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Green corrosion inhibition of carbon steel in acid medium using leaves extract of Moringa oleifera

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بسم الله الرحمن الرحيم

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بسم خالقي و ميسر اموري و عصمت امري لك كل الحمد و الإمتنان

(وَآخِرُ دَعْوَاهُمْ أَنِ الْحَمْدُ لِلَّهِ رَبِّ الْعَالَمِينَ)

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General Introduction

Introduction

Corrosion is an unavoidable fact in everyday life, but it always deserves attention due to its technical, economical, and aesthetic importance. Corrosion inhibitors are one of the most widely used and economically viable methods of protecting metals and alloys against corrosion. Typical corrosion inhibitors are bio-toxic organic compounds, which have serious issues with toxicity. Considering the toxicity of the inhibitors, there is a tremendous interest in searching for an eco-friendly and non-toxic green corrosion inhibitor.**[1]**

Corrosion inhibitors are chemical substances that, when added in small concentrations to corrosive media decrease or prevent the interaction of the metal with the media. Molecules contain heteroatoms with free electrons pairs (O, N, S), aromatic cycles are predicted to be highly effective corrosion inhibitor candidates .**[2]**

Compounds known as organic corrosion inhibitors can be incorporated into aqueous solutions to prevent corrosion of metal surfaces. These inhibitors function by creating a protective layer on the metal surface that obstructs the corrosive atmosphere from reaching the metal . **[3]**

The use of plant extracts as corrosion inhibitors is a topic of research in development if we rely on the number of publications coming out each year.

Indeed, these natural extracts contain numerous families of natural organic compounds (flavonoids, alkaloids, tannins, etc.) that are “ecological,” easily available and renewable. **[4]**

The additive that was examined in our study is an extract from the Moringa oléifera (Moringaceae), one of these significant tropical plants that is well-known for being rich in bioactive phytochemicals and antioxidants. It is occasionally used as a dietary supplement and is used to create medication. **[5]**

As part of this research work, we were interested in studying the corrosion inhibition of X70 steel. According to the World Steel Association, there are more than 3,500 different grades of steel with varying physical, chemical and unique environmental conditions allowing it to be associated with numerous applications. It enjoys a reputation as an “ecological metal”. The corrosion resistance property of steel is so important that almost half of the annual consumption of the metal is used as a protective coating against corrosion of iron and steels. Because of various industrial applications and the economic importance of steel, its corrosion protection has attracted much attention.**[4]**

The aim of this work is to investigate corrosion inhibition characteristics of steel (X70) in chlohydric acid in presence of different concentration of Moringa oleifera extract using the use of the gravimetric method (loss of mass) and electrochemical method.

The work that we present in this manuscript is subdivided into four chapters and a conclusion:

- ✓ The first chapter is devoted to a bibliographical study to metal Corrosion and Its Prevention.
- ✓ The second chapter is devoted to a bibliographical study on of moringa oleifera
- ✓ The third chapter deals with the phytochemical tests applied to moringa oleifera techniques (lost mass technique), as well as devices experiments used for the production of this dissertation.
- ✓ The experimental results and discussions are grouped in the fourth chapter.

Finally, a general conclusion of this work completes this study.

Theoretical part

Chapter One

Metal corrosion and its prevention

I.Generality on the Corrosion of metals

I.1. Historical

For more than 150 years, corrosion has been the focus of scientific study. It is a phenomenon that occurs naturally and is defined as the degradation of a material, typically a metal, or its properties due to its reaction to its environment. It leads to enormous economic losses for the whole world. Apart from the economic consequences, corrosion has resulted in several structural failures that had serious consequences for human health and life and the surrounding environment. Safety and environmental concerns tend to be very difficult to define in terms of cost.

Therefore, corrosion problems must be addressed for safety and to minimize environmental pollution. There has been an inevitable transformation of the subject of corrosion from a condition of isolation and obscurity to a known engineering and science discipline. Over a period of time, considerable developments have taken place in the field of corrosion and corrosion control. There are, however, still many problems that corrosion scientists and engineers need to solve. **[6]**

I.2. Definition of steel

Steel is an iron-based alloy with an additional percentage of carbon (from 0.008% by mass) and its carbon content can be up to 1.2%. An increase in carbon content tends to improve the mechanical strength of the alloy. For ordinary steel, which does not contain alloying elements, we can distinguish between: mild, semi-soft, hard, semi-hard and ultra-hard steels.**[7]**

The main interest of steel lies on the one hand in accumulating high values in basic mechanical properties:

- resistance to forces: modulus of elasticity, elastic limit, mechanical resistance
- hardness
- resistance to shocks (resilience). **[8]**

I.3. Classification of steel

Several classifications of steels are made on the basis of chemical composition , production process and instructions for use. We are interested in a few classifications:

I.3.1. Classification according to their chemical composition

Alloys of iron and carbon are referred to as unalloyed steels or steels .Carbon, more or less carburized iron alloys and other elements, in general metal under the name of alloy steel.

Carbon steel: contains carbon in increasing proportion, magnesium in proportion greater than carbon and traces of silicon phosphorus and sulfur. **[8]**

Table I.1: Classification of steels according to chemical composition.

Quality	C (%)	Mn (%)	P (%)	Si (%)	S (%)	Lengthen (%)
Extra soft	0.15	0.4	0.04	0.03	0.02	30
Soft	0.25	0.4	0.06	0.10	0.03	25
Semi-sweet	0.35	0.4	0.06	0.15	0.03	20
Medium hard	0.45	0.5	0.06	0.20	0.03	15
Hard	0.55	0.6	0.06	0.25	0.03	10
Extra hard	0.65	0.7	0.06	0.3	0.03	5

Alloy steel: Variable composition depending on use, the alloy elements are in proportion generally varying between 0 and 5 (%).**[8]**

Table I.2: alloy elements of alloy steels

Allied element	**Ratio**	**Observations**
Carbon	0.2 ÷ 0.7	
Silicon	0.7÷0.1 2 ÷ 4 (special steels)	Transfer sheet springs
Magnesium	0.8÷0.3 12 ÷ 14 (very hard steels)	Voisferrées
S, P, Cu, Ni, Cr, V, Mo	< 0.1	

I.3.2. Classification by instructions for use

We distinguish the following types:

- **Structural steels:** They are used for construction,
- **Steels with special physical and chemical properties:** they contain steels stainless steels, steels with special magnetic properties, steels with expansion properties, etc…
- **Tool steels:** they are used in the manufacture of instruments (allowing shape materials).

The grades of tool steels are arranged according to the working method of the tool, in four classes as shown in the table. **[8]**

Table I.3: tool steel classes

Classes	Common characters
Carbon tool steels	General purpose steels
Alloy tool steels for cold work	High strength steels
Alloy tool steels for hot work	Steels resistant to thermal shock
High speed steels	Carburized steels

I.4. Definition of Corrosion

Corrosion is an irreversible interfacial reaction of a material (metal, cera- mic, polymer) with its environment which results in consumption of the material or in dissolution into the material of a component of the environment.

Often, but not necessarily, corrosion results in effects detrimental to the usage of the material considered. Exclusively physical or mechanical processes such as melting or evaporation, abrasion or mechanical fracture are not included in the term corrosion .**[9]**

To the great majority of people, corrosion means rust, an almost universal object of hatred. Rust is, of course, the name which has more recently been specifically reserved for the corrosion of iron ,while corrosion is the destructive phenomenon which affects almost all metals. **[10]**

I.5. Corrosion Types

By its definition and mechanism, it can be seen that corrosion is a really complicated process. It can develop in many directions for just one metal, depending on its surrounding environments. Hence ,studying and evaluating different types of corrosion is also a complex task. There are various ways of classification that were discussed among engineers, and it is not easy to tell what is right and the most suitable. **[11]**

I.5.1. Chemical Corrosion

It’s agit a pure chemical reaction on the surface of the material and a gas or un liquid non-electrolyte. For example, the oxidation of the air conditioner at a higher temperature the oxygen in the air is a chemical corrosion. **[12]**

I.5.2. Electrochemistry Corrosion

This is an electrochemical reaction on the surface of the mat and a liquid electrolyte. It is accompanied by the formation of piles that use the electrical circulation fence. **[10]**

I.5.3. Biochemistry Corrosion

This is the attachment of metal materiel bacteria, in particular in the canalizations .enter and serve. In effect, the metabolism of development of certain bacteria provoque the formation of sulfuric acid that affects the metal. **[10]**

I.5.4. Accompanied by wear Corrosion

When the corrosion products appear dry and uniform on the surface metal, it is the result of corrosion. This couch may be limited at certain points for use abrasive or abrasive. It's also accelerating the corrosion on the surface the metal is here. **[10]**

I.6. corrosion forms

Corrosion damage can occur in many ways, for example

I.6.1. Uniform corrosion

Uniform or general corrosion occurs over most (if not all) of a piece of chemical processing equipment, hence the name uniform or general corrosion.Even though general corrosion is often referred to as uniform corrosion, the depth of corrosion on the metal surface is actually not totally uniform as can be seen in Fig.1. Indeed, there are typically peaks and valleys over the metal surface when general corrosion attacks a metal. However, corrosion is considered to be general when there are no small deep corroded areas on the metal. **[13]**

Fig I.1 : General (uniform)

I.6.2. Galvanic corrosion

When two dissimilar conducting materials in electrical contact with each other are exposed to an electrolyte, a current, called the galvanic current, flows from one to the other. Galvanic corrosion is that part of the corrosion that occurs at the anodic member of such a couple and is directly related to the galvanic current by Faraday's law. **[14]**

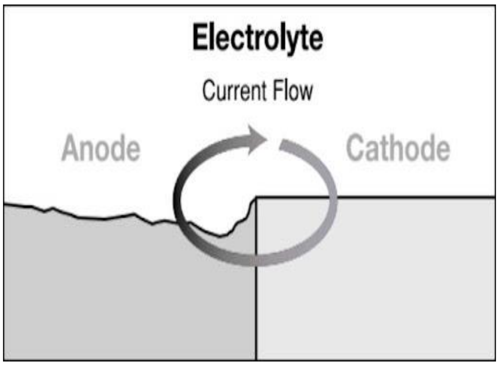


Fig I.2 : Galvonic corrosion

I.6.3. Pitting corrosion

A localized type of corrosion that creates cavities or "holes" in a substance. Pitting is known as more risky than uniform corrosion damage because the identification, estimation and configuration of it is tougher. The pit is also filled by corrosion products. **[15]**

Fig I.3: Pitting corrosion of metal[16]

I.6.4. Stress corrosion cracking

The growth of a crack formed in a corrosive setting is referred to as stress corrosion cracking (SCC). It can result in an unforeseen and unexpected failure of metal alloys that are normally ductile under tensile stress, especially at high temperatures. SCC is very solvent-specific and certain alloys can be subject to SCC only if subjected to a limited number of chemical conditions. The chemical environment that causes SCC for a particular alloy is often the one that causes mild corrosion to the metal. **[17]**

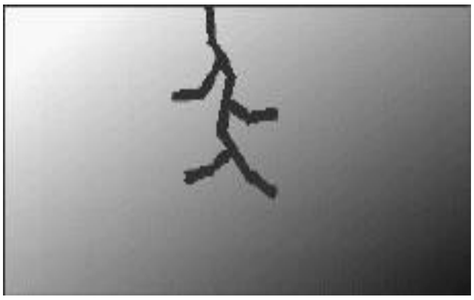


Fig I.4: Stress corrosion of metal

I.6.5. Corrosion fatigue

It occurs in a corrosive fatigue environment. It is the mechanical weakening of a material under corrosion and cyclical loading operating jointly. Almost all engineering systems undergo some form of alternate stress and are subjected to hazardous conditions throughout their lifespan.**[15]**

I.6.6. Ntergranular corrosion

IGC, also known as intergranular attack (IGA), is a type of corrosion in which the boundaries of crystalline materials are more susceptible to erosion than the interiors. **[15]**

Fig I.5: Intergranular corrosion of metal

I.6.7. Crevice Corrosion

Crevice corrosion is a localized form of corrosive attack.Crevice corrosion occurs at narrow openings or spaces between two metal surfaces or between metals and nonmetal surfaces (Figure 6). A concentration cell forms with the crevice being depleted of oxygen. This differential aeration between the crevice (microenvironment) and the external surface (bulk environment) gives the crevice an anodic character. This can contribute to a highly corrosive condition in the crevice.Some examples of crevices are Flanges, Deposits, Washers ,Rolled tube ends, Threaded joints, etc .**[16]**

Fig I.6: Crevice corrosion of metal

I.6.8. Filiform corrosion

It is a particular type of corrosion which occurs in the form of randomly dispersed filaments under some thin coatings. Corrosion of filiforms is often referred to as “underfilm corrosion”. Filiform corrosion is observed on surfaces of painted steel, magnesium, and aluminum with thin layers of tin, silver, gold, phosphate, enamel and varnish. **[15]**

I.6.9. Erosion corrosion

Material surface deterioration due to mechanical activity, often by impaction of material, slurry abrasion, particles trapped in fast-flowing liquid or gas, bubbles or droplets, cavitation...**[13]**

I.6.10. Fretting corrosion

Corrosion impact on touch surface asperities is referred to. In the presence of repetitive relative surface movement, for instance, by vibration, this harm is incurred under load. **[15]**

I.7. Factors Affecting Corrosion. [17]

I.7.1. Environmental Factors :

- ✓ Oxygen: Oxygen is the most common cause of corrosion, as it reacts with metals to form oxides.
- ✓ Moisture: Moisture can dissolve oxygen and other corrosive" substances, making them more likely to come into contact with metals.
- ✓ Temperature: Higher temperatures can increase the rate of corrosion.
- ✓ pH Level: Acids and bases can corrode metals.
- ✓ Salt**:** Salt can accelerate the corrosion process .

