

**A THESIS SUBMITTED TO
THE GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES
OF ÇANKIRI KARATEKİN UNIVERSITY**

**Synthesis of some imidazoline derivatives and study of their anti-corrosion
efficiency**

**IN PARTIAL FULFILLMENT OF THE REQUIREMENTS
FOR
THE DEGREE OF MASTER OF SCIENCE
IN
CHEMICAL ENGINEERING**

BY

Mustafa Salih Mahdi

ÇANKIRI

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**Synthesis of some imidazoline derivatives and study of their anti-corrosion
efficiency**

By Mustafa Salih Mahdi

Martch 2021

We certify that we have read this thesis and that in our opinion it is fully adequate, in scope and quality, as a thesis for the degree of Master of Science

Advisor : Assoc. Prof. Dr. Volkan Eyupoglu

Co-Advisor : Asst. Prof. Dr. Usama Ali Muhsen

Examining Committee Members:

Chairman: Prof. Dr

Chemical Engineering

Çankırı Karatekin University

Member: Assoc. Prof. Dr

Chemical Engineering

Yıldız Technical University

Member: Ast. Prof.Dr.

Chemical Engineering

Çankırı Karatekin University

Approved for the Graduate School of Natural and Applied Sciences

Prof. Dr. İbrahim ÇİFTÇİ

Director of Graduate School

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Mustafa Salih

PREFACE AND ACKNOWLEDGEMENTS

[PREFACE]

It is with great pleasure and a sense of accomplishment that I present this thesis, entitled "[Synthesis of some imidazoline derivatives and study of their anti-corrosion efficiency]," as a culmination of years of dedicated research and academic pursuit. This work represents an in-depth exploration of a subject that has captivated my intellectual curiosity and passion throughout my academic journey.

Undertaking this thesis has been both challenging and rewarding. It has allowed me to delve deep into the realm of [Organic Chemistry], uncovering new insights, and contributing to the existing body of knowledge. Throughout this process, I have had the opportunity to work with esteemed mentors, engage in stimulating discussions with peers, and harness various resources that have enriched my learning experience.

I extend my heartfelt gratitude to all those who have supported and encouraged me along this arduous yet gratifying path. This thesis would not have been possible without their unwavering support.

[ACKNOWLEDGEMENTS]

First and foremost, I dedicate this thesis to the reason for my existence in this life, to those who made a great effort to reach what I am, to the source of my strength, to my father **Prof. Dr Salih Mahdi** and my mother **teacher Khawla Muhammad Ali**

I express my deepest gratitude to my thesis advisor, **Prof. Dr Volkan Eyupoglu**, whose guidance, expertise, and patience have been invaluable throughout this journey. Their encouragement and insightful feedback have shaped the direction of this research and inspired me to strive for excellence.

I would like to thank **Prof. Dr Osama Ali Mohseni** for his smooth support, from scientific matters to resources. His efforts have facilitated an environment conducive to research and learning.

I would like to express my deep appreciation to my family, my brothers and sisters. Their unwavering love, understanding, and encouragement have been my anchor during moments of doubt and exhaustion. Their belief in my abilities gave me the strength to persevere and achieve my academic goals.

I would like to express my deep appreciation to my wife who stood by my side and supported me on this path and contributed directly and indirectly to this achievement.

I am grateful to my friends and fellow researchers for their camaraderie and the exchange of ideas that have challenged and refined my understanding of the subject matter. Their camaraderie has made this academic journey more enjoyable and rewarding.

Last but not least, I would like to thank my friends CPF-1 lab staff who helped me in the practical matters of research

In conclusion, this thesis is a testament to the collective efforts of many individuals who have played a significant role in shaping my academic and personal growth. Their contributions have been immeasurable, and I am truly grateful for their impact on my life.

