



People's Democratic Republic of Algeria  
Ministry of High Education and Scientific  
Research



University of Kasdi Merbah Ouargla  
Faculty of Mathematic and material sciences  
Department of Chemistry

THESIS SUBMITTED TO OBTAIN ACADEMIC MASTER DEGREE IN  
APPLIED CHEMISTRY

**Reproduction and Characterization of a  
Corrosion Inhibitor used in the petroleum  
Industry**

Presented by:

**Lassakeur Wassila**

**Ammari Chaima**

Publicly discussed on **12/06/2024**

In front of the jury members consisting of:

BELFAR Mohamed Lakhdar	Professor	Univ. Ouargla	President
ZAOUI Manel	Professor	Univ. Ouargla	Supervisor
Mekhelfi Tarek	Professor	Univ. Ouargla	<b>Co-Supervisor</b>
Zlkhrai Louiza	Professor	Univ. Ouargla	Examiner

**Academic year:2023/2024**





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## **Abstract**

Several experiments were conducted to obtain chemical inhibitors. In this study we analysed DRX and EDX for Anhydrous sodium sulfate. The results of this analysis proved that this sample was the Anhydrous sodium sulfate phrase. The GC-MS/MS analysis of Diethyl sulfate compound that was manufactured showed that the results were hull.

**Keywords:** corrosion, inhibitor

# ACKNOWLEDEMEN

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Finally, we would like to express our gratitude to all our friends and colleagues, and

all those who helped us directly or indirectly during the completion of this work.

## شكر و عرفان

الحمد لله رب العالمين و الصلاة و سلام على اشرف المرسلين و على اله و صحبه  
أجمعين قال الله تعالى " رب اوزعني ان اشكر نعمتك التي انعمت علي و على والدي و ان  
اعمل صالحا ترضاه "

(19 سورة النمل)

## اعترافا بالحميد

من قال انا لها " نالها "

لم تكن الرحلة قصيرة ولا ينبغي لها ان تكون

لم يكن الحلم قريبا ولا الطريق كان محفوفا بالتسهيلات

لكني فعلتها ونلتها

الحمد لله حبا وشكرا وامتنانا الذي بفضلها ها انا اليوم انظر الى حلما طال انتظاره وقد أصبح واقعا افتخر  
به

الى ملاكي الطاهر، وقوتي بعد الله داعمتي الأولى و الأبدية " امي " **و.شتوي** اهديكي هذا الإنجاز الذي  
لولا تضحياتك لما كان له وجود، ممتنة لان الله قد اصطفاك لي من البشر اما يا خير سند و عوض

الى من دعمني بلا حدود و اعطاني بلا مقابل و امن بقدراتي و شاركني هذه الرحلة خطوة بخطوة كان لي  
خير داعم فاحمد لله على وجودك في حياتي كل الحب والتقدير لك " ابي الغالي " **علي عماري**

الى من قيل فيهم:

{سنشد عضدك بأخيك}

الى من مد يده دون كلل ولا ملل وقت ضعفي و امن بي **اخي رزقي**

والى من رسم بسمه على وجهي وقت حزني وتعبي **اخي مصطفى**

والى صغيري و حبيبي **اخي معتز الله**

الى جميلتي و سر سعادتي صغيرتي **اختي جواهر**

**عماري شيماء**

## DEDICATION

In the name of God the Merciful, to my dear family,

As I stand on the edge of this huge achievement, I am grateful to everyone who has been my rock, strength and support throughout my academic journey.

To those who endowed me with life and hope, growing up with a passion for knowledge and knowledge, and who taught me to rise the ladder of science wisely and patiently, with patience, charity and fulfillment

**"My dear father and dear mother."**

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**Wassila Lassakeur**

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## ***INTRODUCTION***

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## General Introduction

Corrosion is a significant economic issue that affects various industries worldwide. It is a chemical or electrochemical reaction between a material, usually a metal, and its environment that results in the deterioration of the material and its properties. The economic impact of corrosion is substantial, with the United States alone experiencing annual costs of almost \$300 billion.

This cost is attributed to both direct and indirect losses.

Corrosion phenomena are determined by a variety of parameters, including the material's composition and structure, the environment and its chemical properties, temperature, and so on. Corrosion is a collection of physical, chemical, and mechanical events. Understanding these events must allow us to fight more effectively against material deterioration by selecting the best appropriate protection technique.[1]

A corrosion inhibitor may be defined, in general terms, as a substance which when added in a small concentration to an environment effectively reduces the corrosion rate of a metal exposed to that environment. Inhibition is used internally with carbon steel pipes and vessels as an economic corrosion control alternative to stainless steels and alloys, coatings, or non-metallic composites. A particular advantage of corrosion inhibition is that it can be implemented or changed in-situ without disrupting a process. The major industries using corrosion inhibitors are the oil and gas exploration and production industry, the petroleum refining industry, the chemical industry, heavy industrial manufacturing industry, water treatment facilities, and the product additive industries. The largest consumption of corrosion inhibitors is in the oil industry, particularly in the petroleum refining industry. The total consumption of corrosion inhibitors in the United States has doubled from approximately \$600 million in 1982 to nearly \$1.1 billion in 1998.{2}

In this research, we focused on the manufacture of chemical inhibitors that reduce corrosion rates in pipes and petroleum wells and form a protective layer (insulator) on the metal surface.

**Chapter I** reviews the bibliography of the corrosion process, its forms, its types, and the working mechanism, and then provides general information about the inhibitions and their types.

**Chapter II Part I** covers the chemical and physical properties of chemicals that are employed as inhibitors

**Chapter II Part II** Covers the ways and how to manufacture these chemical inhibitors.

**Chapter III** Discussion of search results.

# Chapter I

---

*Corrosion phenomenon review*

---

## **I. Introduction**

Corrosion of metals and alloys is a well-known phenomenon that leads to significant material losses, both direct and indirect, for various industries. More critically, corrosion can have severe impacts on oil installations and production. This includes the costs associated with replacing corroded structures and machinery, and the downtime required to repair these installations. Moreover, corrosion poses risks to human life due to potential pollution, contamination, and other hazards. The degradation caused by corrosion can result in severe deformations, such as a general reduction in thickness, the formation of pits, and issues related to insulating different metals.

### **I.A.1. Definition of corrosion**

Corrosion of a material refers to its deterioration or loss of mechanical properties due to exposure to its immediate environment, which can include soil, atmosphere, water, or other fluids. Given the numerous parameters involved in the electrochemical process, corrosion is a highly intricate phenomenon.

Corrosion can be broadly understood as a spontaneous electron exchange reaction at the interface between metal and its environment. A natural process tends to convert metals back to their oxide state through various levels of attack by the corrosive medium.

The physicochemical interaction between a metal and its surrounding environment, which leads to alterations in the metal's properties and often-functional degradation, defines corrosion. Another viewpoint describes corrosion as the natural transformation of metals and alloys back to their mineral states. Regardless of the specific definition used, corrosion signifies degradation. [3.4.5]

### **I.A.2. Types of corrosion**

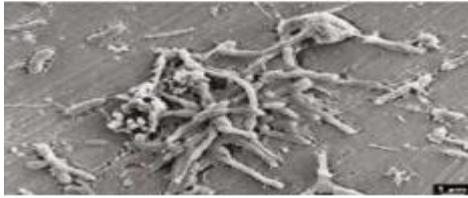
Corrosion can develop according to different processes that characterize each type of corrosion:

#### **I.A.2.1. Chemical corrosion**

This type of corrosion occurs when the metal surface is uniform, and the surrounding reaction mixture is uniformly distributed. The metal undergoes attack uniformly in this scenario, without any electron flow within the metal. For instance, the oxidation of ordinary steel at high temperatures by atmospheric oxygen is a common example of this phenomenon. [6]

#### **I.A.2.2. Biochemical corrosion**

It is the bacterial attack of metallic materials (Figure I.1), especially in underground pipes and tanks. Indeed, the metabolism of the development of certain bacteria causes the formation of sulphuric acid that attacks the metal.[6]



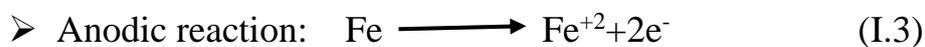
(Figure I.1): Biochemical corrosion

### I.A.2.3. Electrochemical corrosion

Corrosion corresponds to the transformation of metallic atoms into ions in solution (metal dissolution); the dissolved cation can then exist in various forms: hydrated, hydrolyzed, or it can form bonds that are more complex:[7]



These reactions consist of two half-reactions known as the anodic reaction (oxidation reaction) and the cathodic reaction (reduction reaction), respectively. For example, in the case of reaction (I.1)



### I.A.3. The forms of corrosion

The process of corrosion of metals takes a few many forms, which are classified mainly according to the form that manifests itself on the corroded surface.

