



Mohamed Khider University of Biskra
Faculty of Science and Technology
Department of Industrial Chemistry

Master's Thesis

Field: Science and Technology

Program: Process Engineering

Specialization: Chemical Engineering

Réf. : Entrez la référence du document

Submitted and defended by:
ZEKKOUR Hadil / KAB Ala

Le : 18 /06/2025

EXTRACTION CHARACTERIZATION AND APPLICATION OF BIOACTIVE INHIBITORS FROM PLANT-BASED RESOURCES

Jury :

Dr.	GHEBGHOUB Fatima	MCA	University of Biskra	President
Dr.	ADAIKA Kaltoum	MCA	University of Biskra	Examiner
Dr.	HADJEB Rihana	MCA	University of Biskra	Supervisor
Dr.	MENASRA Hayet	Pr	University of Biskra	Supervisor

Academic Year : 2024 / 2025



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Dedication:

اللهم لك الحمد والشكر كما ينبغي لجلال وجهك وعظيم سلطانك

وبكل فخر أهدي تخرجي وفرحتي التي انتظرتها طويلا إلى من كانوا
مصدر الدعم والعطاء دائما.

إلى النور الذي أضاء دربي إلى العزيز الذي حملت اسمه فخرا إلى معلمي
الأول الرجل الذي سعى طوال حياته لتكون الأفضل

أبي الغالي

من كانت الداعم الأول لتحقيق طموحي إلى من كانت ملجأ يدي و
اليمنى في هذه المرحلة ..

إلى من أبصرت بها طريق حياتي واعتزازي بذاتي ... إلى القلب الحنون
إلى من كانت دعواتها تحيطني

أمي الحبيبة

إلى مصدر قوتي الداعمين والساندين إلى خيرة أيامي وصفوتها إلى
ضلعي الثابت وأمان أيامي

أخي الغالي وأخواتي الغاليات

إلى من حبهم يعلو فوق كل حب ... لكل من كان عوننا وسندا في هذا
الطريق ... إلى نوري المضاء الذي لا ينطفئ

إلى صديقات دربي خديجة رفيدة اية شيماء شكرا لدعمكم لي فلقد كنتم
حسن الرفقة في مشواري الجامعي

اهداء

بفضل الله سبحانه وتعالى، حمداً وامتناناً على ما بدأناه وتوَجَّنا به بلطفه ونعمه،

{وَآخِرُ دَعْوَاهُمْ أَنِ الْحَمْدُ لِلَّهِ رَبِّ الْعَالَمِينَ}

أهدي حصيلة إجتهادي المتواضعة

إلى من زرع في قلبي الإيمان، وفي عقلي العزم، أبي العزيز محمد فوزي،

وإلى من علّمتني أن الحنان دعاء، وأن الصبر عطاء، أمي الغالية حاجي غالية

إلى أخواتي العزيزات رفيقات الدرب ضحكاتكنّ كانت موسيقى الطريق، وسندكنّ ظلّ لا يغيّب : نسيبة، تسنيم، دانية، ساجدة و إخوتي : ذاكر و سبيل شكرا لكم

وإلى صغيرتنا إيلاف، حبيبة القلوب وأول فرحتنا

إلى كل أفراد عائلتي كل بإسمه وإلى جدي الحبيب الذي رحل عنا لكنه لا يزال حياً في ذكرياتنا ودعائنا

إلى صديقتي الروح و القلب يسرى وملاك

لكل من تقاسم معي ضحكات مسيرتي و تعب الإجتهد من أصدقاء الرحلة الدراسية

وإلى أساتذتي الأفاضل في كل المراحل، من أشعلوا شموع العلم في دهاليز الجهل، فكنتم منارات الدرب وعنوان الفضل

كعب

الأء

Abstract:

This study explores the use of plant extracts as natural corrosion inhibitors through a comprehensive analysis of extraction yields, phytochemical profiles, antioxidant activity, and anticorrosion performance. The results showed that **Alfalfa extract** had the highest extraction yield (92.66%) and strong antioxidant activity, while **Moringa** exhibited exceptional anticorrosion potential with nearly **99% inhibition at 500 ppm**, attributed to its terpenoids and fatty acids. **Juniper**, though displaying atypical behavior with peak inhibition at **0.5 ppm**, also showed promise, particularly for **protection**. Gravimetric tests confirmed the effectiveness of **Moringa and Alfalfa**, while **SEM observations** revealed well-preserved metal surfaces in samples treated with these extracts. These findings demonstrate that plant extracts, particularly **Moringa and Alfalfa**, can serve as an effective eco-friendly alternative to synthetic corrosion inhibitors, though **concentration and treatment optimizations** are needed to ensure long-term stability under industrial conditions.

Keywords: plant extracts, corrosion inhibitors, Alfalfa, Moringa, Jojoba, Juniper, eco-friendly corrosion inhibitors.

Résumé :

Ce travail explore l'utilisation des extraits de plantes comme inhibiteurs naturels de la corrosion à travers une analyse complète des rendements d'extraction, des profils phytochimiques, de l'activité antioxydante et des performances anticorrosion. Les résultats ont montré que l'extrait d'Alfalfa a présenté le rendement d'extraction le plus élevé (92,66 %) et une forte activité antioxydante, tandis que Moringa a exhibé un potentiel anticorrosion exceptionnel avec une inhibition de près de 99 % à 500 ppm grâce à ses composés terpénoïdes et acides gras. Juniper, bien que montrant un comportement atypique avec une inhibition maximale à 0,5 ppm, a également montré un potentiel, en particulier pour la protection. Les tests gravimétriques ont confirmé l'efficacité de Moringa et Alfalfa, tandis que les observations MEB ont révélé des surfaces métalliques bien préservées pour les échantillons traités avec ces extraits. Ces résultats démontrent que les extraits de plantes, notamment Moringa et Alfalfa, peuvent offrir une alternative écologique efficace aux inhibiteurs de corrosion synthétiques, bien que des optimisations de concentration et de traitement soient nécessaires pour garantir une stabilité à long terme dans des conditions industrielles.

Mots clés: extraits de plantes, inhibiteurs de corrosion, Alfalfa, Moringa, Jojoba, Juniper, bio-inhibiteurs de corrosion.

ملخص:

يستكشف هذا العمل استخدام المستخلصات النباتية كمثبطات طبيعية للتآكل من خلال تحليل شامل لعوائد الاستخلاص، والتركيبات الكيميائية النباتية، والنشاط المضاد للأكسدة، والأداء المضاد للتآكل. أظهرت النتائج أن مستخلص البرسيم (Alfalfa) حقق أعلى عائد استخلاص (92.66%) ونشاطاً مضاداً قوياً للأكسدة، بينما أظهر المورينجا (Moringa) إمكانات استثنائية مضادة للتآكل مع تثبيط يقارب 99% عند تركيز 500 جزء في المليون بفضل مركباته التربينية والأحماض الدهنية. أما العرعار (Juniper) ، فعلى الرغم من نتائجه غير الاعتيادية حيث بلغ التثبيط ذروته عند 0.5 جزء في المليون، إلا أنه أظهر أيضاً إمكانات واعدة، خاصة في الحماية ضد التآكل. أكدت الاختبارات الوزنية فعالية المورينجا والبرسيم، بينما كشفت صور المجهر الإلكتروني الماسح (SEM) عن أسطح معدنية محفوظة جيداً في العينات المعالجة بهذه المستخلصات. تثبتت هذه النتائج أن المستخلصات النباتية، خاصة المورينجا والبرسيم، يمكن أن تكون بديلاً فعالاً وصديقاً للبيئة للمثبطات الصناعية للتآكل، على الرغم من الحاجة إلى تحسين التركيزات وظروف المعالجة لضمان استقرار طويل الأمد في الظروف الصناعية.

الكلمات المفتاحية: المستخلصات النباتية، مثبطات التآكل، البرسيم، المورينجا، الجوجوبا، العرعار، مثبطات التآكل الصديقة

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GENERAL INTRODUCTION

GENERAL INTRODUCTION

The use of plant extracts as natural corrosion inhibitors has garnered increasing interest in recent years. Corrosion of metallic materials, particularly in acidic environments, poses a major challenge for numerous industrial sectors such as energy production, automotive, and shipbuilding. This phenomenon leads to significant economic losses and environmental risks, driving the search for effective and sustainable solutions. Among these solutions, corrosion inhibitors play a key role in slowing metal degradation. However, synthetic inhibitors, while effective, often exhibit high toxicity and prohibitive costs, prompting the exploration of eco-friendly alternatives, particularly plant extracts [1, 2].

In recent years, the use of plant extracts as natural corrosion inhibitors has gained growing interest due to their effectiveness, biodegradability, and low environmental impact. Plants are rich sources of bioactive compounds such as flavonoids, saponins, fatty acids, and terpenoids, which have demonstrated anti-corrosive properties by forming protective films on metal surfaces or acting as free radical scavengers [3, 4]. Among the most studied plant species are **Moringa oleifera**, **Jojoba**, **Alfalfa**, and **Juniperus phoenicea**, whose extracts have shown promising performance in corrosion and antioxidant tests [5, 6].

In this context, this study aims to evaluate and compare the efficacy of various plant extracts as natural corrosion inhibitors, focusing on their extraction, phytochemical profile, and antioxidant activity. A rigorous methodological approach has been adopted, combining advanced analytical techniques such as gas chromatography-mass spectrometry (GC-MS), infrared spectroscopy (IR), antioxidant activity tests (DPPH), gravimetric assays (mass loss), and scanning electron microscopy (SEM) to analyze metal surfaces. These analyses will assess the anti-corrosive potential of the extracts and elucidate their mechanisms of action.

This work is structured into three main chapters:

1. **Theoretical Background:** This chapter presents fundamental principles of corrosion mechanisms, different types of inhibitors (with a focus on bio-sourced inhibitors), and methods for studying corrosion.
2. **Materials and Methods:** This chapter details the experimental protocols, including the extraction of active compounds (Soxhlet method), phytochemical analyses (GC-MS, IR), antioxidant activity tests (DPPH), gravimetric assays (mass loss), and morphological analyses via SEM.

3. **Results and Discussion:** This chapter presents and interprets the findings, highlighting correlations between extract composition and anti-corrosive efficacy, as well as microscopic observations of metal surfaces.

In conclusion, this study contributes to the valorization of plant resources as eco-friendly alternatives to traditional corrosion inhibitors, paving the way for more sustainable solutions in metal protection.

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CHAPTER I

***THEORETICAL BACKGROUND ON
CORROSION INHIBITORS***

I.1. Definition and Key Concepts

Corrosion inhibitors are chemical substances that, when added in small concentrations to an aggressive environment, effectively slow down or prevent metal degradation [1]. These compounds play a vital role in corrosion prevention through three primary mechanisms [2]:

- First, they form a protective barrier on the metal surface either through molecular adsorption (physisorption or chemisorption) or by creating a passive oxide layer.
- Second, they alter the corrosive environment by adjusting pH, removing dissolved oxygen, or chelating aggressive ions.
- Third, they interfere electrochemically by polarizing anodic and/or cathodic sites, thereby increasing corrosion resistance.

I.2. Inhibitory Properties

The performance of a corrosion inhibitor is typically assessed by its inhibition efficiency (% IE), a parameter that quantifies the extent to which the inhibitor reduces the corrosion rate relative to an uninhibited system. A higher % IE indicates better protective action [2].

