical reaction of the metal with its environment, that is, corrosion. In most industries whose facilities are constituted by metallic structures, the phenomenon of corrosion is invariably present.

This problem originates very important material and economic losses due to partial or total replacement of equipment and structures, and plant-repairing shutdowns. Material losses and corrosion consequences are priced so high that in some countries like the U.S. and England these factors have been estimated from 3 to 4% of the GDP. (Koch, *et al*. 2002)

Corrosion not only has economic implications, but also social and these engage the safety and health of people either working in industries or living in nearby towns.

The use of corrosion inhibitors (CIs) constitutes one of the most economical ways to mitigate the corrosion rate, protect metal surfaces against corrosion and preserve industrial facilities (Sastri, 1998).Several different methods can be employed to slow or prevent corrosion of metallic structures. The most commonly used methods are protective coatings on metals using organic molecules, plastics, polymers; and cathodic and/or anodic protection using organic or inorganic inhibitors. The initial report of corrosion inhibition by organic inhibitors is attributed to Speller (Speller *et al*) who examined the corrosion inhibition of scaled water pipes in HCl. Since then, many organic and inorganic compounds that are added to the corrosive fluids have been investigated for this purpose.

There are growing concerns about certain compounds like chromates as inhibitors for corrosion processes, mainly due to the issue of toxicity. (Chatterjee et al, 2000). Green inhibitors like natural products from plant extracts and substances from other renewable sources are of the interest of the researchers who are interested in “green chemistry” or “eco-friendly” technology.

2.2 CORROSION INHIBITORS

Corrosion inhibitors are defined as “chemical substance that decreases the corrosion rate when present in the corrosion system at suitable concentration, without significantly changing the concentration of any other corrosion agent” (ISO 8044:1999). Generally the mechanism of the inhibitor is one or more of three that are cited below:

(1) The inhibitor is chemically adsorbed (chemisorptions) on the surface of the metal and forms a protective thin film with inhibitor effect or by combination between inhibitor ions and metallic surface;

(2) The inhibitor leads a formation of a film by oxide protection of the base metal;

(3) The inhibitor reacts with a potential corrosive component present in aqueous media and the product is a complex (Brostoff 1995).

There are many industrial systems and commercial applications that inhibitors are applicable, such as cooling systems, refinery units, pipelines, chemicals, oil and gas production units, boilers and water processing, paints, pigments, lubricants, etc.(Rahmouni,*et al* 2009)

There are evidences of the use of inhibitor since the early 19century. On that time they were already used to protect metals in processes such as acid picking, protection against aggressive water, acidified oil wells and cooling systems. Since years 1950's and 1960's, there was significant advances in the development of technology for corrosion inhibitor as the application of electrochemistry to evaluate corrosion inhibitors. (D'Ars, *et al* 2007)

Recent studies estimate that the U.S. demand for corrosion inhibitors will rise by 4.1% per year to USD$ 2.5 billion in 2017. In 2012 they estimated that the market demand of inhibitors was divided on 26.6% to refining petroleum, 16.9% utilities, 16.7% gas and oil production, 15.3% chemical, 9.5% metals, 7.1% pulp and paper and 8.0% others.(Liu *et al,* 2017). Nowadays, due to changes occurred on the market of corrosion inhibitors, some industrial corrosion inhibitors are being unused.

Due to high toxicity of chromate, phosphate and arsenic compounds, related to various environmental and health problems, strict international laws were imposed. Reducing the use of these and therefore increasing the need for the development of other inhibitor to supply the lack in this area. Should, however, present a similar anti corrosive properties similar than a chromate inhibitor (Hajjaji *et al*. 2007).

An important number of papers have been published with the intention of developing environmentally friendly corrosion inhibitors (Mohamed, *et al* 2004). Also, there has been increasing research in natural products, such as plant extracts, essential oils and purified compounds to obtain environmentally friendly corrosion inhibitors (Bastidas and Otero, 1996).The first evidence of natural product use as corrosion inhibitors is 1930's, when extract of *Chelidonium majus* (Celadine) and other plants were used on the first time in H_2SO_4 pickling baths. Successful developments of researches to obtain natural corrosion inhibitors are growing as quickly as the environmental consciousness is gaining ground. (Oguzie, *et al*, 2004).

Chromates as active inibitors are being replaced by other components such as molybdate compounds and rare earth metal salt, like cerium chloride. Also, drugs have been studied as corrosion inhibitor (Ding, et al, 2006).

2.3 INHIBITOR CLASSIFICATION

The corrosion inhibitors can be chemicals either synthetic or natural and could be classified by: the chemical nature as organic or inorganic; the mechanism of action as anodic, cathodic or a anodic-cathodic mix and by adsorption action, or; as oxidants or not oxidants. (Gentil and Corrosão, 2003).In general, the inorganic inhibitors have cathodic actions or anodic. The organics inhibitors have both actions, cathodic and anodic and the protective by a film adsorption.

Inorganic corrosion inhibitors

Anodic inhibitors: Anodic inhibitors (also called passivation inhibitors) act by a reducing anodic reaction, that is, blocks the anode reaction and supports the natural reaction of passivation metal surface, also, due to the forming a film adsorbed on the metal. In general, the inhibitors react with the corrosion product, initially formed, resulting in a cohesive and insoluble film on the metal surface (Roberge, 1999).

The anodic reaction is affected by the corrosion inhibitors and the corrosion potential of the metal is shifted to more positive values. As well, the value of the current in the curve decreases with the presence of the corrosion inhibitor. The anodic inhibitors reacts with metallic ions produced on the anode, forming generally, insoluble hydroxides which are deposited on the metal surface as insoluble film and impermeable to metallic ion from the hydrolysis of inhibitors results in OH- ions.

When the concentrations of inhibitor becomes high enough, the cathodic current density at the primary passivation potential becomes higher than the critical anodic current density, that is, shift the potential for a noble sense, and, consequently, the metal is passivated. (Bardal, 2004). For the anodic inhibitors effect, it is very important that the inhibitor concentrations should be high enough in the solution. The inappropriate amount of the inhibitors affects the formation of film protection, because it will not cover the metal completely, leaving sites of the metal exposed, thus causing a localized corrosion. (Dutra, *et al* 2011).Concentrations below to the critical value is worse than without inhibitors at all. In general can cause pitting, due reduction at the anodic area relative to cathodic, or can accelerate corrosion, like generalized corrosion, due to full breakdown the passivity. Some examples of anodic inorganic inhibitors are nitrates, molybdates, sodium chromates, phosphates, hydroxides and silicates.

Cathodic inhibitor: During the corrosion process, the cathodic corrosion inhibitors prevent the occurrence of the cathodic reaction of the metal. These inhibitors have metal ions able to produce a cathodic reaction due to alkalinity, thus producing insoluble compounds that precipitate selectively on cathodic sites. Deposit over the metal a compact and adherent film, restricting the diffusion of reducible species in these areas. Thus, increasing the impedance of the surface and the diffusion restriction of the reducible species, that is, the oxygen diffusion and electrons conductive in these areas. These inhibitors cause high cathodic inhibition (Talbot, 2000). When the cathodic reaction is affected the corrosion potential is shifted to more negative values.

Cathodic inhibitors form a barrier of insoluble precipitates over the metal, covering it. Thus, restricts the metal contact with the environment, even if it is completely immersed, preventing the occurrence of the corrosion reaction. Due to this, the cathodic inhibitor is independent of concentration, thus, they are considerably more secure than anodic inhibitor. (Gentil, 2003).Some examples of inorganic cathodic inhibitors are the ions of the magnesium, zinc, polyphosphates, phosphonates, tannins, lignins.

Organic corrosion inhibitor

Organic compounds used as inhibitors, occasionally, they act as cathodic, anodic or together, as cathodic and anodic inhibitors, nevertheless, as a general rule, act through a process of surface adsorption, designated as a film- forming. Naturally the occurrence of molecules exhibiting a strong affinity for metal surfaces compounds showing good inhibition efficiency and low environmental risk (Yaro,*et al* 2013). These inhibitors build up a protective hydrophobic film adsorbed molecules on the metal surface, which provides a barrier to the dissolution of the metal in the electrolyte. They must be soluble or dispersible in the medium surrounding the metal.(Gentil, 2003).The efficiency of an organic inhibitor depends of the: chemical structure, like the size of the organic molecule; aromaticity and/or conjugated bonding, as the carbon chain length; type and number of bonding atoms or groups in the molecule (either π or σ); nature and the charges of the metal surface of adsorption mode like bonding strength to metal substrate; ability for a layer to become compact or cross-linked, capability to form a complex with the atom as a solid within the metal lattice; type of the electrolyte solution like adequate solubility in the environment.

The efficiency of these organic corrosion inhibitors is related to the presence of polar functional groups with S, O or N atoms in the molecule, heterocyclic compounds and pi electrons, generally have hydrophilic or hydrophobic parts ionisable. The polar function is usually regarded as the reaction centre for the establishment of the adsorption process (Yaro, *et al* 2013)

The organic acid inhibitor that contains oxygen, nitrogen and/or sulphur is adsorbed on the metallic surface blocking the active corrosion sites. Although the most effective and efficient organic inhibitors are compounds that have π-bonds, it present biological toxicity and environmental harmful characteristics. (Mahmoud and El-Haddad, 2013). Some examples are amines, urea, Mercaptobenzothiazole (MBT), benzotriazole toliotriazol, aldehydes, heterocyclic nitrogen compounds, sulphur-containing compounds and acetylenic compounds and also ascorbic acid, succinic acid, tryptamine, caffeine and extracts of natural substances.

There are still some inhibitors that act in vapour phase (volatile corrosion inhibitor). Some examples are: dicicloexilamônio benzoate, diisopropylammonium nitrite or benzoate, etha- nolamine benzoate or carbonate and also the combination of urea and sodium nitrite

2.4 NATURAL PRODUCTS AS ENVIRONMENTALLY FRIENDLY CORROSIONINHIBITORS (EFCIs)

Natural products have been studied extensively as corrosion inhibitors both in product mixtures extracted from natural sources such as plants or essentially pure products derived from animals or plants (i.e. vitamins and aminoacids). From the economic and environmental view points, plant extracts are an excellent alternative as inhibitors because of their availability and biodegradability. These extracts can be obtained in a simple way and purification methods are not required. The extracts are generally obtained from cheap solvents that are widely available, at a low cost and with low toxicity; the aqueous extract is more relieved, but due to the low solubility of many natural products in water, common ethanol extracts are also obtained.

These extracts contain a variety of natural products such as essential oils, tannins, pigments, steroids, terpenes, flavones and flavonoids, among other well-known active substances used as CIs. In general, these compounds present conjugated aromatic structures, long aliphatic chains such as nitrogen, sulphur, and oxygen heteroatom's with free electron pairs that are available to form bonds with the metal surface; in most cases, they act synergistically to exhibit good efficiency regarding the corrosion protection. This can be demonstrated in the case of Ginkgo biloba in which the main components (flavonoids and terpenoids) have been identified (Oguzie *et al,* 2014)

These extracts have demonstrated excellent efficiency as CI with potential applications in the Oil Industry concerning the corrosion inhibition of Q235A steel. The antibacterial activity of the extracts against oil field microorganisms (SRB, IB and TGB) has also been proved (Obot, ***et al,*** 2010).

The main disadvantage of using plant materials as CIs is their frequently low stability, they are readily biodegradable; however, this disadvantage can be minimized or avoided by adding biocides such as N-cetyl-N,N,N-trimethyl ammonium bromide. In the last years, Umoren and Obot's research group has published several papers about the evaluations of plant extracts as CIs, for example, Phyllanthus amarus (Obi-Egbedi,*et al,* 2012) and Spondias mombin L. (Rani and Basu, 2012).They also described the inhibitive action of ethanolic extracts from leaves of Chlomolaena Odorata L. (LECO) as eco-friendly CI of acid corrosion of aluminum in 2 M HCl, using hydrogen evolution and thermometric techniques (Rajendran, 2005) ; and more recently, for corrosion of mild steel in H_2SO_4 solution (Lame,**et al,** 2013).In this last paper, the obtained results showed that LECO functioned as a CI and its efficacy increased with the extract concentration, but decreased with temperature. At a concentration as low as 5 %v/v of the extract, the inhibitory efficiency reached about 95% at 303 K, and 89% at 333 K.

In another interesting work, this group showed the excellent inhibitory properties of Coconut coir dust extract (CCDE) as corrosion inhibitor of aluminum in 1 M HCl, using weight loss and hydrogen evolution techniques at 30 and 60°C by monitoring the volume of evolved hydrogen gas at fixed time intervals.

The representative plots of the volume of the evolved hydrogen gas as a function of the reaction time at 30 and 60°C for Al in 1 M HCl, in the absence and presence of different concentrations of the CCDE, showed a remarkable increase in the volume of evolved H_2 gas in the blank acid solution at both studied temperatures. As for the introduction of CCDE into the corrosive medium, it is seen that there is a considerable reduction in the volume of evolved hydrogen gas, suggesting that the CCDE components were adsorbed onto the metal surface, and blocked the electrochemical reaction efficiently by decreasing the available surface area (Belkhaouda, *et al* 2013)

Several human drugs, including diuretics and barbiturates have shown good performance as CIs of metals in acid media (Oezcan, *et al* 2008).In this sense, two well-known diuretics, Furosemide and Torsemide were recently evaluated as CIs of mild steel in hydrochloric acid medium. From the two inhibitors, Torsemide performance is superior to that of Furosemide due to its high electron density, which favours its adsorption on the metal surface.

Polarization studies revealed that the inhibiting action of the compounds is under mixed control. The free adsorption energy and the temperature influence on the adsorption of inhibitors onto a mild steel surface have been reported. The adsorption of the compounds was found to obey the Langmuir adsorption isotherm. The inhibition and formation mechanisms of the Fe–inhibitor complex were confirmed by FT-IR and UV–visible absorption spectral analysis. The scanning electron microscopy (SEM) and atomic force microscopy (AFM) results established the formation of a protective layer on the mild steel surface. Quantum chemical calculations were applied to correlate the inhibition performance of inhibitors with their electronic structural parameters (Liu, *et al* 2013)

In 2011 the application of drugs as promising novel EFCIs was reviewed. Several synthetic polymers have also been designed as efficient EFCIs for carbon steel in alkaline solution (Khaled, 2003) and for calcium carbonate scale inhibitor for cooling water.

Ionic liquids (ILs) deserve particular attention due to the rapid grown in the number of applications in the Oil Industry and particularly within the topic of synthetic EFCIs; these have shown an effective performance as inhibitors of various metals and alloys (Gabler, et *al* 2011).ILs is ionic compounds, showing anisotropic molecular shape; their structure contains both organic and inorganic type components with various functional groups. They offer novel physical and chemical properties like low toxicity, high chemical stability, low vapour pressure and high electrical conductivity (Martinez-Palou, 2010).

Quite a number of ILs do behave not only as green solvents suitable for the electrochemical devices and methods but also as unique and robust electrolytes with high stability Also, these compounds present structure properties suitable to be absorbed on metal surfaces and some derivatives of these families have proved that they can act as EFCIs in acid and basic corrosive environments. Likhanova et al. have published a paper about the inhibitory action of 1,3-dioctadecylimida- zolium bromide (ImDC18Br), N-octadecylpyridinium bromide (PyC18Br) in 1 M H2SO4 on mild steel at room temperature.