Mustafa Salih Mahdi

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ABSTRACT

Synthesis of some imidazoline derivatives and study of their anti-corrosion efficiency

Mustafa Salih Mahdi

Master of Science in Chemical Engineering

Advisor: Assoc. Prof. Dr. Volkan Eyupoglu

Co-Advisor: Asst. Prof. Dr. Usama Ali Muhsen

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Imidazoline is a class of heterocycles formally derived from imidazoles by the reduction of one of the two double bonds. Three isomers are known, 2-imidazolines, 3-imidazolines, and 4-imidazolines. The 2-imidazolines and 3-imidazolines contain an imine centre, whereas the 4-imidazolines contain an alkene group. The 2-Imidazoline group occurs in several drugs and it is one of the heterogeneous organic compounds used in many pharmaceutical, petroleum and other industries. This thesis focuses on the Synthesis of imidazolinone derivatives using environmentally friendly methods in reducing corrosion in crude oil pipes or other equipment, which causes gas leaks and oil leaks. Imidazoline compound is the most important injected into crude oil in insulation plants to reduce corrosion. Many companies produce imidazoline. However, most of the preparation methods used are harmful to the environment and hazardous to humans, particularly those involved in the synthesis of imidazoline.

Iraq crude oil is one of the acidic substances that cause the corrosion of pipes and other equipment. In such a corrosion, we inject imidazoline into the crude oil. Corrosion is one of

the recurrent problems during crude Oil extraction and one of the persistent problems that has not been finally disposed of. This Thesis will be in four parts in general.

First, the synthesis of imidazole derivatives from fatty acids in an environmentally friendly way (green chemistry).

Secondly, to ensure the purity of the prepared imidazole derivatives and examine them with FT-IR devices and the NMR to ensure the validity of the produced compounds.

Third, studying the effect of imidazole derivatives prepared as corrosion inhibitors by conducting corrosion experiments used in ASTM.

Fourth, discussing the obtained results and explaining how their effect was as corrosion inhibitors.

Through these, the best derivative that works as a corrosion inhibitor was reached according to the obtained results

Keywords: Imidazole, 2-imidazolines, 3-imidazolines, 4-imidazolines, Imidazole Derivatives, Imidazoline, Imidazoline Derivatives, Imidazole reaction, Corrosion, Corrosion Inhibitor, Crude oil, Synthesized, Heterocyclic, 1,3-diazole, fatty acids, Green Chemistry, Imidazole Synthesis, Hantzsch synthesis, Radziszewski synthesis, Ritter reaction, Wallach Synthesis, Dehydrogenation of Imidazoline, Imidazole Alkylation, Imidazole Acylation, Imidazole Ring-opening, Imidazole halogenation,

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chapter1: INTRODUCTION

Any cyclic organic compound containing N, O, or S as a substitute for one or more of a single cyclic carbon atom is called a heterocyclic compound. The rings are either non-aromatic rings or simple aromatic rings. Some examples are - imidazole ($C_3H_4N_2$), pyridine (C_5H_5N), dioxane ($C_4H_8O_2$) and pyrimidine ($C_4H_4N_2$) as shown below: [1]

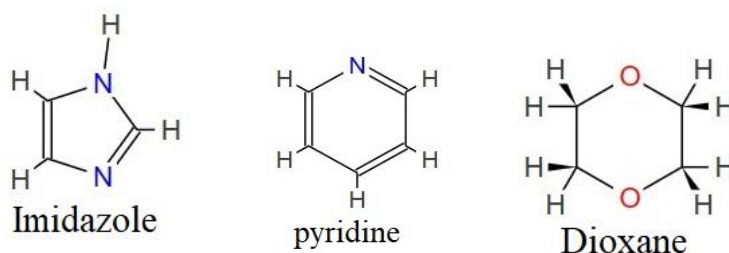


Figure (1) the chemical structure of the heterocyclic compound [2]

Imidazole is a five-membered aromatic heterocycle containing two nitrogen atoms. It exhibits resonance due to the delocalization of electrons within the ring. [1]

In imidazole, the lone pair of electrons on each nitrogen atom can delocalize into the π system of the ring, leading to resonance stabilization. This electron delocalization occurs through the formation of a π bond between the nitrogen atoms and the adjacent carbon atoms in the ring. As a result, the electrons are shared between multiple atoms, creating resonance structures. [2]