#### I.A.3.1. Uniform corrosion (generalized)

Uniform corrosion occurs when the entire surface of a metal exposed to a solution is attacked uniformly. This results in a consistent dissolution of the metal surface in contact with the corrosive agent.[7]

This type of material corrosion typically occurs in acidic or alkaline environments.



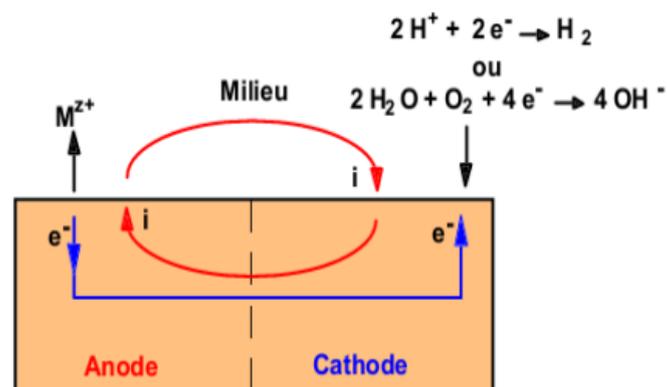
(Figure I.2): Uniform corrosion (generalized) of a steel part

### I.A.3.2. Localized corrosion

This refers to a type of corrosion where metal loss occurs only in specific areas of the material, particularly affecting passivable materials. This type of corrosion is commonly known as localized corrosion.[7]

### I.A.3.3 Galvanic corrosion

Galvanic corrosion occurs when two dissimilar metals are in electrical contact with each other. This can also be triggered by factors that alter electrochemical potential, such as temperature variations, exposure to different chemicals, and so on. For galvanic corrosion to happen, both the anode and cathode must be in contact and exposed to a continuous electrolytic environment. The most typical electrolytic environments are water and moist soil.[7]



(Figure I.3): A diagram of a pile of corrosion

#### **I.A.3.4 Pitting corrosion**

Pitting corrosion is a prevalent type of corrosion in oilfields, second only to general attack. Unlike general corrosion, which can be anticipated and monitored, pitting corrosion can begin and spread rapidly within short timeframes, causing substantial damage. Monitoring for pitting corrosion necessitates frequent inspections or sampling because extended periods of no corrosion activity may be followed by sudden and aggressive initiation and growth of pits.[7]

#### **I.A.3.5 Intergranular corrosion**

Grain boundaries typically exhibit higher reactivity compared to the surrounding base metal due to impurities and defects. However, this increased reactivity is generally minimal and of little consequence within crystal matrices. Intergranular corrosion occurs when significant variations in alloy composition near grain boundaries lead to substantial attack and deterioration of the alloy.

This type of corrosion can be particularly problematic in stainless steels and certain aluminum alloys. Similarly, highly alloyed austenitic iron-chromium-nickel alloys may also experience this issue.[7]

#### **I.A.3.6 Selevtive corrosion**

This refers to a form of corrosion where one or more alloying elements dissolve preferentially. One of the alloy constituents is preferentially attacked, leading to a total loss of mechanical strength without apparent material loss. Examples include dezincification of brass and graphitic corrosion.[8]

#### **I.A.3.7 Cavernous corrosion**

These are attacks caused by the confinement of the environment, which makes a certain metal portion anodic within enclosed areas. This differential aeration creates a galvanic cell where the least exposed part of the metal structure becomes the site of corrosion.[9]

#### **I.A.3.8 Stress corrosion**

Stress corrosion is a cracking of the metal (Figure I.4) that results from the joint action of mechanical stress and an electrochemical reaction. This particularly dangerous phenomenon occurs by the combined effects of three parameters [10.11]:

- Temperature: stress corrosion rarely develops below 50°C.
- The applied or residual stresses experienced locally by piece.

- Corrosivity of the environment: presence of Cl, H<sub>2</sub>S or NaOH.



**(Figure I.4):** Stress corrosion

#### **I.A.4 Mechanisms of corrosion**

When we consider the corrosion of copper or iron, the reactions we see the results of, in the form of corrosion products, are galvanic cell or electrochemical reaction. As in all chemical reactions, corrosion reactions occur through an exchange of electrons.

In electrochemical reactions, the electrons are produced by a chemical reaction in one area, the anode area. The electrons travel through a metallic path, usually the parent metal, and are consumed through a different chemical reaction in another area, the cathodic area.[12]

##### **I.A.4.1 Anodic reactions**

The generic chemical reaction for this metal loss at anodic sites is:



Where M is an uncharged metal atom at the metal surface, M<sup>+</sup> is a positively charged metal ion in the electrolyte and e<sup>-</sup> is an electron that remains in the metal.

This type of chemical reaction is called metal oxidation, even though it does not directly involve oxygen, but only results in an increase in positive charge on the atom undergoing oxidation or loss of electrons. This resultant ion is dissolved into the water.

More than one electron can be lost in the reaction, as in the case of iron where the most common anodic reaction is:



Where Fe is metallic iron.  $\text{Fe}^{+2}$  is a ferrous ion that carries a double positive charge.

#### I.A.4.2 Cathodic reactions

At cathodic sites, electrons are consumed by chemical reactions that consume electrons.

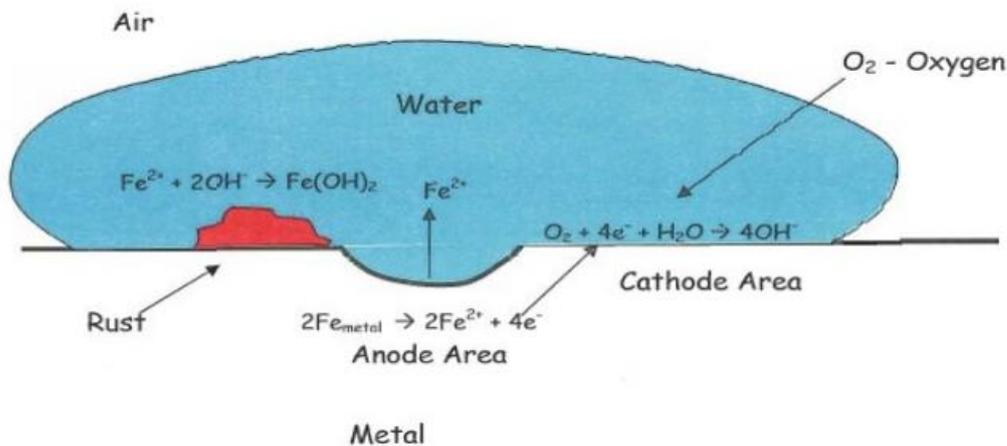
The reduction of oxygen is a common cathodic reaction:



Further to this, the four  $\text{OH}^{-}$  ions are free in the water to combine with the  $\text{Fe}^{+2}$  ions to form  $\text{Fe}(\text{OH})_2$  following the equation:



This whole process can be seen in the following diagram (**Figure I.5**) where the product is iron hydroxide, or what we commonly call “rust”.



(**Figure I.5**): reaction with water as the solvent.