The effect of the concentration of inhibitor compounds was investigated by electrochemical tests, whereas the surface analysis was performed at 100 ppm for both compounds. In the case of ImDC18Br, corrosion products were additionally studied by X-ray diffraction and Mössbauer spectroscopy. The results revealed that ILs act as corrosion inhibitors with 82-88% at 100 ppm to protect the mild steel corrosion in the aqueous solution of sulfuric acid; their efficiencies are increased with the inhibitor concentration in the range 10–100 ppm range. SEM-EDX, XRD and Mössbauer analysis indicated the presence of carbon species and iron sulfates in the presence of ILs; whereas corrosion products such as iron oxyhydroxides were present in the absence of the ILs; this behavior was described by the proposed corrosion inhibition mechanism (Tuken,*et al*,2012).

The same research group published a related paper in 2011 where five imidazolium-type ILs containing N1-vynil and N3-long alkyl saturated chains as cation and bromide as anion were synthesized under microwave irradiation and evaluated as CIs for acid environment. Weight loss and electrochemical polarization techniques were used to test the inhibitory properties of these compounds in AISI 1018 carbon steel immersed in 1.0 M H_2SO_4. All the studied ILs showed inhibitory properties dependent on the chain length linked to N3. The highest efficiency of IL4 was confirmed by Scanning Electron Microscopy (SEM)/Energy-Dispersive X-ray spectrosco- py (EDX) and Atomic Force Microscopy (AFM) images of film formation. SEM revealed revealed that the surface morphology was strongly damaged in the CI absence, but in the presence of 100 ppm of CI, damage was considerably diminished, which confirmed the high efficiency of 1-Vinyl-3-octadecylimidazolium bromide at this concentration.

2.5 CONSIDERATION TO EMPLOY INHIBTORS

For all types of inhibitors, we should consider some environmental actions factors because some elements such as metals, pH, composition, impurities, agitation, and temperature, geometry of the system, the concentration of inhibitor and the mixture of one or more inhibitors may change the anti-corrosive mechanism. (Yohai,*et al* 2013). To employment of the inhibitors, it is quite satisfying that certain factors should be seen as the real cause of the corrosion, the cost X benefit and possible interactions of the inhibitor with the environment, such as the influence of a catalyst, deposition or contamination.

2.6 INDUSTRIAL APPLICATION OF INHIBITORS

Acid pickling: Prevent the attack in the metal due to the acid solution in which metal gets cleaned of mill scale (bark lamination), and also prevented the subsequent hydrogen evolution. Inhibitors are added, typically organic, must be soluble or dispersed in the solution. Examples: thiourea and amino and its derivatives, propargyl alcohol (Gentil, 2003.)

Oil industry: sodium carbonates or organic amines complex are employed to reduce the corrosive effect of CO_2, H_2S and organic acids, enabling the use of more cheaper materials and less resistant to corrosion in wells extracting crude oil. Pipes for gasoline and kerosene are employed sulphonated oils, sodium nitrite. Oil well uses up fatty amines, fatty acids, imida- zolines and quaternary ammonium salts. Internal pipe corrosion occurs in wet gas transportation due to condensation of water containing dissolved corrosive gases. Corrosion is caused by the dissolution of the corrosive gases, such as carbon dioxide and hydrogen sulfide as well as condensation of acid vapours.

Water transmission and distribution systems: is used corrosion inhibitor in combination with pH adjusters and alkalinity control towards an efficient protection (Koch, *et al*, 2013]

2.7 INHIBITORS EVALUATION

The most usefully technique to analysis the effectiveness of an inhibitor are weight loss experiment and electrochemical measurements, like polarization curve method and the impedance measurement analyzing. In addition, microscopy techniques are used to characterize the corrosion process

The parameter commonly used to quantify the corrosion inhibition properties of a substance is the inhibitor efficiency (IE), defined as:

$$IE\ (\%) = \frac{CR_{abs} - CR_{pre}}{CR_{abs}} \times 100 \qquad 2.1$$

Where CR_{abs} and CR_{pre} are the metal corrosion rate in the absence and presence of inhibitor, respectively. Corrosion rates can be obtained by different ways, being the most used gravimetric and electrochemical techniques. Gravimetric measurements are usually carried out using coupons and measuring the weight before and after exposure to the corrosive environment, without the inhibitor and with the inhibitor added to the solution or the metal pre-treated. Electrochemical techniques, such as polarization resistance, calculation of Tafel slopes from voltametries and electrochemical impedance spectroscopy (EIS), allow for an indirect calculation of corrosion rates and have the advantage of providing information on the mechanisms of the corrosion and inhibition processes (Dowsett, et al 2009).

Since the adsorption of the inhibitor molecules on the metal surface is a fundamental step in the inhibition process, the study of the adsorption process can also provide useful information of the inhibition mechanisms. The study of the adsorption isotherms is a classical method for studying this process and they have the general form:

$$kc = g(\theta, \chi)\exp(-f\theta) \qquad 2.2$$

were k is the equilibrium binding constant of the adsorption reaction, c is the inhibitor concentration; g(θ,χ) is the configurational term parameter, in which χ is the number of water molecules replaced by one molecule of organic inhibitor and θ is the degree of coverage of the metallic surface; and *f* is the interaction term parameter (*f*> 0 lateral attraction, and *f*< 0 lateral repulsion between the adsorbed inhibitor molecules) (Bastidas, et al 2002).

These models assume that: the adsorption sites on the metal surface are homogeneous, a mono-layer inhibitor adsorption is formed, and corrosion is uniform and no localised attack takes place (Bastidas, et al, 2005) which is not always the case, especially in heritage objects. They also consider a thermodynamic equilibrium between the inhibitors in the environment and the adsorbed layer, thus in those cases where the concentration of the inhibitor changes or they are applied in solvents and then exposed to a different environment, these models are not useful.

The use of quantum chemical calculations for the evaluation of inhibition properties is not a new tool, but has gained a huge popularity in the last years due to the improvement of the calculation capabilities of personal computers. A recent review of the use of these techniques has been recently published by Gece (Gece, 2008).

These calculations allow to correlate inhibitor efficiencies with molecular properties such as orbital energies (mainly highest occupied molecular orbital energy, E_{HOMO}, and lowest unoccupied molecular orbital energy, E_{LUMO}), dipole moment, charge density, heat of formation and ionization potential (Khalil, 2003).

Quantum chemistry calculations can be very helpful to study fundamental inhibition mechanisms and have shown a good correlation with experimental data in some cases, for simple corrosion systems. However, in others, the correlation is not so clear, since the assumptions and simplifications needed to allow the computing of the models might neglect important factors in the corrosion inhibition process (Gece, 2008).

Surface analysis techniques have also been extensively used for the characterization of the inhibitor layers formed on the metals (Fang, et al, 1986). The use of these techniques allows the study of the layer composition formed on metals following the procedures used by conservators-restorers, and the study after exposure of the coated metals to the atmospheric environment, closer to the real life of the objects than the immersion tests necessary for electrochemical measurements (Cano, et al, 2004). The main disadvantage is that sometimes the efficiencies observed by electrochemical or gravimetric measurements are difficult to correlate with the surface characterization results (Galtayries, et al 2007)

CHAPTER THREE

MATERIALS AND METHODS

3.1 MATERIALS PREPARATION

3.1.1 SPECIMEN PREPARATION

Mild steel specimens with composition C = 0.01 %, Mn = 0.34 %, P = 0.08 % and Fe = 99.51 % were used for this study. Each sheet, which was 0.1 cm in thickness, was mechanically pressed-cut into coupons of dimension 3 cm x 3 cm. The coupons were first mechanically polished using sis emery paper, degreased with ethanol or acetone and cleansed with deionized water. It was later dried in vacuum desiccators at room temperature and weight.

3.1.2 REAGENTS

All reagents were of analytical grade, and used as source without further purification. Distilled water was used for all solutions preparations. 1 M HCl and 0.5 M H_2SO_4 solutions were employed as corrodents. The concentrations of the plant solution used were 50 mg L^{-1}, 200 mg L^{-1}, 400 mg L^{-1}, 600 mg L^{-1}, 800 mg L^{-1} and 1000 mg L^{-1}.

3.1.3 PREPARATION OF *MIMOSA PUDICA* EXTRACT

The leaf and stem of *Mimosa pudica* were cut and washed with sterile water, sun dried to a constant weight and ground into fine powder. The extract was prepared by refluxing 0.04kg of *Mimosa pudica* leaf and stem separately in 2 L of ethanol for 3 h after which it was filtered using cloth filter. The concentration of the filtrate (stock solution) was determined by drying and weighing the weight of residue in relation to the volume of the sample taken. Using dilution, stock with different extract concentrations was prepared for both stem and leaf extract.

3.2 CORROSION EXPERIMENTS

3.2.1 WEIGHT LOSS/GRAVIMETRIC EXPERIMENTS

The pre weighed mild steel specimen was immersed in 0.3 L acid solution without and with *Mimosa pudica* extract of different concentrations. The specimens were left immersed between 24 to 168 hrs at room temperature. The weight loss was taken as the difference in weight of the specimens before and after immersion determined using digital balance with sensitivity of ±1 mg. Before taking weight loss measurement, the metal specimens were washed thoroughly with water and ethanol and then dried before reading their weight differences.

The corrosion rate was obtained using the equation bellow:

Corrosion Rate (CR) = W/At (gcm^{-2}/h^{-1}) (3.1)

Where W is the weight loss (g),A is th area of mild steel coupon (cm^{2}) and t is the time of immersion (h).

Also, the inhibition efficiency for the additive was calculated using the equation below:

$$IE(\%) = \left(1 - \frac{Winh}{Wblank}\right) \times 100 \quad (3.2)$$

Where:

W_{inh} = *weight loss in inhibitor solution*

W_{blank} = *weight loss in blank solution*

IE (%) = inhibition efficiency

3.2.2 POTENTIODYNAMIC POLARIZATION MEASURENTS.

A conventional three electrode cell consisting of a saturated calomel (reference) electrode, a platinum auxiliary (counter) electrode and the working electrode with 1cm^{2} exposed was used for potentiodynamic polarization (PDP).

Before any measurement was made, the working electrode was introduced in a test solution and kept to attain the Open Circuit Potential (OCP) to stabilize the solution. The working electrode was coated thoroughly with epoxy resin keeping surface area of 1cm^2 only for the study. The surface was degreased with acetone and washed with distilled water. The measurement was made using computer controlled electrochemical workstation VERSASTAT 400 Complete DC Voltammeter with studio software Power suite.

Potentoidynamic polarization measurements were carried out in the range of potential from -250 to +250V with a scan rate of 0.3660V/S. The range of potential from -0.3 to -0.7 was used for potentiodynamic polarization curve.

$$IE\% = \left(\frac{I_{corr(bl)} - I_{corr(inh)}}{I_{corr(bl)}}\right) x\ 100 \qquad (3.3)$$

Where 1corr (bl)and 1corr (inh)are corrosion current density value without and with extract respectively. Extrapolation of Tafel straight line will lead to the calculation of corrosion potential, cathodic and anodic Tafel slope (B_c andB_a)

3.3 DESIGN OF EXPERIMENT

Based on the choice of response variable and proper consideration of potential design factors and sample size, a central composite factorial design was made using Design Expert 9.0 software in order to select a suitable run order for the experimental trials, number of replicates and determine whether or not blocking or other randomization restrictions will be involved. The matrix for the two variables was varied at two levels (-α, and +α) and the experiments were performed as specified by the software in order to avoid systematic error.

3.4 DATA ANALYSIS

VARYING OF SELECTED INPUT PARAMETERS TO THEORETICALLY OPTIMIZE THE CORROSION INHIBITION PROCESS USING CENTRAL COMPOSITE DESIGN

The generated experimental data were inputted into the Design Expert 8.0 software to examine the combined effect of the potential design factors (inhibitor concentration, acid concentration and exposure time) on inhibition efficiency (% I.E) and derive a model (quantitative relationship (equation) between the response and the important design factors) and evaluate optimum conditions for the inhibition efficiency. This was done to ensure so that results and conclusions are objective rather than judgmental in nature. The quadratic and linear interactive effects of the process variables on the percentage inhibition efficiency were calculated and their respective Significance evaluated by ANOVA test. The *p*-value was used as the yardstick for measuring the significance of the regression coefficients, values of $p<0.05$ signified that the coefficient is significant. The adequacy of the model was tested by the coefficient of determination (R^2) value as compared to the adjusted R^2 value.

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 RESULTS

From the basic standard gravimetric and electrochemical analysis adopted to investigate the suitability of *Mimosa pudica* (Mp) extract as an inexpensive and non toxic corrosion inhibitor for mild steel in acidic environment, various distinct modifications on corrosion inhibition properties where obtained as presented bellow.

4.1.1 EFFECT OF MP CONCENTRATION AND EXPOSURE TIME ON WEIGHTLOSS.

The non-electrochemical technique of weight loss was done in order to determine the corrosion rate and percentage of inhibition. In 1.0 M HCl solution, MPS extract of 200Mg/L concentration recorded a weight loss value of 0.6817g after 168 h exposure, while 1000Mg/L concentration of the same extract gave a weight loss value of 0.1077g after the same period as shown in table 4.1a and 4.2a bellow. In the same solution, 0.8448g was lost at 200Mg/L MPL extract concentration in

168h while 1000Mg/L of the same extract recorded weight loss of 0.3399g after the same period as shown in table 4.1b and 4.2b.

In 0.5 M H_2SO_4 solution, MPS extract of 200 Mg/L concentration recorded a weight loss value of 1.1102 g after 168 h exposure, while 1000 Mg/L concentration of the same extract gave a weight loss value of 0.1196g after the same period as shown in table 4.3a and 4.4a bellow. In the same solution, 1.8445 g was lost at 200 Mg/L MPL extract concentration in 168 h while 1000 Mg/L of the same extract recorded weight loss of 0.4744 g after the same period as shown in table 4.3b and 4.4b bellow.

Figure 4.1a to 4.1d show the weight loss of mild steel in the absence and presence of different concentration of *Mimosa pudica* leaves and stem extract in both 1.0 M HCl and 0.5 M H_2SO_4 at different time intervals. The figures clearly illustrate a reduction in weight loss of the metal coupons in the presence of these inhibitors compared to the blank solution. The figures also revealed that the loss in weight of the coupons decreases as the concentration of the inhibitors increases and increases with increase in time. From the data, both inhibitors tend to perform more in 1.0 M HCl than in 0.5 M H_2SO_4 owing to the presence of chlorine which has the ability to detach hydroxyl ion in metal thereby increasing the negativity of the metal, to that, neutral inhibitors will act more on more negative solution.

Table 4.1a. Effects of concentration on the corrosion inhibition performance of *Mimosa pudica* stem extract in 1.0 M HCl after 24h

MPS (Mg/l)		Initial weight (g)	final weight (g)	Weight loss (g)	Average (g)	EI (%)
50	A	4.7241	4.6213	0.1028	0.1027	86
	B	4.7846	4.6820	0.1026		
200	A	4.6699	4.6182	0.0517	0.0613	94
	B	4.8703	4.7994	0.0709		
400	A	4.9080	4.8672	0.0408	0.0454	95
	B	4.5954	4.5455	0.0499		
600	A	4.8413	4.8247	0.0166	0.0233	97
	B	5.1554	5.1254	0.0300		
800	A	4.9211	4.9035	0.0176	0.0153	98
	B	5.0503	5.0373	0.0130		
1000	A	4.7770	4.7660	0.0110	0.0110	99
	B	4.9033	4.8923	0.0110		

Table 4.1b. Effects of concentration on the corrosion inhibition performance of *Mimosa pudica* leaf extract in 1.0 M HCl after 24h.