I.7.2. Metallurgical Factors:

- ✓ Composition: The composition of a metal can affect its resistance to corrosion.
- ✓ Grain Structure: The grain structure of a metal can affect its corrosion resistance.
- ✓ Surface Condition: The surface condition of a metal can affect its corrosion resistance .

I.7.3. Mechanical Factors :

- ✓ Stress: Stress can cause cracks in a metal, which can allow corrosive substances to enter.
- ✓ Abrasion: Abrasion can remove the protective oxide layer from a metal, making it more susceptible to corrosion .

I.8. Corrosion as a Chemical Reaction

The corrosion process involves acid-base reactions as well as electrochemical reactions. It is the acid-base reactions that make metallic corrosion diversified depending not only on the electrode potential but also on the acid-base species present in the environment.

Localized corrosion results from the growth of electrochemical perturbation into the formation of a dissipative structure, which begins when the perturbation exceeds a marginal size. Selective ion transport through corrosion precipitates on the metal surface often plays a decisive role in the development of localized corrosion and passivation **.[15]**

I.8.1. Corrosion in Acids

One of the common ways of generating hydrogen in a laboratory is to place zinc into a dilute acid, such as hydrochloric or sulfuric. When this is done, there is a rapid reaction in which the zinc is attacked or “dissolved” and hydrogen is evolved as a gas :

$$Zn + 2HCl \rightarrow ZnCl_2 + H_2\ (g)$$
$$Zn + 2H+ + 2Cl^- \rightarrow Zn^{2+} + 2Cl^- + H_2(g)$$

Many other metals are also corroded by acids, often yielding soluble salts and hydrogen gas, such as iron and aluminum. **[18]**

I.8.2. Corrosion in natural and alkaline solution

The corrosion of metals can also occur in fresh water, seawater, salt solutions, and alkaline or basic media. In almost all of these environments, corrosion occurs importantly only if dissolved oxygen is also present. Water solutions rapidly dissolve oxygen from the air, and this is the source of the oxygen required in the corrosion process.

The most familiar corrosion of this type is the rusting of iron when exposed to a moist atmosphere. **[16]**

$$4Fe + 6H_2O + 3O_2 \rightarrow 4Fe(OH)_3 \downarrow$$

II. Corrosion inhibitors

II.1. Historic

There are evidences of the use of inhibitor since the early XIX century. 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 applica- tion of electrochemistry to evaluate corrosion inhibitors.

Now a days, 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 develop-ment of other inhibitor to supply the lack in this area. Should, however, present a similar anti corrosive properties similar than a chromate inhibitor.

An important number of papers have been published with the intention of develop an environmentally friendly corrosion inhibitors and a lot of research has been doing to devel-opment of the called “green” corrosion inhibitors. Also, has been increasing research in natural products, such as plant extracts, essential oils and purified compounds to obtain environmentally friendly corrosion inhibitors. The first evidence of natural product use as corrosion inhibitors is 1930’s. When extracts of chelidonium majus (Celadine) and other plants were used on the first time in H_2SO_4 pickling baths. **[19]**

II.2. Definition of inhibition

Inhibition is a process of preventive measure against corrosive attack on metallic materials. Chemical compounds may be used which, when added in small concentrations to an aggressive environment, are able to decrease corrosion of the exposed metal. **[20]**

Corrosion inhibitors are one of the most practical and cost-effective methods of anticorrosion protection. They are widely used to protect metals and alloys from the effects of corrosive environments. Corrosion inhibitors can be organic or inorganic in nature and are classified depending on the method of use and the specificity of the inhibitory effect. However, a significant part of them can pose a danger to the environment and people. Therefore, there is a need to minimize the use of such harmful substances and to replace them with environmentally safe alternatives. Great examples of alternative inhibitors are substances based on natural products, e.g., plant extracts . In addition to being environmentally friendly, such inhibitors have high solubility and effectiveness. Besides, they are readily available on the market and are cost-efficient, which altogether makes them very attractive for industrial use. **[21]**

Corrosion inhibitors act by one or more of the following mechanisms:

- They form precipitates, which visibly coat and protect metal surfaces.
- They adsorb on metal surfaces to form protective films .
- They combine with corrosion product to protect metal surfaces. **[22]**

II.3. Classification of Corrosion Inhibitors

Corrosion inhibitors are substances that are added in small amounts to the corrosive medium to stop or slow down electrochemical corrosion reactions on a metal surface are an attractive area of research because of their usefulness in various industries. The efficiency of the inhibitor depends on the stability of the formed chelate, and the inhibitor molecule must have centers capable of forming bonds with the metal surface by electron transport. Most organic inhibitors are absorbed onto the metal surface by displacing water molecules on the surface and forming a pressurized barrier.

The availability of non-bonding electrons (a single pair) and p electrons in the inhibitor molecules facilitate the transfer of the electron from the inhibitor to the metal. Inhibitors can be divided into two main categories inorganic and organic. **[22.23]**

II.3.1. Depending on the nature of the chemical

II.3.1.1 Organic inhibitors

Organic Corrosion Inhibitors are an attractive area of research because of their usefulness in various industries. The efficiency of the inhibitor depends on the stability of the formed chelate, and the inhibitor molecule must have centers capable of forming bonds with the metal surface by electron transport. Most organic inhibitors are absorbed onto the metal surface by displacing water molecules on the surface and forming a pressurized barrier. The availability of non-bonding electrons (a single pair) and p electrons in the inhibitor molecules facilitate the transfer of the electron from the inhibitor to the metal. The efficiency of theinhibitor depends on the stability of the chelate formed, so it mainly depends on the type and nature of the alternatives present in the inhibitor molecule. Organic inhibitors are used mainly in oil field systems. **[23]**

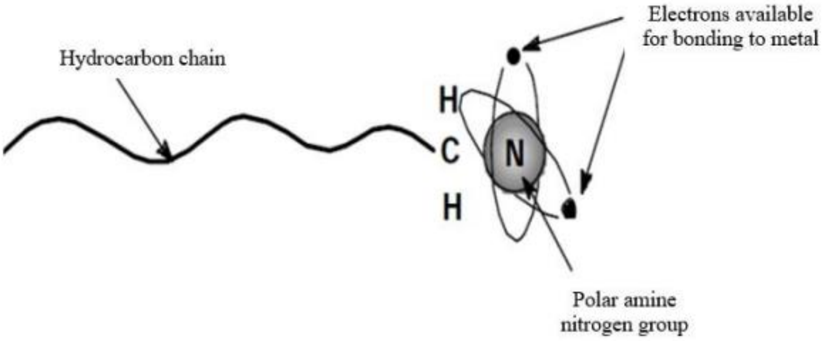


Fig I.7 : Organic inhibitors molecule. [21]

II.3.1.2. Inorganic inhibitors

Inorganic molecules are most often used in environments close to the neutrality or in an alkaline environment, and more rarely in an acidic environment. Almost all of these Molecules contain oxygen in their formula. Depending on whether they require more or less oxygen dissolved in the corrosive environment to be effective, they are classified into non-passivating inhibitors (weak acid and strong base salts) or passivating inhibitors (nitrites and oxo-anions of MeO_4 x- type).

The first act rather by forming a protective compound, precipitated on the surface, while the latter act by helping to formation of a homogeneous and insulating oxide layer. The number of molecules in use at the current times are becoming more restrictive, because most effective products have a side harmful to the environment. Inorganic inhibitors are used mainly in boilers, cooling towers, and fractionation units. **[24]**

II.3.2. Depending on the electrochemical nature of the process

II.3.2.1. Anodic inhibitors

Anodic inhibitors are very effective and often used, they generally have one undesirable property: if the anodic content inhibitor is/or becomes gradually lowered, the metal surface is no longer completely covered, and it will function as an anode, thus obtaining a dangerous combination, a small anode and a large cathode, leading to pitting corrosion. In this case, the inhibitor does more harm than good. This is why anodic inhibitors are often designated as dangerous. Another mechanism involves a modification of the kinetics of anodic dissolution by absorption of the inhibitor. There is no simple blocking of sites preferential dissolution of the metal, there is also an intervention of the inhibitor at the level of the reaction intermediates accompanying the different stages of the dissolution of the metal. **[25]**

II.3.2.2. cathodic inhibitors

The action of these inhibitors results in a reduction of the speed of the cathodic reaction and therefore by a shift in the corrosion potential towards less noble values. These are generally cations that can migrate to the surface cathodic, where they precipitate in the form of basic salts or hydroxides, forming films adherent and compact. Cathodic inhibitors include combinations of zinc, nickel ,magnesium, alkaline phosphates, arsenic combinations. According to their mode of action , they are observed to be useful in very acidic environments. **[25]**

II.3.2.3. Mixed inhibitors

Only a few of the inhibitors mentioned above can become fully effective in connection with dissolved oxygen or with calcium salts .Simultaneous addition of two inhibitors can result in increased inhibitory effect and it can also eliminate the risk of pitting corrosion at low concentrations. Such inhibitor often consists of a combination of oxidizing agent, such as nitrate or chromates, and a non-oxidizing agent, but which precipitates as orthophosphate or silicate. Of the Examples of such inhibitors are the nitrate and benzoate mixture, which is the most common inhibitor and effective for automobile radiators and the orthophosphate and chromate mixture, which is very effective even in salt water. In other cases, the mixed inhibitor is a mixture between a cathodic inhibitor and an anodic inhibitor, such as polyphosphates and chromate. The Evans diagram summarizes the various cases of these types of inhibitors. **[25]**

III. Methods of corrosion evaluation

Corrosion evaluation can be done by various methods. **[25]**

III.1 Gravimetry method

This method has the advantage of being simple to implement, of not require significant equipment, but does not allow the approach of the mechanisms implemented play during corrosion. Its principle is based on the measurement of the weight loss Δm suffered by a sample of surface S, during the time t of immersion in a corrosive solution maintained at constant temperature. The corrosion rate is given by the relation next:

$$\boldsymbol{V_{corr}} = \Delta \boldsymbol{m} / \boldsymbol{S}.\, \boldsymbol{t}$$

V_{corr} can be expressed in $mg.cm^{-2}.h^{-1}$

The inhibitory effectiveness of an organic compound is given by the following relationship:

$$\varepsilon(\%) = \frac{\boldsymbol{Vcorr - Vinh}}{\boldsymbol{Vcorr}}\, \boldsymbol{x100}$$

Where V_{corr} et $Vinh$ and Vinh are the corrosion rates in the solution without and with inhibitor. **[26.]**

III.2 Electrochemical method

The electrochemical methods used to study the corrosion phenomenon can be divided into two categories:

- Stationary methods,
- Transitional methods. **[26]**

III.2.1. Stationary method: polarization curves

The polarization curve of the metal-solution interface is a characteristic fundamental of electrochemical kinetics, but only accounts for the most slow of the overall process at the electrochemical interface. To determine a curve of potentiostatic polarization, we apply, using a potentiostat, different potentials between the working electrode and a reference electrode. We measure the stationary current which is established after a certain time in the electrical circuit between this working electrode and a counter electrode.