Several parameters influence this efficiency:

- First, the chemical resilience of the inhibitor under operating conditions is essential to ensure long-term protection [3, 4].
- Second, adequate solubility and uniform dispersion in the corrosive medium are crucial for maintaining consistent surface coverage [5].
- Third, the adsorptive interaction between inhibitor molecules and the metal substrate governs the formation of a stable protective layer [6].
- Moreover, the Eco toxicological profile must be considered, as modern regulations increasingly favor environmentally benign compounds [7].
- Lastly, the inhibitor's compatibility with other chemical agents present in the system, such as biocides or scale inhibitors, determines its practical applicability [8].

To evaluate inhibitor performance, several methodologies are employed. Gravimetric techniques, which involve measuring mass loss over time, provide a direct and simple assessment of corrosion rate. Electrochemical methods, including potentiodynamic polarization and electrochemical impedance spectroscopy (EIS), offer deeper insight into the inhibition mechanism and kinetics. Additionally, surface analysis tools such as scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR),

and X-ray photoelectron spectroscopy (XPS) help characterize the nature and morphology of the protective film formed on the metal surface.

In general, an inhibitor must:

- Reducing the rate of corrosion of a metal, without affecting its physico-chemical characteristics, in particular its mechanical strength (for example, risk of hydrogen embrittlement in an acid medium).
- Be stable in the presence of the other constituents of the medium, particularly with regard to oxidants.
- Be stable at operating temperatures.
- Be effective at low concentrations.
- Be consistent with non-toxicity standards.
- Be inexpensive [9].

I.3. Applications of Corrosion Inhibitors in Industry

Corrosion inhibitors are integral to numerous industrial processes where metal components are exposed to aggressive environments (Figure I.1) [10]. Their use not only extends the lifespan of equipment but also reduces maintenance costs and improves safety. Below are key sectors where inhibitors play a critical role, along with specific examples of their implementation.

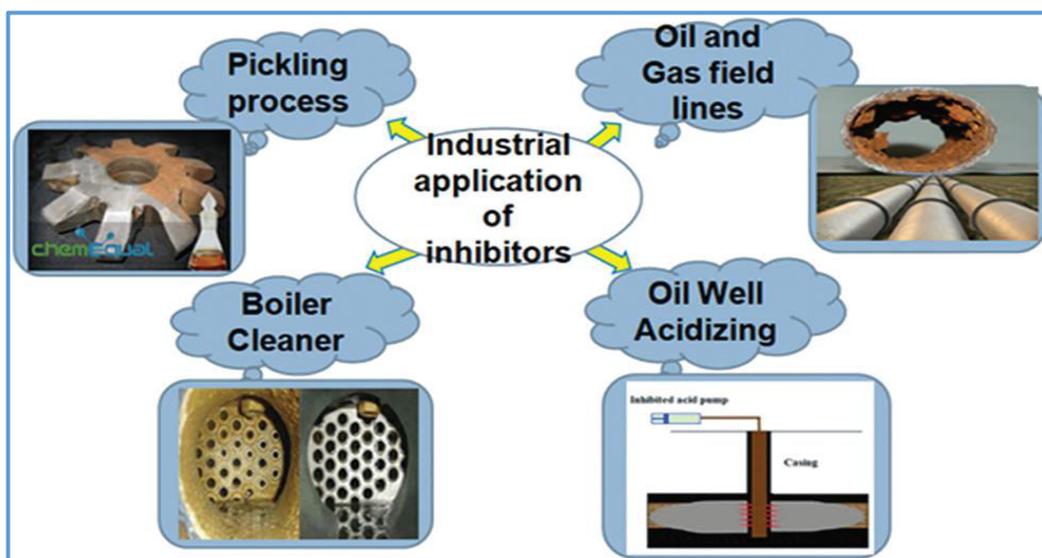


Figure I.1: Industrial Applications of Organic Corrosion Inhibitors [10].

I.3.1. Oil and Gas Industry

In this sector, corrosion is a major concern due to the presence of CO₂, H₂S, chlorides, and acidic fluids. Inhibitors are widely used in both upstream (exploration and production) and downstream (refining and distribution) operations [11].

- In acidizing treatments, where hydrochloric acid (HCl) is injected into wells to dissolve rock and improve flow, film-forming organic inhibitors (e.g., quaternary ammonium salts or acetylenic alcohols) are added to protect tubular steel from acid attack [11].
- During gas transmission, inhibitors such as imidazolines or fatty amine derivatives are injected into pipelines to prevent internal corrosion caused by water-condensed CO₂ (Figure I.2)[12, 13].
- In refinery units, inhibitors are used in desalting, fractionation columns, and overhead systems to counteract naphthenic acid or sulfur-induced corrosion.



Figure I.2: role of corrosion inhibitors in oil and gas pipelines [14].

I.3.2. Water Treatment Systems

In water systems, corrosion and scale formation are persistent challenges, particularly in industrial cooling circuits, steam boilers, and desalination plants [15 - 18].

- In cooling towers, where water recirculates and evaporates, inhibitors like polyphosphates, zinc salts, or benzotriazole (for copper alloys) are used to minimize general and localized corrosion.
- In boiler systems, where high-pressure steam is generated, volatile corrosion inhibitors (e.g., hydrazine, morpholine) are dosed to neutralize acidic components and protect condensate lines.
- In reverse osmosis desalination units, organic inhibitors prevent biofouling and corrosion of metallic housings and membranes, maintaining efficiency and preventing early degradation.

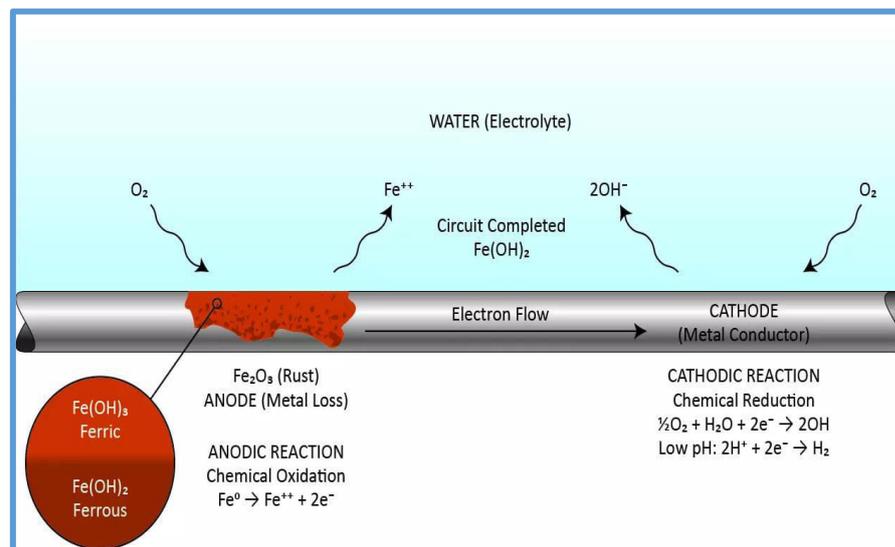


Figure I.3: Applications of Corrosion Inhibitors in Industry Water Treatment Systems [19].

I.3. 3. Metallurgy and Manufacturing Industry

In metal-related industries, inhibitors are essential during processing, surface treatment, storage, and transportation stages to protect against both chemical and atmospheric corrosion.

- In pickling operations, where acids (like HCl or H₂SO₄) are used to clean metal surfaces, acid corrosion inhibitors such as thiourea derivatives or alkynol-based compounds are added to protect the underlying metal without hindering the cleaning process [20].

- In metal cutting and machining, oil-based inhibitors prevent corrosion during and after the operation, particularly for high-precision components [21].
- In storage and shipment, volatile corrosion inhibitors (VCI) are used in the form of impregnated papers, foams, or powders that release protective vapors into enclosed spaces (e.g., packaging of spare parts or military hardware)[22].
- In coating formulations, inhibitors such as zinc phosphate or cerium salts are embedded into paints and primers to provide passive and active protection over long durations [23].

I.4. Classification of Inhibitors

Corrosion inhibitors can be classified based on various criteria, including their chemical nature, the type of electrochemical reaction they influence, and their mechanism of action. Understanding these categories helps in selecting the most appropriate inhibitor for a given system.

I.4.1. Classification by Nature

I.4.1.a. Organic Inhibitors

Organic inhibitors are compounds containing carbon, usually with heteroatoms like nitrogen, oxygen, sulfur, or phosphorus that facilitate adsorption onto the metal surface. These molecules often form a protective film that blocks the access of corrosive agents [24].

Common types include [25-27]:

- Amines: They work primarily by forming a barrier through electron donation to the metal surface.
- Heterocyclic compounds: These include imidazoles, pyridines, triazoles, and others that offer multiple adsorption sites, enhancing efficiency.

The performance of organic inhibitors is largely determined by the molecular structure particularly the presence of π -electrons and lone pairs that enhance surface interaction.

I.4.1.b. Inorganic Inhibitors

Inorganic inhibitors do not contain carbon as their main component and are often salts or oxoanions. They usually alter the redox potential of the metal surface or form insoluble precipitates that block active sites. Examples include:

- Chromates and dichromates: Very effective anodic inhibitors, although highly toxic and environmentally regulated [28].
- Phosphates, molybdates, silicates: These are considered safer and are widely used in water treatment and cooling systems [29].

I.4.2. Classification by Reaction Type

Inhibitors can also be classified according to the part of the corrosion reaction they affect [30, 31]:

- Anodic Inhibitors: These slow down the oxidation reaction (metal dissolution). They often form passive films on the metal surface but may risk localized corrosion if used improperly (e.g., chromates, phosphates).
- Cathodic Inhibitors: These target the reduction reaction, such as the reduction of oxygen or hydrogen ions. They can act by precipitating over the cathodic sites or by reducing their catalytic activity (e.g., zinc salts, certain polyphosphates).
- Mixed-type Inhibitors: These affect both anodic and cathodic processes simultaneously. Many organic inhibitors fall into this category due to their ability to adsorb on the entire metal surface.

I.4.3. Classification by Mechanism of Action

This classification is based on how the inhibitor interacts with the metal surface [32-34]:

- **Adsorption inhibitors:** Most organic inhibitors fall under this category. They adhere to the metal surface via physical or chemical adsorption, creating a protective film that hinders corrosive species from accessing the substrate.
- **Film-forming inhibitors:** These substances promote the formation of a stable, often insoluble, layer on the metal surface. This film can be either a precipitate or a polymeric layer that isolates the metal from the environment.

- **Passivating inhibitors:** These induce the formation of a passive oxide layer, particularly effective on metals like iron and aluminum. For instance, nitrates or chromates can enhance passivity in aggressive conditions.

I.5. Corrosion Inhibition in Acidic Media

Acidic environments, particularly those involving strong acids like hydrochloric acid (HCl) or sulfuric acid (H₂SO₄), are highly aggressive toward metals. Such media are commonly encountered in industrial processes such as acid pickling, cleaning, descaling, and oil-well acidizing. In these contexts, metals such as steel, copper, and aluminum are vulnerable to rapid corrosion unless adequately protected. One of the most effective strategies to mitigate this issue is the use of organic corrosion inhibitors [35].

Organic inhibitors function primarily through adsorption onto the metal surface, forming a protective barrier that isolates the metal from corrosive species (e.g., H⁺ ions or Cl⁻) [36]. The adsorption can be [36, 37]:

- Physical (physisorption): Involving electrostatic attraction between the inhibitor and the charged metal surface.
- Chemical (chemisorption): Based on the formation of coordinate covalent bonds between the metal atoms and electron-donating groups of the inhibitor (e.g., N, O, S atoms, π -electrons).