#### I.A.5 Corrosion factors

The phenomenon of corrosion is influenced by a wide range of factors and can be categorised into four main groups: **Table I.1**

<b>Corrosive environment invoices</b>	<b>Metallurgical fractures</b>	<b>Factors defining employment conditions</b>	<b>Factors depending on exposure time</b>
<ul style="list-style-type: none"> <li>- Reactant concentration</li> <li>-Oxygen content</li> <li>-PH of the medium</li> <li>-Temperature</li> <li>-Pressing</li> </ul>	<ul style="list-style-type: none"> <li>- Chemical composition of the alloy.</li> <li>- Development processes.</li> <li>- Impurities.</li> <li>- Heat treatment.</li> <li>- Mechanical treatment of the elements of addition.</li> </ul>	<ul style="list-style-type: none"> <li>-Surface condition</li> <li>-Coin shape.</li> <li>- Employment.</li> <li>-Inhibitors.</li> <li>-Assembly processes.</li> </ul>	<ul style="list-style-type: none"> <li>-Mechanical stress</li> <li>-Modification of protective coatings</li> <li>-formation of depositions corrosion</li> </ul>

(Table I.1): Corrosion factors[13]

## **I.B.2 Definition inhibition**

An inhibitor is a substance that, when introduced in small quantities to an environment, effectively reduces metal deterioration. It helps minimize metal loss, decreases the risk of hydrogen embrittlement, protects against pitting, reduces excessive etching, and mitigates the emission of acid fumes from highly reactive acidic and basic metals. Corrosion inhibitors function by forming a protective barrier, creating an adsorbed layer, or slowing down the processes occurring at the cathode and anode. Corrosion inhibitors facilitate any method that delays corrosion or reduces the rate of metal oxidation through chemical addition. These inhibitors are typically easy to apply and can be used on-site without significant disruption to the process. Utilizing corrosion inhibitors is one of the most effective strategies to combat corrosion.[14]

### **I.B.2.1 Corrosion inhibitors**

An inhibitor is a chemical compound that, when added at low levels to the corrosive medium slows down or even stops the corrosion process of a metal in contact with this medium.

### **I.B.2.2 Properties**

To meet this definition, a corrosion inhibitor must verify a number of fundamental aspects:[15]

- ✓ Minimize the metal's corrosion rate while maintaining its physicochemical properties.
- ✓ Stability is essential in the presence of other constituents.
- ✓ Must be stable in the temperature range used.
- ✓ Low concentration levels require effective use.
- ✓ It is necessary to be effective in the conditions of use.
- ✓ The savings it makes make it inexpensive.

The current standards for non-toxicity and environmental protection must be met.

Make sure that it is in line with the current standards for non-toxicity and environmental protection.

### **I.B.2.3 Uses**

- ✓ There are several traditional fields where water inhibitors can be utilized.
- ✓ Oil treatment (including sanitary, water, industrial process water, boiler water, and others)
- ✓ Paints where industry: drilling, extraction, refining, storage and transport, corrosion inhibitors are additives that provide corrosion protection to metals.[16]

### **I.B.3 Classes of inhibitors\_[17]**

Different methods exist for classifying inhibitors, which can be distinguished from each other.

- The products' nature (organic or mineral inhibitors) is the reason.
- Either their electrochemical mechanism of action (cathode inhibitors, anodic or mixed)
- Their interface mechanisms and principles of action (adsorption to the metal surface and/or formation of a protective film) are involved.
- Either from the area of application.

#### **I.B.3.1 Classification according to the nature of the inhibitor**

- **Organic inhibitors**

The intention of organic molecules is to be more than just corrosion inhibitors. Their current preference over inorganic inhibitors is due to reasons of ecotoxicity, mainly.

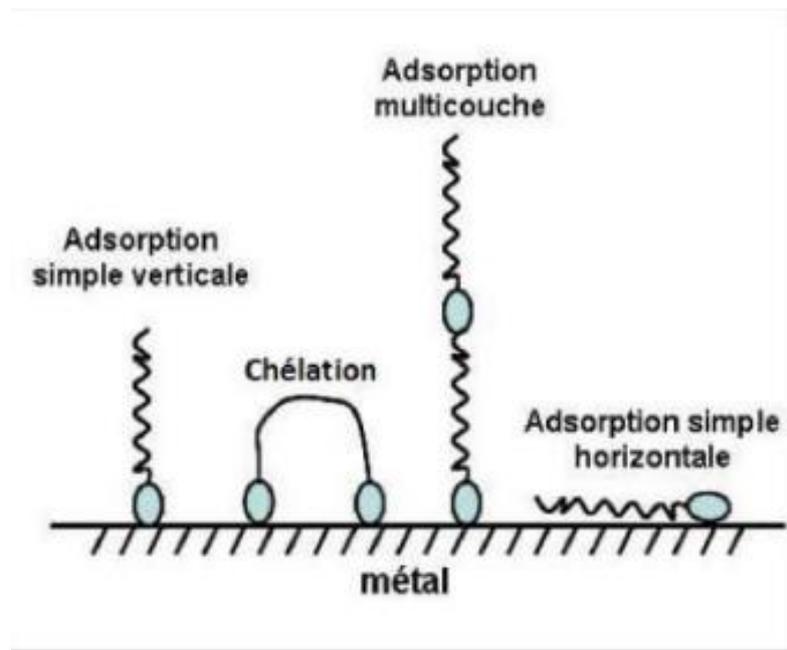
The inhibitory action of these organic compounds is related to the formation (by adsorption) of a more or less continuous barrier, but of finite thickness, which prevents the access of the solution to the metal.

Organic compounds used as inhibitors must have at least one heteroatom serving as an active centre for their fixation on metal such as nitrogen (amines, amides, imidazolines, triazoles, etc.), oxygen (acetylene alcohols, carboxylates, oxadiazoles, etc.), sulphur (derived from thiourea, mercaptans, sulfoxides, thiazoles, etc.) or phosphorus (phosphonates). Organic molecules can become unstable at high temperatures, which can limit the use of these products.

The molecule binds to the surface by its functional group, while its largest non-polar part partially blocks the active surface (**Figure I.6**). Other structural parameters that may influence inhibitor effectiveness include:

- The projection of the inhibitor's molecular area onto the metal surface is influenced by the arrangement of organic ions or molecules at the interface (metal/solution).
- The effect of molecular weight.
- The importance of the molecular configuration, descriptors of the molecule, namely HOMO energy, LUMO energy and dipole moment  $\mu$ ...

The impact of replacing nature.



(Figure I.6): Schematic representation of adsorption modes of inhibitory organic molecules on a metal surface.

### ➤ Mineral inhibitors

Mineral molecules are most often used in near-neutral environments, even in alkaline environments and more rarely in acidic environments. The products dissociate in solution, and it is often their dissociation products that ensure the phenomena of inhibition (anions and cations). Inhibitory cations are essentially  $\text{Ca}^{+2}$  and  $\text{Zn}^{+2}$  and those that form insoluble salts with certain anions such as hydroxyl ( $\text{OH}^-$ ). The main inhibitory anions are  $\text{XO}_{4n}^-$  type oxo-anions such as chromates, molybdates, phosphates, silicates.

The number of molecules currently in use is becoming more limited, as most effective products have a negative impact on the environment. However, new Organic complex of chromium III and other cations ( $\text{Zn}^{+2}$ ,  $\text{Ca}^{+2}$ ,  $\text{Mg}^{+2}$ ,  $\text{Mn}^{+2}$ ,  $\text{Sr}^{+2}$ ,  $\text{Al}^{+2}$ ,  $\text{Zr}^{+2}$ ,  $\text{Fe}^{+2}$ ....) effective against corrosion and non-toxic have been developed

### I.B.3.2 Classification according to the mechanism of action

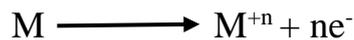
Corrosion inhibitors operate differently based on the specific corrosion system, including the type of metal and solution involved. A single compound can exhibit various mechanisms of action depending on these factors. Generally, the mechanism of an inhibitor's action occurs near the metal surface. However, in a closed system, eliminating oxygen can simplify corrosion control through pH

adjustment to a relatively high level. In such cases, chromates, amines, and nitrites are particularly effective.