MPL (Mg/l)		Initial weight (g)	final weight (g)	Weight loss (g)	Average (g)	EI (%)
50	A	4.9065	4.7532	0.1533	0.1846	81
	B	4.9505	4.7347	0.2158		
200	A	4.6734	4.5514	0.1220	0.1033	86
	B	4.9978	4.9132	0.0846		
400	A	4.9509	4.8104	0.1405	0.1160	88
	B	4.8919	4.8005	0.0914		
600	A	4.8582	4.8032	0.0550	0.0809	92
	B	4.8188	4.7121	0.1067		
800	A	4.9990	4.9451	0.0539	0.0506	95
	B	4.9378	4.8905	0.0473		
1000	A	4.9420	4.8994	0.0426	0.0440	95
	B	5.0503	5.0049	0.0454		

Table 4.2a. Effects of concentration on the corrosion inhibition performance

Blank	A	4.8948	3.8915	1.0033	0.9844	
	B	4.8122	3.8467	0.9655		

MPS Mg/l		Initial weight (g)	final weight (g)	Weight loss (g)	Average (g)	EI (%)
50	A	4.7514	3.5611	1.1903	1.2415	34
	B	4.7941	3.5014	1.2927		
200	A	4.6907	4.2402	0.4505	0.6817	65
	B	4.6366	3.7237	0.9129		
400	A	4.7534	4.4837	0.2697	0.2435	87
	B	4.7287	4.5114	0.2173		
600	A	4.9578	4.6726	0.2852	0.2284	88
	B	4.6082	4.4367	0.1715		
800	A	4.9482	4.7942	0.1540	0.1719	91
	B	4.9059	4.7162	0.1897		
1000	A	5.0639	4.9474	0.1165	0.1077	94
	B	4.8593	4.7605	0.0988		

Table 4.2b.Effects of concentration on the corrosion inhibition performance of *Mimosa pudica* leaf extract in 1.0 M HCl after 168h.

MPL (Mg/l)		Initial weight (g)	final weight (g)	Weight loss (g)	Average (g)	EI (%)
50	A	4.6742	3.6401	1.0341	1.2299	37
	B	5.0203	3.5946	1.4257		
200	A	4.7829	3.8658	0.9171	0.8448	56
	B	4.7522	3.9797	0.7725		
400	A	4.4470	3.7739	0.6731	0.7081	63
	B	5.0434	4.3003	0.7431		
600	A	4.6251	4.0334	0.5917	0.5985	69
	B	4.7023	4.0971	0.6052		
800	A	5.0648	4.6406	0.4242	0.3766	81
	B	4.7062	4.3773	0.3289		
1000	A	4.8786	4.5800	0.2986	0.3399	82
	B	4.7152	4.3341	0.3811		

Blank	A	4.6753	2.8206	1.8547	1.9378	
	B	4.8680	2.8471	2.0209		

Table 4.3a. Effects of concentration on the corrosion inhibition performance of *Mimosa pudica* stem extract in 0.5 M H_2SO_4 after 24h.

MPS (Mg/l)		Initial weight (g)	Final weight (g)	Weight loss (g)	Average (g)	IE (%)
50	A	4.8757	4.5068	0.3689	0.3525	80
	B	4.9548	4.6188	0.3360		
200	A	4.8652	4.7398	0.1254	0.1060	94
	B	4.4018	4.3152	0.0866		
400	A	5.0477	4.9799	0.0678	0.0661	96
	B	4.9903	4.9260	0.0643		
600	A	4.9395	4.9021	0.0374	0.0416	98
	B	4.7533	4.7241	0.0458		
800	A	4.7699	4.7515	0.0184	0.0189	99
	B	4.8058	4.7868	0.0194		
1000	A	4.7118	4.6952	0.0166	0.0164	99
	B	4.8049	4.7887	0.0162		

Table 4.3b. Effects of concentration on the corrosion inhibition performance of *Mimosa pudica* leaf extract in 0.5 M H_2SO_4 after 24h.

MPL (Mg/l)		Initial weight (g)	Final weight (g)	Weight loss (g)	Average (g)	IE (%)
50	A	4.7000	3.9672	0.7328	0.7850	55
	B	4.9173	4.0802	0.8371		
200	A	4.8786	4.5378	0.3408	0.3556	79
	B	4.7630	4.3927	0.3703		
400	A	5.0890	4.9240	0.1650	0.1605	91
	B	4.9641	4.8082	0.1559		
600	A	5.2097	5.0613	0.1484	0.1129	93
	B	4.9082	4.8308	0.0774		
800	A	4.5788	4.5115	0.0673	0.0628	96
	B	4.8628	4.8044	0.0584		
1000	A	4.8680	4.8362	0.0318	0.0441	97
	B	5.0092	4.9528	0.0564		

Blank	A	5.2691	3.4478	1.8213	1.7316	
	B	4.8909	3.2490	1.6419		

Table 4.4a Effects of concentration on the corrosion inhibition performance of *Mimosa pudica* stem extract in 0.5 M H_2SO_4 after 168h.

MPS Mg/l		Initial weight (g)	Final weight (g)	Weight loss (g)	Average (g)	IE (%)
50	A	5.0000	2.9980	2.0020	1.9113	47
	B	4.9311	3.1106	1.8205		
200	A	4.7170	3.7024	1.0146	1.1102	70
	B	4.8971	3.6913	1.2058		
400	A	5.0555	4.4946	0.5609	0.5447	85
	B	5.1351	4.6067	0.5284		
600	A	5.6771	5.3899	0.2872	0.2712	93
	B	4.9888	4.7337	0.2551		
800	A	4.7615	4.5676	0.1939	0.2501	93
	B	4.8738	4.5676	0.3062		
1000	A	4.5434	4.4273	0.1161	0.1196	97
	B	4.7654	4.6424	0.1230		

Table 4.4b Effects of concentration on the corrosion inhibition performance of *Mimosa pudica* leaf extract in 0.5 M H_2SO_4 after 168h.

MPL (Mg/l)		Initial weight (g)	Final weight (g)	Weight loss (g)	Average	IE (%)
50	A	4.7539	2.9489	1.8050	1.8916	48
	B	4.7830	2.8048	1.9782		
200	A	4.5616	2.7946	1.7670	1.8445	49
	B	4.8563	2.9344	1.9219		
400	A	4.2124	2.7075	1.5049	1.6348	55
	B	4.8590	3.0943	1.7647		
600	A	5.2562	3.7181	1.5381	1.4519	60
	B	4.7729	3.4072	1.3657		
800	A	5.0082	4.4721	0.5361	0.5376	85
	B	5.2100	4.6709	0.5391		
1000	A	4.8166	4.3352	0.4814	0.4744	87
	B	4.9450	4.4776	0.4674		

Blank	A	4.7181	0.5869	4.1312	3.6406	
	B	4.4234	1.2735	3.1499		

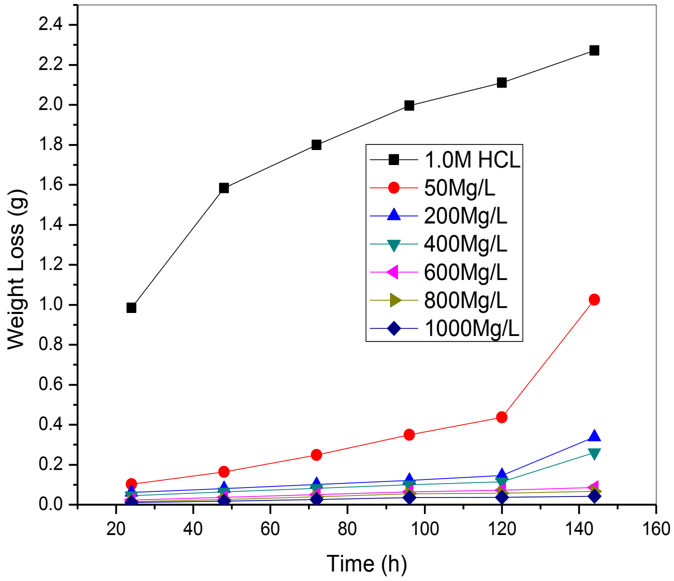


Figure 4.1a: Variation of weight loss with time for the corrosion of mild steel in 1.0M HCl solution containing various concentration of *Mimosa pudica* stem extract.

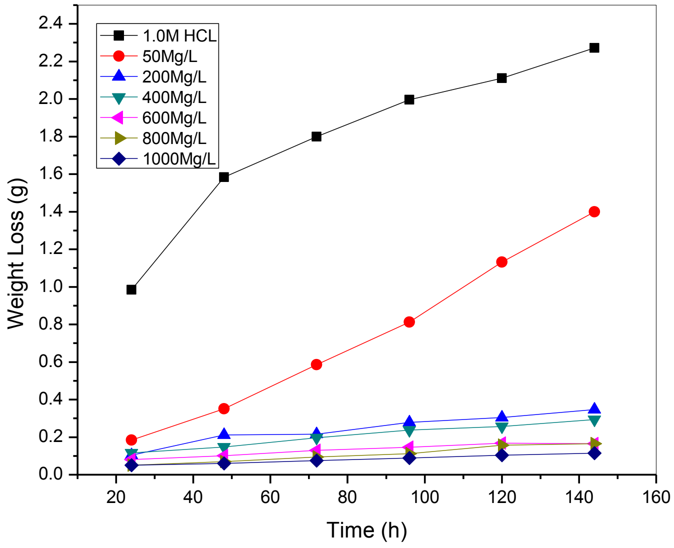


Figure 4.1b: Variation of weight loss with time for the corrosion of mild steel in 1.0M HCL solution containing various concentration of *Mimosa pudica* leaves extract.

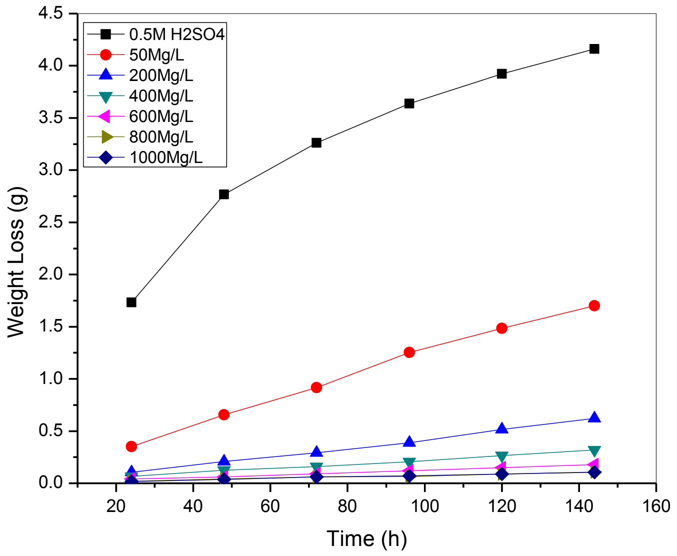


Figure 4.1c: Variation of weight loss with time for the corrosion of mild steel in 0.5 M H_2SO_4 solutions containing various concentration of *Mimosa pudica* stem extract.

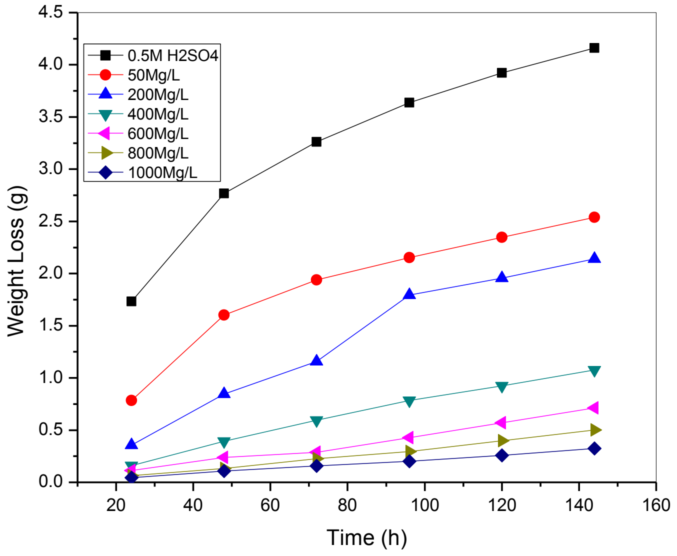


Figure 4.1d: Variation of weight loss with time for the corrosion of mild steel in 0.5 M H_2SO_4 solution containing various concentration of *Mimosa pudica* leaves extract.

4.1.2 INHIBITION EFFICIENCY

Table 4.1a to 4.4d show the inhibition efficiency of mild steel in different concentrations of *Mimosa pudica* leaf and stem extract in 1.0 M HCl and 0.5 M H_2SO_4 solution at different time of immersion. The results show that the inhibition efficiency increases with increase in the concentration of the extract of *Mimosa pudica*, probably due to an increase in the metal surface area covered by the extract.

The optimum inhibition efficiency was found to be 99% at 1000Mg/L concentration of MPS inhibitor in 0.5 M HCL solution and 96% at the same concentration of MPL in the same acidic solution. In 0.5M H_2SO_4, the optimum inhibition efficiency was found to be 99% at 1000Mg/L concentration of MPS extract and 97% at the same concentration of MPL in the same acidic solution.From figure 4.2a to figure 4.2d bellow, it can be deduced that 1000Mg/L of *Mimosa pudica* extract serves as an optimum concentration that exhibit higher efficiency of corrosion inhibition. An increase of inhibitor concentration beyond 1000Mg/L resulted in a steady to diminished corrosion protection. It can also be deduced that inhibition efficiency decreases with time as it is illustrated in figure 4.2e to figure 4.2h. This may be attributed to weakening of metal inhibitor- binding force as a result of biodegradation of the natural extract.

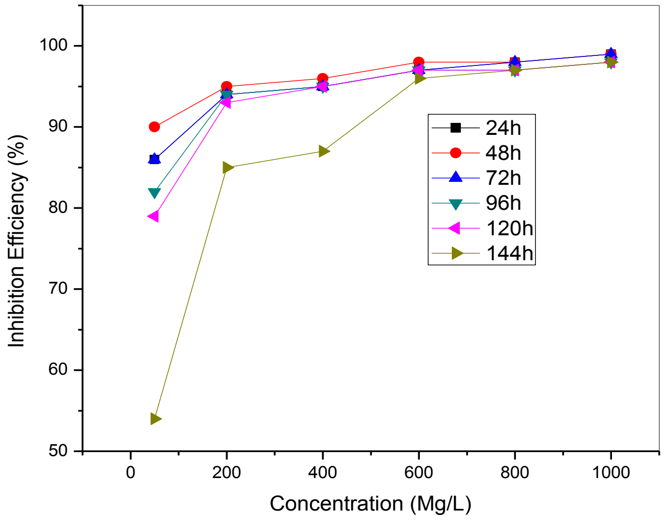


Figure 4.2a: Variation in inhibition efficiency with varying concentration of extract of *Mimosa pudica* stem in 1.0M HCL at various times.

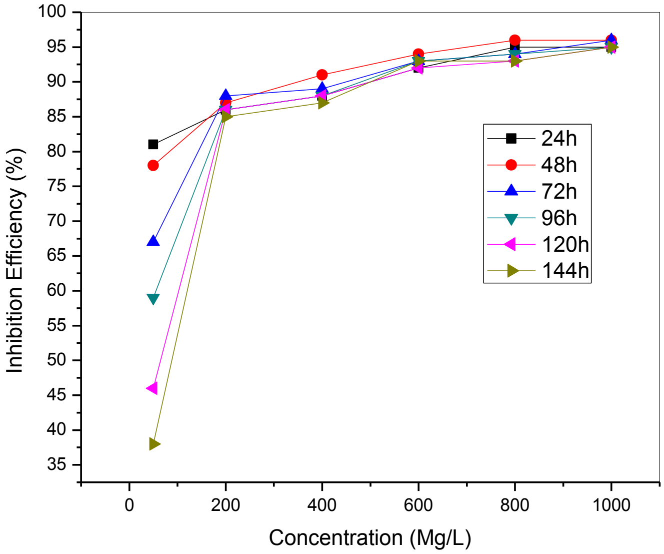


Figure 4.2b: Variation in inhibition efficiency with varying concentration of extract of *Mimosa pudica* leaf in 1.0 M HCl at various times.