This film decreases the active surface area available for corrosion reactions and/or retards the anodic and/or cathodic reaction kinetics.

I.6. Plant Extracts as Corrosion Inhibitors

In recent years, plant-based extracts have emerged as a promising class of eco-friendly corrosion inhibitors, particularly in response to growing environmental regulations and the need to replace toxic synthetic chemicals. These green inhibitors are derived from naturally occurring substances found in leaves, seeds, roots, bark, and peels, and are rich in organic compounds with corrosion-inhibiting properties [38, 39].

I.6.1. Why Use Plant Extracts

The appeal of plant extracts as corrosion inhibitors lies in several advantages [40]:

- Biodegradability: They degrade naturally, leaving no long-term toxic residues.
- Low toxicity: Safer for both the environment and human health compared to conventional inhibitors like chromates or nitrites.
- Renewable and abundant: Sourced from agricultural or food waste, making them cost-effective and sustainable.
- Chemical richness: Contain a variety of active compounds (alkaloids, flavonoids, tannins, saponins, polyphenols) known for their ability to adsorb onto metal surfaces and block corrosion reactions.

I.6.2. Mechanism of Inhibition

Plant extracts act predominantly via adsorption of their active constituents onto the metal surface, forming a protective layer that minimizes direct contact with the corrosive medium. The inhibition is typically of mixed-type, affecting both anodic metal dissolution and cathodic reduction processes.

The efficiency of inhibition depends on [41]:

- The presence of heteroatoms (O, N, S) in the bioactive molecules,
- The molecular structure (planarity, conjugation),
- The synergistic effect of multiple phytochemicals acting together.

I.6.3. Examples of Effective Plant Extracts

Numerous plant extracts have demonstrated high inhibition efficiencies, particularly in acidic environments (HCl, H₂SO₄) [42]:

- **Azadirachta indica (Neem):** Rich in azadirachtin and limonoids, neem leaf and seed extracts have shown up to 90% efficiency in protecting mild steel in HCl solutions [43].

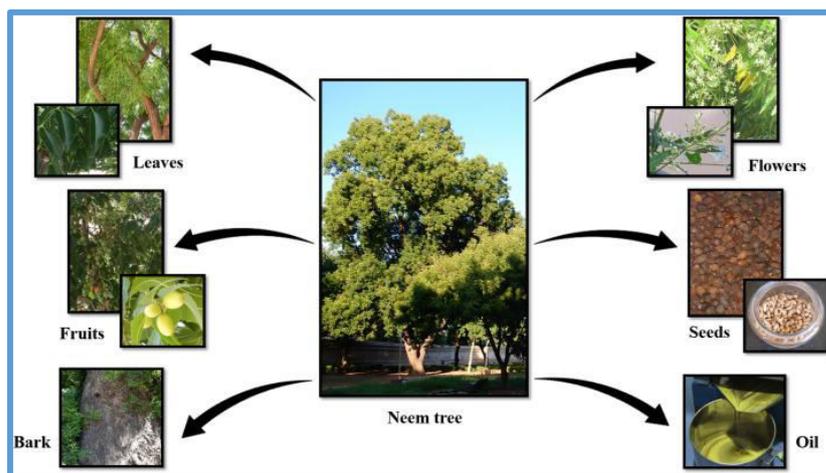


Figure I.4: *Azadirachta indica* (neem) [43].

- ***Ziziphus mauritiana***: Leaves and fruit extracts have been studied as inhibitors in sulfuric acid, owing to their flavonoid and saponin content [44].



Figure I.5: a) *Ziziphus mauritiana* Leaves b) fruit [44, 45].

- ***Lawsonia inermis* (Henna)**: Contains lawsone (2-hydroxy-1, 4-naphthoquinone), which adsorbs effectively on metal surfaces [46].



Figure I.6: Lawsonia inermis (Henna) [47].

- **Garlic and onion extracts:** Rich in sulfur compounds like allicin, these extracts form strong chemisorbed layers on steel and copper [48].
- **Pomegranate peel extract:** High in tannins and polyphenols, it has been used successfully for mild steel corrosion inhibition in acidic environments [49].
- **Ginger, turmeric, and green tea extracts:** These contain phenolic compounds and antioxidants that contribute to efficient film formation [50].

I.6.4. Factors Influencing Efficiency

Several factors govern the performance of plant extracts [51]:

- **Extraction method:** The type of solvent (ethanol, methanol, water) and temperature used during extraction affects the concentration of active compounds.
- **Dosage:** Like synthetic inhibitors, plant extract performance improves with concentration, up to a limit.
- **Temperature:** Higher temperatures may enhance adsorption but can also cause desorption of weakly bound molecules.
- **Metal type and surface roughness:** The metal's affinity for phytochemicals influences the protective film's uniformity and stability.

I.6.5. Analytical and Electrochemical Evaluation

The corrosion inhibition properties of plant extracts are typically evaluated through [52]:

- Weight loss measurements over time.
- Electrochemical tests like Tafel polarization and impedance spectroscopy (EIS).
- Surface analysis (SEM, FTIR, AFM) to visualize and identify protective films.
- Phytochemical screening and GC-MS to identify active constituents responsible for inhibition.

I.7. Methods for the Study of Corrosion Inhibitors

Experimental and theoretical approaches are commonly used to study corrosion inhibitors. Laboratory tests provide practical insights into inhibitor efficiency under different conditions, while theoretical methods help predict inhibitor behaviour and interaction mechanisms at the atomic level. Combining both approaches enhances the understanding and development of effective corrosion inhibitors.

I.7.1. Experimental Methods

Researchers use various lab tests to evaluate how well corrosion inhibitors work. These experiments help understand their performance and protection mechanisms.

I.7.1.1. Gravimetric Analysis

Gravimetric analysis is one of the most traditional, straightforward, and widely used experimental techniques for studying corrosion processes and evaluating the efficiency of corrosion inhibitors. It involves measuring the **mass loss** of a metallic specimen after immersion in a corrosive medium, with or without the presence of an inhibitor.

In this method, pre-weighed metal coupons of known surface area are immersed in a corrosive solution for a specified time under controlled conditions. After the exposure period, the samples are removed, cleaned to eliminate corrosion products (typically following standards such as **ASTM G1-03 or ISO 8407** [53], dried, and reweighed. The mass loss corresponds to the extent of corrosion.

The **corrosion rate** (CR) is calculated using the following equation [54]:

$$CR = \frac{W}{A \times t \times \rho} \quad (\text{Eq. I.1})$$

Where:

- W : Weight loss (g)
- A : Surface area of the coupon (cm²)
- T : Immersion time (h)
- ρ : Density of the metal (g/cm³)

The **inhibition efficiency** (%IE) is determined by comparing the corrosion rate in the absence and presence of the inhibitor:

$$IE\% = \left(\frac{CR_0 - CR_{inh}}{CR_0} \right) \times 100 \quad (\text{Eq. I.2})$$

Where:

- CR₀= Corrosion rate without inhibitor
- CR_{inh}= Corrosion rate with inhibitor

In gravimetric analysis, two key parameters are typically measured:

- ✓ The **corrosion rate**, usually expressed in units such as mg·cm⁻²·h⁻¹ or mm/year, and
- ✓ The **inhibition efficiency** (%), which quantifies the effectiveness of a corrosion inhibitor by comparing corrosion rates with and without the inhibitor.

This method offers several notable advantages. It is simple, cost-effective, and provides a direct, quantitative assessment of metal degradation. Moreover, it is applicable to a broad range of metals and corrosive environments, making it highly versatile.

However, gravimetric analysis also presents certain limitations. It does not yield detailed information about the electrochemical mechanisms involved in corrosion processes, and its accuracy depends heavily on the thoroughness of the cleaning procedure, which must remove all corrosion products without altering the metal surface. Furthermore, the method is relatively time-consuming compared to electrochemical techniques [54].

To ensure reliable and reproducible results, several best practices should be followed. Metal samples must be polished and standardized, and rigorous cleaning protocols—such as those described in **ASTM G1-03**—must be applied both before and after immersion. Performing tests in triplicate is also strongly recommended to account for variability and confirm data consistency. Gravimetric analysis is widely used in applications such as evaluating environmentally friendly (green) corrosion inhibitors derived from plant extracts, studying corrosion behavior in acidic, saline, or industrial environments, and conducting comparative assessments of different inhibitor formulations under similar conditions [55].

I.7.1.2. Electrochemical Techniques

Electrochemical techniques are among the most widely used methods in corrosion science due to their high sensitivity, rapid measurement, and ability to provide mechanistic insight into corrosion and inhibition processes. These techniques are based on measuring the electrochemical response of a metal surface immersed in an electrolyte under controlled potential or current conditions. They allow for real-time monitoring of corrosion kinetics and evaluation of inhibitor efficiency with great precision.

a) Potentiodynamic Polarization (Tafel Curves)

Potentiodynamic polarization is employed to determine the corrosion kinetics and to identify the type of inhibition mechanism—whether anodic, cathodic, or mixed. In this method, the potential of the working electrode (typically a metal specimen) is scanned linearly in time, starting from a potential well below the open circuit potential (OCP) and extending to above it. The resulting current-potential plot, known as the **Tafel plot**, exhibits linear regions where the anodic and cathodic Tafel slopes (β_a , β_c) can be extracted. From this, the corrosion potential (E_{corr}) and corrosion current density (i_{corr}) can be determined using extrapolation techniques [56].

The corrosion current density is directly related to the corrosion rate, and the **inhibition efficiency** can be calculated as:

$$IE\% = \left(\frac{i_{corr}^0 - i_{corr}^{inh}}{i_{corr}^0} \right) \times 100 \quad (\text{Eq. I.3})$$

Where, I_{corr} and $I_{\text{corr}}^{\text{inh}}$ are the corrosion current densities without and with the inhibitor, respectively.

This technique is particularly useful for understanding how an inhibitor influences the anodic metal dissolution and the cathodic hydrogen evolution reactions **Figure I.**

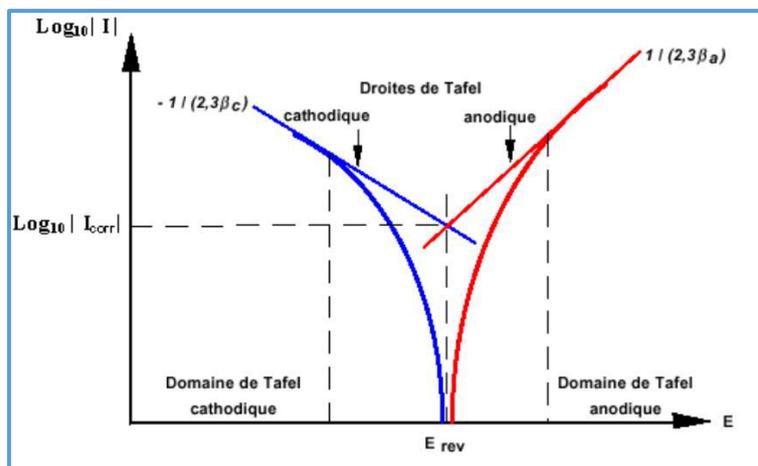


Figure I.7: schematic representation of a typical Tafel curve can be provided on request [56].

b) Electrochemical Impedance Spectroscopy (EIS)

EIS is a powerful non-destructive technique used to evaluate the electrochemical behavior of metal surfaces over a wide frequency range. It is particularly effective for studying the **stability and integrity of protective films** formed by corrosion inhibitors on the metal surface. In this method, a small AC potential (typically 5–10 mV) is applied at the open circuit potential, and the resulting current response is measured as a function of frequency [59].