This method makes it possible to precisely determine the parameters electrochemical effects of a metal in contact with an electrolyte, namely: the instantaneous speed of corrosion (icorr), the corrosion potential (E_{corr}), the Tafel slopes, the resistance of polarization (R_p), the diffusion limit currents.It gives rapid measurements and its implementation is relatively simple. There determination of the corrosion rate from polarization curves is closely linked to the kinetics governing the electrochemical process, we distinguish three main types of kinetics:

- Pure activation kinetics
- Mixed kinetics (activation-diffusion)
- Pure diffusion kinetics

To experimentally determine these electrochemical parameters, a presentation logarithmic current density is generally preferable, because it highlights the linear relationship between the logarithm of the current density and the potential (figure 8). **[27]**

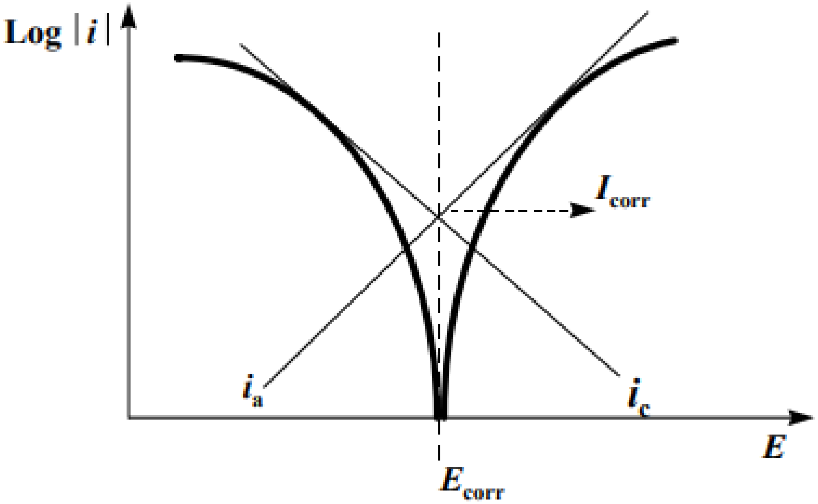


Fig I. 8: Determination of electrochemical parameters from Tafel lines

III.2.2. Transient method: electrochemical impedance spectroscopy

Non-stationary methods can be classified into two categories, namely :

Large amplitude disturbance methods (cyclic voltammetry) and the low amplitude method amplitude (electrochemical impedancemetry). Previous work has shown that the electrochemical impedance spectroscopy (EIS) can identify the stages elementary elements involved in the overall process taking place at the metal/solution interface ,in the form of various time constants. The S.I.E. is used today in following areas:

- photoelectrochemistry,
- the semiconductor/electrolyte interface
- organic electrochemistry and the study of adsorption phenomena
- bio electrochemistry
- corrosion and its inhibition. **[27]**

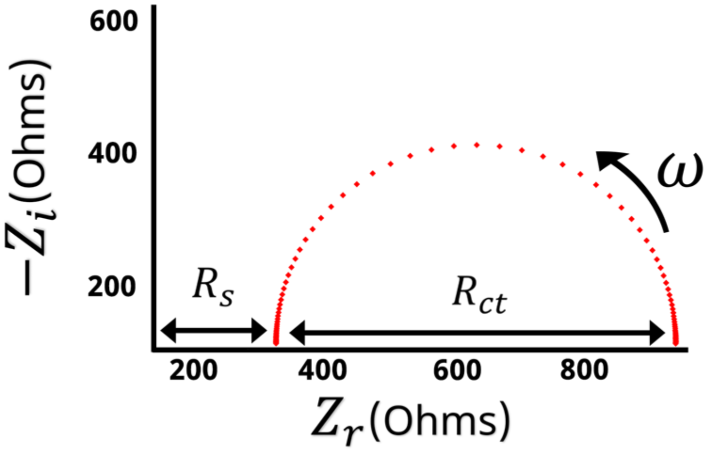


Fig I.9: Spectroscopie d'impédance électrochimique

Chapter Two

General information of moringa oleifera

Introduction

Plants are unique source of food, medicinal artifacts, energy and shelter for both human and animal., many useful harvests obtained from plants directly or indirectly validate their importance to the human and other living organisms. **[28.29]**

I. General information of moringa oleifera

I.1. Definition

Moringa oleifera lam. (Synonym: Moringa pterygosperma Gaertner) belongs to the monogeneric family of shrubs and trees of Moringaceae. This family is said monogeneric because there is only one genus “Moringa”, which includes approximately 13 species. It is called “the tree of life”, “the tree miracle” or divine plant .Is one of the best known species and most used in the world because of its numerous medicinal, industrial, nutritional and environmental . **[30]**

Fig II.10: Moringaoleifera

I.2. Geographical distribution

Moringa oleifera is one of the species of family Moringaceae, native to, Africa, Arabia, South Asia, South America, Himalaya region, India ,Pakistan, the pacific and Caribbean Islands. Moringa oleifera has been naturalized in many tropic and subtropics regions worldwide ,the plant is referred to number of names such as horseradish tree ,drumstick tree, ben oil tree, miracle tree, and “Mothers best friend ”Moringa oleifera is commonly known as

"Drumstick". It is a small or medium sized tree, about 10m height, found in the sub-Himalayan tract. Moringa oleifera is a small, fast-growing evergreen or deciduous tree that usually grows up to 10 to 12m in its height, open crown of drooping fragile branches, feathery foliage of trip innate leaves and thick corky, whitish bark. **[31]**

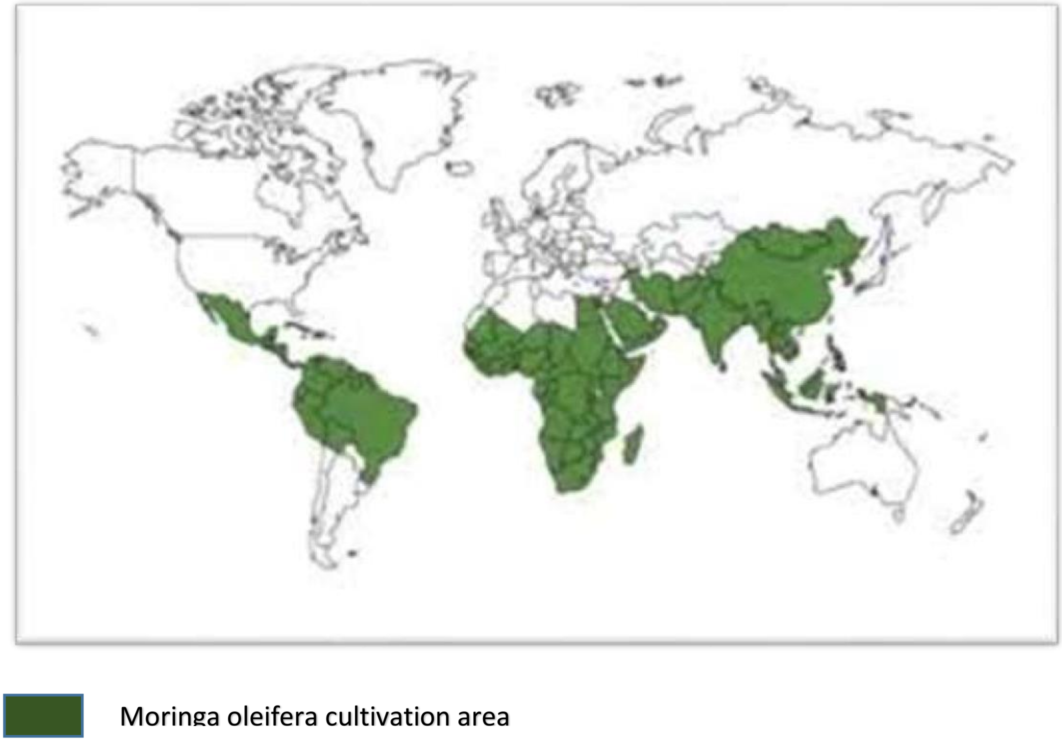


Fig II.11: Geographic distribution of Moringa oleifera in the world

I.3. Systematic classification of the plant

The classification of the Moringa oleifera species is presented in (Table).

Table II.4: Botanical classification of Moringa oleifera according to Lamarck.(1785) . **[30]**

Reign	Plantae
Subkingdom	Tracheobionta
Division	Magnoliophyta
Class	Magnoliopsida
Subclass	Dilleniidae
Order	Capparales
Family	Moringaceae
Genus	Moringa
Species	Moringa oleifera

I.4. Botanical description of the plant M. oleifera

Botanical data is summarized in the table below

Table II.5: Botanical appearance of Moringa oleifera . **[30]**

Organ	Description	Photographs and references
Tree	Moringa oleifera is a perennial tree, fast-growing, which can reach a height of 7 to 12 meters and whose trunk measures 20 to 40 cm in diameter and 1.5to 2 meters high before branch out; she has several branches. The barrel has a diameter measuring between 9 - 20 cm and 1.3 m in length	
Leaves	The leaves are alternate and imparipinnate, compound and bi or tripinnate, develop mainly at the end of the branches. They have a long petiole (20 to 70 cm) with 8 to 10 pairs of pinnae, each composed of two pairs of opposite leaflets, plus one at the apex, oval to elliptical, and measuring 1 to 2 cm long	

Flowers	The flowers are 2.5 cm wide and appear as axillary, drooping panicles of 10 to 25 cm.They are white in color, with yellow dots at the base.the sepals, five in number, are symmetrical and lanceolate. the five petals are thin and spatulate, symmetrical except for the lower petal, and surround five stamens	
Fruits	The fruits are made of three-lobed pods, dark green in color and become light brown or brown when ripe, measuring 20 to 60 cm in length. Each pod contains between 12 and 35 seeds. They have constrictions between the seeds that they open in three parts and are released at over-maturity by dehiscence.	
Seeds	The seeds are round, with a semi-permeable brown shell. The shell has three white wings and contains an almond. A tree can produce 15,000 to 25,000 seeds per year. A seed weighs on average 0.3 g and the hull represents 25% of the weight of the seed	

Roots	The root system has a tubular structure, it is formed of a central pivot which can sink into the ground up to 1.30 m deep; which gives it its great resistance to drought. Secondary roots then branch laterally from the latter until they form a dense head of hair.	

I.5. medicinal properties of moringa

Medicinal potential of MO is vast. All parts and the components derived from plant viz. root, bark, gum, leaf, fruit pods, flowers, seed, and seed oil have been used for alleviating various ailments in traditional medicine. Moringa is a cornucopia of diverse activities, of which some have been reviewed below.

1. **anti-inflammatory activity:** Several parts of Moringa plant have been shown to possess anti-inflammatory activity. Poultice of leaves helps in glandular swellings.
2. **antioxidant activity**: Mo has antioxidant galore. Aqueous extracts of leaf, fruit and seed of Mo act as an antioxidant.
3. **anticancer activity:** Mo has several bioactive compounds showing antitumor activity. Niazimicin, is a bioactive ompounds found in Mo leaves, has anticancer activity.
4. **protection in eye diseases**: Vitamin A deficiency is a major cause of blindness. Consumption of Mo leaves, and pods and leaf powder, rich source of vitamin A, can prevent night blindness and eye problems in children.
5. **antidiabetic activity:** Mo potential as a therapeutic agent for diabetes has been explored. are potent source of polyphenols, responsible for hypoglycemic activity. The extract from Moringa leaf decreases sugar levels in the blood within 3 h after intake.
6. **reduce blood pressure:** Moringa powder has anti-hypertensive properties because it contains quercetin.

7. **lower cholesterol:** Both animal- and human-based studies have shown that Moringa oleifera have similar cholesterol-lowering effects..
8. **Protecting the liver:** Moringa might help protect the liver against nonalcoholic fatty liver disease.
9. **very nutritious:** Moringa leaves are an excellent source of many vitamins and minerals. One cup of fresh, chopped leaves (21 grams) contains:
   - Protein: 2 grams
   - Vitamin B6: 19% of the RDA
   - Vitamin C: 12% of the RDA
   - Iron: 11% of the RDA
   - Riboflavin (B2): 11% of the RDA
   - Vitamin A (from beta-carotene): 9% of the RDA
   - Magnesium: 8% of the RDA

However, there is one downside: Moringa leaves may also contain high levels of antinutrients, which can reduce the absorption of minerals and protein. **[32]**

II. Phytochemical study

In plant metabolism,we differentiate between primary metabolites and secondary metabolites. Primary metabolites include molecules synthesized by the body which play a direct role in vital functions such as nutrition, growth and reproduction. Made up of atoms such as carbon, oxygen and nitrogen, primary metabolites are classified into four main categories according to their properties biochemical: carbohydrates, lipids, amino acids and nucleic acids.