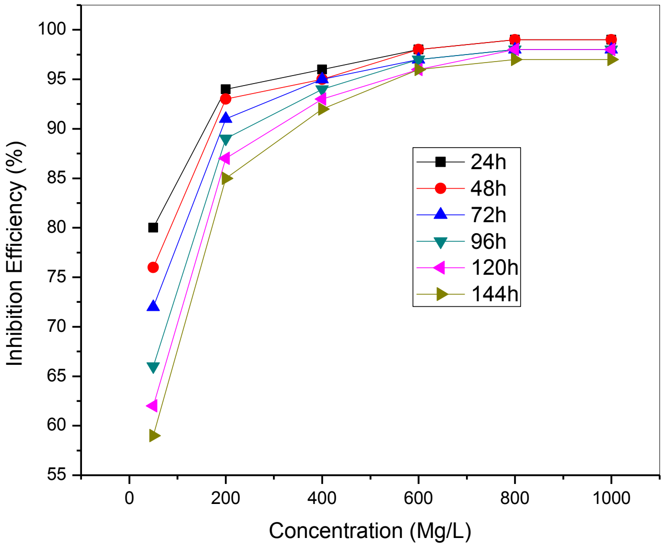


Figure 4.2c: Variation in inhibition efficiency with varying concentration of extract of *Mimosa pudica* stem in 0.5 M H_2SO_4 at various times.

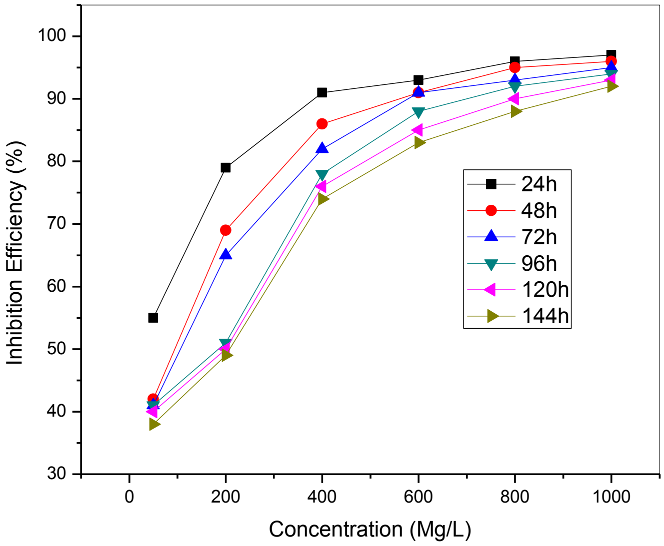


Figure 4.2d. Variation in inhibition efficiency with varying concentration of extract of *Mimosa pudica* leaf in 0.5M H_2SO_4 at various times

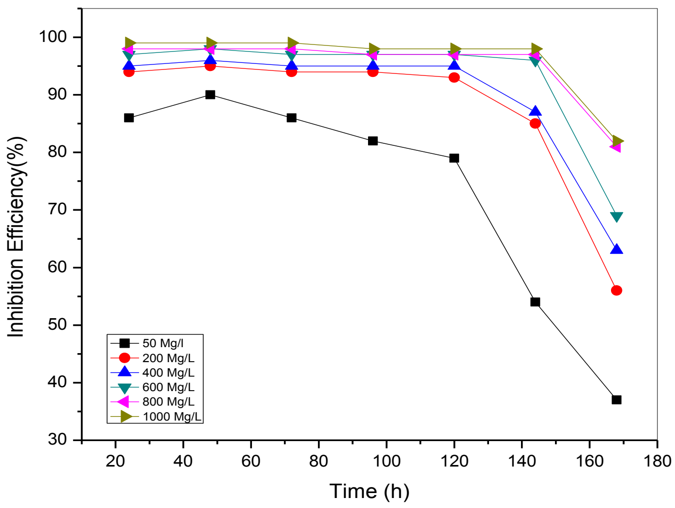


Figure 4.2e. Variation in inhibition efficiency with time at various concentration of extract of *Mimosa pudica* stem in 1.0 M HCl.

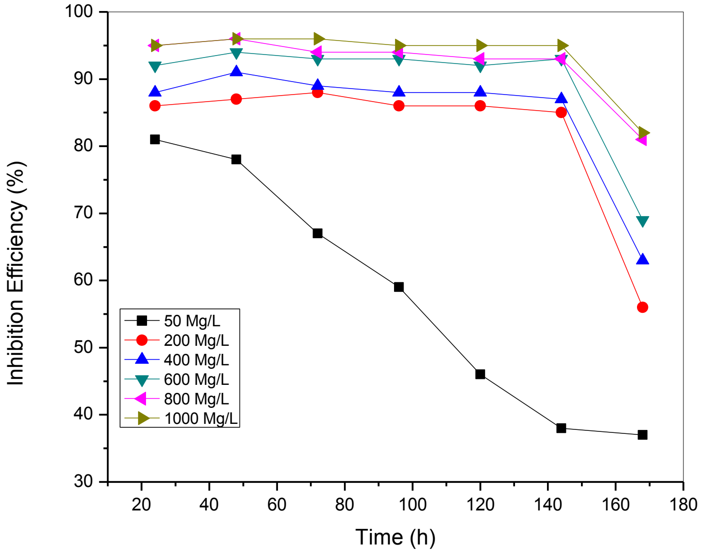


Figure 4.2f. Variation in inhibition efficiency with time at various concentration of extract of *Mimosa pudica* leaf in 1.0 M HCl.

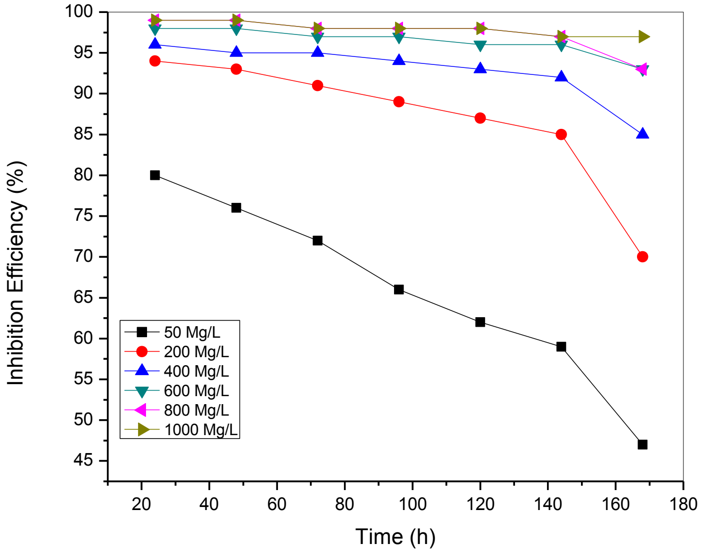


Figure 4.2g. Variation in inhibition efficiency with time at various concentration of extract of *Mimosa pudica* stem in 0.5 M H_2SO_4.

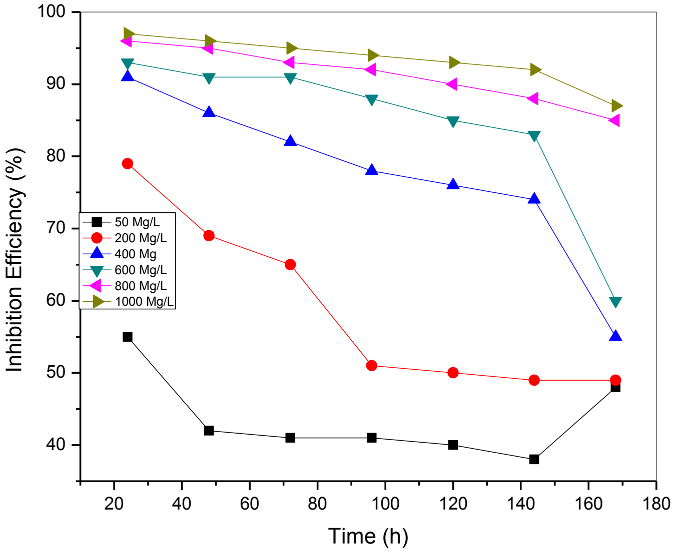


Figure 4.2h. Variation in inhibition efficiency with time at various concentration of extract of *Mimosa pudica* leaf in 0.5M H_2SO_4.

4.1.3 POTENTIODYNAMIC POLARIZATION RESULTS

The effect of *Mimosa pudica* leaf and *Mimosa pudica* stem extract concentration on the anodic and cathodic polarization behavior of mild steel in 1.0 M HCl and H_2SO_4 solution was studied by polarization measurements and the recorded Tafel plots are shown in Figure 4.4a to 4.4d

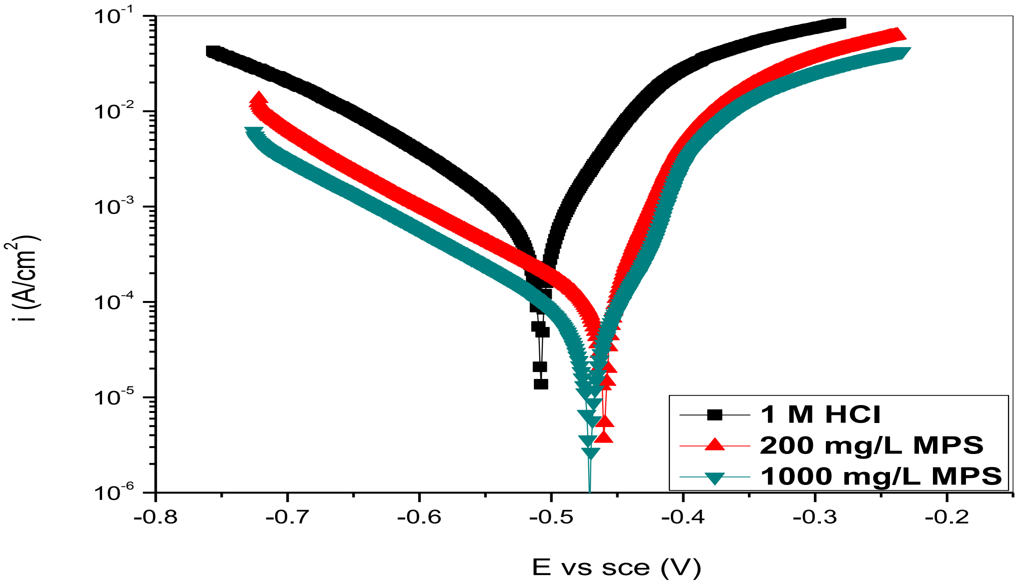


Figure 4.3a: Polarization curves for mild steel in 1.0 M HCl at various concentrations of MPS

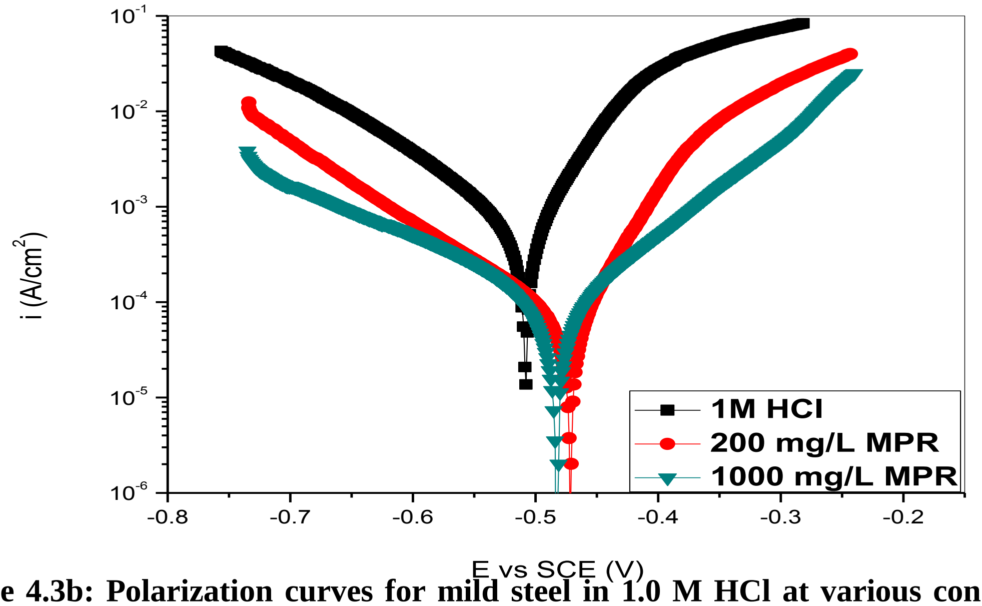


Figure 4.3b: Polarization curves for mild steel in 1.0 M HCl at various concentrations of MPL.

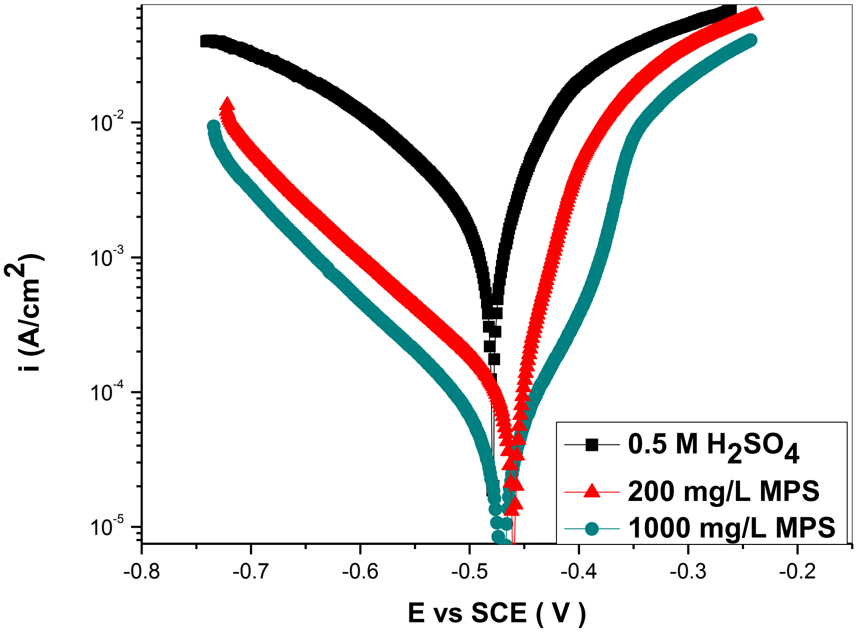


Figure 4.3c: Polarization curves for mild steel in 0.5 M H_2SO_4 at various concentrations of MPS.