The data are represented in the form of **Nyquist** or **Bode plots**, from which key parameters such as the charge transfer resistance (R_{ct}) and double-layer capacitance (C_{dl}) are extracted by fitting the data to an appropriate equivalent circuit. A higher R_{ct} value in the presence of an inhibitor indicates better corrosion protection.

EIS is highly sensitive to interfacial processes and allows continuous monitoring of film formation and degradation over time.

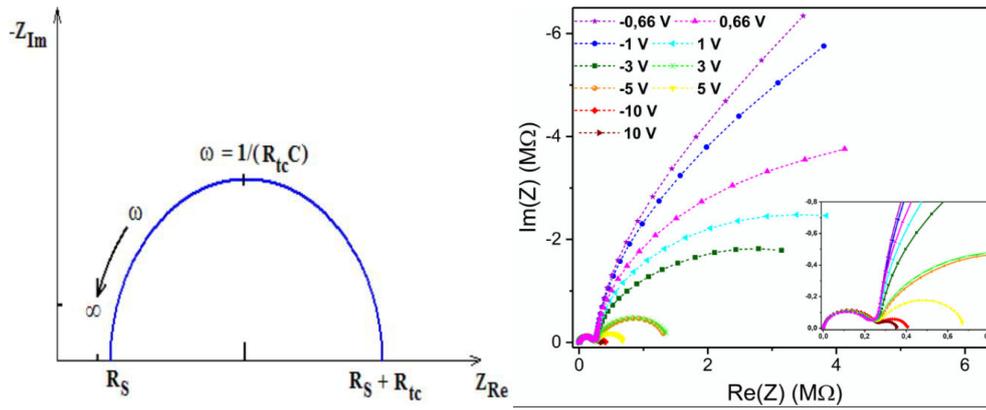


Figure I.8: A Nyquist plot example and equivalent circuit model can be generated on request.

c) Linear Polarization Resistance (LPR)

Linear polarization resistance is a rapid and widely used method to estimate the corrosion rate of metals. It involves applying a small perturbation (typically $\pm 10\text{--}20$ mV) around the open circuit potential and measuring the resulting current. The slope of the resulting linear region of the polarization curve yields the **polarization resistance** (R_p), which is inversely proportional to the corrosion rate via the Stern–Geary equation[60,61]:

$$i_{corr} = \frac{B}{R_p} \quad (\text{Eq. I.4})$$

Where B is a constant that depends on the Tafel slopes:

$$B = \frac{\beta_a \cdot \beta_c}{2.303(\beta_a \cdot \beta_c)} \quad (\text{Eq. I.5})$$

This method is particularly suitable for **field applications and long-term corrosion monitoring**, especially when fast results are needed. Although it does not provide mechanistic information, it is highly effective for quantifying the corrosion rate in real time.

I.7.1.3. Surface Characterization Techniques

While electrochemical and gravimetric methods provide essential quantitative information about corrosion and inhibition efficiency, surface characterization techniques offer invaluable insights into the morphological, structural, and chemical changes occurring on metal surfaces before and after corrosion. These analyses help to understand the protective film formation,

adsorption behavior of inhibitors, and corrosion product composition. The following techniques are commonly used (SEM, XPS or FTIR):

➤ **Scanning Electron Microscopy (SEM)**

Scanning Electron Microscopy (SEM) is widely employed to analyze the surface morphology of corroded or inhibited metal specimens. It provides high-resolution micrographs that reveal important features such as pitting, cracking, surface roughness, and film uniformity. In corrosion studies, SEM is used to compare the surface degradation of untreated metals with those protected by corrosion inhibitors, thus visually confirming their protective action [39].

When coupled with Energy Dispersive X-ray Spectroscopy (EDS or EDX), SEM can also provide elemental analysis of surface deposits or corrosion products.

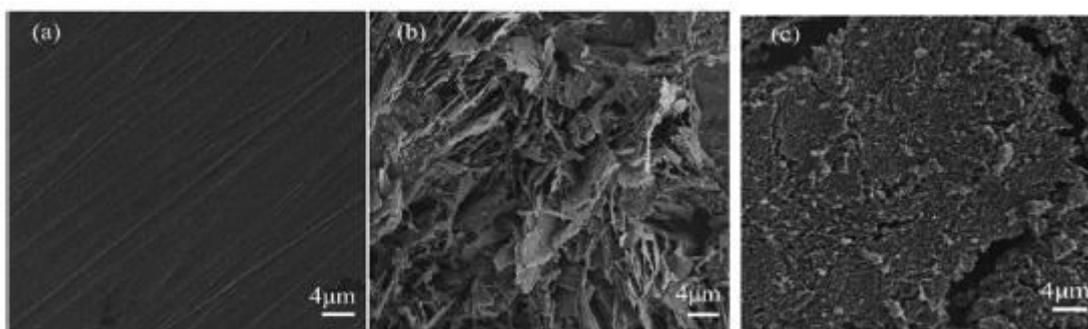


Figure I.9: SEM micrographs of Q235 steel (a) before and (b) after 2 h corrosion in 0.5 M HCl. after 2 h corrosion in 0.5 M HCl with inhibitor [39].

➤ **X-ray Photoelectron Spectroscopy (XPS)**

XPS is a powerful surface-sensitive technique used to determine the **elemental composition** and **chemical states** of atoms on the outermost 5–10 nm of the surface. In corrosion inhibitor research, XPS provides direct evidence of inhibitor adsorption, metal–inhibitor bond formation, and oxide or corrosion product composition.

It is particularly useful for confirming whether specific atoms from the inhibitor molecule (e.g., N, O, S) are coordinated to the metal surface [62].

➤ **Fourier Transform Infrared Spectroscopy (FTIR)**

FTIR spectroscopy is used to identify functional groups and study the interaction between inhibitor molecules and the metal surface. The comparison of FTIR spectra before and after interaction with the metal can indicate adsorption behavior, such as shifts or disappearance of characteristic peaks (e.g., C=O, N–H, or OH), suggesting chemical bonding.

This method is frequently applied to verify the presence of adsorbed organic films and to support proposed inhibition mechanisms based on molecular structure [63].

I.7.2. Theoretical Methods

In addition to experimental investigations, theoretical and computational methods play a crucial role in understanding the mechanisms of corrosion inhibition at the molecular level. These approaches provide insights into the electronic properties, adsorption behavior, and structure–activity relationships of inhibitor molecules, offering predictive power and helping to design more efficient compounds. The following are the most commonly used theoretical techniques in corrosion inhibitor studies (DFT and MD):

I.7.2. 1. Density Functional Theory (DFT)

Density Functional Theory (DFT) is one of the most widely used quantum chemical approaches for investigating the electronic structure and reactivity of corrosion inhibitor molecules. DFT allows the calculation of molecular orbitals (HOMO–LUMO), charge distributions, dipole moments, Fukui functions, and global reactivity descriptors such as electronegativity (χ), hardness (η), and electrophilicity index (ω).

These parameters are used to evaluate the tendency of a molecule to donate or accept electrons, which is critical for predicting its interaction with metal surfaces. A higher HOMO energy, for instance, indicates a greater ability to donate electrons to vacant d-orbitals of the metal, suggesting stronger adsorption [64, 65].

DFT calculations are usually carried out using software such as Gaussian, DMol³, or ORCA, employing functionals like B3LYP with appropriate basis sets (e.g., 6-31G(d)) [64].

I.7.2.2. Molecular Dynamics (MD) Simulations

Molecular Dynamics (MD) simulations offer an atomistic view of the dynamic interaction between inhibitor molecules and the metal/solution interface. Unlike static DFT calculations, MD simulations consider the time-dependent behavior of atoms and molecules under realistic conditions, including solvent effects and temperature.

These simulations provide information about adsorption configurations, binding energy, diffusion behavior, and film stability on surfaces like Fe (110), Fe (111), or Cu (111). They help visualize how inhibitor molecules align, aggregate, and interact with the metal substrate and surrounding water molecules or ions.

MD simulations are typically conducted using force fields such as COMPASS, OPLS, or CHARMM, and software packages like Materials Studio, LAMMPS, or GROMACS [66, 67].

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CHAPTER II

METHODS AND CHARACTERIZATION TECHNIQUES

II.1. Introduction

This chapter outlines the experimental methodology employed in this study, including the selection of plant materials, extraction of bioactive compounds, formulation of inhibitors, and characterization techniques. The chapter is divided into two main sections:

- ✓ **Part 1: Methods** – Describes the procedures for plant selection, extraction, formulation, and antioxidant activity assessment.
- ✓ **Part 2: Characterization Techniques** – Covers analytical methods used to evaluate the extracts and their inhibitory effects.

Part 1: Methods

II.2. Selection of Plant Materials

In this study, four plant species were selected for their anticorrosive potential, local availability, and richness in bioactive secondary metabolites (Table II.1). The selection criteria were based on documented botanical, chemical, and toxicological properties from the scientific literature:

a) Alfalfa (*Medicago sativa* L., Fabaceae)[1]



- ✓ **Origin:** Widely cultivated forage plant in Mediterranean and temperate regions.
- ✓ **Used Part:** Seeds, rich in triterpenoid saponins (e.g., medicagosides) and flavonoids (e.g., vicenin). These compounds form stable complexes with metal ions, inhibiting electrochemical corrosion reactions.
- ✓ **Toxicity:** No acute toxicity reported; saponins are biodegradable and non-toxic at tested concentrations (<1000 ppm) .

b) **Jojoba Leaves** (*Simmondsia chinensis* (Link) C.K. Schneid., Simmondsiaceae)[2]

- ✓ **Origin:** Xerophytic shrub native to North American deserts, adapted to arid climates.
- ✓ **Used Part:** Mature leaves, containing long-chain fatty alcohols (C20–C22, e.g., simmondsin) and wax esters. These lipids adsorb onto metal surfaces via polar groups, forming a hydrophobic barrier .
- ✓ **Toxicity:** Classified as non-toxic (LD50 > 2000 mg/kg); widely used in cosmetics and food industries .

c) **Moringa Leaves** (*Moringa oleifera* Lam., Moringaceae)[3]

- ✓ **Origin:** Tropical tree native to India, cultivated in Africa and Asia for nutritional uses.
- ✓ **Used Part:** Dried leaves, a major source of polyphenols (gallic acid, quercetin) and alkaloids (e.g., moringine). These molecules chelate Fe²⁺ ions and passivate metal surfaces .
- ✓ **Toxicity:** No adverse effects reported at industrial doses; approved as a dietary supplement

d) **Phoenician Juniper** (*Juniperus phoenicea* L., Cupressaceae)[4]

- ✓ **Origin:** Drought-resistant Mediterranean conifer, widespread in North Africa.
- ✓ **Used Part:** Berries, containing essential oil rich in monoterpenes (α -pinene, limonene) and sesquiterpenes. These volatile compounds inhibit corrosion via an adsorbed film mechanism.
- ✓ **Toxicity:** Essential oil is irritating at high doses (>5% v/v) but safe at tested concentrations (0.1–1%).