The resonance structures of imidazole show that the electrons are not localized on specific atoms but are spread out over the entire ring system. This delocalization of electrons enhances the stability of the molecule. It also influences the reactivity of imidazole, making it more nucleophilic and capable of participating in various chemical reactions. [2]

The resonance in imidazole also affects its acidity. The presence of the lone pair of electrons on one of the nitrogen atoms makes imidazole a weak base. However, due to resonance, the electrons can be partially shared with the neighbouring atoms, reducing the availability of the lone pair and decreasing its basicity. [3]

Overall, resonance plays a significant role in determining the electronic structure, stability, and reactivity of imidazole. It contributes to its unique properties and makes it a versatile building block in various organic synthesis and biological processes as shown below: [3]

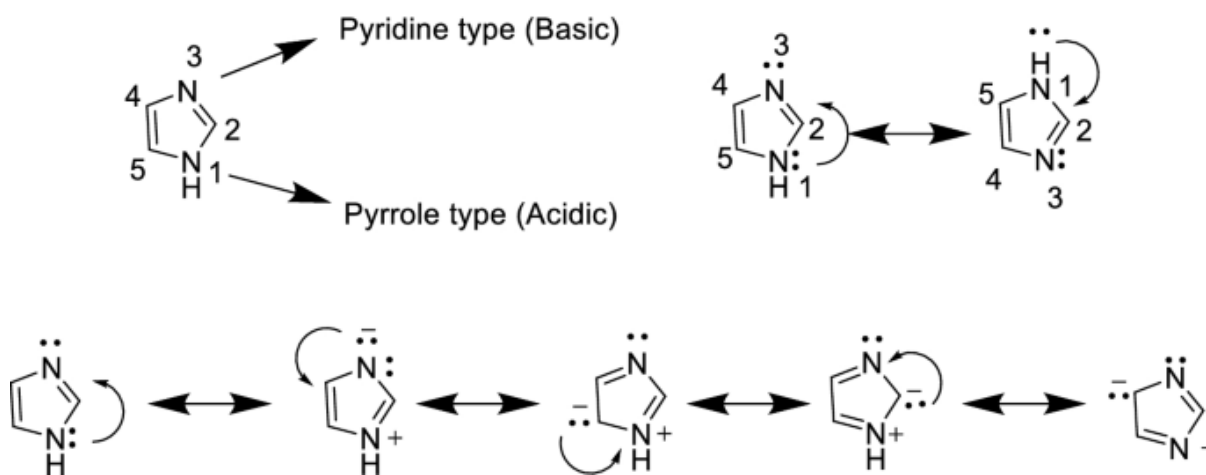


Figure 2 Resonance in Imidazoles [4]

Imidazole derivatives can be synthesized by various methods, including the condensation of aldehydes or ketones with primary amines or amino acids, the cycloaddition of nitriles with alkenes or alkynes, and the oxidative coupling of primary amines. The resulting compounds can be further modified by various functional groups to enhance their biological activities and pharmacokinetic properties. [4]

In the field of medicinal chemistry, imidazole derivatives have been extensively studied as potential drug candidates. For example, imidazole-containing compounds such as histamine H₂ receptor antagonists and proton pump inhibitors are commonly used for the treatment of gastroesophageal reflux disease, peptic ulcers, and other gastrointestinal disorders. Other imidazole-based drugs, such as benzimidazoles and triazoles, are used as antifungal and antiprotozoal agents. [5]

In addition to their medicinal properties, imidazole derivatives have also been studied for their applications in materials science. For example, imidazole-based metal-organic frameworks (MOFs) have been developed as potential candidates for gas storage and separation, catalysis, and drug delivery. These materials exhibit high surface area, tunable

pore size, and unique chemical properties that make them attractive for a variety of applications. [6]