The secondary metabolites, for their part, play several roles such as serving as defense for plant or attract certain pollinating species. They also enable communication from plants to plants. **[33.34]**

II.1. Phytochemical screening of the plant

II.1.1. Alkaloids

Alkaloids are complex nitrogen-containing heterocyclic organic molecules,of natural origin, basic in nature, generally presenting intense activity pharmacological. For the most part, they are very active plant poisons, with an action specific. Medicine most often uses them in their pure state. Morphine was the first alkaloid isolated from opium (in 1805). Then we discovered strychnine (1818), caffeine (1819), etc.. refers to their “alkaline” or “basic” character .Alkaloids are found as secondary metabolites mainly in plants, fungi and a few small animal groups. Usually the alkaloids are derivatives of amino acids. **[35]**

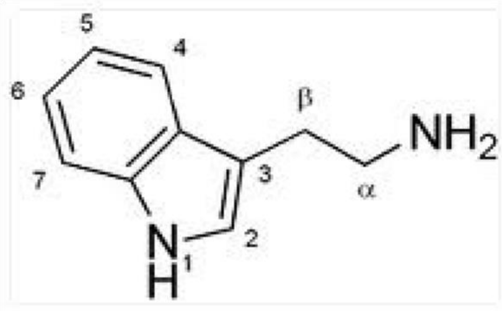


Fig II.12: structure of alkaloids [36]

II.1.2. Flavonoids

Present in most plants, are polyphenolic pigments which contribute, among other things, to coloring flowers and fruits yellow or white. They have an important field of action and possess numerous medicinal antioxidant properties. They are particularly active in maintaining good circulation. Certain flavonoids also have anti-inflammatory and antiviral properties, and protective effects on the liver. Flavonoids such as hespendin and rutin, present in several plants, including buckwheat (Fagopyrum esculentum) and lemon (Citrus limon), strengthen the walls of capillaries and prevent infiltration into neighboring tissues. Isoflavones, found for example in red clover (Trifolium pratense), and lemon (Citrus limon) have estrogenic effects, are effective in the treatment of disorders linked to menopause. **[24]**

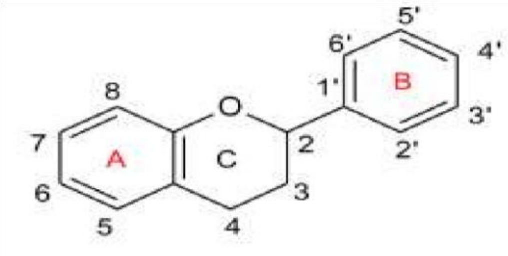


Fig II. 13: Basic structure of flavonoids

II.1.3. Tannins

Tannins are complex phenolic compounds, found in many plant species, with a molecular weight ranging from 500–3000 Da. They are found in fruits, berries, chocolates, and other dietary components, and because of their ability to bind and precipitate proteins, tannins were considered antinutrients. **[37]**

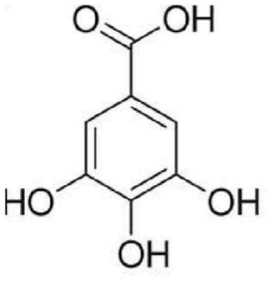


Fig II.14: structure of tannins

II.1.4. Saponins

Saponins compounds are a large group of secondary metabolites occurring in significant amounts in many plant species. Plants can synthesize and accumulate a comprehensive spectrum of these compounds in response to physiological stimuli and stress. **[39]**

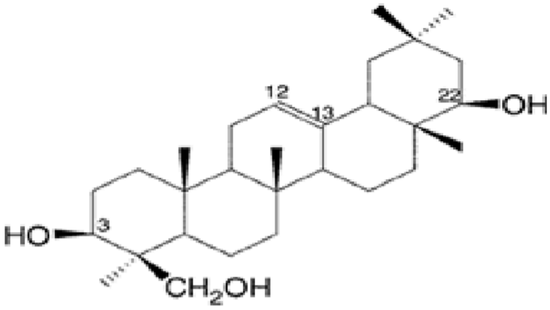


Fig II.15: structure of saponins

II.1.5. Anthocyanins

Anthocyanin is a particular variety of phenolic compounds which is associated to the purple color of açai and has exhibited important therapeutic effects such as anti-inflammatory, UV radiation-protective, chemoprotective properties, as well as the reduction of the risk of cardiovascular diseases. **[33]**

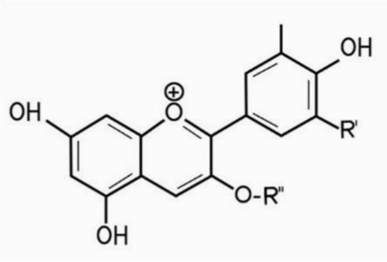


Fig II.16: General structure of anthocyanins

II.1.6. Glucosides

Glucosides are carbohydrate analogues resulting from the condensation of aglycones and non-carbohydrate substances. Glucosides are molecules made up of a carbohydrate function associated with various other types of molecules. Many plants store these glycosides in an inactive form. These molecules are made up of 2 distinct parts: the glycone, which corresponds to the carbohydrate group (made up of one or more carbohydrate functions), and the aglycone, which is the non-carbohydrate part of the molecule. Glucosides can be classified according to the chemical function of the aglycone part. **[30]**

II.1.7. Terpenes and sterols

Terpenes constitute an important group of natural products, comprising approximately 30,000 compounds. Steroids constitute a group of lipids derived from triterpenoids (lipids with 30 carbon atoms). They are described as compounds that are essentially constituents of an essential oil and contain carbon and hydrogen atoms with or without oxygen. They have received considerable interest in dermal and transdermal delivery of drugs with a wide range of physicochemical properties. **[30]**

II.2 Extraction methods

II.2.1. Definitions

Man uses colorings, perfumes, flavorings, and product extracts natural since ancient times. Extraction is a unit operation used to extract selectively one or more compounds from an initial mixture, on the basis of properties chemical or physical.

Extraction consists of transferring a compound from one phase to another:

- From one liquid phase to another liquid phase.
- From a solid phase to a liquid phase. **[39]**

II.2.2. Types d'extraction

Extraction is an operation which consists of separating certain compounds from a plant organism using various techniques. The choice of the extraction procedure is based on the physicochemical characteristics of the compounds to be extracted.. Deux procédures d'extraction sont généralement utilisées the presence of interfering substances. Two extraction procedures are generally used: solid-liquid extraction and liquid-liquid extraction used: solid-liquid extraction and liquid-liquid extraction. **[38]**

II.2.3. Solvent extraction

The choice of extraction method is based on prior data on the physicochemical characteristics of the metabolites to be extracted. We distinguish numerous extraction methods (solid-liquid) of phytochemical compounds:

II.2.3.1. Maceration

Maceration has been known and used at least since antiquity and just like decoction or infusion this is a solid-liquid extraction technique intended to remove of a solid substance the chemical species it contains by dissolving them in a liquid. This technique is most often implemented with plant parts (leaves, flower, root, bark etc.) using a solvent which may be water, alcohol and often a oil or other fat. Maceration is done by directly plunging solid substances into a liquid.

The latter are generally left in suspension for a certain time which can range from a few hours to a few days. Maceration is in fact a ―cold‖ extraction, which does not does not mean that it is accompanied by cooling but quite simply that it occurs at ambient temperature without benefiting from a rise in temperature which accelerates most chemical phenomena. At the end of the process it is necessary to remove the solid residues from the solvent, they are in generally eliminated by filtration. **[38]**

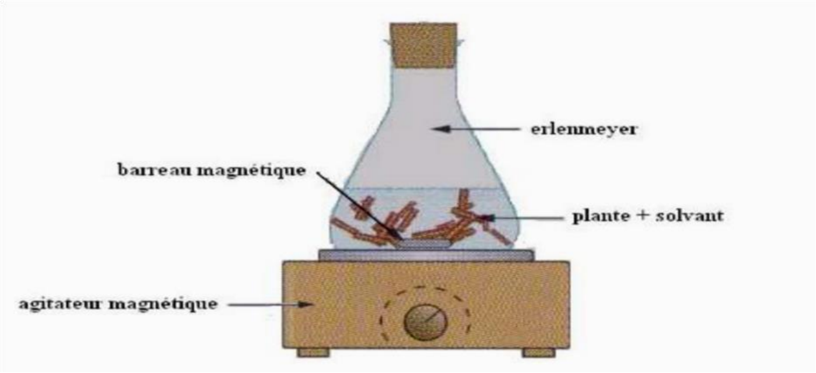


Fig II.17: Diagram of the Maceration technique

II.2.3.2. Soxhlet

The Soxhlet extraction is a combination of both, percolation and maceration methods. The extraction is carried out in a special apparatus known as Soxhlet apparatus that was designed by Franz von Soxhlet in 1879. It has been one of the most widely used extraction method which is still used extensively.

The major advantages of Soxhlet extraction is the increase in mass transfer rate due to use of high temperatures and recycling of fresh solvent (which further improves the transfer equilibrium). **[40]**

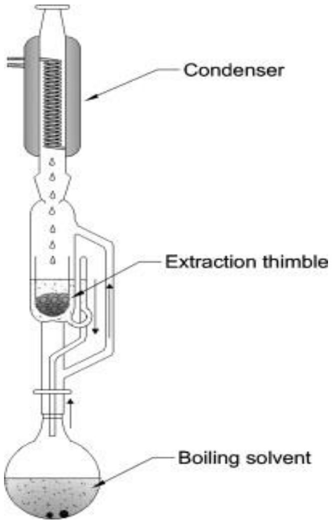


Fig II.18 : Conventional Soxhlet extractor

II.2.3.3. Hydrodistillation

This is the simplest method and therefore the oldest used. The principle of hydrodistillation is that of the distillation of immiscible binary mixtures. It consists of immersing the plant biomass in a still filled with water, which is then brought to the boil. The water vapor and the gasoline released by the plant material form a mixture not miscible. The components of such a mixture behave as if each were alone at the same time. temperature of the mixture, i.e. the partial vapor pressure of a component is equal to the vapor pressure of the pure substance. This method is simple in principle and does not does not require expensive equipment. However, due to water, acidity, temperature of the medium, hydrolysis, rearrangement, racemization reactions can occur, oxidation, isomerization, etc. which can very significantly lead to denaturation.

The duration of a hydrodistillation can vary considerably, being up to several hours depending on the equipment used and the plant material to be treated. The duration of the distillation influences not only the yield but also the composition of the extract. **[38]**

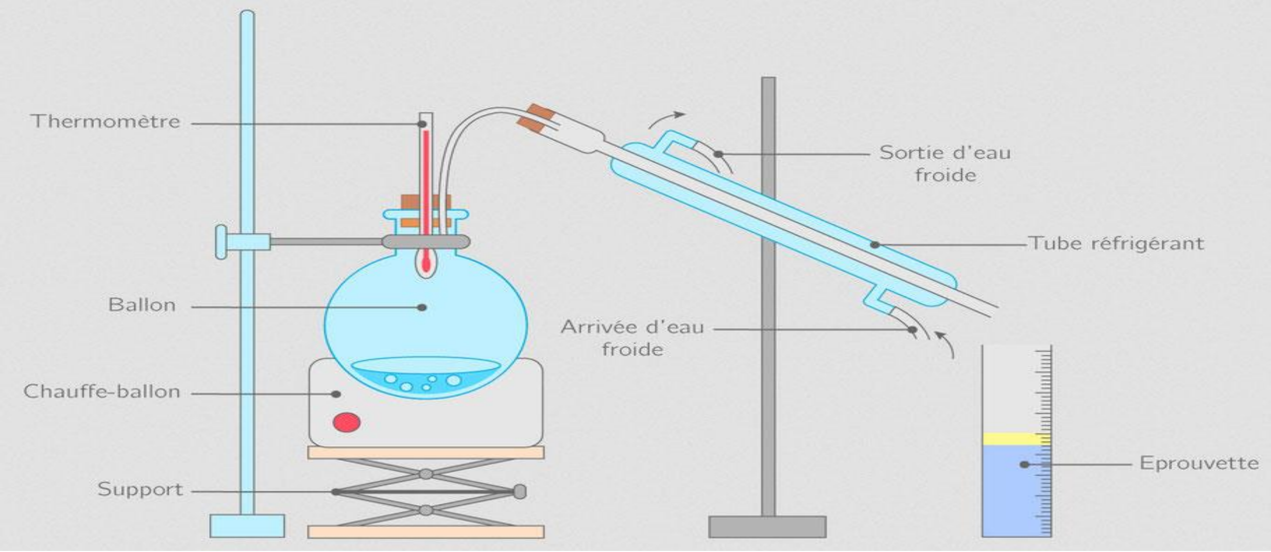


Fig II.19: Extractions by hydrodistillation

Experimental part

Chapter Three

Materials and methods

I. Phytochemical evaluating for the plant

Phytochemical screening is a bunch of tests completed on the powder. These tests permit us to have a thought of the presence or nonattendance of specific essential metabolites also, auxiliary in the plant.

The phytochemical bunches are various, yet we can refer to the fundamental ones: alkaloids, polyphenols (flavonoids, anthocyanins, tannins, saponosides,coumarins, sterols, terpenes, natural ointments).