Table II.1: Plants and Anticorrosion Mechanisms

Plant	Family	Origin	Used Part	Active Compounds	Anticorrosive Mechanism	Toxicity
Alfalfa	Fabaceae	Temperate regions	Seeds	Saponins, flavonoids	Metalion chelation	Non-toxic (<1000 ppm)
Jojoba	Simmondsiaceae	North American deserts	Leaves	Fatty alcohols (C20–C22)	Hydrophobic film formation	LD50 > 2000 mg/kg
Moringa	Moringaceae	India, Africa, Asia	Leaves	Polyphenols, alkaloids	Surface passivation	Food-grade safe
Juniper	Cupressaceae	Mediterranean Basin	Berries	α -Pinene, limonene	Volatile adsorption	Irritant (>5% v/v)

II.3. Extraction of Bioactive Compounds

Extraction is the process of separating bioactive compounds from plant materials using a solvent or physical method. The goal is to isolate desired molecules (e.g., polyphenols, terpenes, alkaloids) while preserving their chemical structure and biological activity[5].

The selection of extraction method depends on[6]:

- ✦ Compound properties (**polarity, volatility, thermal stability**).
- ✦ Plant matrix (**leaves, seeds, bark**).
- ✦ Solvent affinity (**water, ethanol, hexane**).
- ✦ Environmental & safety considerations (**green chemistry principles**).

In this study, two methods were chosen based on these criteria: Soxhlet extraction and Hydrodistillation (Clevenger).

II.3.1. Soxhlet Extraction

The Soxhlet extraction method was selected for its high efficiency in extracting thermally stable, non-volatile compounds such as saponins from alfalfa seeds, fatty alcohols from jojoba leaves,

and polyphenols from moringa leaves[7] (**Figure.II.1**). This method is particularly suitable for solid plant matrices due to its continuous extraction mechanism, which ensures thorough compound recovery. The use of 70% ethanol as a solvent provides an optimal balance between polar and non-polar compound extraction, making it ideal for broad-spectrum bioactive recovery. Additionally, the controlled temperature of 70°C prevents degradation of heat-resistant compounds while maximizing extraction yield.

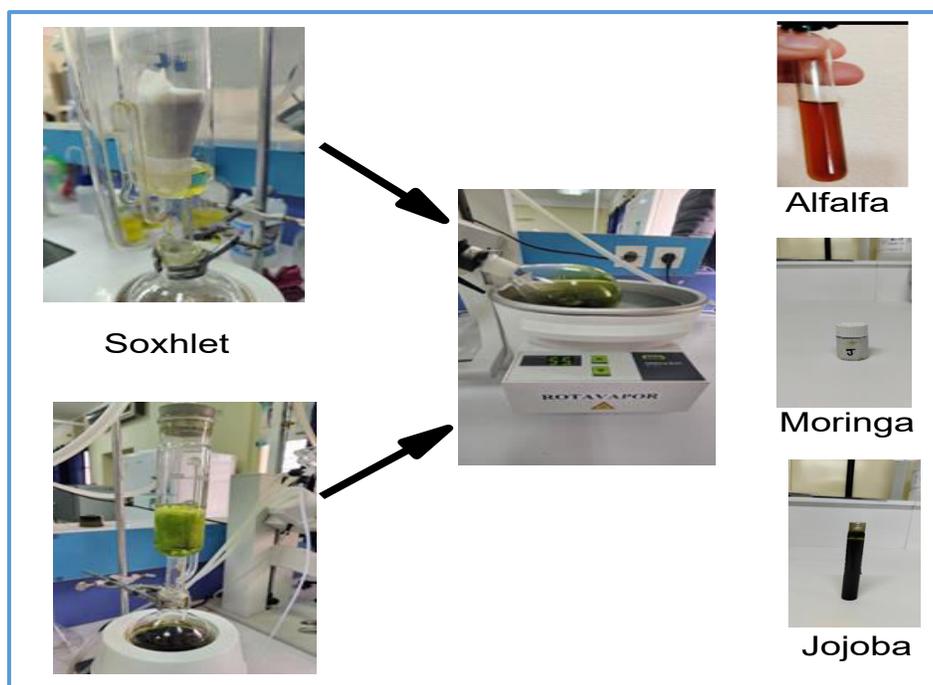


Figure II.1: Extraction from alfalfa seeds and jojoba and moringa leaves by soxhlet.

II.3.2. Hydrodistillation (Clevenger)

For volatile and heat-sensitive compounds, such as the essential oils (e.g., α -pinene and limonene) present in juniper berries, hydrodistillation using a Clevenger apparatus was employed[8](**Figure II.2**). This method avoids the thermal degradation risks associated with Soxhlet extraction by utilizing water vapor to gently carry volatile compounds. Being a water-based process, it eliminates the need for organic solvents, aligning with green chemistry principles. The relatively low processing temperature (100°C, from boiling water) and shorter extraction time (4 hours) help preserve the integrity of delicate terpenes, ensuring high-quality essential oil recovery.

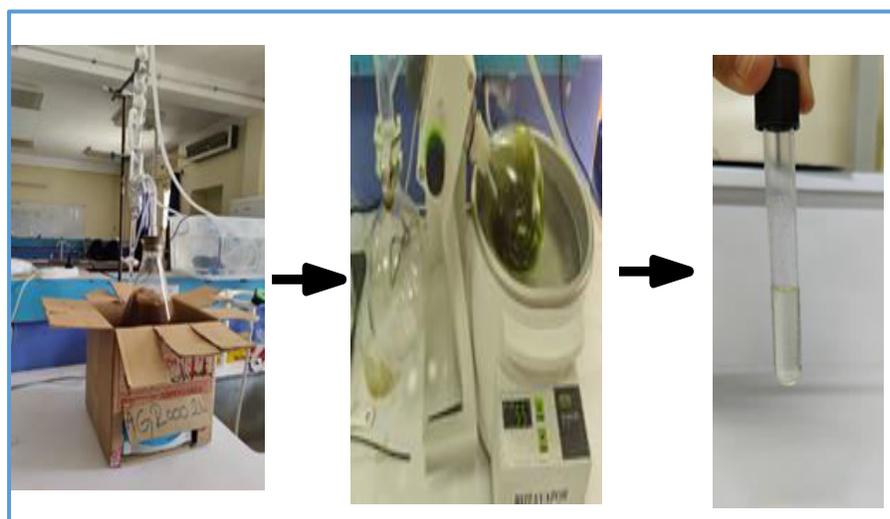


Figure II.2: Extraction from juniper berries by hydrodistillation (Clevenger).

II.3.3. Justification of Protocol Parameters

The extraction parameters were carefully optimized to maximize efficiency and compound stability.

- For Soxhlet extraction, a plant-to-solvent ratio of 1:10 (w/v) was maintained to prevent saturation, while a temperature of 70°C and an 8-hour duration ensured complete extraction without degradation.
- In hydrodistillation, the same 1:10 (w/v) ratio was used, with boiling water (100°C) facilitating efficient essential oil release within 4 hours.

These conditions were selected based on preliminary trials and literature evidence, ensuring reproducibility and high yields.

The extraction yield was calculated using the following formula to quantify the efficiency of the extraction process:

$$\text{Extraction Yield}(\%) = \left(\frac{\text{Dry Extract Mass (g)}}{\text{Dry Plant Material Mass (g)}} \right) \times 100 \quad (\text{Eq. II.1})$$

Where:

Dry Extract Mass: Weight of the obtained extract after solvent evaporation

Dry Plant Material Mass: Initial weight of the dehydrated plant sample.

II.4. Formulation of Corrosion Inhibitors

The crude plant extracts were used directly as corrosion inhibitors without synergists or additives to evaluate their intrinsic effectiveness. The formulation process was conducted as follows in section (II.7.1.b.).

II.5. Evaluation of Antioxidant Activity (DPPH Method)

The antioxidant activity of the extracts was evaluated through their free radical scavenging capacity using the DPPH (2,2-diphenyl-1-picrylhydrazyl) method[9]. This indirect measurement correlates the reducing power of phytochemicals (polyphenols, flavonoids) with their corrosion inhibition potential, via their ability to stabilize free radicals involved in metal oxidation-reduction reactions.

Table II.2: Optimized Protocol.

Step	Parameters	Controls/Justifications
1. Solution Prep		
- DPPH	0.04 mg/mL in ethanol (2mg/50mL)	Amber vial, dark storage
- Extract stocks	<ul style="list-style-type: none"> • Moringa: 1.12 mg/mL • Jojoba: 0.8 mg/mL • Alfalfa: 0.8 mg/mL • Juniper: 0.86 mg/mL (in methanol) 	Methanol chosen for DPPH compatibility
2. Incubation	<ul style="list-style-type: none"> • 1 mL DPPH + 1 mL extract • 30 min at 25°C in darkness • n=3 replicates 	Negative control: DPPH + methanol
3. Measurement	UV-Vis spectrophotometry at 517 nm	Blank correction (methanol alone)
4. Calculations	$\% \text{ Inhibition} = \left(\frac{\text{Absorbance control} - \text{Absorbance Sample}}{\text{Absorbance control}} \right) \times 100$ <p style="text-align: center;">(Eq. II.2)</p>	Dose-response curve with ≥ 2 dilutions

Part 2: Characterization Techniques

II.6. Characterization of Extracts

II.6.1. GC-MS Analysis

The chemical composition of plant extracts was characterized using gas chromatography-mass spectrometry (GC-MS), which separates volatile compounds by gas chromatography and identifies them through mass spectral fragmentation patterns[10].

Filtered extracts (0.22 μm) were injected in splitless mode onto a DB-5MS capillary column (30 m \times 0.25 mm) with helium carrier gas (1.2 mL/min).

The oven temperature was programmed from 50°C to 300°C at 10°C/min.

Electron ionization (70 eV) generated characteristic mass spectra (40-600 m/z range), with compounds identified by matching against the NIST 2020 library (similarity >85%) and quantified via peak area normalization. (System without calibration standards).

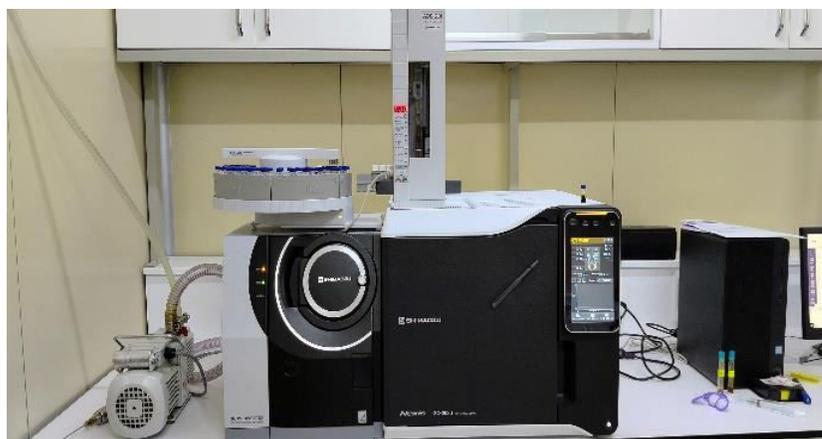


Figure II.3: Gas Chromatograph coupled with Mass Spectrometer (GC-MS).

II.6.2. Fourier-Transform Infrared Spectroscopy (FTIR) Analysis

Fourier-transform infrared spectroscopy (FTIR) was employed to characterize the functional groups present in our plant extracts. This analytical technique is based on the absorption of infrared radiation by chemical bonds, which vibrate at characteristic frequencies[11]. Unlike traditional

methods, FTIR systems utilize a Michelson interferometer to modulate the light beam, enabling simultaneous acquisition of all frequencies and significantly improving analysis sensitivity and speed.