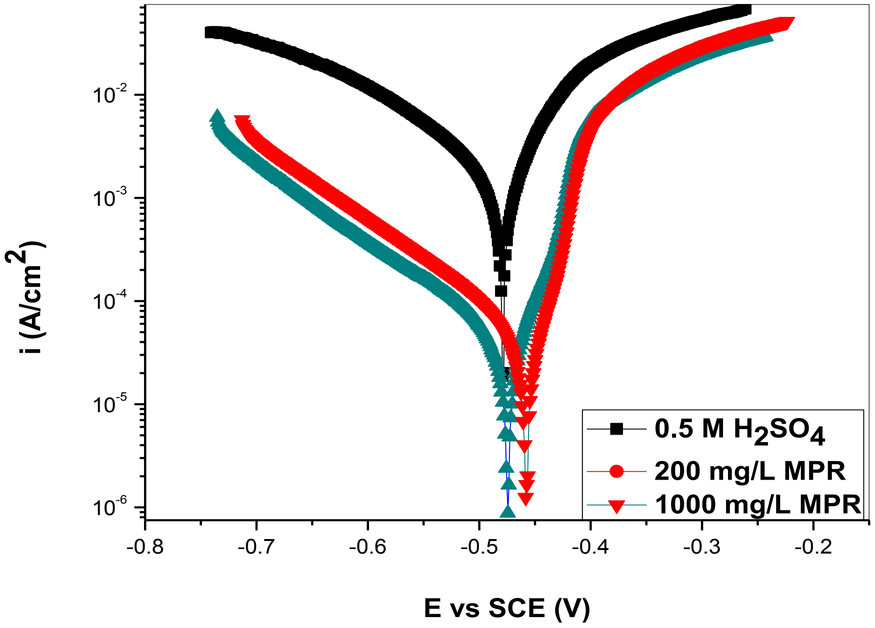
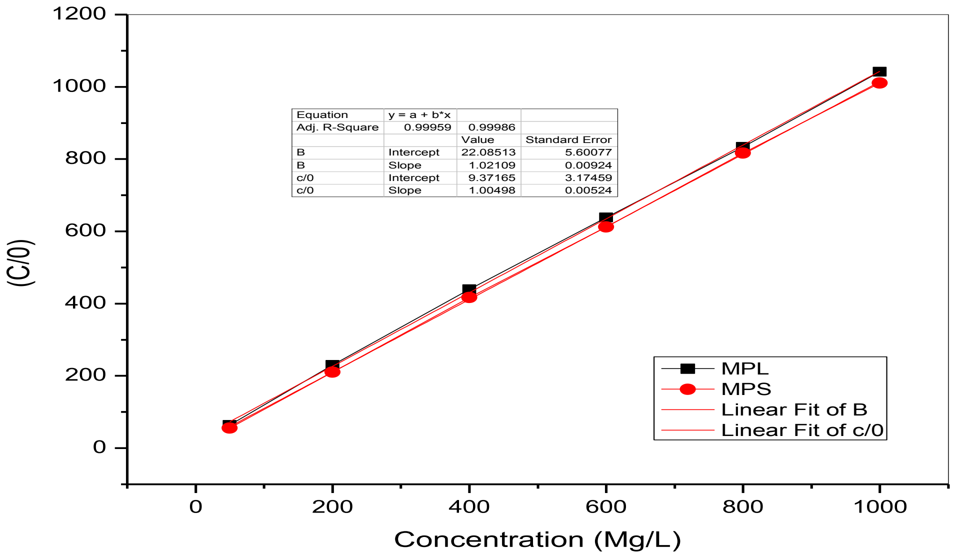


Figure 4.3d: Polarization curves for mild steel in 0.5 M H_2SO_4 at various concentrations of MPL.

It is illustrated from the figure 4.3 that both anodic metal dissolution of iron and cathodic hydrogen evolution reaction were inhibited after the addition of MPS and MPL in 1.0 M HCl and 0.5 M H_2SO_4 solution. The inhibition of these reactions was more pronounced on increasing MPS and MPL concentration. In presence of MPS and MPL, the corrosion potential of mild steel shifted 2-10 mV anodically compared to the blank and also small change in cathodic Tafel slopes were noticed. An inhibitor can be classified as cathodic or anodic type if the displacement in corrosion potential is more than 85 mV with respect to corrosion potential of the blank (Yan *et al,* 2008).This indicates that both MPS and MPL act as mixed-type inhibitor. The cathodic current-potential curves gave rise to parallel lines indicating that the addition of MPL and MPS to both acidic solution did not modify the hydrogen evolution mechanism and the reduction of H + ions at the mild steel surface taken place mainly through a charge transfer mechanism. Although both inhibitors exhibited mixed inhibition(cathodic and anodic),the inhibition of MP is more of anodic than cathodic protection because concentration was noticed as the major factor that controlled its efficiency which does not has much effect on cathodic protection.

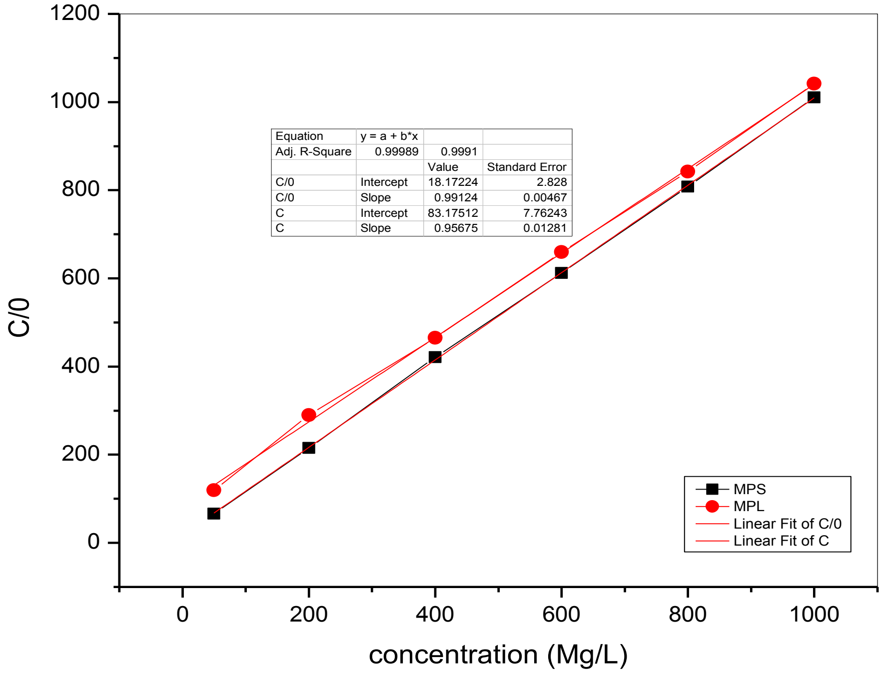
4.1.4 ADSORPTION ISOTHERM

Adsorption of molecules of phytochemical constituents present in the plant extracts on the surface of the metal is responsible for the inhibitive properties of some natural inhibitors (Zakvi and Mehta, 1988). Therefore, it is critical to determine the mechanism of interaction between the phytochemicals in the MPS and MPL extract and the mild steel sample surfaces in the current study. The plot of C/θ versus C is shown in Figure 4.3a to 4.3b, bellow which clearly revealed that the experimental data fitted with the Langmuir adsorption isotherm for the adsorption of MPS and MPL extract on mild steel surface. The results show a clear linear relationship with a regression(R^2)close to unity of 0.9999 and0.9996 for the adsorption of the MPS and MPL respectively in 1.0M HCL solution and a regression of 0.9999 and 0.9991 for the adsorption of MPS and MPL extract respectively in 0.5M $H_2SO_{4.}$. This proves that Langmuir isotherm model is appropriate for the adsorption of the extract of MPS and MPL onto the metal surface in both acidic solutions. A higher coverage of the MPS and MPL on the surface was obtained in solutions with the higher concentrations.



Equation	y = a + b*x		
Adj. R-Square	0.99959	0.99986	
		Value	Standard Error
B	Intercept	22.08513	5.60077
B	Slope	1.02109	0.00924
c/0	Intercept	9.37165	3.17459
c/0	Slope	1.00498	0.00524

Figure.4.4a: Langmuir adsorption isotherm plot of C/θ against C for MPL and MPS 1.0MH HCl.



Equation	y = a + b*x		
Adj. R-Square	0.99989	0.9991	
		Value	Standard Error
C/0	Intercept	18.17224	2.828
C/0	Slope	0.99124	0.00467
C	Intercept	83.17512	7.76243
C	Slope	0.95675	0.01281

Figure 4.4b: Langmuir adsorption isotherm plot of C/θ against C for MPS and MPL in 0.5 M H_2SO_4.

4.1.5 EVALUATION OF REGRESSION MODEL FOR INHIBITION EFFICIENCY (%IE)

The correlation between the experimental process variables and the inhibition efficiency was evaluated using the central composite modeling technique. A linear regression equation fitted between the response (inhibition efficiency) and the process variables: extract concentration and time.

For MPS and MPL extract in 1.0 M HCl as indicated in ANOVA tables in appendix X and XI, the model F-valve of 7.31 and 6.11 imply that the models are significant. Also the Prob >F less than 0.0500 indicates model terms are significant. In this case, concentration is significant model term as value greater than 0.1000 indicate the model terms are not significant.

Final equation in terms of actual factors

MPS EI = +88.12495+0.015477A- 0.032329B 4.1

MPL EI =+78.39453+0.0020080A - 7.81250B-003 4.2

For MPS and MPL extract in 0.5 M H_2SO_4 as indicated in ANOVA tables in appendix XII and XIII, the model F-valve of 4.71 and 17.11 imply that the models are significant. Also the Prob >F less than 0.0500 indicates model terms are significant. In this case, concentration is significant model term as value greater than 0.1000 indicate the model terms are not significant

Final equation in terms of actual factors

$$\text{MPS IE} = +80.36122 + 0.025512A - 0.033407B \qquad 4.3$$

$$\text{MPL IE} = +61.26986 + 0.048392A - 0.099328B \qquad 4.4$$

The interactive effects of the process variables on the percentage inhibition efficiency were studied by plotting three dimensional surface curves against any two independent variables, while keeping other variables at their central (0) level. The 3D curves of the response (% inhibition efficiency) and contour plots from the interactions between the variables are shown for each inhibitor on mild steel in different acidic solution in figure 4.6a to 4.6d. From the interactive effects of extract concentration and time, it was observed that inhibition efficiency of mild steel increase in both solutions with increase in extract concentration. The optimum predicted values with the experimental data agree closely as shown in figure 4.5a to 4.5d. The high level of correlation between the predicted and experimental response proved the regression model to be adequate explaining the variations in the experimental data.

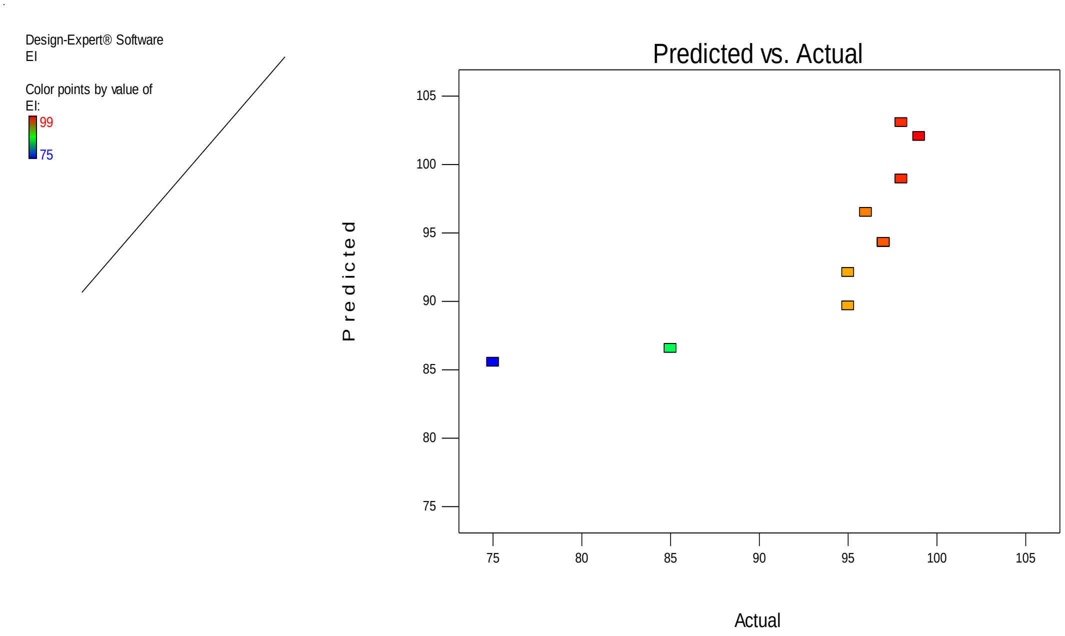


Figure 4.5a- Plot of predicted values versus the actual experimental values for inhibition of MPS on Mild steel in 1.0M HCl

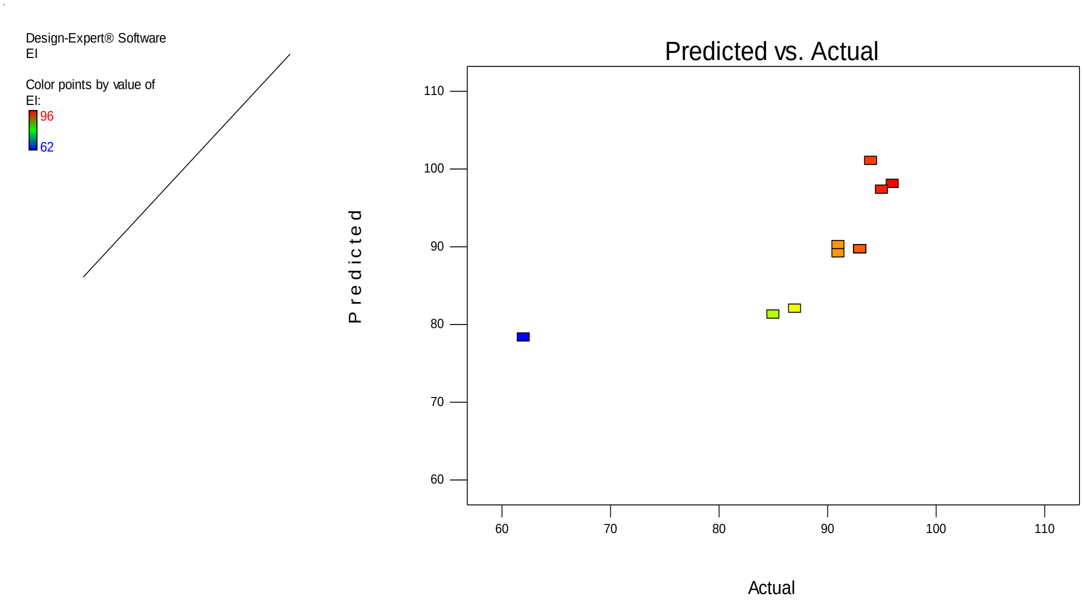


Figure 4.5b- Plot of predicted values versus the actual experimental values for inhibition of MPL on Mild steel in 1.0M HCl

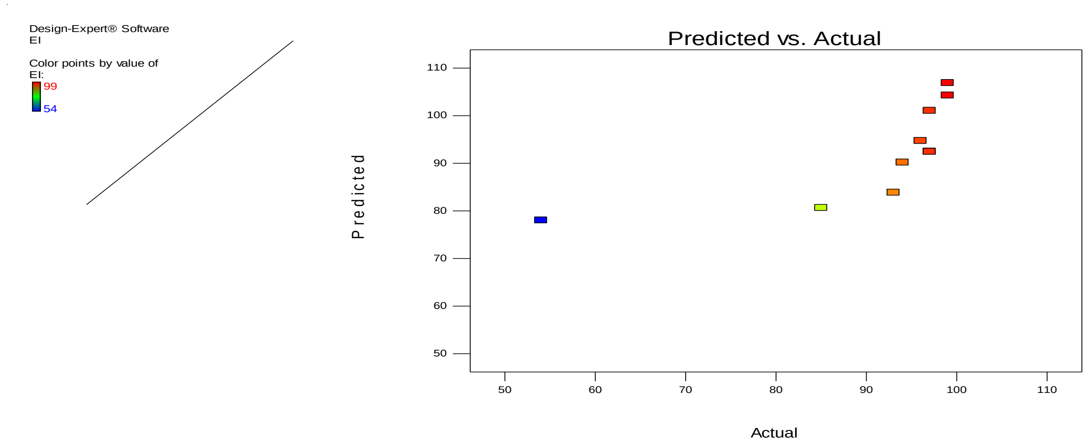


Figure 4.5c Plot of predicted values versus the actual experimental values for inhibition of MPS on Mild steel in 0.5M H_2SO_4