I.1. Sample preparation

After being sorted and cleaned, the leaves are left to dry in the open air; they are then crushed using an electric grinder. The powder obtained was then filtering to obtain a powder and stored in glass jars protected from light to avoid any deterioration.

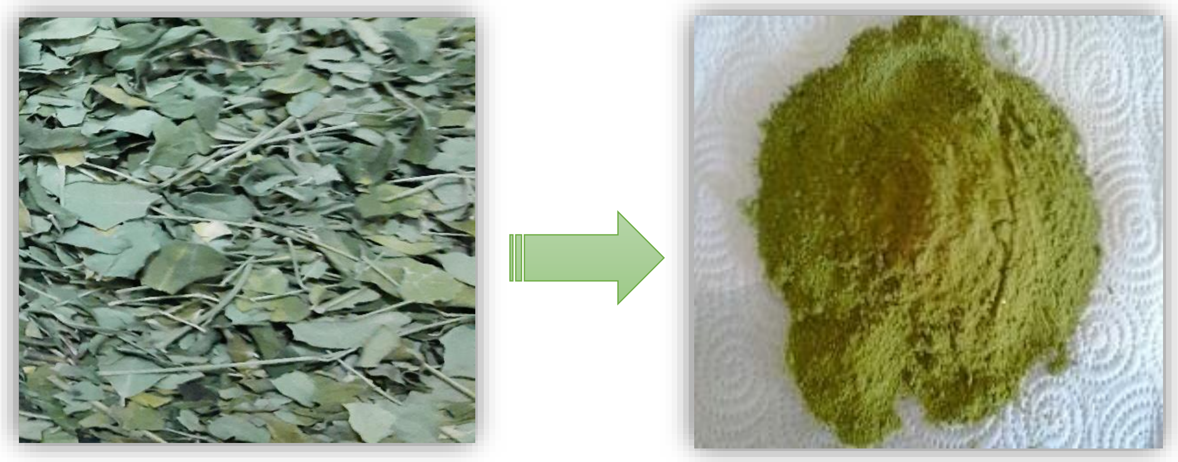


Fig III.20: Moringa oleifera leaves before and after grinding

I.2. Chemical composition of Moringa oleifera

I.2.1. Alkaloid Test

5g of the dried and squashed plant are blended in with 50ml of 1% HCl in a container. After 30 minutes of maceration, the combination is sifted and added to the filtrate. a couple of drops of Mayer's reagent (25g of KI dissolved in 20ml of distilled water added to 13.5g of $HgCl_2$ dissolved in 20ml of distilled water then completed with distilled water up to 1L).

- The appearance of a yellowish-white precipitate shows the presence of alkaloids.

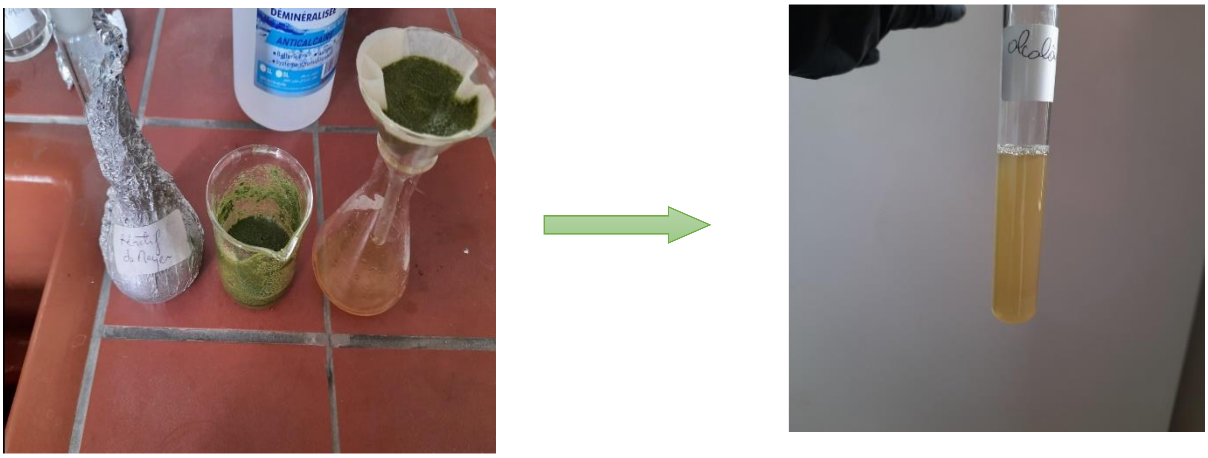


Fig III.21 : Detection of alkaloids

I.2.2. Test of Flavonoids

10 g of plant material are macerated in 150 ml of 1% HCL for 24 hours. After filtering the mixture, a 10 ml aliquot of the filtrate is taken, to which a few drops of NH_4OH (to make it basic) are added.

- The appearance of a yellow color in the upper part of the test tube indicates the presence of flavonoids.

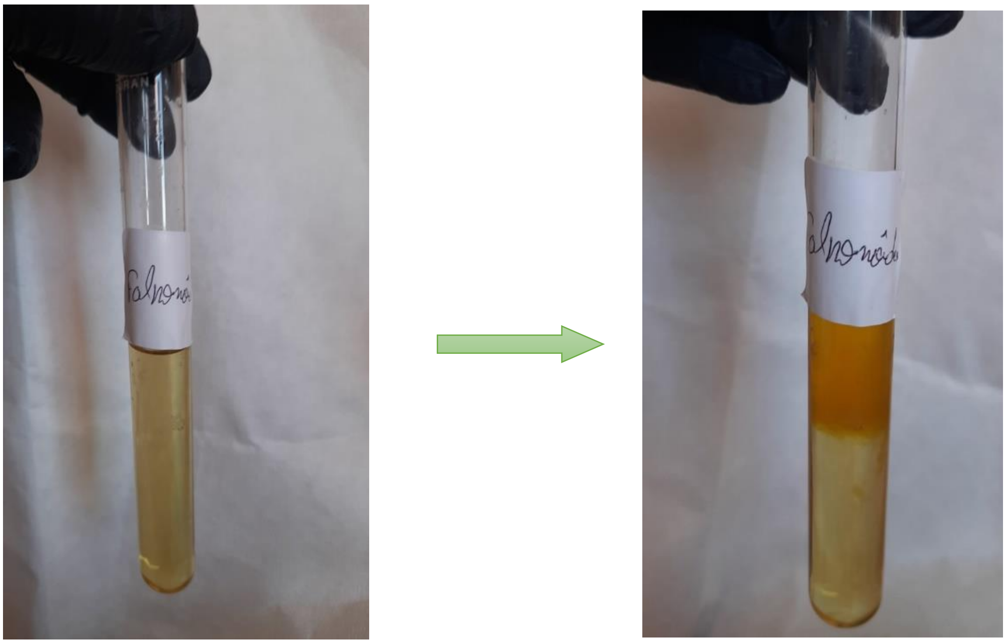


Fig III.22 : Detection of Flavonoids

I.2.3. Test for Tannins

Take 5g of the powder, extract with 20 ml of ethyl alcohol C_2H_5OH, under agitation for 15 minutes, then filter. Then test the filtrate with a few drops of $FeCl_3$.

- The appearance of a green color indicates the presence of tannins.

Fig III.23: Detection of Tannins

I.2.4. Test for Saponins

A mass of 2g of the plant powder was heated in 80ml of distilled water until boiling, after cooling, the mixture is filtered, and in a test tube, the tube is shaken lengthwise.

- The appearance of stable foam after 15 minutes indicates the presence of saponins.

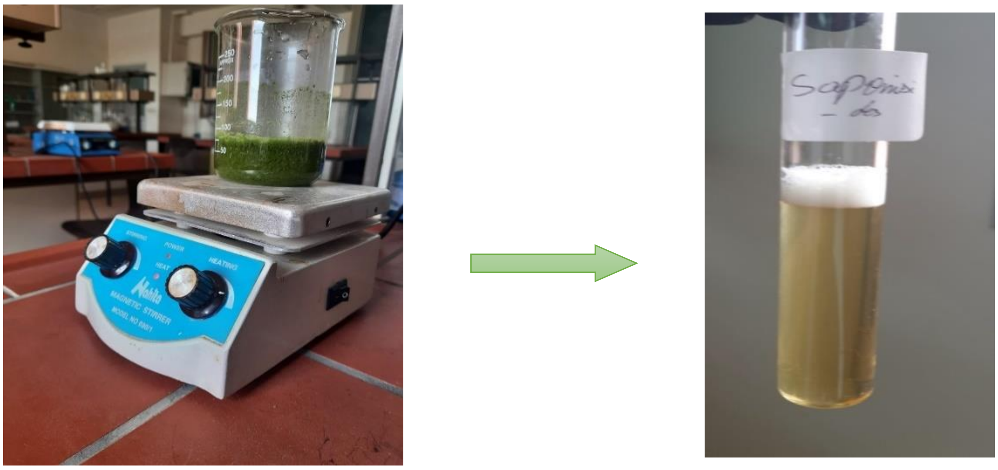


Fig III.24: Detection of Saponins

I.2.5. Test for Unsaturated Sterols and Terpenes

A 5g mass of powder was macerated in 20ml of chloroform. After filtration, 1ml of concentrated sulfuric acid (H_2SO_4) was added to the filtrate, carefully along the walls of the test tube. The point of contact between the two phases .

- The appearance of a violet or brown color turning towards gray indicates the presence of unsaturated sterols and terpenes.

Fig III.25: Detection of unsaturated sterols and terpenes

I.2.6. Test for anthocyanins

1g of dry powder is macerated in 10ml of distilled water, brought to a boil for 15 minutes, then filtered. A few drops of HCl and (NH4OH) are added .

- A change in color indicates the presence of anthocyanins.

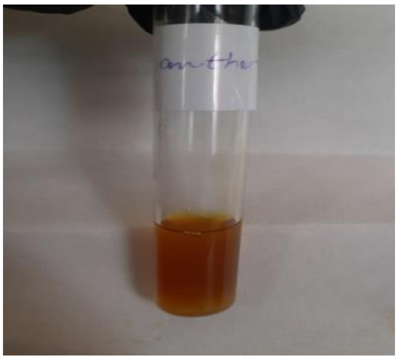


Fig III.26 : Detection of anthocyanins

I.2.7. Test for leucoanthocyanins

First, 1g of powder is placed in 5 ml of propanol, then drops of concentrated HCl are added. The mixture is heated for 3 to 5 minutes in a water bath.

- After heating, the appearance of a red color indicates the presence of leucoanthocyanins.

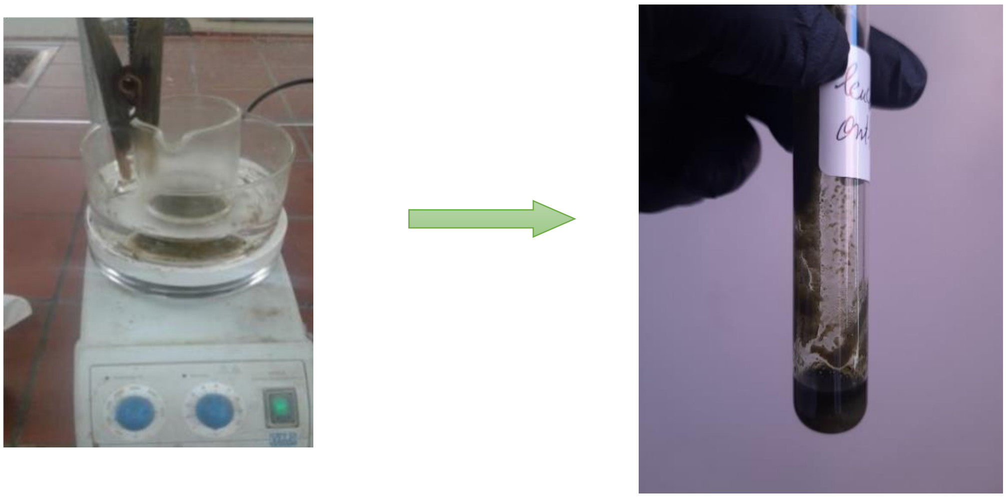


Fig III.27: Detection of leucoanthocyanins

I.2.8. Starch Test

Take 1g of powder in a test tube, add a few drops of iodine (I_2) until a change in color to purple-blue indicates the presence of starch.

Fig III.28 : Starch detection

I.2.9. Test for Glucosides

Put two drops of concentrated sulfuric acid on one gram of plant powder.

- The appearance of a brick-red color, then purple, indicates the presence of glucosides.

Fig III.29 : Detection of glycosides

I.3. Green inhibitors use

In our study, we used green inhibitors, namely Moringa oleifera extract.