For our investigations, we used an FTIR spectrometer equipped with a diamond crystal Attenuated Total Reflectance (ATR) accessory. This configuration offers the distinct advantage of analyzing liquid samples directly without extensive preparation. Each extract was carefully deposited on the crystal surface and gently dried under nitrogen flow to remove excess solvent while preserving compounds of interest.

Spectra were acquired over the range of $4000\text{--}400\text{ cm}^{-1}$ with a resolution of 4 cm^{-1} , averaging 32 scans per sample to ensure optimal signal-to-noise ratio. The ATR technique provided enhanced sensitivity for our liquid samples while eliminating the need for KBr pellet preparation.



Figure II.4: Agilent Cary 630 FTIR spectrometer.

II.7. Evaluation of Inhibitory Activity

II.7.1. Gravimetric Tests (Mass Loss Method)

II.7.1.a. Material Preparation

- ✓ *Steel Specimens*: XC48 carbon steel coupons (composition detailed in Table II.1) were cut into identical dimensions (typical size: $2.5 \times 2.0 \times 0.2\text{ cm}$).
- ✓ All sides except one test face were coated with chemically resistant epoxy paint.
- ✓ Exposed surfaces were progressively polished using SiC abrasive papers (120 to 1200 grit) to achieve a mirror finish, followed by ultrasonic cleaning in acetone for 10 min (**Figure II.5**).

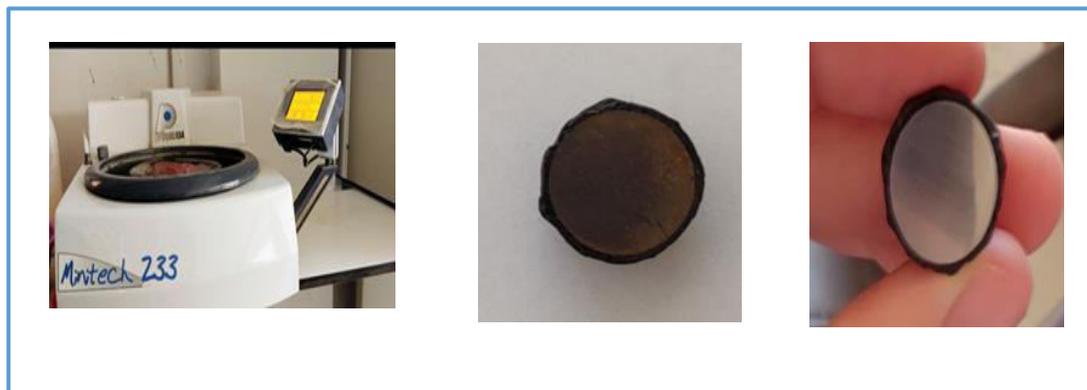


Figure II.5: Preparation of steel specimens by the polisher.

Table II.3: Chemical composition (wt%) of XC48 steel

Element	C	Si	Mn	P	S	Mo	Ni	Cr
%	0.45	0.27	0.69	0.008	0.008	0.002	0.02	0.13

II.7.1.b. Corrosion Testing Protocol

- ✓ Test Medium: 50 mL of 1 M HCl (prepared from 37% analytical-grade HCl) at 25°C.
- ✓ Inhibitor Concentrations: 0.5, 1, 10, 25, 50, 100, 200, 300, 400, and 500 ppm. These ranges were selected based on reported effective concentrations for similar plant-derived inhibitors in literature [12, 13].
- ✓ Experimental Setup:
 - Triplicate specimens were immersed for 24 h in: Blank solution (no inhibitor), Inhibitor-containing solutions
 - Containers: 100-mL glass beakers with PTFE lids to minimize evaporation.



Figure II.6: Experimental setup for the mass loss method.

II.7.1.c. Post-Exposure Analysis

- ✓ Cleaning: Removed corrosion products by scrubbing with nylon brush under running water, followed by ethanol rinse and drying in desiccator.
- ✓ Weighing: Measured mass loss (Δm) to ± 0.1 mg precision using analytical balance.

II.7.1.d. Calculations of Corrosion Rate (V) and Inhibition Efficiency

The gravimetric corrosion rate (V) was determined using the mass loss method with the following equation[14]:

$$V = \frac{\Delta m}{S \cdot t} \quad (\text{Eq. II.3})$$

Where:

- Δm = Initial mass (M_1) - Final mass (M_2) (mg)
- S = Exposed surface area (cm^2)
- t = Immersion time (24 h)

The corrosion inhibition efficiency (E%) was calculated using the following formula[14]:

$$E(\%) = \left(1 - \frac{V_{inh}}{V_{blank}}\right) \times 100 \quad (\text{Eq. II.4})$$

Where :

- V_{inh} : Corrosion rate in inhibitor-containing solution ($\text{mg} \cdot \text{cm}^{-2}$)

- V_{blank} : Corrosion rate in blank (inhibitor-free) solution ($\text{mg} \cdot \text{cm}^{-2}$)

II.7.2. SEM/EDX Analysis

The surface morphology of XC48 steel specimens was examined using scanning electron microscopy (SEM) to evaluate corrosion inhibition mechanisms. After gravimetric testing, samples were carefully cleaned with deionized water and ethanol, then dried. High-resolution micrographs ($5,000\times$ to $20,000\times$ magnification) were acquired at 15 kV accelerating voltage (Hitachi SU3500 SEM), focusing on:

- ✓ Pitting density **in uninhibited samples,**
- ✓ Surface coverage uniformity **in inhibitor-treated specimens,**
- ✓ Crack formation **at grain boundaries.**

Elemental composition analysis was performed concurrently with SEM using **EDX**. Three 1×1 mm areas were scanned per sample (live time = 60 s) to detect:

- ✓ Key elements: **Fe, O, C (steel substrate and corrosion products),**
- ✓ Inhibitor markers: **P, S, or N (from plant extract adsorption),**
- ✓ Contaminants: **Cl residues from HCl medium.**

Semi-quantitative results (atomic %) were compared between inhibited and blank surfaces to verify protective film formation.



Figure II.7: Scanning Electron Microscopy (SEM).

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GENERAL CONCLUSION

GENERAL CONCLUSION

The aim of this study was to explore and characterize the properties of plant extracts as natural corrosion inhibitors in acidic environments, specifically in a 1M HCl medium. Through a comprehensive experimental approach, including extraction yields, phytochemical analysis (GC-MS and FTIR), antioxidant activity tests (DPPH), and gravimetric corrosion analysis, we evaluated the potential of four plants—Alfalfa, Jojoba, Moringa, and Juniper—as eco-friendly alternatives to conventional corrosion inhibitors.

Extraction yields showed significant variations among the plant species. Alfalfa exhibited an exceptional extraction yield of 92.66%, much higher than the other plants. In contrast, Juniper showed the lowest yield (0.65%), which is understandable given the volatile nature of its components. However, despite its low yield, the Juniper extract demonstrated significant corrosion inhibition efficiency, notably due to its high α -pinene content (37.06%), a terpene with anti-inflammatory and antiviral properties.

GC-MS analysis of the extracts revealed distinct and complementary phytochemical profiles. For example, the Moringa extract showed a high concentration of 1H-Naphtho [2,1-b]pyran (62.96%), a polycyclic terpenoid whose conjugated double bond system facilitates strong adsorption onto metal surfaces. This characteristic contributed to exceptional corrosion inhibition rates, reaching 99.19% at a concentration of 500 ppm—a remarkable result that rivals that of synthetic inhibitors. Alfalfa, with a corrosion inhibition rate of 98.39% at 500 ppm, showed similar protective performance, highlighting the importance of its compounds, such as fatty acids and flavonoids, which form stable protective layers on metal surfaces.

Antioxidant activity tests, measured by the DPPH method, also confirmed the ability of the extracts to neutralize free radicals, with IC₅₀ values of 0.40 mg/ml for Alfalfa, 0.8 mg/ml for Jojoba, 1.0 mg/ml for Juniper, and 1.12 mg/ml for Moringa. These results highlight that Alfalfa extracts have stronger antioxidant activity than the others, which could also contribute to their effectiveness in preventing corrosion by neutralizing the free radicals that trigger metal degradation.

Gravimetric corrosion analysis provided crucial data on the effectiveness of the extracts in reducing the corrosion rate of metal specimens. The Moringa and Alfalfa extracts showed high corrosion inhibition at 500 ppm, with inhibition efficiencies of 99.19% and 98.39%, respectively. Jojoba, although effective, showed slightly lower performance with a maximum efficiency of

91.3% at 500 ppm. In contrast, Juniper exhibited an inverse concentration dependence, with peak efficiency (75.01%) at 0.5 ppm, but a decline to 21.77% at 500 ppm, suggesting competitive adsorption phenomena between its multiple terpenes.

Finally, SEM observations provided images showing remarkable preservation of metal surfaces treated with Moringa and Jojoba, contrasting with the untreated control samples, which showed severe degradation. The metal surfaces treated with Moringa and Jojoba exhibited minimal pitting and well-preserved topography, directly correlating with the gravimetric results and confirming the extracts' ability to form stable protective films on metal surfaces.

In conclusion, the results of this study show that plant extracts, particularly those from Moringa and Alfalfa, exhibit high potential as natural corrosion inhibitors, with inhibition rates comparable to or higher than those of synthetic corrosion inhibitors. These extracts offer significant environmental benefits due to their natural origin, biodegradability, and low toxicity. However, further research is needed to optimize extraction conditions, optimal concentrations of the extracts, and assess their long-term stability under industrial conditions. A blend of Moringa and Alfalfa extracts could offer an effective and sustainable solution for corrosion protection in acidic environments, while reducing the environmental impact associated with traditional chemical inhibitors.

الجمهورية الجزائرية الديمقراطية الشعبية

وزارة التعليم العالي والبحث العلمي

جامعة محمد خيضر بسكرة

عنوان المشروع:

BioBlock Labs – Next-Gen Natural Inhibitors for Chemical-Free Preservation.

مشروع لنيل شهادة مؤسسة ناشئة في إطار القرار الوزاري 1275.

صورة العلامة التجارية:



الاسم التجاري:

Biocorrshield

السنة الجامعية:

2025/2024

بطاقة المعلومات:

حول فريق الإشراف وفريق العمل.

1-فريق الإشراف:

التخصص: هندسة كيميائية	المشرف الرئيسي (01): حاجب ريحانة
التخصص: هندسة كيميائية	المشرف الرئيسي (02): مناصرة حياة

2-فريق العمل:

الكلية	التخصص	فريق المشروع
العلوم وتكنولوجيا	هندسة كيميائية	الطالبة: زكور هديل
العلوم وتكنولوجيا	هندسة كيميائية	الطالبة: كعب آلاء

عنوان المشروع:

BioBlock Labs – Next-Gen Natural Inhibitors for Chemical-Free Preservation.

فهرس المحتويات:

المحور الأول: تقديم المشروع.

المحور الثاني: الجوانب الابتكارية.

المحور الثالث: التحليل الاستراتيجي للسوق.

المحور الرابع: خطة الإنتاج والتنظيم.

المحور الخامس: الخطة المالية.

المحور السادس: النموذج الأولي التجريبي

عنوان المشروع :

BioBlock Labs – Next-Gen Natural Inhibitors for

.Chemical-Free Preservation

المحور الأول: تقديم المشروع.