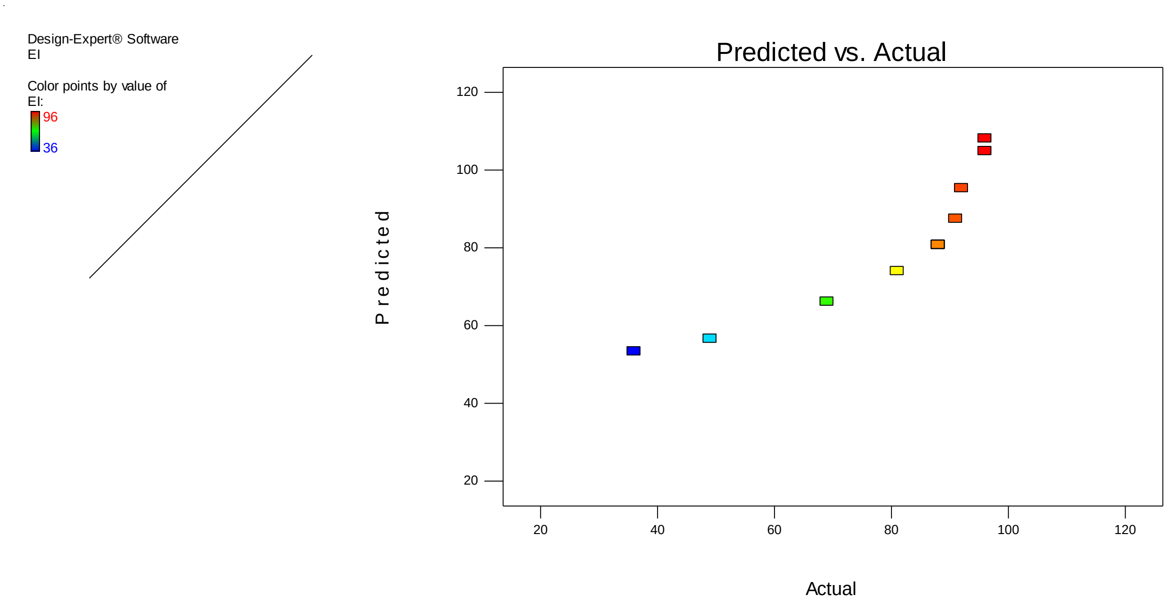


Figure 4.5d- Plot of predicted values versus the actual experimental values for inhibition of MPL on Mild steel in 0.5M H_2SO_4

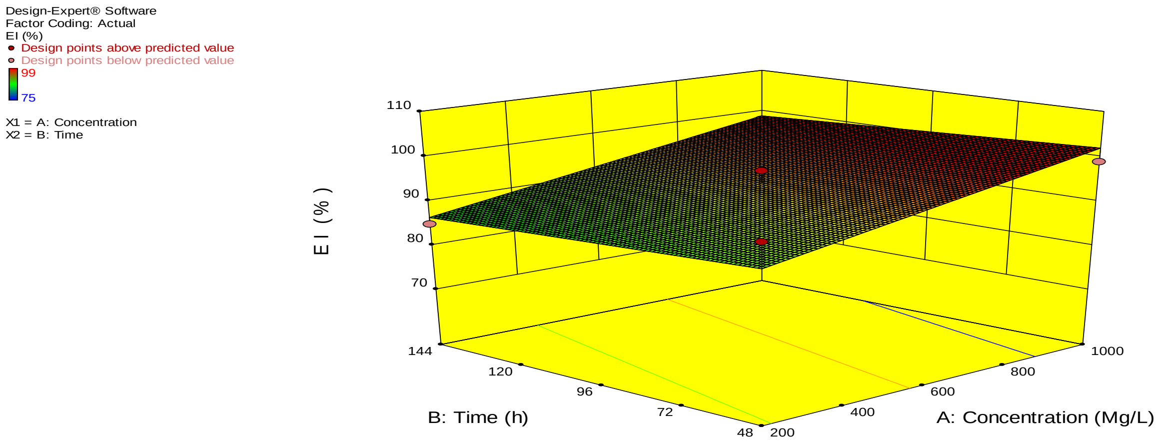


Figure 4.6a: Effects of extract concentration and time on inhibition efficiency of MPS extract on mild steel in 1.0 M HCl.

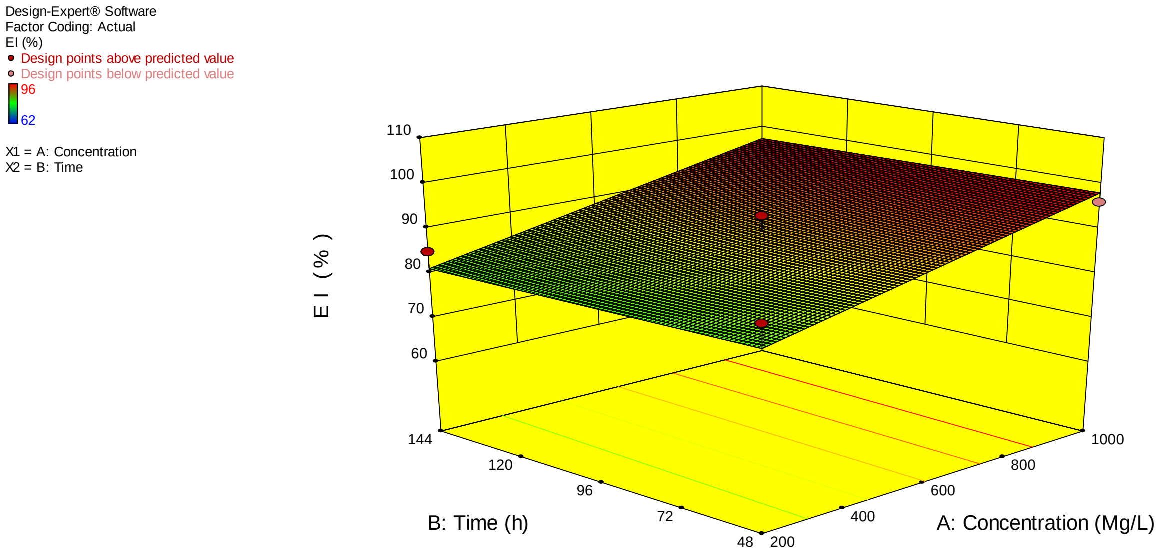


Figure 4.6b: Effects of extract concentration and time on inhibition efficiency of MPL extract on mild steel in 1.0 M HCl.

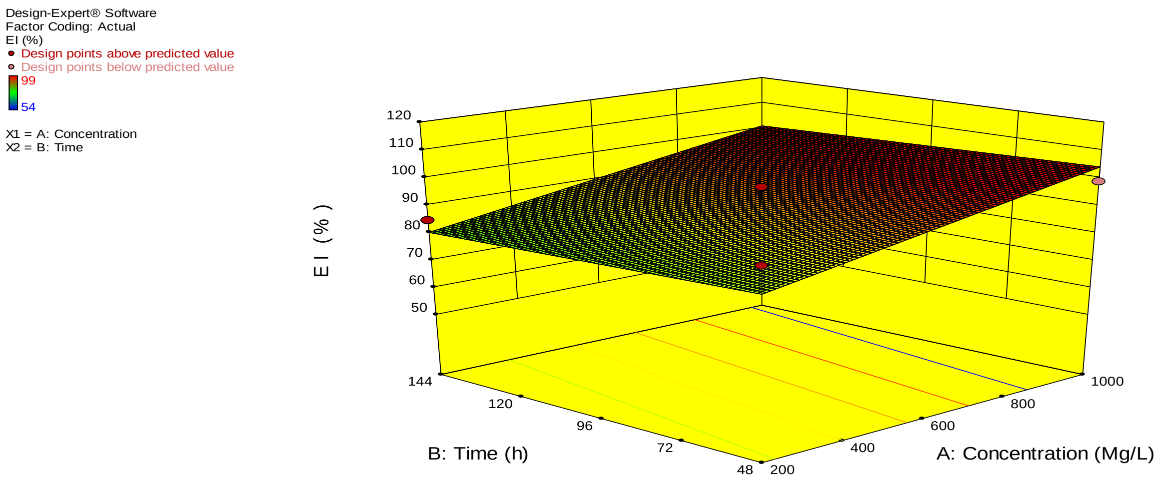


Figure 4.6c: Effects of extract concentration and time on inhibition efficiency of MPS extract on mild steel in0.5M H_2SO_4

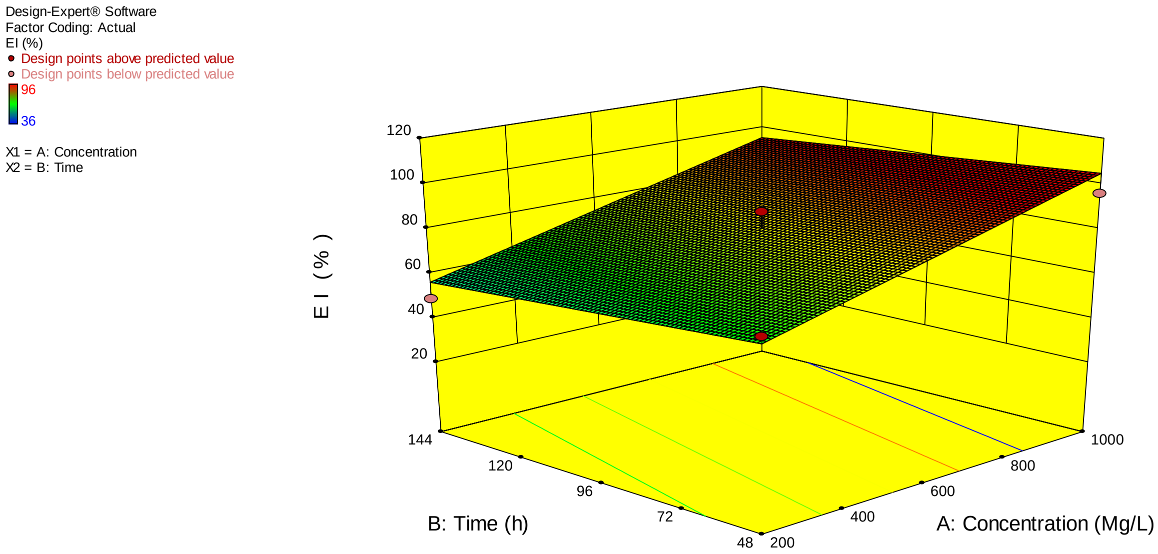


Figure 4.6d: Effects of extract concentration and time on inhibition efficiency of MPL extract on mild steel in 0.5M H_2SO_4.

4.2 DISCUSSION

WEIGHT LOSS RESULTS

The weight loss results clearly show a reduction in weight loss of the metal coupons in the presence of these inhibitors compared to the blank solution. It also revealed that the loss in weight of the coupons decreases as the concentration of the inhibitors increases and increases with increase in time. This observation is in line with the findings of several authors on the anti corrosion performance of plant extracts (Obi-Egbedi,*et al,* 2012, Rani and Basu, 2012, Belkhaouda, *et al* 2013,Bardal, 2004 and Dutra, *et al* 2011).it is also in line with a work according to Lame *et al.* (2013) which revealed that ethanolicextracts of *Chlomolaena odorata* functioned as corrosion inhibitor for mild steel in H_2SO_4 solution and its efficacy increased with the extracts concentration.

The result of this research clearly revealed that both inhibitors tend to perform more in 1.0 M HCl than in 0.5 M H_2SO_4 owing to the presence of chlorine which has the ability to detach hydroxyl ion in metal thereby increasing the negativity of the metal, to that, neutral inhibitors will act more on more negative solution (Gunasekaran and Chauhan, 2004).

A high significance inhibitory property shown by both inhibitors is due to formation of passive film on the metal surface. The synergistic combination of all the phytochemical constituents could be responsible for the passivity and hence very good inhibition in the test medium. (Salami *et al*, 2012).

INHIBITION EFFICIENCY

The results show that the inhibition efficiency increases with increase in the concentration of the extract of *Mimosa pudica*, probably due to an increase in the metal surface area covered by the extract. As the concentration of the inhibitor increases, the amount of phytochemical constituents absorbed onto the surface of the metal also increases, thereby increasing the efficiency of the inhibitor at higher concentrations (Rani and Selvara, 2011).Low concentration of inhibitor affects the formation of film protection because it will not cover the metal completely, leaving sites of the metal exposed, thus causing localized corrosion (Dutrae *etal*, 2011). An increase of inhibitor concentration beyond 1000Mg/L resulted in a steady to diminished corrosion protection. This may be due to the withdrawal of adsorbate (inhibitor) back into the bulk solution when the concentration of inhibitor closed to or beyond the critical concentration (Satapathy *etal*, 2009). The above effect leads to the weakening of metal-inhibitor interactions, resulting in the replacement of inhibitor by water or chloride ions (Cl^-) with decrease in inhibition efficiency (Gunasekaran and Chauhan, 2004)

From this research results, it can be deduced that inhibition efficiency decreases with time. This may be attributed to weakening of metal inhibitor- binding force as a result of biodegradation of the natural extract. This observation is in accordance with the work by Obi-Egbedi et al. (2012) which revealed that most natural corrosion inhibitors have low stability and are readily biodegradable.

This study also revealed that *Mimosa pudica* stem (MPS) exhibits higher corrosion protection than Mimosa pudica leave (MPL).This may be attributed to higher electron density of the former than the later as it is revealed according to Oezcan et al. (2008) that inhibition efficiency may be affected by electron density which favours its adsorption on the metal surface.

POTENTIODYNAMIC POLARIZATION RESULTS

The polarization curves illustrate that both MPS and MPL exhibited a mixed protection (anodic and cathodic protection).This observation is in accordance with the work according to Gentil and Corrsao,(2003) which revealed that in general, inorganic inhibitors have cathodic or anodic action but organic have both actions. Although both inhibitors exhibited mixed inhibition(cathodic and anodic),the inhibition of MP is more of anodic than cathodic protection because concentration was noticed as the major factor that controlled its efficiency which does not has much effect on cathodic protection. According to Gentil,(2003) cathodic inhibitors is independent of concentration. The cathodic current-potential curves gave rise to parallel lines indicating that the addition of MPL and MPS to both acidic solution did not modify the hydrogen evolution mechanism and the reduction of H + ions at the mild steel surface taken place mainly through a charge transfer mechanism. The MP molecules were first adsorbed on the mild steel surface and blocked the reaction sites of the mild steel surface. In this way, the surface area available for H + ions was decreased while the actual reaction mechanism remains unaffected. These observations are in line with the findings of several authors on the anti corrosion performance of plant extracts (Solmaz *et al*, 2008, Liu, *et al* 2013 ,Khaled, 2003 Galtayries, et al 2007, Sastri, 2011,Bastidas, et al 2002 andBastidas, et al, 2005).

ADSORPTION ISOTHERM

Adsorption analysis results show a clear linear relationship with a regression (R^2) close to unity. High adsorption potential of the two inhibitors are attributed to the presence of polar functional groups, S, O_2 and or N atoms in the MP molecules which is usually regarded as the reaction centre for the establishment of adsorption process (Apreal et al, 2013).The compounds interact by mutual repulsion or attraction and this may be advocated as the reason for the departure of the slopes values from unity. A higher coverage of the MPS and MPL on the surface was obtained in solutions with the higher concentrations.