➤ **Electrochemical mechanism of action**

This classification of inhibitors takes into account the electrochemical nature of corrosion in the liquid phase, which involves at least two reactions:

-An anodic metal dissolution reaction (oxidation reaction):

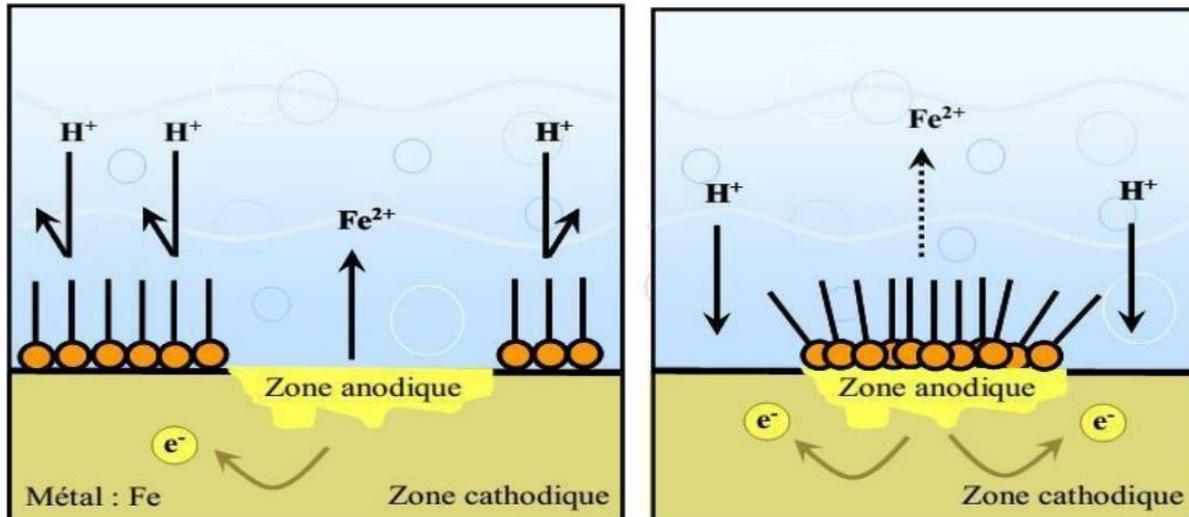


-A cathodic reaction of reduction of an oxidant in the solution:



If the inhibitor decreases the rate of the oxidation reaction by blocking the anodic sites (site of metal oxidation), it is called an anodic inhibitor.

If, on the other hand, it slows down the reduction reaction by blocking the cathodic sites (site of the reduction of dissolved oxygen in a ventilated environment or site of the reduction of the proton  $H^{+}$  in an acidic medium), it is called a cathode inhibitor (**figure I.7**). Mixed inhibitors work to both decrease the rate of the anodic reaction and that of the cathodic reaction



(**figure I.7**): : Formation of cathodic (A) and anodic (B) barrier layers, in an acidic medium.

➤ **Interfacial mechanisms of action**

This other method of classifying inhibitors classifies them by taking into account their mode of attachment to the metal substrate. Thus we distinguish:

- Adsorption or "interface" inhibitors, which appear in an acidic environment (mono or two-dimensional film).
- So-called "interphase" inhibitors, which appear in an alkaline medium (three-dimensional films)

### I.B.3.3 Classification according to the field of application Identify

- Inhibitors in an acidic environment. They are used to prevent electrochemical attack on steel during pickling.
- Inhibitors in neutral environments, which are mainly used to protect cooling circuits.
- Inhibitors in organic media (in engine lubricants and in gasoline).
- Gas phase inhibitors which are generally used for temporary protection of various packaged objects during transport (example: amines)

### I.B.4 Mechanism of inhibitor[18]

Corrosion has a multifaceted mechanism that depends on the metal's chemical make-up, the surrounding environment, and the presence or absence of inhibitors. Corrosion inhibitors are chemicals that work by blocking or slowing the electrochemical reactions taking place on a metal's surface, therefore preventing or delaying corrosion. Inhibitors often take the form of chemicals that coat the metal's surface and keep it from coming into touch with the corrosive environment. Substances that alter the pH of the surrounding environment, making it less corrosive, and those that can prevent the flow of electrons in the electrochemical reaction are two further categories of inhibitors. The concentration of the inhibitor, the type of metal being inhibited, and the conditions to which the metal is exposed are all crucial considerations in determining the inhibitor's efficacy

To be considered sustainable, corrosion inhibitors need to be able to keep up with both current and future demands without compromising on any of the aspects that make them effective. Physisorption or chemisorption processes are hypothesized to underlie the inhibitory effect. As shown in **(Figure I.8)**, physisorption occurs when inhibitor molecules have a weak polar connection with the charged metal surface. In contrast, chemisorption occurs when the molecules are held onto the metal surface by strong electrostatic forces. In the corrosive media, the cathodic reactions are given by Equations (1) and (2). And the reduction reaction of hydrogen gas is given by Equation (3).



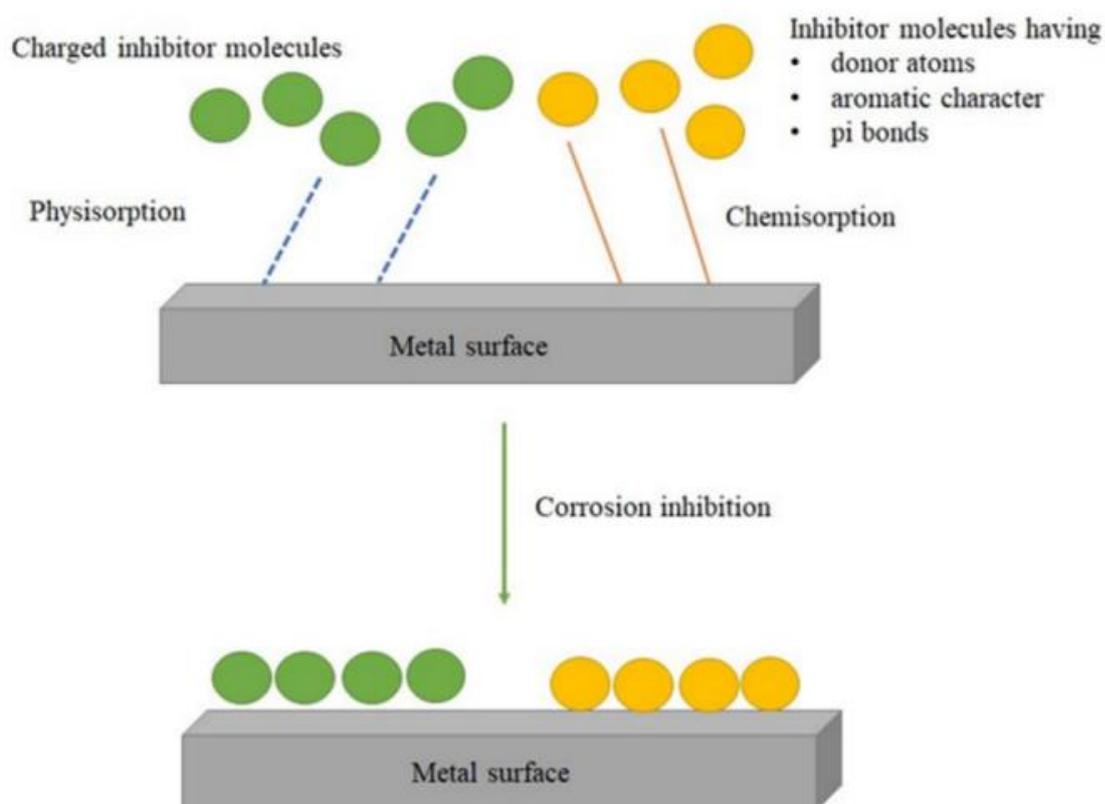


Adsorbed hydrogen ions on the metal surface catalyse reactions involving multiple hydrogen ions. The cathode surface then releases the hydrogen gas that has been holding it in place. Adsorption on an exposed metal surface

causes inhibitor molecules to act as neutral molecules rather than hydrogen ions, as shown in Equation (4).



According to Equation (2), by displacement of water molecules on the metal surface as site blocking elements, green inhibitors have adsorption properties.