I.3.1. Preparation of the inhibitor

A quantity of 50 g of plant powder is macerated in 300 ml of 70% methanol for 24 hours under agitation. The hydro-methanolic filtrate obtained is filtered using a vacuum flask and filter paper. The separation of the solvent from the extract is done using a device called a Rotavapor.

In the Rotavapor, we perform vacuum evaporation using a vacuum pump with a control valve. During evaporation, the flask is rotated and immersed in a heated liquid bath. The device is equipped with a condensate collector flask with a refrigerant. The rotation of the flask creates a larger and renewed exchange surface, allowing for rapid evaporation. The reduction of pressure allows the solvent to evaporate at a reduced temperature, thus avoiding potential thermal degradation of the compounds. It's a simple, useful, gentle, and rapid evaporation method.

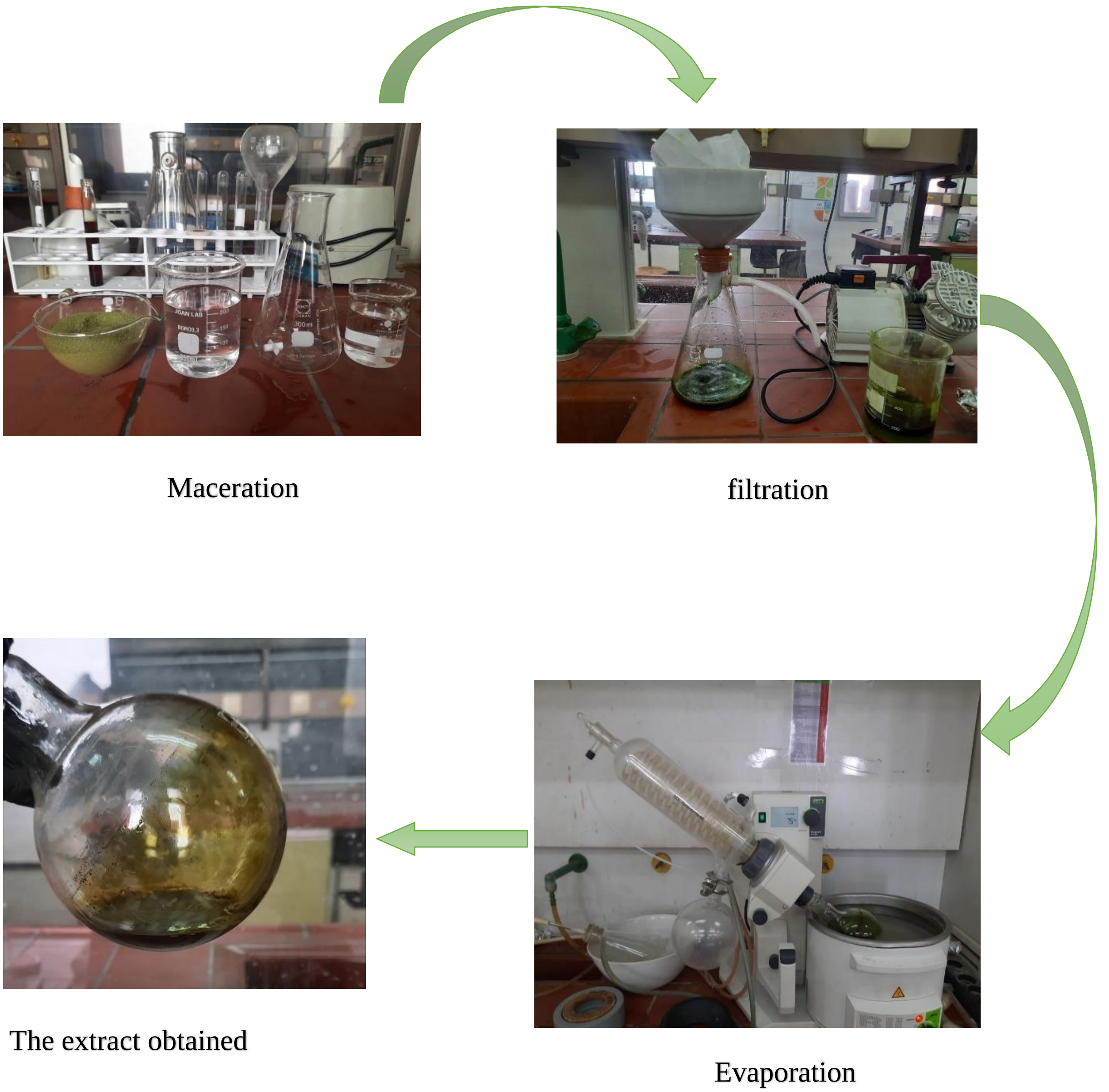


Fig III.30 :The steps of preparing the inhibitor

II. Gravimetric study of corrosion inhibition

II.1. Studied Material

The study material is micro alloy steel of API 5L X70 standard this material (X70) is used widely in petroleum and gas industry. Distilled and deionizer water was used for the preparation of the different solutions and purification of X70 .**[41]**

II.1.1. Characteristics of X70 Steel [42]

The material used as the working electrode is a carbon steel used for hydrocarbon transportation called API 5L X70, which meets the specification imposed by the API standard.

The API (American Petroleum Institute) standard is designed to be compliant with the oil industry's requirements regarding legislation and the environment. API 5L X70 steel is designated by its yield strength (70).

- API 5L stands for: pipeline.
- X70 stands for: the grade of the steel. Other grades include, for example, A, B, X60, X70.
- The number 70 signifies: 70,000 psi, which is the yield strength of the steel in pounds per square inch.

II.1.2. Chemical properties

Table III.6 : Chemical composition of the steel used. **[43]**

Element	C	Mn	Si	Cr	Ni	Mo
X70 %	0.125	1.68	0.27	0.051	0.04	0.021
Element	S	Cu	Ti	Nb	Al	p
X70 %	0.005	0.045	0.003	0.033	0.038	0.012

II.2. Sample preparation

II.2.1. Cutting

We prepare two types of samples. The first type is square-shaped with a diameter of 3cm and a height of 1cm for gravimetric studies. The second type of samples has been cut into cylindrical shapes with a diameter of 1.40 cm and a height of 1 cm for electrochemical studies.

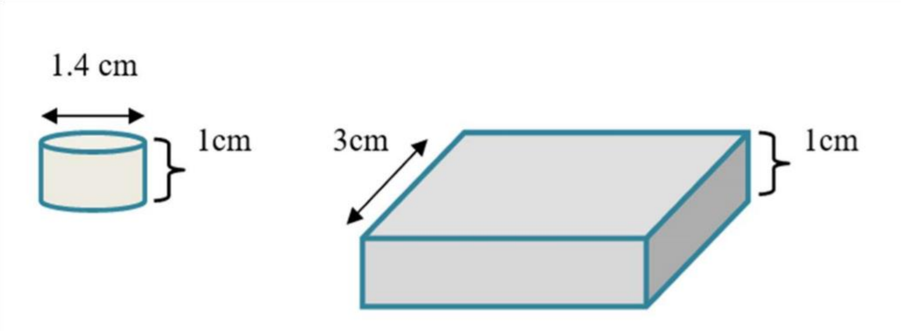


Fig III.31: Shape and dimensions of samples in cm

II.2.2. Polishing

The preparation of sample surfaces is carried out using abrasive papers of decreasing grain size: from 400 to 1200 mesh. Then, the piece is rinsed with distilled water and dried using an electric dryer to achieve a smooth surface, resembling a mirror finish. Subsequently, the samples are rinsed with acetone, washed with distilled water, dried for a few seconds, and immediately immersed in the corrosive solution.

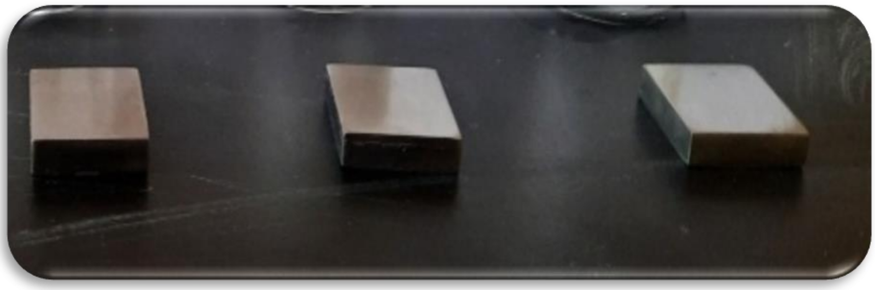


Fig III.32 :The sample after polishing

II.2.3. Preparation of corrosive environments

Corrosive environments are divided into two categories. They can either be inhibitor free (blank) or with inhibitor.From a concentrated solution of hydrochloric acid (mother) a daughter solution with a concentration of 1M was prepared**.** 1M HCl is obtained by diluting hydrochloric acid with distilled water.

II.3. Testing techniques (gravimetric study)

II.3.1 Experimental apparatus

In this study, mass loss tests were conducted in two different environments. The first one is HCl (without inhibitor) with a concentration of (1M), and the second one is HCl with inhibitor. Once the preparation of the sample surfaces is completed, each sample is weighed and immediately placed into beakers containing an HCl solution of varying concentrations.

Without inhibitor :

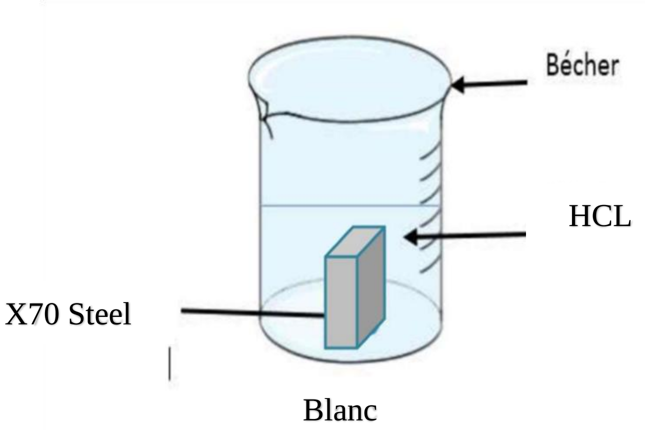


Fig III.33: Steel x70 in HCl acid medium without inhibitor

With inhibitor:

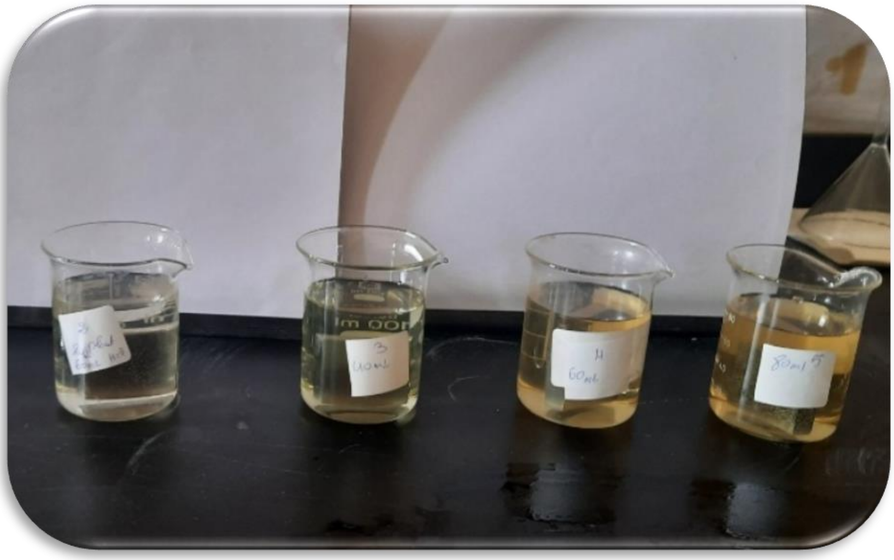


Fig III.34: Steel x70 in acidic HCl medium with inhibitor

After week :

- Take out the sample is washed, dried and weighed again.
- The corrosion rate (or corrosion velocity) is calculated using equation (1).
- Each product's inhibitory is determined using equation (2)

The major drawback of this method lies in the difficulty of completely removing corrosion products without attacking the base metal.

The values of the corrosion rate V_{corr} are estimated by the following formulas:

$$\mathbf{V=\Delta M/S\ .\ t\ (mg\ cm^{-2}\ d^{-1})} \qquad \mathbf{(1)}$$

Where Δm = (mi - mf) being the mass loss expressed in mg, S being the surface of the substrate and t the residence time of the substrate in the solution.

$$\boldsymbol{\varepsilon(\%) = (V - V_{inh})/V \times 100\%} \qquad \mathbf{(2)}$$

where V and Vinh stand for the steel's corrosion rate values during immersion in the absence and presence of the inhibitor, respectively. **[44]**

III. Electrochemical Study

III.1. Electrochemical Setup

The electrochemical study of the behavior of steel in corrosive environments is based on plotting polarization curves i=f(E) and the Tafel line. The experimental setup used consists of a potentiostat-galvanostat of type PGZ 301 (Radiometer brand) and a microcomputer equipped with VOLTAMASTER 4 software, along with a three-electrode electrolysis cell.