CHAPTER FIVE

CONCLUSION AND RECOMMENDATION

5.1 SUMMARY OF FINDINGS

From the results obtained from this research, the protection efficiency of the inhibitors was found to increase with increase in concentration of the inhibitor showing an optimum inhibition efficiency of 99% at 1000Mg/L concentration of MPS inhibitor in 1.0 M HCl solution and 96% at the same concentration of MPL in the same acidic solution .In 0.5M H_2SO_4, the optimum inhibition efficiency was found to be 99% at 1000Mg/L concentration of MPS extract and 97% at the same concentration of MPL in the same acidic solution.

Also from the results, MPS tends to be more efficient than MPL which can be attributed to MPS having higher electron density than MPL which favors its adsorption to the metal surface. The results obtained from the polarization studies reveal that the extracts behaved as a mixed type of inhibitor with the displacement in corrosion potential less than 85 mV with respect to corrosion potential of the blank. The adsorption behavior of the extract is consistent with Langmuir adsorption model with a regression (R^2) close to unity of 0.9999 and 0.9996 for the adsorption of the MPS and MPL respectively in 1.0M HCL solution and a regression of 0.9999 and 0.9991 for the adsorption of MPS and MPL extract respectively in 0.5M H_2SO_4..

The study also reveals that inhibition efficiency decreases with time which maybe as a result of weakening of metal- inhibitor binding force as a result of biodegradation of the natural extract.

CONCLUSION

This study has revealed that the ethanol extract of *mimosa pudica* leaf and stem behaved as potent inhibitors to mild steel corrosion in 1.0 M HCl and 0.5M H_2SO_4.

A number of alternatives of environmentally friendly corrosion inhibitors are currently emerging oriented towards minimizing environmental impact providing effective corrosion inhibition. *Mimosa pudica* extract has a promising potential as an alternative eco-friendly, non-toxic, readily available inhibitor to replace the non-biodegradable, toxic and expensive synthetic chemicals, which are currently being in use**.** I hope that these products will be able to replace, in the near future, the toxic commercial products that are still being used by many industries worldwide.

5.2 RECOMMENDATION

In order to maintain the promising potential offered by Mimosa pudica leaf and stem extract as good alternative eco-friendly corrosion inhibitor, there is the need for further research on a way to increase its stability as it has been observed from this study that it loses its efficiency with time. Consequently, the addition of biocides such as N-cetyl-N,N,N-trimethyl ammonium bromide to reduce its rate of degradation is recommended.

There is also the need for further research on the effect of temperature on Mimosa pudica extract corrosion inhibition efficiency.

REFERENCES

Ahmad, I. and Beg, A. Z. J. (2001) “Antimicrobial and phytochemical studies of 45 Indian medicinal plants against multidruges resistance human pathogens” *J. Ethnopharmaco;* 74:113-23.

Ajayi, O.O., Omotosho, O.A., Ajanaku, K.O. and Olawore, B.O. (2011). “Failure evaluation of aluminium alloy in 2M HCL in the presence of Cola acuminate,” *Environmental Research Journal*; 5(4):455-462.

Applewhite, P.B. (1972), “Behavioral plasticity in the sensitive plant, *Mimosa”,*Behavioral Biol, 7:47.

Aprael, S., Yaro, Anees, A., Khadom, Rafal, K., and Wael, (2013). “Apricot juice as green corrosion inhibitor of mild steel in phosphoric acid,” *Alexandria Engineering Journal*, vol 52, issue 1, pp. 129-135.

Argyropoulos, V., Giannoulaki, M., Michalakakos, G.P.and Siatou, A (Eds) (2007). “Strategies for saving our cultural heritage”,166-170; Cairo, Athens, TEI of Athens.

Bastidas, J.M and Otero, E.(1996). “*Materials and Corrosion - Werkstoffe und Korrosion”*, 1996, **47**, (6), 333-337; Scotland UK.

Bastidas, D.M., Cano, D and Mora, E.M. (2005), “*Anti-Corrosion Methods and Materials”*, 2005, **52**, (2), 71-77; Emerald group.

Bastidas, D.M., Gómez, P.P.and Cano, E. (2005), “*Revista de Metalurgia (Madrid)”*,2005, **41**, (2), 98-106.

Belkhaouda M., Bammou L., Zarrouk A., Salghi R., Ebenso E.E., Zarrok H., Hammouti B, Bazzi. L., Warad, I. (2013). “Inhibition of C-steel corrosion in hydrochloric solution with *Chenopodium ambrorsioides* extract”, *Int. J. Electrochem.* Sci. 8:7425-7436.

Brostoff, L.B. (Eds) (1995). “Metal 95. Proceedings of the International Conference on Metals Conservation”, 99-108;Semur-en-Auxois, France:James and James.

Cano, E., Polo, J.L., Iglesia, A.L.A. and Bastidas, J.M. (2004). “Adsorption study of the benzotriazole on copper in hydrochloric acid using inflection point of the isotherm,*”Solid State Electrochem*, **10**, (3), 219-225.

Cano, E., Lafuente, E. and Bastidas, D.M. (2010). *“*Use of EIS for the evaluation of the potential properties of coating for metallic cultural heritage*,”Solid State Electrochem.*, **14**, (3), 381-391.

Cano, D. M. Bastidas, J. Simancas, J. A. López-Caballero and J. M. Bastidas. (2004).“ Chemical composition of the copper/benzotrazole interface in the presence of acetic acid vapoure at low humudity”,*Sage journal ofAdsorption Science and Technology”*, **22**, (7), 565-582.

Carla, W. M. (2002). “Environmental geology” fifth edition. McGraw Hill Higher Education, U.S A.308p.

Chauhan, R., Garg, U. and Tak, R.K. (2011). “Corrosion inhibition of aluminium in acidic media by *Citrullus colocynthesis* extract,”*E-Journal of Chem*, 8(1), 85-90.

Chatterjee, J. and Chatterjee, C. (2000). “Phytotoxicity of cobalt, chromium in cauliflower”, Environmental pollution; vol 109, No: 1, pp 69-74.

Cho, Y., Seo, S., Choi, S.H., Lee, S., Kim, K. and Kim, H.J. (2013). “ Association of arsenic level in soil and water with urinary arsenic concentration 0f residents in the vicinity of close metal mines,” *International journal of hygiene,environment and health* ;216:255-262.

Cutler, C. and Saundry, P.(Eds) (2008). “Exxon Valdez Oil Spill”, In Encyclopaedia of Earth,Washington, D.C. Environmental Information Coalition, National Council for Science and Environment.

Das, S., Pal, S., Mujib, A. and Dey, S. (1999). “Biotechnology of medicinal plants-recent advances and potential,” first edition. Vol II (UK992 Publications, Hyderabad), 126-139.

D'Ars,F.J.,De Bellis,V.M.,Lins,Vanessa,F.C and Souza,A.L.C. (2007). “A note on the products of the reaction of ATM with bronze and with three corrosion products of broze”,*Studies in Conservation”*, **52**, (2), 147-153.

Ding, Y.M., Xu, C.C.and Wang, J.L. (2006), “Beijing huagong daxue xuebao*”, (Ziran Kexueban)/Journal of Beijing University of Chemical Technology (Natural Science Edition)*, **33**, (2), 68-71.

Dowsett, M., Adriaens, A., Schotte, B., Jones. and Bouchenoire, L.(2009). “Surface and Interface Analysis*”*, Journal of Analytical Atomic Spectrometry,**41**, (7), 565-572.

Dutra e, A.C and Nunes, L.D.P.(2011). "Proteção catódica técnicas de combate a corrosão", 5 ed.Rio de Janeiro: interciências, 2011.

Enujiugha, V.N. and Nwanna, I.C. (2004). "Aquatic Oil Impact Indicators", *Journal of Applied Sci. Environ. Mgt*. Vol. 8(2) 71-75.

Fang,B.S., Olson, C.G. and Lynch,D.W. (1986)."A photoemission study of benzotraizole on clean copper and cuprous oxide",*Surface Science*,**176** (3), 476-490.

Gabler, C., Tomastik, C., Brenner J, Pisarova, L., Derr, N. and Allmaier, G. (2011). "Corrosion properties of ammonium based ionic liquids evaluated by SEM-EDX, XPS and ICP-OES". *Green Chemistry*,13 (**10**) 2869-2877.

Galtayries, A., Mongiatti,A., Marcus, P. and Chiavari, C. (Eds)(2007). "Corrosion of metallic heritage artefacts," Vol. , 335-351;Cambridge,England: Woodhead Publishing.

Gandhiraja, N., Sriram, S., Meenaa, V., Srilakshmi, J.K., Sasikumar, C., Rajeswari, R. (2009). "Phytochemical sreening and antimicrobial activity of the plant extract of Mimosa pudicaleaf against selected microbes," Ethnobotanical *Leaflets*, 13: 618-24.

Gece, G. (2008). "The use of quantum chemical methods in corrosion inhibitor studies,"*Corrosion Science*, 2008, **50**, (11), 2981-2992.

Gentil, V.(2003), "Corrosão" 4th ed., Rio de Janeiro: LTC.

Gordon, J. J. and Quastel, G. H. (1948). "Effect of organic arsenicals on enzyme system*" Biochem. J.* 42, pp 337-350.

Gunasekaran, G. and Chauhan, L.R. (2004)."Eco-friendly inhibitor for corrosion inhibition of mild steel in phosphoric acid medium*," Electrochim Act*, 49, 4387.

Hajjaji, N. (Eds) (2007).*"*Strategies for Saving our Cultural Heritage", 138-142; Cairo, Athens: TEI of Athens.

Human Rights Watch, (HWR). (1999), "Cooperate responsibility and human rights violation in Nigeria's oil producing communities," New York, America.

IPCS. (2001). “Arsenic and arsenic compounds”, 2nd ed. Geneva, World Health Organization, International Programme on Chemical Safety (Environmental Health Criteria 224; http://whqlibdoc.who.int/ehc/WHO_EHC_224.pdf).

ISO 8044. (1999). “Corrosion of metals and alloys” -- Basic terms and definitions'.

Khaled, K.F. (2003). “The inhibition of benzimidazole derivatives on corrosion of iron in 1 M Hcl solutions”. *Electrochimica Acta*;48: 2493-2503.

Khalil, N.(2003).“Quantum chemical approach of corrosion,”*Electrochimica Acta*,**48**, (18), 2635-2640.

Kokalari E. and Jano A. (2013). “Use of green inhibitors for concrete armor protection Against H_2SO_4 Corrosion”, *Asian Journal of Chemistry*;25(7) 4017-4021.

Koch, G.,Varney, J., Thompson, N., Moghissi, O., Gould, M. and Prayer, J. (2016). “International measures of the prevention, application and economics of corrosion technologies study. NACE International.

Koch, G.H., Brongers, M.P.H., Thompson, N.G., Virinani, Y.P. and Prayer, J.H. (2002). “Corrosion costs and prevention strategies in the United State. Federal Highway Administration, Mclean, V.A.

Krishnaraju, A.V., Rao, T.V.N., Sundararaju, S., Vanisree, M., Tsay, H.S., Gottumukkala, V. and Subbaraju, G. (2006).“Assessment of bioactivity of Indian medicinal plant using brime shrimp(*Artemia salina*)lethality assay,” *Int. J. Appl. Sci. Eng*., 3(2): 125-134.

Lame, A. and Oezcan, M. (2008). “AC impedance measurements of cysteine adsorption at mild steel/sulphuric acid interface as corrosion inhibitor”,*J. Solid State Electrochemistry*; 12(12) 1653-1661.

Likhanova, N.V., Martínez-Palou, R., Veloz, M.A., Matías, D.J., Reyes-Cruz, V.E and Olivares- Xometl, O. (2007). “Microwave-assisted synthesis of 2-(2-pyridyl) azoles: Study of their corrosion inhibiting properties”, *Journal of Heterocyclic Chemistry;* 44(1) 145-153.

Liu, G., Huang, J., Zhou, Y., Yao, Q., Wang, H, Ling, L., Zhang, P., Cao, K., Liu, Y., Wu, W. and Sun,W. (2013). “Carboxylate-terminated doublehydrophilic block copolymer containing fluorescent groups: An effective and environmentally friendly inhibitor for calcium carbonate scales. *International Journal of Polymeric Materials and Biopolymeric Materials* 62(13) 678-685.

Lozoya, M. and Lozaya, X. (1989). “Weed plants and its uses”, *Tepescohuite Arch*”. Invest. Mex., pp. 87-93.

Marchiset, F.N., Savanovitch, C. and Sauvant R.M.P. (2012). “What is the best biomarker to assess arsenic exposure through drinking water? *International journal on environment;* 39(1) :150 -171.

Markin, V.S., Volkov A.G. and Jovanor, E. (2008). “Active movements in plant: Mechanism of trap closure by *Dionaea muscipula ellis”* .Plant signalling and behaviour. 3:778-783.

Martinez, S. and Stern, I. (2002). “Thermodynamic characterization of metal dissolution and inhibitor adsorption processes in the low carbon steel/mimosa tannin/sulfuric acid system”, *Applied Surface Science,* **199**, (1–4), 83-89.

MERCK, (2007). Indigenous Plants to the Rescue. Science in Africa. Working paper 3.

Mohamed, W.A., Rateb, N.M. and Shakour, A.A.(2004).“Metal 04: Proceedings of the International Conference on Metals Conservation”, 369-378; Canberra, Australia, National Museum of Australia.

Mahmoud, N. and El-Haddad (2013). “Chitosan as a green inhibitor for copper corrosion in acid- ic medium,” *International Journal of Biological Macromolecules*, vol. 55, pp 142-149, 2013.

Nwilo, P.C. and Badejo, O. (2007). “Impact of oil spills Along the Nigerian Coast”.Dept. of Surveying and Geo informatics, Faculty of Engineering, University of Lagos Akoka-Lagos, Nigeria.

Oguzie, E.E.,Onuchukwu, A.I., Okafor,P.C. and Ebenso, E.E.(2006). “Corrosion inhibition and adsorption behaviour of *ocimum basilicum* extract on aluminium”,Pigment and resin technology,vol 35 issue 2 pp 63-70.

Oguzie,E.E., Okolie,B.N., Ebenso, E.E., Onuoha, G.N. and Onuchukwu, A.I. (2004). "Evaluation of the inhibitory effect of methylene blue dyes on the corrosion of aluminium in hydrochloric acid",Matter chem. Phys;87:pp 394-401.

Oguzie, E.E. (2008). "Evaluation of the inhibitive effect of some plant extracts on the acid corrosion of mild steel", Corrosion science; 50(11) pp 2993-2998.

Oguzie, E.E., Enenebeaku, C.K., Akalezie, C.O., Okoro,S.C., Ayuk,A.A. and Ejike,E.N. (2010). "Adsorption and corrosion inhibition effect of *Dacryodis edulis* extract on low carbon- steel corrosion in acidic media";*International journal of colloid interface science*,34:283-292.

Oguzie,E.E.,Chidiebere,M.A.,Oguzie,K.L.,Adindu,C.B andMomoh-Yahaya,H.(2014), "Biomass extracts for materials protective corrosion inhibition of mild steel in acedic media by terminalia chebula extracts",Chemical engineering communication,vol 201,No 6,pp.790-803.

Oguzie,E.E.,Adindu C.B.,Enenebeaku,C.K.,Ogukwe, C.E. and Chidiebere, M.A. (2012). "Natural products for materials protection: Mechanism of corrosion inhibition of mild-steel by acid extracts of *Piper guineense",Journal of physical chemistry,*C 116(25);13603-13615.

Obi-Egbedi, N.O., Obot, I.B., Obot, I.B and Umoren, S.A.(2012). "*Spondias mombin L.* as a green corrosion inhibitor for aluminium in sulphuric acid: Correlation between inhibitive effect 455 and electronic properties of extracts major constituents using density functional theory". *Arabian Journal of Chemistry*;5(3) 361-373.