**(Figure I.8) :** The mechanism for corrosion inhibition

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# Chapter II:

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*Chemical and physical properties of  
inhibitors*

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## Part One :

### II.A.1 Introduction

Extremely large quantities of water are used daily by the oil industry. The production of one ton of piltole uses several tons of water. Due to its use as an injection fluid for maintaining pressure, water is increasingly used as a cooling fluid This wide use of water is justified by its reliable cost, its availability in sufficient volumes. But this use is systematically compromised by two major drawbacks: corrosion and deposits. - Corrosion degrades the material and reduces its lifespan - The deposit following the designations, it has the disadvantages: \*

- \* Constitution of a film or an insulating crust which slows down thermal exchange
- \* Reduction of the passage section, therefore reduction in water flow (increase in its temperature) or increase in pressure losses.
- \* Possibility of corrosion under the scale
- \* Clogging of the rock constitutes the oil reservoir and restrictions on the flow of fluids within this environment.

Previous studies show that the water used is responsible for scourge, This water is therefore treated by adding organic complexes. In this study we propose to synthesize inhibitors capable of preventing the deposition of these salts at industrial installations and at oil wells

The definition of a corrosion inhibitor is not unique. The one chosen by the American association "National Association of Engainées Corrosion (NACE)" is the following: "an inhibitor is a substance that delays corrosion when added to a low concentration environment [19] .

### II.A.2 Anhydrous sodium sulphate

Anhydrous sodium sulfate is a chemical compound with the formula  $\text{Na}_2\text{SO}_4$ . It's a white, crystalline solid commonly used as a drying agent in organic synthesis. When it comes to inhibition, anhydrous sodium sulfate doesn't typically function as an inhibitor itself. Instead, it's often used in conjunction with inhibitors or stabilizers to prevent unwanted chemical reactions or degradation processes, particularly in aqueous solutions or during sample preparation in analytical chemistry. In this context, anhydrous sodium sulfate helps to absorb water, keeping the system dry and stable, which indirectly inhibits certain reactions or unwanted changes.

An inhibitor in the context of anti-corrosion refers to a substance that is added to a system to slow down or prevent corrosion reactions. Anhydrous sodium sulfate is a chemical compound that can act as a corrosion inhibitor in certain applications. It works by forming a protective layer on the surface of metals,

hindering the access of corrosive agents like water and oxygen to the metal surface, thus reducing the rate of corrosion [20] .

## II.A.3 The physical and chemical properties of sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>)

### II.A.3.1 Physical Properties

- **Appearance:** Sodium sulfate exists as white crystalline solid.
- **Density:** The density of sodium sulfate varies depending on its form, but typically ranges from 2.664 to 2.7 (g/cm<sup>3</sup>).
- **Melting Point:** Sodium sulfate has a melting point of 884°C (1,623°F).
- **Solubility:** It is highly soluble in water, with a solubility of approximately 42.7 g/100 mL at 20°C.

### II.A.3.2 Chemical Properties

- **Structural formula:** Na<sub>2</sub>SO<sub>4</sub>
- **Molecular Weight :**The molecular weight of sodium sulphate is 142.04 (g/mol).
- **pH:** Aqueous solutions of sodium sulfate are neutral, with a pH close to 7.
- **Hygroscopicity:** Sodium sulfate is hygroscopic, meaning it can absorb water vapor from the air.
- **Stability:** It is stable under normal conditions, but may decompose at high temperatures.
- **Reactivity:** Sodium sulfate is not highly reactive with most substances under standard conditions. However, it can react with strong acids to form sodium bisulfate and with alkalis to form sodium hydroxide and sodium carbonate.

## II.A.4 Diethyl sulfate

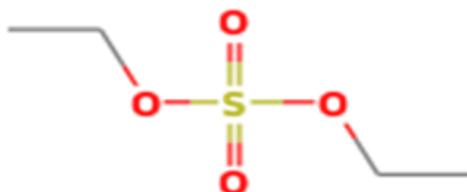
Inhibitor of corrosion with diethyl sulfate" is a chemical compound used as a corrosion inhibitor, typically part of anti-corrosion formulations used in industries dealing with materials prone to corrosion such as steel and other

metals. The inhibitor works to reduce or prevent corrosion by reacting with the metal surface, limiting its interaction with chemical or environmental agents that cause corrosion.

Diethyl sulfate corrosion inhibitor is usually added to anti-corrosion chemicals to enhance their performance in protecting metal surfaces from corrosion. The inhibitor reacts with the metal surface to form a thin layer that acts as a protective barrier, shielding the surface from corrosion resulting from external factors such as moisture and active chemicals .

#### II.A.4.1 The physical and chemical properties of Diethyl sulfate

Diethyl sulfate, often abbreviated as DES, is a chemical compound with the molecular formula  $(C_2H_5)_2SO_4$ . Here are its physical and chemical properties[21] :



(Figure II.A.1 the diethyl sulphate formula)

#### II.A.4.2 Physical Properties

- **Appearance:** Diethyl sulphate is a clear, colourless to pale yellow liquid.
- **Other names::** Sulphuric acid, diethyl ester; DES; Ethyl sulphate; Sulphuric acid diethyl ester; Diethyl sulphirate; Diaethy sulphate
- **Odor:** It has a pungent, unpleasant odor, often described as similar to mustard gas or sulfur dioxide.
- **Density:** The density of diethyl sulfate is about  $(1.13 \text{ g/cm}^3)$
- **Melting Point:** It has a relatively high melting point of around  $-32^\circ\text{C}$  ( $-26^\circ\text{F}$ ).
- **Boiling Point:** Diethyl sulfate has a boiling point of approximately  $208^\circ\text{C}$  ( $406^\circ\text{F}$ ).
- **Solubility:** It is soluble in water, alcohol, and ether.

#### III.A.4.3 Chemical Properties:

- **Reactivity:** Diethyl sulfate is a highly reactive compound. It is an alkylating agent, meaning it can transfer its ethyl groups to other molecules.

- **Acidity/Basicity:** It is weakly acidic due to the presence of the sulfuric acid functional group (SO<sub>4</sub>), but it is not considered a strong acid.
- **Stability:** Diethyl sulfate is relatively stable under normal conditions, but it can decompose when exposed to heat, light, or moisture, producing toxic and corrosive fumes.
- **Flammability:** It is flammable and can form explosive mixtures with air.
- **Toxicity:** Diethyl sulfate is highly toxic and can cause severe burns upon contact with skin or eyes. Inhalation or ingestion of the vapor or liquid can lead to serious health effects, including respiratory tract irritation, nausea, vomiting, and damage to internal organs.
- **Alkylating Properties:** Its ability to alkylate nucleophiles, such as DNA and proteins, makes it extremely hazardous and potentially carcinogenic.

Due to its toxicity and reactivity, diethyl sulfate is handled with extreme caution in laboratory settings and industrial processes. Proper safety measures, including personal protective equipment and engineering controls, should be implemented when working with this compound.

**The prior research on these producers is compiled in this table:**

### Diethyl sulfate :

	References	Method	Yield
Intratec Solutions LLC	(Allison L Hurley , Mark E Welker,,2000)	Sulfur Trioxide Addition to Diethyl Ether	80-90%
Primary Information Services	(Remi Ayu Pratika and all)	Sulfuric Acid Addition to Diethyl Ether or Ethanol	85-90%
Intratec Solutions LLC	(Erkan Ertürk , Tolga A. Yeşil ,,2022)	Reaction of Ethanol with Sulfuryl Chloride	75-85%
Intratec Solutions	(Pouya Ghamari kargar and all ,,2023)	Reaction of Absolute Alcohol with Chlorosulfonic Acid	70-85%

(Table II.A.1 some results of previous studies of diethyl sulphate) [22]

Anhydrous sodium sulfat :

	References	Method	Yiled
<b>Materials</b>	( Lei Wan and al ,,2021)	Sodium chloride (NaCl) and sulfuric acid (H <sub>2</sub> SO <sub>4</sub> )	95-98%
ELSEVIER	( Wenwei Zhang and al ,,2023)	Evaporation and Crystallization	90%
OL Organic Letters	( Huaran Zhang and al)	Mining, purification, and conversion	Depends on mineral purity

(Table II.A.2 some results of previous studies of anhydrous sodium sulphate)

[23]

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## Part two :

### II.B .1 Introduction

In the previous chapter, we conducted a theoretical study on the problems of erosion in industrial establishments, especially in oil wells.