Fig III.35 : Electrochemical Setup

III.2. Electrochemical Cell

Fig-36 shows the three-electrode electrochemical cell (Working electrode; Reference electrode; Auxiliary electrode).

1- Working electrode: This is an electrode made from an X70 steel sample in cylindrical form, 1 cm in height and with a diameter of 1.4 cm (active surface).

2- Reference electrode: This is a saturated calomel electrode in potassium chloride. This reference electrode, noted as ECS, is unpolarizable, with a rigorously constant potential.

3- Auxiliary electrode or counter electrode: The auxiliary electrode is made of platinum (with a 1 cm active surface), intended to facilitate the passage of electrical current in the electrolysis cell.

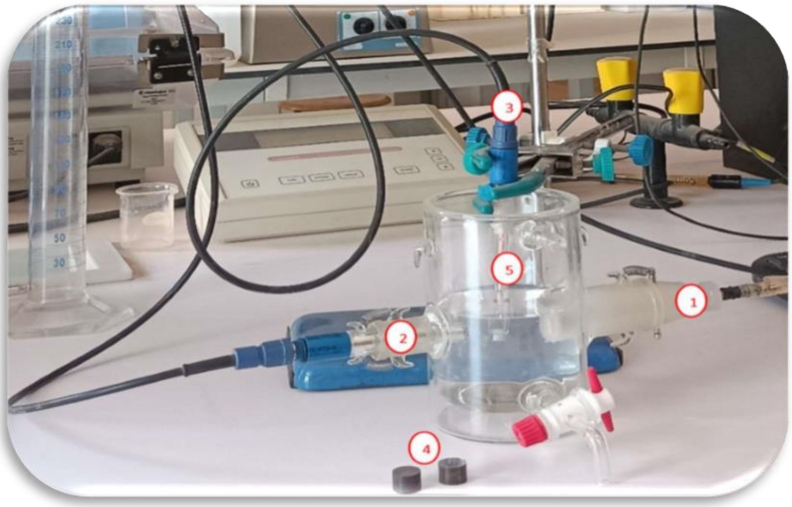


Fig III.36 : Electrochemical Cell

1- Working electrode

2- Auxiliary electrode or counter electrode

3- Reference electrode

4- Samples

5- The cell containing 250 ml of hydrochloric acid

Chapter Four

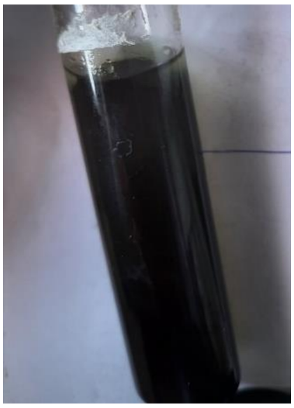
Results and discussions

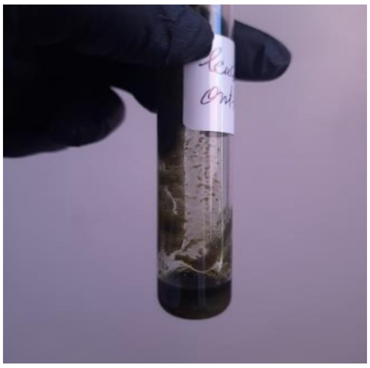
I. Phytochemical screening

To detect the presence of certain chemical compounds in the moringa plant, we conducted several phytochemical tests. This study allowed us to highlight the presence of some chemical compounds as represented in the table below:

Table IV.7: results of phytochemical tests of the moringa plant

Chemical composition	Result	
Alkaloid	++	 Yellowish white precipitate
Flavonoids	+++	 yellow
Tannins	+++	 Green

Saponins	++	 appearance of foam
Unsaturated Sterols and Terpenes	+++	 a violet or brown color towards gray
Anthocyanins	+++	 A change in color

Leucoanthocyanins	-	 The color has not changed
Starch	+	 a change in color to purple-blue
Glucosides	++	 The appearance of a brick-red color, then purple

- The sign (+): low presence.

- The sign (++): moderate presence.

- The sign (+++): strong presence.

- The sign (-): absence.

This table shows that there are considerable amounts of nitrogenous and occasionally oxygenated organic compounds in moringa extract.

Because these substances which include tannins, saponosides, and flavonoids are often heterocyclic aromatic, their extracts or essential oils have been employed as corrosion inhibitors in harsh conditions.

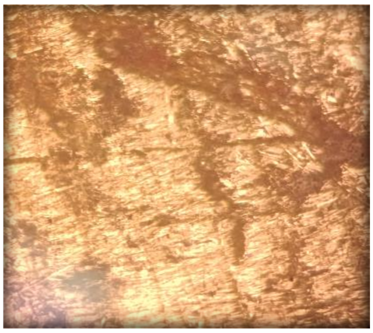
II. Gravimetric study

II.1. Microscopic observation of steel

Observation by optical microscope of X70 steel surfaces before and after immersion in acidic environments of 1M HCl, without and with inhibitor is shown in the following images:

1- Before immersion in acidic mediumimer

2- After immersion in acidic mediumimer **+ inhibitor**



3- After immersion in acidic mediumimer

We can observe fig (2) the formation of a thin layer that protects the steel surface from corrosion when using the Moringa plant extract as a corrosion inhibitor in acidic media, unlike the obvious corrosion of the steel surface seen in the fig (3) in acidic media.

II.2. Effect of inhibitor concentration in 7 days of immersion

In order to determine the optimal concentration in 7 days of immersion , we have carried out gravimetric measurements of X70 steel in HCl medium at different concentration (0, 0.75 ,1.5 ,2.25and 3)* 10^{-3}M

The table gives the values of mass loss (Δm), corrosion speed (V) of X70 steel after 7 days of immersion

Table IV.8 : Variations in mass loss, corrosion rate

C (g/l)	M before	M after	Δm (g)	Vcorrosion (g/cm^2 day)	ε%
Blank	53.829	52.422	1.387	6.605	**-**
0.75	51.936	51.322	0.614	2.924	**55.73**
1.5	53.950	53.469	0.481	2.290	**65.33**
2.25	55.399	55.037	0.362	1.724	**73.90**
3	54.018	53.878	0.140	0.667	**89.90**

The variation of the inhibitory efficiency (ε%) of this green inhibitor with the concentrations, is reported in (Figure 38) this figure shows that the inhibitory efficiency increases with the increase in the concentration of the inhibitor so there is a proportionality between these two parameters and we achieve an inhibitory efficiency of 89.90 % in the presence of a quantity of our inhibitor corresponding to a concentration of 3 $\times 10^{-3}$ g/l.

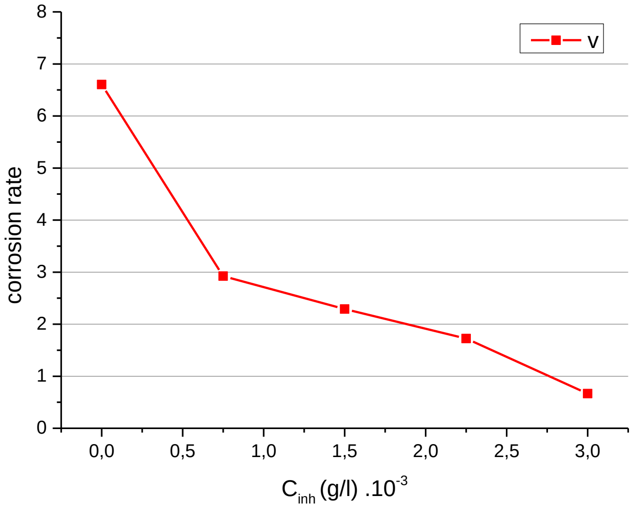


Fig IV.37 : Variations in V corrosion at different inhibitor concentrations

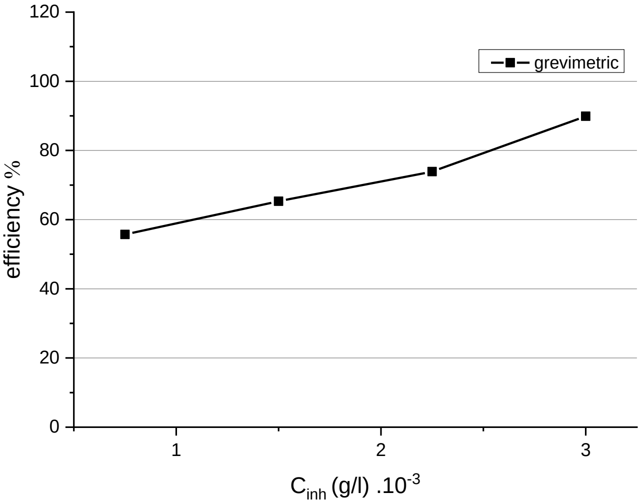


Fig IV.38 : Variations of inhibitory efficiency

III. Electrochemical method

III.1. Free potential measurement

The polarization curves i =f(E) require knowledge of the free potential. After different tests, we opted for immersion of the sample for a period of 30 minutes. This time is sufficient to obtain satisfactory stabilization of the potential free. In hydrochloric acid solution (HCl 1M). Before each manipulation the electrode work is polished with abrasive paper of different grain sizes.

III.2. Polarization curve

The polarization curve was drawn in a potential range corresponding to [-700 mV to -200 mV], and we opted for a scanning speed equal to 30 mV/min. This speed allows us to obtain good reproducibility of the results without hiding the phenomena to be studied. The cathodic and anodic polarization curves of steel in 1 M HCl medium at 25°C, in the absence and presence of inhibitor, at different concentrations, are presented in (Figure 39).

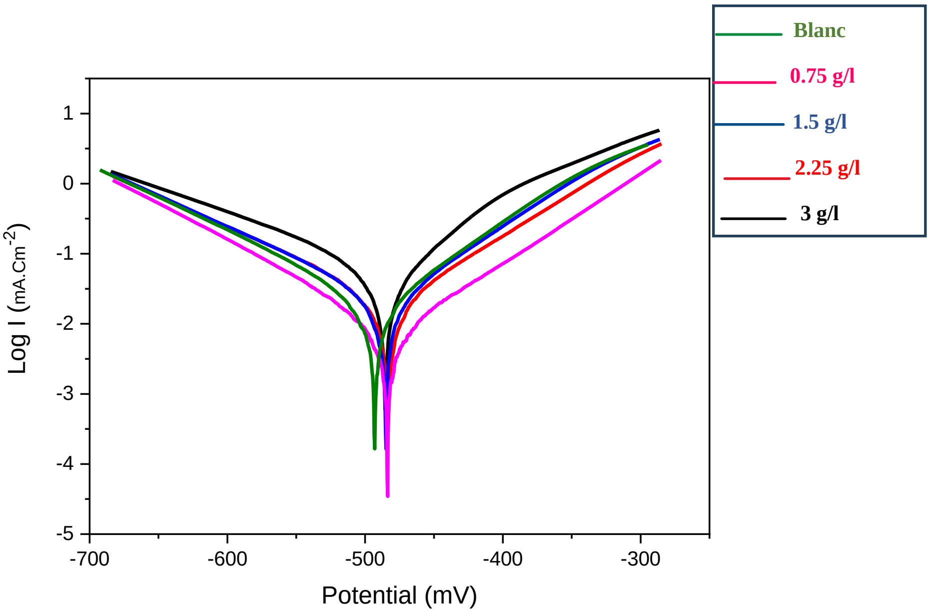


Fig IV.39 : Tafel curve of X70 steel in an acidic environment without and with inhibitor

The figure presents the polarization curves obtained for the X70 steel. The density of passivation current, corrosion potential and current, etc. are determined from these curves. Only the linearity domain of the cathode and anodic branches will be taken into consideration in the plot of the Tafel polarization curve. The corrosion rate is calculated by the VOLTA MASTER 4 software, using Tafel lines.