Obot, I.B., Obi-Egbedi, N.O., Umoren, S.A. and Ebenso, E.E. (2010). "Synergistic and antagonistic effects of onions and Ipomoea invulcrata as green corrosion inhibitor for aluminium dissolution in acidic medium",*International Journal of Electrochemical Science*;5(7) 994-1007.

Obot, I.B., Umoren, S.A. and Obi-Egbedi, N.O. (2011). "Corrosion inhibition and adsorption behavior for aluminium by extract of Anigeria robuster in HCl solution:Synergistic effects of iodie ions",Journal of Materials and Environmental, 2(1): 60-71.

Okafor, P.C., Ekpe, U.J., Ebenso, E.E., Umoren, E.M and Leizou, E. (2005).“Inhibition of mild steel corrosion in acidic medium by *Allium sativa* extract”, *Bulletin Electrochem*. 21(8) 347.

Otero, E and Bastidas, J.M. (1996). “Materials and Corrosion - Werkstoffe und Korrosion”, Scotland.**47**, (3), 133-138.

Pande, M. and Pathak, A. (2010). “ Preliminary pharmacognostic evaluations and phytochemical studies on roots of *Mimosa pudica (Laajvanti)”. International journal of pharm. Sci. reve. Resources;1:50-52.*

Rahmouni, K., Takenouti, H., Hajjaji, N., Srhiri, A. and Robbiola, L. (2009). “Roman investigation of artificial patinas on recent bronze part II:Urban air exposure”,*Electrochim. Acta*, 2009, **54**, (22), 5206-5215.

Raja, P.B. and Sethuraman, M.G. (2008).“Natural products as corrosion inhibitors for metals in corrosive media - a review”, *Materials Letters*, **62**, (1), 113-116.

Rajendran, S., Sri, V.G., Arockiaselvi, J. and Amalraj, A.J. (2005). “Corrosion inhibition by plant extracts” - an overview”,*Bulletin of Electrochemistry*; 21(8) 367-377.

Rani, P.D. and Selvaraj, S. (2011).“Inhibitive and adsorption properties of *Punica gratum* extract on brass in acid media”, *Journal of phytology,* 2(11):58-64.

Rani, B.E.A. and Basu, B.B.J. (2012). Green inhibitors for corrosion protection of metals and alloys: An overview”,*International Journal of Corrosion*; article ID: 380217.

Renner, K.O., Pedro, K.N. and Nubi, O.A. (2008). “Oil spillage and its impact on the edible mangrove periwinkle *Tympanotomus fuscantus var radular”*; *Science world journal.* 3 (3); 13-16.

Roberge, P.R., Handbook of corrosion engineering, New York: Mc Graw Hill Handbook, 1999.

Salami, L., Wewe, T.O.Y., Akinyemi, O. P. and Patinvoh, P.J; (2012). “Global engineers and technology review”, 2(12), 6.

Sastri, V.S.(1998). “Corrosion Inhibitors :Principles and applications”, New York, Wiley.

Sastri, V.S.(2011). “Green Corrosion Inhibitors: Theory and Practice”, Wiley Series in Corrosion;Wiley.

Satapathy, A.K., Gunasekaran, G., Sahoo, S.C., Amit, K. and Rodrigues, P.V. (2009). “Inhibition of C-steel corrosion by green tea extract in hydrochloric solution”, *International Journal of Electrochemical Science*, 12(12): 2848-3283.

Shelef, L.A. (1983). “Antimicrobial effects of spices”,*J. Food Safety* 6: 29-U S Forest service.

Solmaz, R., Kardas, G., Ulha, M.C., Yazıcı, B. and Erbil, M. (2008). “Citric acid as a natural corrosion inhibitor for aluminum protection”,*Corrosion Engineering Science and Technology*;43(2) 189-191.

Uken, T., Demir, F., Kicir, N., Sigircik, G. and Erbil, M. (2012).“Inhibition effect of 1-ethyl-3-methyli-midazolium dicyanamide against steel corrosion”,*Corrosion Science*;59: 110-118.

Ueda, M. and Yamamura, S. (1999). “The chemistry of leaf movement in *Mimosa pudica* L”,*Tetrahedron*.55:10937–48.

Uchegbu, S.N; (2002). “Issues and strategies in environmental planning and management in Nigeria”, Spotlite Pub. Enugu. Pp. 177.

Wormald, P.A. (1976). “Effect of spilled marine diesel oil on the micro fauna bay Hong Kong. Environmental Pollution 11; 117; 130.

Yan, Y., Li, W., Cai, L. and Hou, B. (2008) “Electrochemical and quantum chemical study in purine as corrosion inhibitors for mild steel in 1M HCl solution”,*Electrochim. Acta*, 53, 5953).

Zaika, L. J. (1988). “Spices and herds: There antimicrobial activity and its determination”,Journal of Food Safety, 9: 97-117.

Zakvi, S.J. and Mehta, G.N.(1988). “Inhibition of corrosion of mild steel in acid by *Mahasudarshana churna* extract”, *Trans. SAEST* 23, 4.

APPENDIX I

Inhibition efficiency of mild steel in different concentrations of *Mimosa pudica* stem extract in 1.0 M HCL at different time of immersion

STD	Run	Factor 1A:Concentation(Mg/L)	Factor 2B:time(h)	Response1 IE (%)
7	9	600	28.1177	96
1	1	200	48	95

2	2	1000	48	99
10	3	600	96	97
12	6	600	96	97
11	7	600	96	97
6	8	1165.69	96	98
9	10	600	96	97
5	11	34.3146	96	75
13	12	600	96	97
3	4	200	144	85
4	13	1000	144	98
8	5	600	163.882	95

APPENDIX II

Inhibition efficiency of mild steel in different concentrations of *Mimosa pudica* leaf extract in 1.0 M HCL at different time of immersion.

STD	Run	Factor 1 A:Concentation (Mg/L)	Factor 2 B:time (h)	Response 1 IE (%)
7	9	600	28.1177	91

1	1	200	48	87
2	2	1000	48	96
10	3	600	96	93
12	6	600	96	93
11	7	600	96	93
6	8	1165.69	96	94
9	10	600	96	93
5	11	34.3146	96	62
13	12	600	96	63
3	4	200	144	85
4	13	1000	144	95
8	5	600	163.882	89

APPENDIX III

Inhibition efficiency of mild steel in different concentrations of *Mimosa pudica* stem extract in 0.5M H_2SO_4 at different time of immersion

STD	Run	Factor 1 A:Concentation (Mg/L)	Factor 2 B:time (h)	Response 1 IE (%)
7	9	600	28.1177	96

1	1	200	48	93
2	2	1000	48	99
10	3	600	96	97
12	6	600	96	97
11	7	600	96	97
6	8	1165.69	96	99
9	10	600	96	97
5	11	34.3146	96	54
13	12	600	96	97
3	4	200	144	85
4	13	1000	144	97
8	5	600	163.882	95

APPENDIX IV

Inhibition efficiency of mild steel in different concentrations of *Mimosa pudica* leaf extract in 0.5M H_2SO_4 at different time of immersion.

STD	Run	Factor 1 A:Concentation (Mg/L)	Factor 2 B:time (h)	Response 1 IE (%)
7	9	600	28.1177	91

1	1	200	48	69
2	2	1000	48	96
10	3	600	96	88
12	6	600	96	88
11	7	600	96	88
6	8	1165.69	96	96
9	10	600	96	88
5	11	34.3146	96	36
13	12	600	96	88
3	4	200	144	49
4	13	1000	144	92
8	5	600	163.882	81

APPENDIX V

Adsorption isotherm table for 0.5M H_2SO_4

	Concentration (Mg/L)	Inhibition Efficiency (%)	Surface Coverage (θ)	C/θ
MPS	50	90	0.90	55.56

	200	95	0.95	210.53
	400	96	0.96	416.67
	600	98	0.98	612.24
	800	98	0.98	816.33
	1000	99	0.99	1010.10
MPL	50	78	0.78	64.10
	200	87	0.87	229.89
	400	91	0.91	439.56
	600	94	0.94	638.30
	800	96	0.96	833.33
	1000	96	0.96	1041.67

APPENDIX VI

Adsortion isotherm table for 0.5M H_2SO_4

	Concentration (Mg/L)	Inhibition Efficiency (%)	Surface Coverage(θ)	C/θ
MPS	50	76	0.76	65.79

	200	93	0.93	215.05
	400	95	0.95	421.05
	600	98	0.98	612.24
	800	99	0.99	808.08
	1000	99	0.99	1010.10
MPL	50	42	0.42	119.05
	200	69	0.69	289.86
	400	86	0.86	465.12
	600	91	0.91	659.34
	800	95	0.95	842.11
	1000	96	0.96	1041.67

APPENDIX VII

Calculated values of Langmuir Adsorption isotherm parameters of *Mimosapudica* leaf and stem extract in 1.0M HCL and 0.5M H_2SO_4.

Plant Extract	Intercept	Slope	R^2

1.0M HCL			
MPS	9.37165	1.00498	0.99986
MPL	22.08513	1.02109	0.99959
$0.5MH_2SO_4$			
MPS	18.17224	0.99124	0.99989
MPL	83.1751	0.95675	0.9991

APPENDIX III

Inhibition efficiency of mild steel in different concentrations of *Mimosa pudica* stem extract in 0.5M H_2SO_4 at different time of immersion

STD	Run	Factor 1 A:Concentation (Mg/L)	Factor 2 B:time (h)	Response 1 IE (%)

7	9	600	28.1177	96
1	1	200	48	93
2	2	1000	48	99
10	3	600	96	97
12	6	600	96	97
11	7	600	96	97
6	8	1165.69	96	99
9	10	600	96	97
5	11	34.3146	96	54
13	12	600	96	97
3	4	200	144	85
4	13	1000	144	97
8	5	600	163.882	95

APPENDIX IV

Inhibition efficiency of mild steel in different concentrations of *Mimosa pudica* leaf extract in 0.5M H_2SO_4 at different time of immersion.

STD	Run	Factor 1 A:Concentation (Mg/L)	Factor 2 B:time (h)	Response 1 IE (%)

7	9	600	28.1177	91
1	1	200	48	69
2	2	1000	48	96
10	3	600	96	88
12	6	600	96	88
11	7	600	96	88
6	8	1165.69	96	96
9	10	600	96	88
5	11	34.3146	96	36
13	12	600	96	88
3	4	200	144	49
4	13	1000	144	92
8	5	600	163.882	81

APPENDIX V

Adsorption isotherm table for 0.5M H_2SO_4

	Concentration (Mg/L)	Inhibition Efficiency (%)	Surface Coverage (θ)	C/θ

MPS	50	90	0.90	55.56
	200	95	0.95	210.53
	400	96	0.96	416.67
	600	98	0.98	612.24
	800	98	0.98	816.33
	1000	99	0.99	1010.10
MPL	50	78	0.78	64.10
	200	87	0.87	229.89
	400	91	0.91	439.56
	600	94	0.94	638.30
	800	96	0.96	833.33
	1000	96	0.96	1041.67

APPENDIX VI

Adsortion isotherm table for 0.5M H_2SO_4

	Concentration (Mg/L)	Inhibition Efficiency (%)	Surface Coverage(θ)	C/θ

MPS	50	76	0.76	65.79
	200	93	0.93	215.05
	400	95	0.95	421.05
	600	98	0.98	612.24
	800	99	0.99	808.08
	1000	99	0.99	1010.10
MPL	50	42	0.42	119.05
	200	69	0.69	289.86
	400	86	0.86	465.12
	600	91	0.91	659.34
	800	95	0.95	842.11
	1000	96	0.96	1041.67

APPENDIX VII

Calculated values of Langmuir Adsorption isotherm parameters of *Mimosapudica* leaf and stem extract in 1.0M HCL and 0.5M H_2SO_4.

Plant Extract	Intercept	Slope	R^2
1.0M HCL			
MPS	9.37165	1.00498	0.99986
MPL	22.08513	1.02109	0.99959
0.5MH_2SO_4			
MPS	18.17224	0.99124	0.99989
MPL	83.1751	0.95675	0.9991

APPENDIX VIII

Electrochemical parameters for mild steel in 1.0 M HCl at various concentrations of *Mimosa pudica* leaf and stem extract respectively.

System	**E_{corr}(mV/SCE)**	**j_{corr}(μA/cm2)**	**η(%)**
Blank			
1 M HCl	-470.38	269	
MPS			
200mg/L	-483.1	38.9	85.5
1000mg/L	-489.3	4.6	98.3
MPL			
200mg/L	-481	39.8	85.2
1000mg/L	-488	8.9	96.6

APPENDIX IX

Electrochemical parameters for mild steel in 0.5M H_2SO_4 at various concentrations of Mimosa pudica leaf and stem extract respectively.

System	E_{corr} (mV/SCE)	j_{corr} (μA/cm 2)	H (%)
Blank 0.5M H_2SO_4	-485.29	591.29	
MPS			
200mg/L	-489	98.3	83.4
1000mg/L	-469	27.2	95.4
MPL			
200mg/L	-491	289.7	51
1000mg/L	-480	32.1	94.6

MPL= *Mimosa pudica* leaf, MPS=*Mimosa pudica* stem, η= Inhibition Efficiency.

APPENDIX X

ANOVA for Response Surface Linear model, Analysis of MPS in 0.1M HCL

Source	**Sum of Squares**	**df**	**Mean Square**	**F Value**	**p-value Prob > F**	
Model	325.88	2	162.94	7.31	0.0111	Significant
A-Concentration	*306.61*	*1*	*306.61*	*13.76*	*0.0040*	
B-Time	*19.26*	*1*	*19.26*	*0.86*	*0.3744*	
Residual	222.89	10	22.29			
Lack of Fit	*222.89*	*6*	*37.15*			
Pure Error	*0.000*	*4*	*0.000*			
Cor Total	548.77	12				

APPENDIX XI

ANOVA for Response Surface Linear model

Analysis of variance table of MPL in 1.0M HCL

Source	**Sum of Squares**	**df**	**Mean Square**	**F Value**	**p-value Prob > F**
Model	517.21	2	258.61	6.11	0.0185 Significant
A-Concentration	*516.09*	*1*	*516.09*	*12.18*	*0.0058*
B-Time	*1.12*	*1*	*1.12*	*0.027*	*0.8738*
Residual	423.56	10	42.36		
Lack of Fit	*423.56*	*6*	*70.59*		
Pure Error	*0.000*	*4*	*0.000*		
Cor Total	940.77	12			

APPENDIX XII

ANOVA for Response Surface Linear model
Analysis of variance table of MPS in 0.5M H_2SO_4

Source	**Sum of Squares**	**df**	**Mean Square**	**F Value**	**p-value Prob > F**	
Model	853.70	2	426.85	4.71	0.0361	significant
A-Concentration	*833.13*	*1*	*833.13*	*9.20*	*0.0126*	
B-Time	*20.57*	*1*	*20.57*	*0.23*	*0.6439*	
Residual	905.53	10	90.55			
Lack of Fit	*905.53*	*6*	*150.92*			
Pure Error	*0.000*	*4*	*0.000*			
Cor Total	1759.23	12				

APPENDIX XIII

ANOVA for Response Surface Linear model

Analysis of variance table of MPL in 0.5M H_2SO_4

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	3179.28	2	1589.64	17.11	0.0006	Significant
A-Concentration	*2997.42*	*1*	*2997.42*	*32.26*	*0.0002*	
B-Time	*181.85*	*1*	*181.85*	*1.96*	*0.1920*	
Residual	929.03	10	92.90			
Lack of Fit	*929.03*	*6*	*154.84*			
Pure Error	*0.000*	*4*	*0.000*			
Cor Total	4108.31	12				