In the course of this research, we've created two products that are used in a measured proportion and concentrations to reduce and reduce this natural phenomenon.

### II.B.2.The materials and tools employed

Material	Objects
Beaker	Sodium hydroxide
Erlenmeyer Flask ,Separatory	Sulphuric acid
Funnel	sodium bicarbonate
Distillation Flask ,Condenser	rock salt
Spatula ,Water Bath	distilled water
Hot Plate , Drying Oven	ethanol
	Anhydrous sodium sulphate

(Table II.B.1 Table of materials and tools)

### II.B.3 Part one Preparation of sodium sulphate

#### II.B.3.1 Mode of operation

##### ➤ From NaOH

The synthesis of sodium sulphate from sodium hydroxide is usually carried out in several stages. Here is a detailed summary of this process in the laboratory:

1. To make a sodium hydroxide solution: we dissolved a precise amount of sodium hydroxide in distilled water for a concentrated solution
2. Sulphuric acid can be used to react with sodium hydroxide solution by slowly adding concentrated sulphuric acid. The reaction will result in the production of sodium sulphate and water.



3. Filter the mixture after the reaction is finished to separate the solid sodium sulfate from the other products of the reaction.
4. To get an immaculate product, you can recrystallize the sodium sulphate by dissolving it in hot water and after that letting it crystallize.
5. Four a moufle (250-300 °C) was used for sodium sulfate drying process eliminating any moisture effects.

The production of sodium sulphate from sodium hydroxide was possible through these steps

➤ **From NaHCO<sub>3</sub>**

1. To make a sodium bicarbonate solution: we dissolved a precise amount of sodium bicarbonate in distilled water for a concentrated solution
2. Sulphuric acid can be used to react with sodium bicarbonate solution by slowly adding concentrated sulphuric acid. The reaction will result in the production of sodium sulphate, carbon dioxide and water.

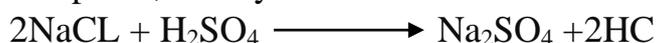


3. Filter the mixture after the reaction is finished to separate the solid sodium sulphate from the other products of the reaction.
4. To get an immaculate product, you can recrystallize the sodium sulphate by dissolving it in hot water and after that letting it crystallize.

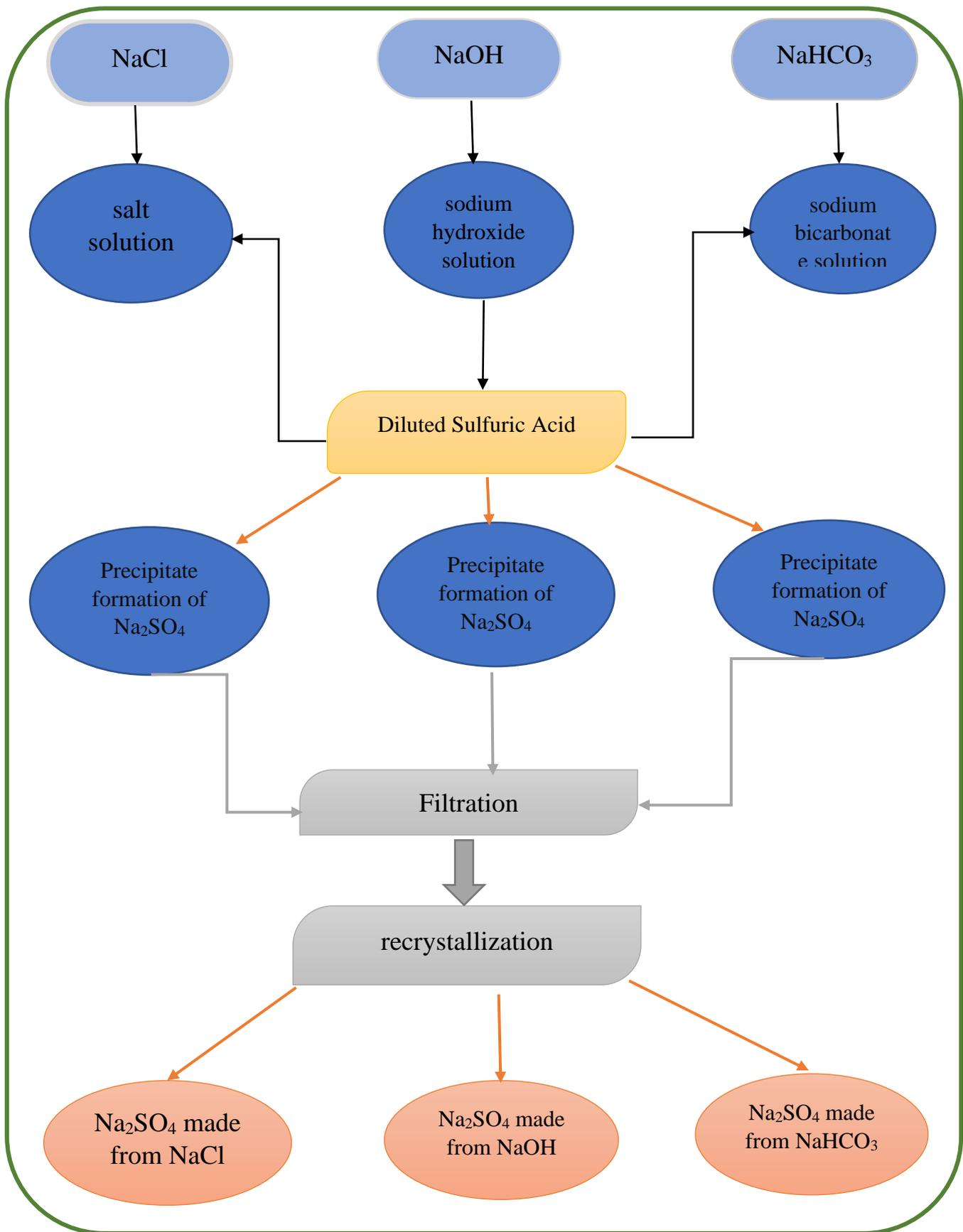
➤ **From NaCl**

Considering the presence of rock salt in nature, we can create sodium sulfate by following the following steps:

1. To make a salt solution: we dissolved a precise amount of bicarbonate in salt water for a concentrated solution.
2. Sulphuric acid can be used to react with salt solution by slowly adding concentrated sulphuric acid. The reaction will result in the production of sodium sulphate, and hydrochloric acid.



3. Filter the mixture after the reaction is finished to separate the solid sodium sulphate from the other products of the reaction.
4. To get an immaculate product, you can recrystallize the sodium sulphate by dissolving it in hot water and after that letting it crystallize.
5. The crystallization of sodium sulphate necessitates filtering and drying, which ultimately leads to the extraction of sodium sulphate from natural rock salt.

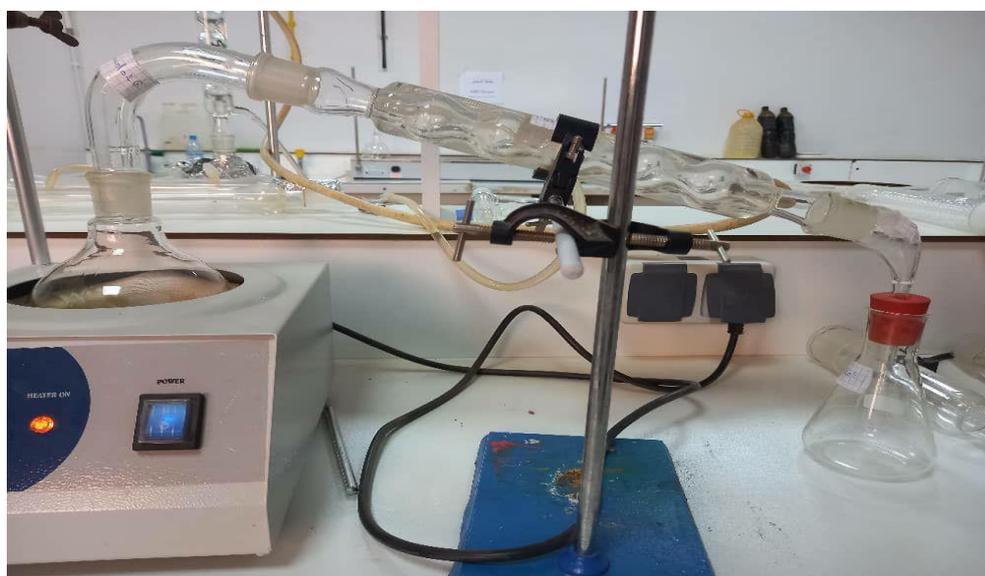
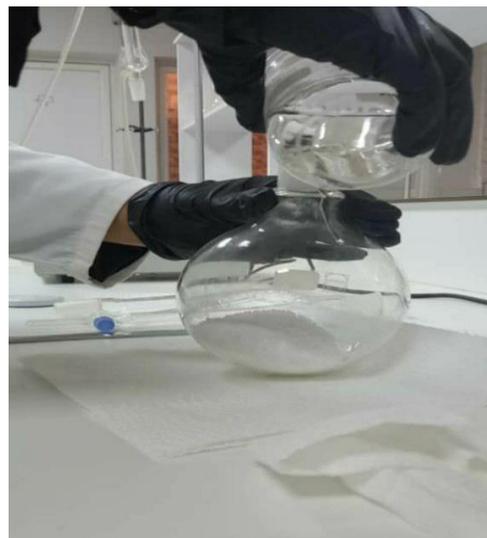