تُعد مثبطات التآكل الطبيعي حلاً مبتكراً وصديقاً للبيئة، لا غنى عنه للمؤسسات التي تعتمد في عملياتها على أنظمة التسخين والمياه. فهي تحمي الأنابيب المستخدمة في نقل السوائل الحرارية أو المياه الساخنة، سواءً كانت قيد التشغيل أو مخزنة، مما يطيل عمرها التشغيلي ويقلل التكاليف. كما أنها حل آمن يحافظ على كفاءة المعدات ويُقلل من المخاطر البيئية، مما يجعلها اختياراً مثالياً للصناعات التي تهدف إلى الاستدامة.

1-فكرة المشروع (الحل المقترح):

انبثقت فكرة هذا المشروع البحثي خلال تقدم أحد مسؤولي لشركة حيث تقوم هذه الأخيرة على إنتاج الاجر لمواد البناء حيث تعاني هذه المؤسسة الصناعية من التآكل بسبب استعمالها كل من الحرارة والمياه حيث سببت هذه الأخيرة في تلف العديد من المعدات مما أدى الى تكاليف كبيرة لهذه الخسارة فأردنا إنتاج مثبطات طبيعية تعمل على الحد من التآكل والتقليل من الاضرار التي تلحق بهذه المؤسسات التي تعتمد على الحرارة والمياه في انتاجها.

2-القيم المقترحة:

الاداء:

توفير منتج طبيعي غير مضر وأحسن من المتاح حالياً.

السعر:

سعر اقل من المستورد وهو منتج كيميائي.

سهولة الوصول:

توفير منتج محلي بدل الاستيراد بصفات مختلفة على الكيميائي جدا وتوفير

مصاريف لأصحاب الشركات.

خفض التكاليف بالنسبة للمستهلكين:

توفير المنتج محليا يقلل من مصاريف شحن المنتج.
منتج طبيعي صديق للبيئة

3-فريق العمل:

الطلبة	التخصص	الدورات التكوينية
زكور هديل	هندسة كيميائية	تقنيات البحث عن وظيفة المسار المقاولاتي أحسن فكرة مشروع مبتكر التفكير التصميمي والابتكاري الذكاء الاصطناعي تطبيقات المالية والمحاسبية اعداد نموذج اعمال أي اساسيات و تطبيقات على: BMC 1275 مشاريع ورشات حوا تطبيقات واستخدامات التسويق الرقمي في المشاريع. كيفية إعداد الدراسة التقنية والاقتصادية للمشاريع المبتكرة.
كعب الاء	هندسة كيميائية	تقنيات البحث عن وظيفة المسار المقاولاتي أحسن فكرة مشروع مبتكر التفكير التصميمي والابتكاري الذكاء الاصطناعي

<p>تطبيقات المالية والمحاسبية</p> <p>اعداد نموذج اعمال أي اساسيات و تطبيقات على :BMC</p> <p>مشاريع 1275</p> <p>ورشات حوا تطبيقات واستخدمات التسويق الرقمي في المشاريع.</p> <p>كيفية إعداد الدراسة التقنية والاقتصادية للمشاريع المبتكرة</p>		
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4-اهداف المشروع:

- الهدف الرئيسي للمشروع هو تحويل المنتجات الثانوية الزراعية إلى مثبتات تآكل طبيعي صديق للبيئة كبديل آمن للمثبتات الكيميائية الضارة مما يساهم في حماية المنشآت والبنية التحتية مع تقليل الأثر البيئي.
- استغلال المنتجات الثانوية الزراعية وتحويلها إلى منتج فعال وصديق للبيئة، مما يقلل الاعتماد على المواد الكيميائية الضارة.
- تحقيق حصة سوقية تنافسية من خلال تقديم منتج مبتكر وغالي الجودة يلبي احتياجات الصناعات المختلفة.
- تعزيز الإنتاج المحلي وتقليل الاستيراد مما يدعم الاقتصاد الوطني ويحقق الاكتفاء الذاتي في مجال مثبتات التآكل.
- تلبية توقعات العملاء من خلال منتج آمن وموثوق، مع تركيز على جودة وأداء المتميز.
- بناء سمعة قوية للمشروع كرائد في مجال المثبتات الطبيعية مع التركيز على الابتكار والاستدامة.
- تغطية السوق المحلية بكفاءة، ثم التوسع نحو الأسواق الأفريقية والعالمية، نظرا للطلب المتزايد على الحلول البيئية البديلة.
- دعم التحول نحو الاقتصاد الأخضر من خلال تقليل الاعتماد على الكيماويات الصناعية الضارة، وتعزيز الممارسات المستدامة.

5-جدول زمني لتحقيق المشروع:

الزمن (بالأشهر)												الاعمال	
12	11	10	9	8	7	6	5	4	3	2	1		
													مقر اختيار الوحدة الإنتاجية وتجهيز الوثائق المطلوبة
													طلب التجهيزات
													إيجار وتهيئة مقر الإنتاج
													تركيب المعدات
													اقتناء المواد الأولية
													ترويج عينات من المنتج واستقطاب بالمزبانن
													بداية انتاج اول منتج.

عنوان المشروع :

BioBlock Labs – Next-Gen Natural Inhibitors for Chemical-Free Preservation.

المحور الثاني: الجوانب الابتكارية

ابتكار السوق: الدخول إلى السوق الجزائرية بمنتج جديد وحيوي.
ابتكار متزايد: تطوير وتحسين المنتج للحصول على عدد كبير من الزبائن.

تتمثل الجوانب الابتكارية في:

- التنوع في المواد الأولية المستخدمة في إنتاج مثبت التآكل الطبيعي.
- بساطة طريقة الإنتاج.
- التركيز على الدول التي تعاني من تآكل البنية التحتية بسبب الرطوبة أو المياه المالحة

عنوان المشروع :

BioBlock Labs – Next-Gen Natural Inhibitors for Chemical-Free Preservation.

المحور الثالث: التحليل الاستراتيجي للسوق

أولاً: تحليل التغيرات الكلية (PESTEL)

1-العوامل البيئية: صديق للبيئة.

2-العوامل السياسية: تواجه الدولة الجزائرية إلى تشجيع المؤسسات الناشئة.

قرارات رئيس الجمهورية إلى منع الإستيراد.

3-العوامل الاقتصادية: إمكانية الحصول على قروض عن طريق مصادر الدعم وصناديق ضمان الاجتماعي.

ثانياً: تحليل القوى التنافسية (PORTER)

1-حدة المنافسين:

منافسين مباشرين: لا توجد منافسة في السوق نظراً لعدم وجود شركات متخصصة في هذا الإنتاج.

منافسين غير مباشرين: مصانع لصناعة مثبت تآكل كيميائي.

2-قوة التفاوضية مع العملاء:

توفر مثبت طبيعي محلياً يعطي فرصة للدخول في الأسواق واحتكارها ذلك لعدم وجود منافسين وكذلك بموجب القوانين

الجديدة التي تمنع الاستيراد وبالتالي محاولة الاستغناء عن المثبت الكيميائي.

3-قوة الموردين:

توفر مصانع آخرين في مختلف المجالات الصناعية يستعملون المنتجات الثانوية للزراعة مما يؤدي إلى حدوث تنافس

عليها وبالتالي إمكانية غلاءها.

4-تهديدات المنتجات البديلة:

ما يتم طرحه الآن من منتجات في الأسواق هو المثبت الكيميائي الذي له أخطار على مستعمليه وعلى المحيط فإنه تم

التخمين في إيجاد بديل طبيعي غير ضار وذلك باستغلال المنتجات الثانوية للزراعة.

عنوان المشروع:
**BioBlock Labs – Next-Gen Natural Inhibitors for
Chemical-Free Preservation.**

ثالثا: تحليل SWOT

1-نقاط الضعف:

توفير المواد الأولية بشكل قليل.
عدم وجود راس مال للمشروع.
انخفاض ميزانية للترويج.

2-نقاط القوة:

يمكن توفير المنتجات الثانوية للزراعة بحكم انها غير موسمية.
تمكن أصحاب المشاريع للقيام به (أعضاء الفريق).
قوة تنافسية لبعض المؤسسات.

3-الفرص:

منتج محلي ومناصب عمل وتقليص الوقت وتكاليف للشركات.
نمو سريع في السوق نظرا لسلامة المنتج.
غلق الاستيراد
دعم الدولة للمؤسسات الناشئة
شغور وفراغ في السوق (لعدم توفر هذا المنتج من قبل ولحد الان)
4-التحديات:

تقليد المنتج ودخول منافسين وهذا ان لم تكن هناك حماية للمنتج.
عدم مواكبة التطورات التكنولوجية مما يفقد المؤسسة قدرتها التنافسية.
رابعا تحليل المنتج التسويقي :

1.المنتج:

(XXXالاسم):

(XXXالوزن):

(XXX/XX/XXXXتاريخ الإصدار):

طريقة الحفظ: يحفظ في درجة حرارة منخفضة اقل من 4°.

2.السعر.

3.الترويج:

منصات التواصل الاجتماعي

انشاء منصة خاصة أي موقع مخصص للمنتج.

المشاركة في المعارض والاحتكاك بمدراء الشركات الاقتصادية.

البيع الشخصي.

4.التوزيع:

تعتمد الوسيط للتوزيع وإيصال المنتج للزبائن.



خامسا: الاستراتيجيات التسويقية:

استراتيجية التميز: منتج جديد مع جودة عالية وهذا ما يجعل تسعيرة المنتج متميزة.

سادسا: تقدير المبيعات:

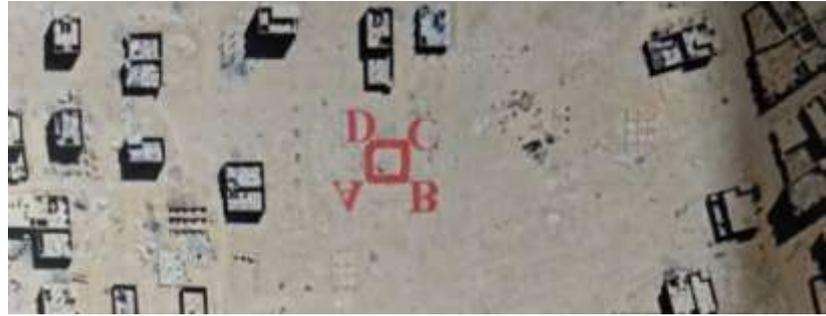
نظرا للمنتج جديد في سوق كما يعتبر ذا جودة عالية مما يمكن المؤسسة من بيع نسبة كبيرة من حجم الإنتاج السنوي .

عنوان المشروع:

BioBlock Labs – Next-Gen Natural Inhibitors for Chemical-Free Preservation.

المحور الرابع : خطة الإنتاج و التنظيم.