The current density values corrosion (Icorr), the corrosion potential (Ecorr), the cathodic and anodic Tafel slopes (bc and ba) and the inhibition efficiency ε (%) for different concentrations of the inhibitor in HCl medium are reported in the following:

Table IV.9 : Values of electrochemical parameters

C (g/l)	E (i=0) [mV]	Rp [ohm.cm^2]	icorr [mA/cm^2]	Ba [mV]	Bc [mV]	Vcorrosion [g / Cm^2 day]	ε%
Blanc	-485.3	410.40	0.0675	84.8	-148.3	789.8	**-**
0.75	-483.5	982.45	0.0245	89.6	-116.3	287.0	**63.70**
1.5	-492.9	976.81	0.0241	83.8	-110.0	281.5	**64.30**
2.25	-484.9	907.08	0.0236	81.6	-113.2	276.3	**65.04**
3	-483.1	715.06	0.0115	86.6	-100.6	134.4	**82.96**

- **Rp**, Polarization resistance
- **Corrosion current**: obtained on the ordinate axis at the intersection of the tangents.
- **βa and βc**: Anodic and cathodic Tafel coefficient.
- **V$_{\text{Corrosion}}$** (Corrosion or corrosion rate)

The analysis of these curves shows that there is an improvement in the corrosion potential as the inhibitor concentration increases. we can deduce that the densities of the corrosion current (Icorr) decrease as the concentration of the inhibitor increases.

The same is true for corrosion rates. The inhibitory efficiency increases with the concentration of the inhibitor and reaches a maximum value of 82.96 % at a concentration of 3 10^{-3} g/l. These results highlight the mixed nature of the inhibitor used and are in good agreement with those obtained by the mass loss method.

III.3. Electrochemical impedance curves

The Nyquist diagrams of the steel immersed in the acid solution without and with the addition of different concentrations of the inhibitor are shown in the figure 41

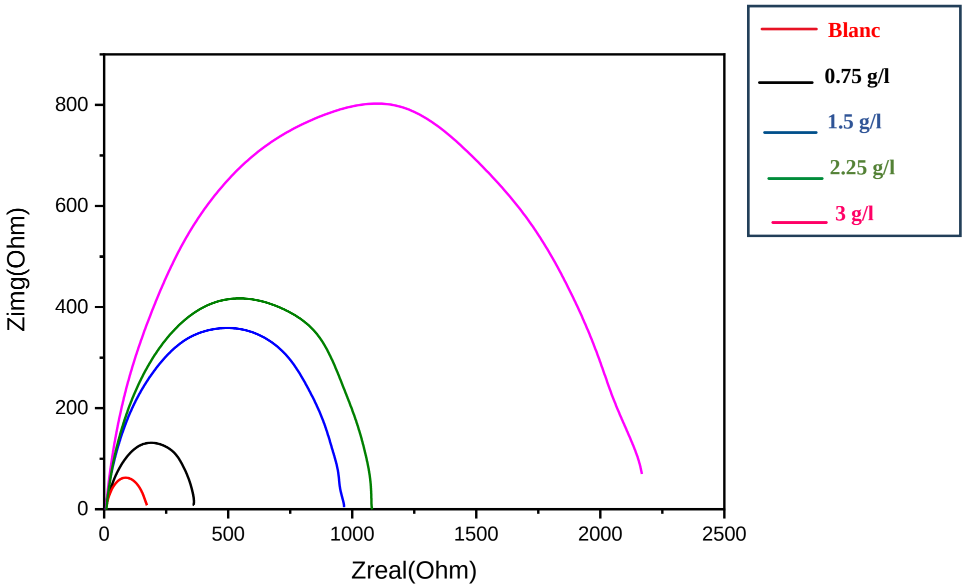


Fig IV.41 : Diagrams of electrochemical impedance spectroscopy in an acidic medium without and with inhibitor.

The resulting impedance patterns are not perfect semicircles, and this is attributed to the frequency dispersion of the interfacial impedance, generally due to the heterogeneity of the electrode surface.

The corrosion inhibitory effectiveness of steel is calculated from the load transfer resistance according to the relationship:

$$\mathcal{E}\% = \frac{R_{tinh} - R_t}{R_{tinh}} \times 100$$

where Rt and Rt (inh) are respectively the values of the load transfer resistances of the steel after immersion without and with addition of the inhibitor. The values of the electrochemical parameters and the inhibitory efficiency for different concentrations of our inhibitor for the corrosion of steel in 1M HCl medium obtained by electrochemical impedance spectroscopy (ESI) are gathered in the following table.

Table IV.10: Values of the electrochemical parameters and the inhibitory efficiency for different concentrations

C_{in} 10^{-3} (g/l)	R1(Ω.cm^2)	C (µF/cm^2)	R2(Ω.cm^2)	ε%
Plank	7.502	609.0	165.1	-
0.75	8.058	44.17	360.2	**54.16**
1.5	5.973	16.53	962.5	**82.84**
2.25	8.859	14.77	1077	**84.67**
3	1.292	18.46	2154	**92.33**

The analysis of these results leads us to draw the following remarks:

- The Rt values increase considerably with increasing inhibitor concentration while the capacitance of the film formed decreases to reach a value of 17.44 µF/cm^2.
- Increasing the concentration of the inhibitor seems to reduce the surface area of active sites present on the surface of the metal. This reduction may be due to the blocking of the active surface by the adsorption of the inhibitor on the surface of the metal and consequently the increase in the transfer resistance Rt which gives a maximum inhibitory efficiency of 92.33% at 3 10^{-3}g/L.

III.4. A comparative study of the influence of the inhibitor concentration on the three analysis methods

The variation in the inhibitory efficiency of the inhibitor as a function of the inhibitor concentration in 1M HCl of the different methods is illustrated in (Figure 40). The inhibitory efficiency increases with increasing concentration. We note that there is good agreement between the values of inhibitory efficiency determined by gravimetry, polarization curve and that resulting from impedance measurements.

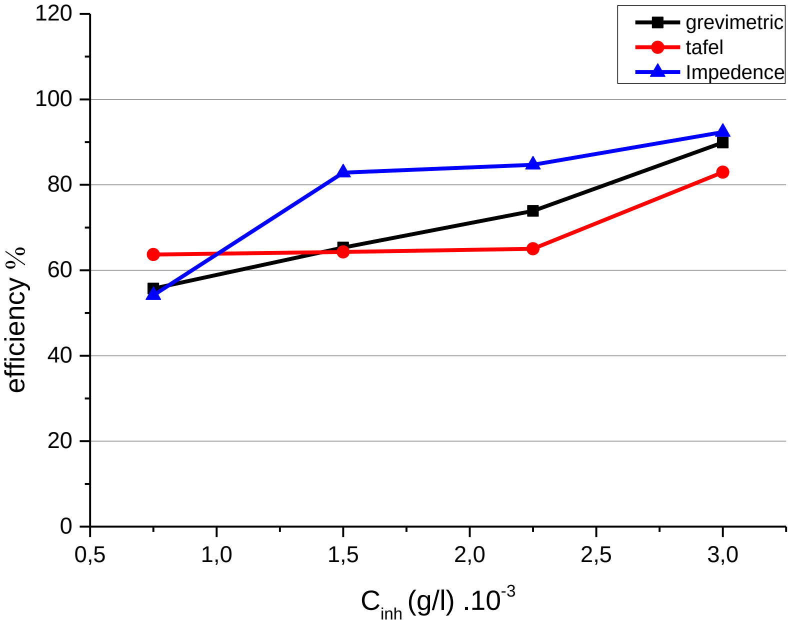


Fig IV.41: Variation in the inhibitory efficiency of the inhibitor as a function of its concentration in 1M HCl, of the three analysis methods

General conclusion

General conclusion

The work presented in this dissertation is focused on the study of the corrosion inhibition of X70 steel in an aggressive medium of hydrochloric acid HCl 1M, using Moringa oleifera leaf extracts as a green inhibitor Followed by a comparative study of the inhibitory efficiency using gravimetric method and electrochemical methods (polarization and impedance).

The workflow proceeded as follows: First, we confirmed the presence of the chemical compounds (tannins, saponicides, flavonoids, alkaloids, etc...) in the Moringa oleifera extracts. After that, the plant extract was prepared by soaking in order to use it as an inhibitor in acidic media HCl 1M. We also prepared X70 steel samples in square and cylindrical shapes. Finally, we tracked the corrosion process in acidic media using an optical microscope and calculated the inhibitory efficiency using different measurement techniques (gravimetric method and electrochemical method).

The results that arise from this work are the following:

- Phytochemical screening of plants confirms the richness of the extract in large quantities of nitrogenous organic matter and sometimes oxygen. These are generally heterogeneous aromatic substances such as: tannins, saponisides, flavonoids and alkaloids, Therefore, it was chosen in this study to apply it as a corrosion inhibitor in aggressive environments.
- The mass loss of X70 steel in hydrochloric acid medium decreases rapidly with increasing inhibitor concentration in acidic medium.
- The rate of steel corrosion decreases with an increase in the inhibitor ratio in the acidic medium. It reaches a maximum value of 82.96% at a concentration of 3×10^{-3} g/L, using the gravimetric method.
- Examination using an optical microscope confirms the formation of a protective layer on the steel surface that protects it from corrosion.
- We also achieved similar results using electrochemical methods (polarization and electrochemical impedance), where the inhibitory efficiency was 82.96% and 92.33%, respectively.

Finally, this work made it possible to highlight and promote green inhibitors (environmentally friendly materials) for use in the indust

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Summary :

Through this work, we aim to study the corrosion of X70 steel in acidic medium HCl, and to study the inhibitory effect of steel corrosion using Moringa oleifera leaf extracts.

We initially conducted a chemical examination to determine the most important active elements present in the plant extract, and the results obtained confirmed that the plant extracts have a good effect against the corrosion of steel. We found, using the gravimetric method, that the inhibitory efficiency increases with increasing inhibitor concentration and reaches a maximum value of 82.96% at a concentration of 3.10^{-3} g/L.

We also achieved similar results using electrochemical methods (polarization and electrochemical impedance), where the inhibitory efficiency was 82.96% and 92.33%, respectively.

Keywords: Steel corrosion, Green inhibitor, Gravimetric method, Electrochemical method.

Résumé :

A travers ce travail, nous visons à étudier la corrosion de l'acier X70 en milieu acide HCl, et à étudier l'effet inhibiteur de la corrosion de l'acier à l'aide d'extraits de feuilles de Moringa oleifera.

Nous avons d'abord procédé à un examen chimique pour déterminer les éléments actifs les plus importants présents dans l'extrait végétal, et les résultats obtenus ont confirmé que les extraits végétaux ont un bon effet contre la corrosion de l'acier. Nous avons constaté, en utilisant la méthode gravimétrique, que l'efficacité inhibitrice. augmente avec l'augmentation de la concentration d'inhibiteur et atteint une valeur maximale de 82,96 % à une concentration de 3.10^{-3} g/L.

Nous avons également obtenu des résultats similaires en utilisant des méthodes électrochimiques (polarisation et impédance électrochimique), où l'efficacité inhibitrice était respectivement de 82,96 % et 92,33.%

Mots clés : Corrosion de l'acier, Inhibiteur de vert, Méthode gravimétrique, Méthode électrochimique.

الملخص:

نهدف من خلال هذا العمل إلى دراسة تآكل الفولاذ X70 في الوسط الحمضي HCl ، ودراسة التأثير التثبيطي لتآكل الفولاذ باستخدام مستخلصات أوراق نبات المورينجا اوليفيرا، حيث قمنا في البداية بالفحص الكيميائي لمعرفة أهم العناصر الفعالة الموجودة في المستخلص النباتي.

أكدت النتائج المتحصل عليها أن للمستخلصات النباتية تأثيرا جيدا ضد تآكل الفولاذ، حيث وجدنا باستخدام الطريقة الوزنية أن الكفاءة التثبيطية تزداد بزيادة تركيز المثبط وتصل إلى قيمة قصوى تبلغ 82.96% عند تركيز $3\ 10^{-3}$ غ/لتر.

كما توصلنا إلى نتائج مقاربة باستخدام الطرق الكهروكيميائية (الاستقطاب والمعاوقة الكهروكيميائية) حيث كانت الكفاءة التثبيطية 82.96% و 92.33% على الترتيب.

الكلمات المفتاحية: تآكل الفولاذ، المانع الأخضر، الطريقة الوزنية، الطريقة الكهروكيميائي...

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تصريـــح شرفـــي

خاص بالالتزام بقواعد النزاهة العلمية لإنجاز بحث

(ملحق القرار 1082 المؤرخ في 2021/12/27)

قسم علوم المادة

أنا الممضي أسفله،

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المسجل بكلية: علم المادة قسم: علوم المادة

والمكلف بانجاز أعمال بحث : مذكرة ماستر في الكيمياء

عنوانها: Green Corrosion inhibition of carbon steel in acid medium using leaves extract of Moringa oléfera

أصرح بشرفي أني ألتزم بمراعاة المعايير العلمية والمنهجية ومعايير الأخلاقيات المهنية والنزاهة الأكاديمية المطلوبة في انجاز البحث المذكور أعلاه وفق ما ينص عليه القرار رقم 1082 المؤرخ في 2021/12/27المحدد للقواعد المتعلقة بالوقاية من السرقة العلمية ومكافحتها.

التاريخ: 04/06/2024

إمضاء المعني بالأمر