(Figure II.B.2)

## II.B.4 Part two Preparation of diethyl sulphate

### ➤ Method one by Fractional distillation

1. We first made a solution of ethanol and sulphuric acid by cooling it, because this reaction is a heat diffuser.
2. Sodium sulphate is added to the distillery by first weighing it, then adding ethanol solution and sulphuric acid and repairing the device at 80C
3. Diethyl sulphate production begins after 4 hours.



(Figure II.B.2 Steps to prepare diethyl sulphate)

# Chapter III

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*Results and discussion*

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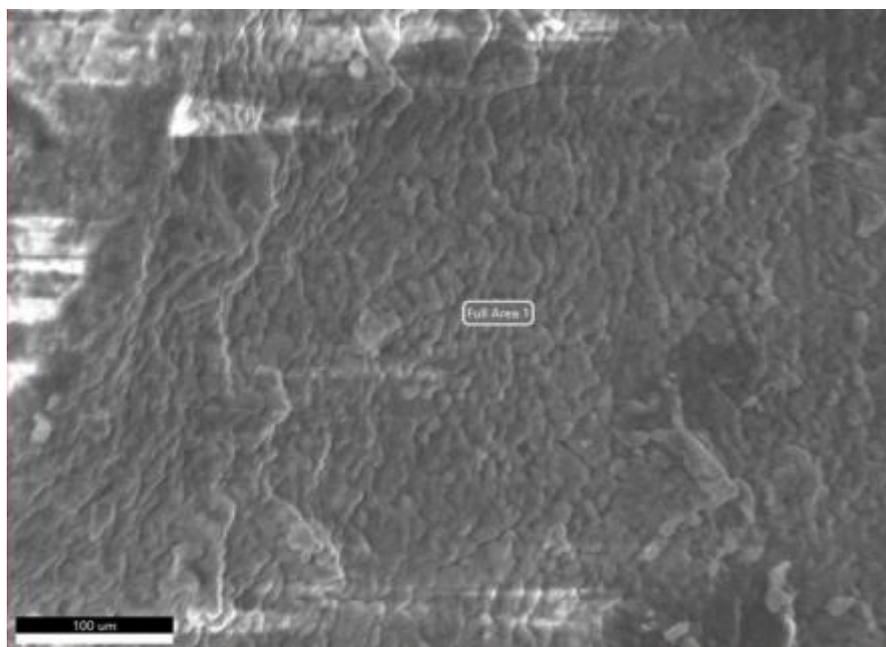
### III. Part one results of Na<sub>2</sub>SO<sub>4</sub>

(Table III.1 Comparative evaluation of different production methods)

	NaOH	NaCl	NaHCO <sub>3</sub>
<b>Yield</b>	68.09%	-	3.2%
<b>Time</b>	24h	-	48h
<b>Cost</b>	Lower cost	More expensive	Lower cost
<b>Wated material</b>	Less	More	Less
<b>Industrially</b>	Suitable	Unsuitable	Suitable
<b>Laboratory</b>	Suitable	Unsuitable	Suitable
<b>Risks</b>	Heat hazards and fumes	Heat hazards and fumes	No risks

#### III.1 EDX/SEM

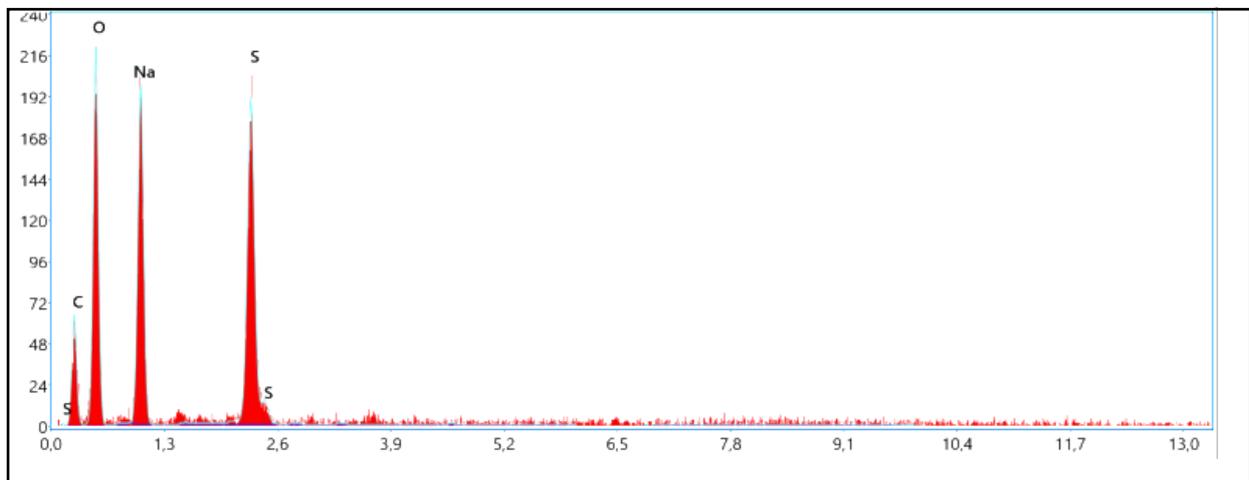
##### III.1.1 SEM/EDX analysis



(Figure III.1 images taken using EDX/SEM for NaOH's Na<sub>2</sub>SO<sub>4</sub> sample)

#### ➤ Interpretation

From this image, we note that its morphological shape is small, combined granules are close to each other, and its size is 100mm



(Figure III.2 EDX for Na<sub>2</sub>SO<sub>4</sub> record NaOH)

Element	% de masse	% Atomique
C K	<b>25.69</b>	<b>36.21</b>
O K	<b>37.63</b>	<b>39.81</b>
Na K	<b>22.17</b>	<b>16.33</b>
S K	<b>14.50</b>	<b>7.65</b>

(Table III.2 EDX's Quantitative Results)

### ➤ Interpretation

EDX analysis is used to identify elements such as carbon (C), oxygen (O), sodium (Na), and sulphur (S).