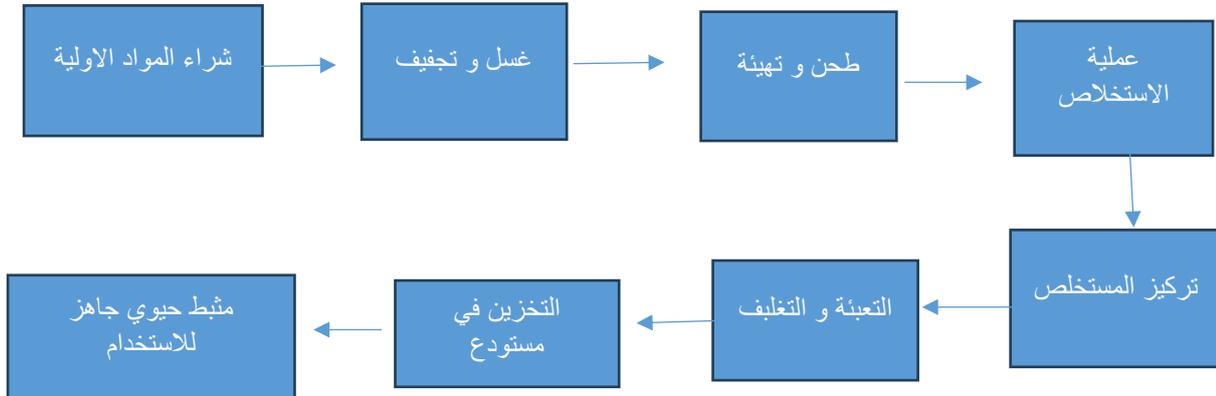
موقع المشروع (مساحة 5 هكتار) .



طبيعة لانتاج:

يهدف المشروع الى تحويل مصادر نباتية طبيعية (بذور البرسيم ,العراعر, أوراق الجوجوبا و المورينغا) الى مستخلصات نباتية حيوية تعمل كمثبطات طبيعية للتآكل تستخدم في التطبيقات الصناعية .

مخطط الإنتاج:



احتياجات المشروع :

المواد الأولية : لتجهيز الكمية الأولى في اليوم الواحد فقط , هذا الجدول قابل للتغيير من ناحية الكمية .

النوع	الكمية المطلوبة	سعر الوحدة	المصدر
بذور البرسيم	5 كغ	800 دج\كغ	أسواق الجملة
العرعار	5 كغ	1000 دج\كغ	جمع مباشر او سوق محلي
أوراق الجوجوبا	5 كغ	1200 دج\كغ	مشاتل او تعاونيات زراعية
أوراق المورينغا	5 كغ	1500 دج\كغ	مشاتل او جمع طبيعي
كحول ايثانولي 96%	10 لتر	1700 دج\لتر	مورد كيميائي
زجاجات تعبئة	100 قارورة	140 دج\قارورة	متاجر الجملة
عبوات تغليف كرتونية	100 وحدة	40 دج\وحدة	مورد التعبئة

الالات و المعدات:

اسم الآلة و وظيفتها	مدة الاستهلاك	سعرها (DA)
حوض غسيل و تجفيف اولي	\	400,000
فرن تجفيف كهربائي (تجفيف النباتات)	20 سنة	220,000
جهاز استخلاص	10 سنوات	350,000
جهاز تركيز (Rotary Evaporator)	10 سنوات	1,200,000
مطحنة كهربائية	10 سنوات	150,000
جهاز تقطير ماء مقطر	10 سنوات	150,000
معدات التعبئة اليدوية	10 سنوات	100,000
المجموع	-	2,570,000

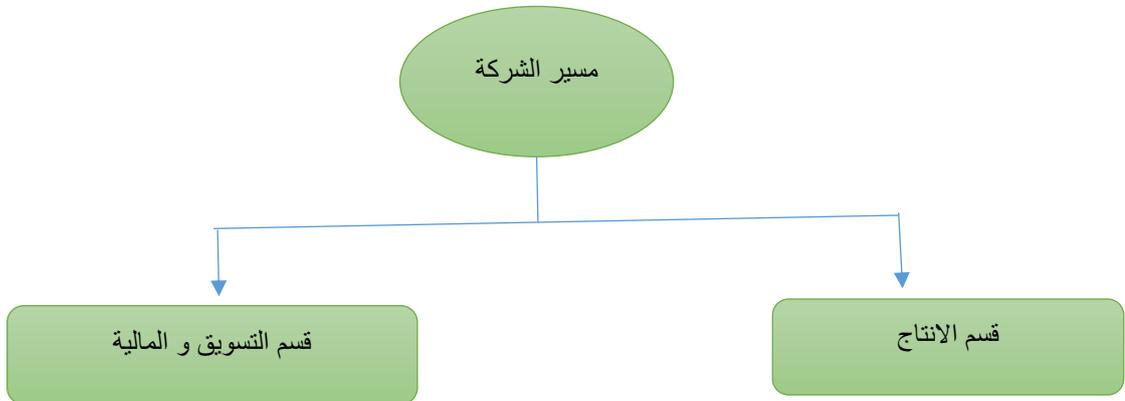
الموارد البشرية اللازمة للمشروع:

عدد المناصب	دور المنصب	الاجر الشهري لكل عامل (DA)
2 مهندس كيميائي	الاستخلاص و الترشيح.	50,000
2 عامل مخبري	غسل و تجفيف. تعبئة و تغليف. التخزين.	40,000
1 حارس ليلي		30,000

الأثاث و التجهيزات :

الاسم	الكمية المطلوبة	سعر الوحدة (DA)
طاوولات مخبرية	3	250,000
خزانات	2	100,000
كراسي	5	75,000
مكيفات 24000W	2	200,000
حواسيب	2	120,000
طابعة	1	48,000

المخطط التنظيمي:



عنوان المشروع :

BioBlock Labs – Next-Gen Natural Inhibitors for Chemical-Free Preservation.

المحور الخامس : الخطة المالية PLAN FINANCIER

أولا تكاليف المشروع و اجمال الاستثمار:

تكاليف المشروع: تتمثل التكاليف الاجمالية للمشروع في التكاليف الاستثمارية و التكاليف التشغيلية.

التكاليف الاستثمارية:

الأصول	التكلفة
الالات و المعدات	2,570,000
التجهيزات	793,000
المجموع	3,363,000

التكاليف التشغيلية (الميزانيات التقديرية لسنة واحدة):

الأصول	التكلفة
مشتريات لمواد الاولية	57,500
أجور العمال	210,000
ايجار المحل	100,000
مصاريف اخرى	40,000
الاشهار	400,000
المجموع	807,500

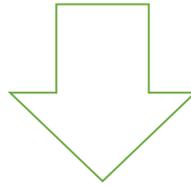
الهيكل التمويلي:

سنعتمد في مشروعنا على القروض بنسبة 100% في التمويل.

عنوان المشروع :

BioBlock Labs – Next-Gen Natural Inhibitors for Chemical-Free Preservation.

المحور السادس: النموذج الاولي التجريبي.



عنوان المشروع :

BioBlock Labs – Next-Gen Natural Inhibitors for Chemical-Free Preservation.

الجدول و الاشكال:

	Preview					
Product A intended for Customer	N	N+1	N+2	N+3	N+4	N+5
Quantity product A	10,000	11,000	12,000	12,500	13,000	13,500
Price HT product A	3,300	3,400	3,500	3,600	3,700	3,800
Sales product A	33,000,000	37,400,000	42,000,000	45,000,000	48,100,000	51,300,000
SALES TURNOVER	33,000,000	37,400,000	42,000,000	45,000,000	48,100,000	51,300,000

ملحق : نموذج العمل التجاري

<p>الشركات الرئيسية</p> <p>مخابر. مكتب محاسبة معتمد. شركات التوصيل. الموردون المحليون للمواد الخام.</p>	<p>الأنشطة الرئيسية</p> <p>استخلاص مثبتات التآكل الطبيعية. البحث والتطوير لتحسين جودة المنتج. التسويق والترويج عبر القنوات الرقمية والمعارض. التواصل مع العملاء وتقديم الدعم الفني.</p>	<p>القيمة المقترحة</p> <p>منتج طبيعي وآمن وصديق للبيئة. سعر تنافسي أقل من المنتجات المستوردة. منتج محلي بجودة عالية. تقليل المخاطر البيئية مقارنة بالمثبتات الكيميائية.</p>	<p>العلاقة مع الزبائن</p> <p>بناء الثقة مع العملاء من خلال الدعم المستمر. التواصل عبر المنصات الرقمية . تقديم عينات مجانية لجذب العملاء الجدد. إنشاء علاقة طويلة الأمد مع الشركات الصناعية.</p> <p>القنوات</p> <p>البيع المباشر للشركات الصناعية. منصات التواصل الاجتماعي والموقع الإلكتروني. شركات التوصيل والتوزيع (وسطاء).</p>	<p>شرايح العملاء</p> <p>المصانع التي تعاني من مشاكل التآكل. شركات البناء والصناعات الحرارية. الصناعات التي تعتمد على أنظمة المياه والحرارة.</p>
<p>هيكل التكاليف</p> <p>تكلفة شراء المواد الخام. أجور العمال. إيجار مقر الإنتاج. مصاريف الترويج والإعلانات. تكاليف الصيانة .</p>		<p>مصادر الإيرادات</p> <p>مبيعات المنتج (مثبط التآكل الطبيعي).</p>		

بسكرة في:

إذن بإيداع مذكرة الماستر بعد التصحيحات

أنا المضي أسفله الأستاذ: حاجب ريجانة

الرتبة: أستاذ محاضر قسم أ

أستاذ مشرف على مذكرة ماستر - للطالب (ة):

زكور حديد و كعب آلاء

الشعبة: كيمياء صناعية

التخصص: هندسة كيميائية

بعنوان:

*Extraction, characterisation and application of bioactive
inhibitors from plant-based resources.*

أرخص بإيداع المذكرة المذكورة.

رئيس لجنة المناقشة

الأستاذ المشرف

الجمهورية الجزائرية الديمقراطية الشعبية

وزارة التعليم العالي والبحث العلمي

بسكرة في:

جامعة محمد خيضر - بسكرة

كلية العلوم والتكنولوجيا

قسم الكيمياء الصناعية

إذن بإيداع مذكرة الماستر بعد التصحيحات

أنا الممضي أسفله الأستاذ: مناصرة حياة

الرتبة: أستاذ

أستاذ مشرف على مذكرة ماستر - للطلاب (ة):

زكور حديد و كعب آلاء

الشعبة: كيمياء صناعية

التخصص: هندسة كيميائية

بعنوان:

Extraction, characterisation and application of bioactive inhibitors from plant-based resources

أرخص بإيداع المذكرة المذكورة.

رئيس لجنة المناقشة

الأستاذ المشرف

P. Mansour Hayet 

ملحق القرار رقم: 933 المؤرخ في: 20 جمادى الأولى 2016

الذي يحدد القواعد المتعلقة بالوقاية من السرقة العلمية ومكافحتها

الجمهورية الجزائرية الديمقراطية الشعبية

مؤسسة التعليم العالي:

نموذج التصريح الشرفي

خاص بالالتزام بقواعد النزاهة العلمية لانجاز بحث

أنا الممضي أدناه،

السيد: كحي... الصفة: طالب، أستاذ باحث، باحث دائم: ...

الحامل لبطاقة التعريف الوطنية رقم: ... والصادرة بتاريخ: ...

المسجل بكلية العلوم والتكنولوجيا... الصناعية

و المكلف بإنجاز أعمال بحث (مذكرة التخرج ، مذكرة ماستر ، مذكرة ماجستير ، أطروحة

دكتوراه)، عنوانها: Extraction... characterization... and... Application... of bioactive... inhibitors... from... plant... based... resources.

أصرح بشرفي أنني ألتزم بمراعاة المعايير العلمية والمنهجية ومعايير الأخلاقيات المهنية والنزاهة الأكاديمية المطلوبة في انجاز البحث المذكور أعلاه.

التاريخ: 06/06/2022

إمضاء المعني