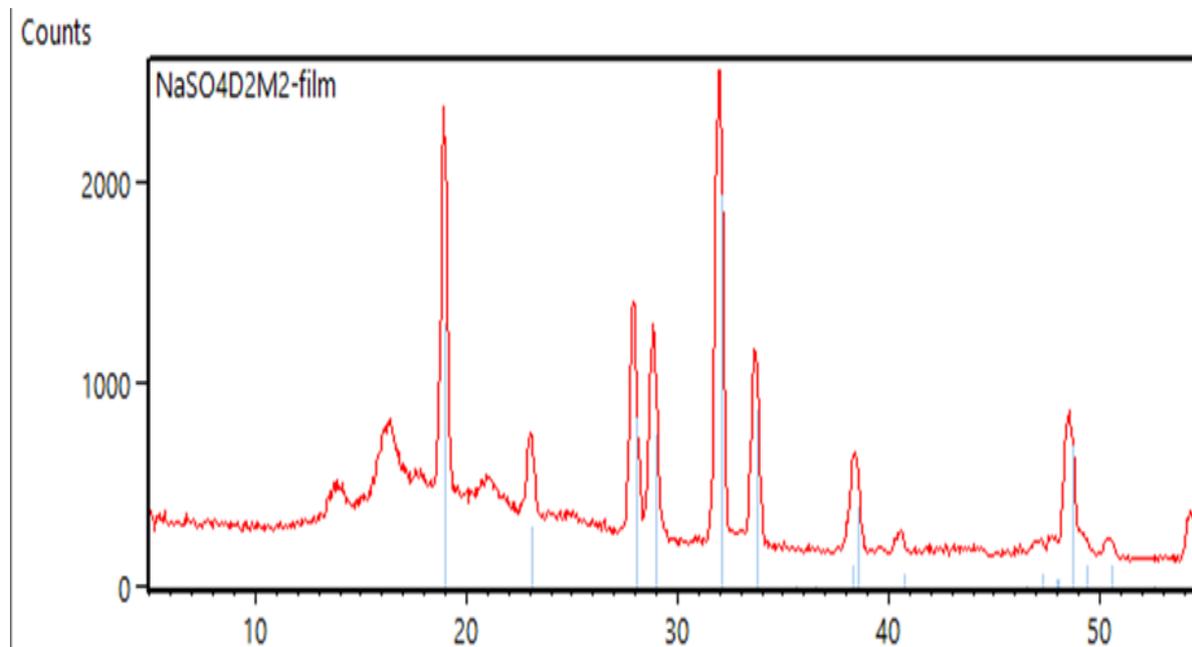
The high concentration of oxygen is caused by the production of oxygen particles in the analyser material.

And the existence of sodium and carbon in regular quantities (22.17% and 25.69%, respectively), such that carbon returns to the cell content in which it was analysed and sodium to the analyser material.

On the other hand, support the presence of sulphur by 14.50% owing to the analyser material.

### III.1.2 DRX

The DRX results for the samples were processed using HighScore Plus software. After inspection and verification, we obtained the following results:



(Figure III.3 XRD spectrum of Na<sub>2</sub>SO<sub>4</sub>)

The spectrum of this sample, especially from card number 01-074-2036, matches the CSD database table after analysis.

**Empirical formula:** Na<sub>2</sub>O<sub>4</sub>S

**Chemical formula:** Na<sub>2</sub>SO<sub>4</sub>

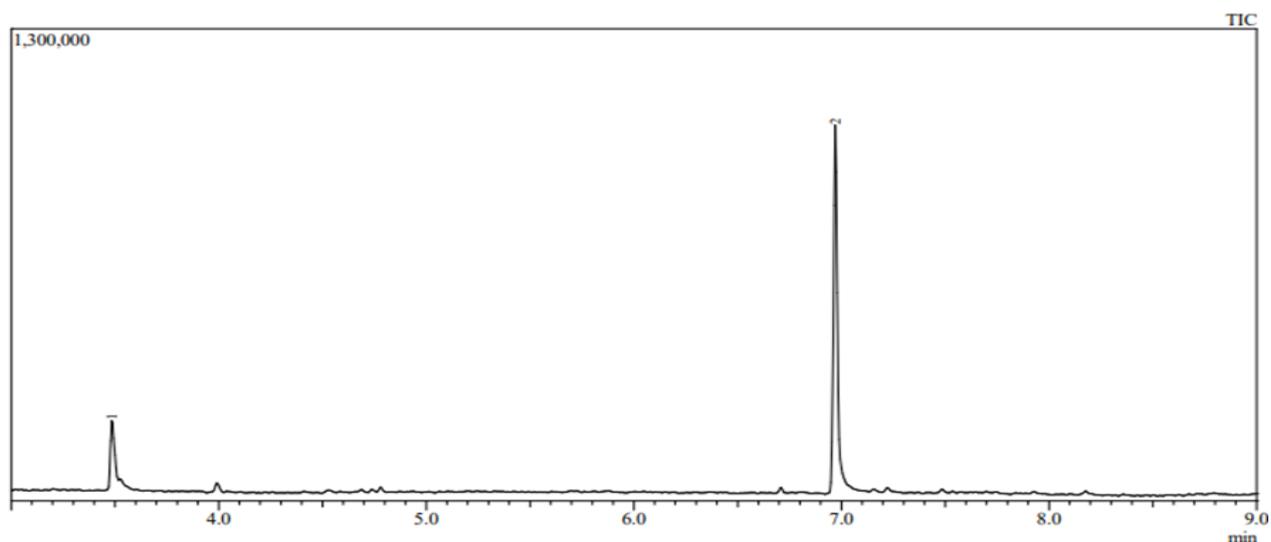
### III.Part two results of diethyl sulphate

The conditions acquired during the manufacture of diethyl sulphate are summarised in the table.

	<b>Distillation</b>
<b>Yield</b>	25.10%
<b>Time</b>	4h
<b>Cost</b>	Lower cost
<b>Wated material</b>	Less
<b>Industrially</b>	Suitable
<b>Laboratory</b>	Suitable
<b>Risks</b>	No risks

(Table III.3 Results of preparation method)

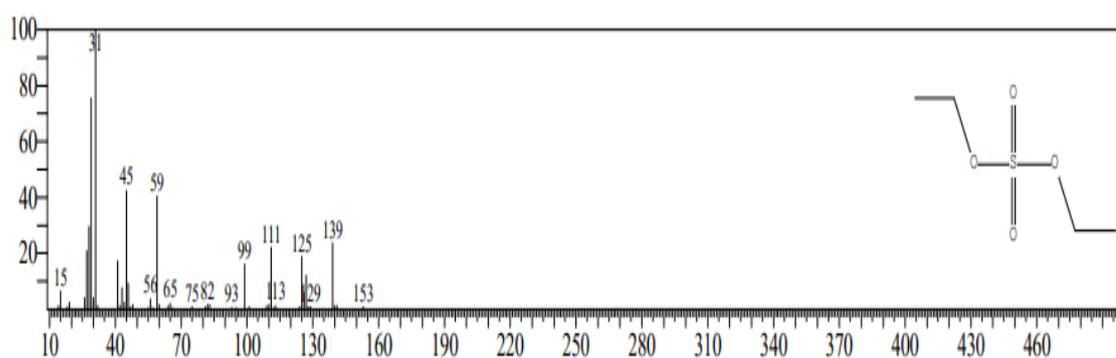
### III.1 GC-MS/MS



(Figure III.4 GC chromatogram of diethyl sulphate)

Peak	R.Time	Area%	Height%	Name
1	3.487	16.96	15.89	1,1-Diethoxyethane
2	6.971	83.04	84.11	Diethyl sulphate

(Table III.4 GC chromatogram Results)



(Figure III.5 spectrum of prepared diethyl sulphate)



### III.2 Physicochemical characteristic

Characteristic	Value
Refractive index	1.395.
Viscosity dyn(25°C)	2.3 mPa·s
Viscosity cin(25°C)	1.81 mm <sup>2</sup> /s
Density(25°C)	1.177 g/mL

(Table III.6 Physicochemical characteristics Results)

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## *General Conclusion*

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## *General Conclusion*

This study represents an important step in the field of manufacturing and characterizing corrosion inhibitors used in the petroleum industry, focusing on developing effective materials to mitigate the adverse effects of corrosion on equipment and infrastructure in this vital sector. Through research and laboratory experiments, positive results were obtained demonstrating the effectiveness of some new chemical compounds in reducing corrosion rates.

These findings not only contribute to improving performance and operational efficiency in the petroleum industry but also help reduce costs related to maintenance and repairs, positively impacting the overall economy. Furthermore, the optimal use of these inhibitors aids in environmental conservation by reducing the environmental risks associated with chemical and petroleum product leaks.

We hope this study has provided a comprehensive overview of the importance of corrosion inhibitors, their manufacturing processes, and characterization methods, and that it serves as an impetus for further research in this field to foster innovation and sustainable development.