Another important application of imidazole derivatives is as corrosion inhibitors in various industries, including oil and gas, water treatment, and metal processing. Imidazoline derivatives, which are imidazole derivatives containing a long alkyl chain, have been shown to be effective in inhibiting the corrosion of metal surfaces by forming a protective film on the metal surface. [7]

Imidazole and its derivatives are versatile scaffolds that have found numerous applications in various fields of chemistry, including medicinal chemistry, biochemistry, materials science, and corrosion science. The unique properties of these compounds, combined with their potential for modification and functionalization, make them attractive candidates for further study and development. [8]

1.1 Physical Properties

Imidazole is an aromatic heterocyclic compound with interesting physical properties that stem from its molecular structure. Here are some key physical properties of imidazole:

1. **Molecular Formula:** Imidazole has the molecular formula $C_3H_4N_2$, representing three carbon atoms, four hydrogen atoms, and two nitrogen atoms in its structure.
2. **Molecular Weight:** The molecular weight of imidazole is approximately 68.07 g/mol.
3. **State at Room Temperature:** Imidazole is typically a white to off-white crystalline solid at room temperature.
4. **Melting Point:** The melting point of imidazole ranges from approximately 87 to 88 degrees Celsius (189 to 190 degrees Fahrenheit).

5. Boiling Point: Imidazole has a relatively high boiling point, which is around 256 degrees Celsius (493 degrees Fahrenheit). This high boiling point is attributed to the aromatic nature of the compound and the presence of strong intermolecular forces.
6. Solubility: Imidazole is moderately soluble in water, ethanol, and methanol. Its solubility in water is enhanced by the presence of polar functional groups, such as hydroxyl or amino groups, which can form hydrogen bonds with water molecules.
7. Aromaticity: Imidazole exhibits aromaticity due to its conjugated π -electron system resulting from alternating single and double bonds within the ring. This aromatic character contributes to its stability and influences its reactivity in various chemical reactions.
8. Acid-Base Properties: Imidazole is a weak base because of the lone pair of electrons on one of its nitrogen atoms. It can form salts with strong acids, and the basicity can be further influenced by substituents on the imidazole ring.
9. UV Absorption: Imidazole absorbs ultraviolet (UV) light with a maximum absorption wavelength around 230 nm, making it useful in UV spectrophotometry and as a chromophore in some molecules.
10. Hygroscopicity: Imidazole has some hygroscopic properties, meaning it can absorb moisture from the air, particularly when in a humid environment.

These physical properties of imidazole contribute to its wide-ranging applications, including its use as a building block in pharmaceuticals, agrochemicals, and coordination complexes. The combination of its aromaticity, solubility, and basicity makes it a versatile and important compound in various chemical and biological processes.

1.2 Natural Products of Imidazole

Natural imidazole products are a fascinating and diverse group of compounds found in various living organisms, ranging from plants and animals to microorganisms. Imidazole is a five-membered aromatic heterocycle consisting of two nitrogen atoms at positions 1 and 3 and three carbon atoms. It is an essential building block for numerous bioactive molecules, contributing to their biological activities and therapeutic potential.

One of the most well-known natural imidazole products is histamine, a crucial molecule involved in the immune response and inflammation regulation. Histamine is synthesized and stored in specialized cells called mast cells and basophils. When these cells are triggered by allergens or pathogens, they release histamine, leading to allergy symptoms like sneezing, itching, and inflammation. Histamine also plays a role in regulating stomach acid secretion and neurotransmission in the central nervous system.

Another significant class of natural imidazole products is found in the realm of antibiotics and antifungal agents. Some microorganisms, particularly bacteria and fungi, produce imidazole-containing compounds as part of their defense mechanisms against other competing organisms. For example, miconazole and clotrimazole are synthetic imidazole-based antifungal drugs derived from natural sources. They target fungal cell membranes, disrupting their integrity and leading to their death.

Marine organisms are also a rich source of natural imidazole products. Sponges, in particular, have been found to contain various imidazole alkaloids. These compounds often exhibit potent biological activities, including antimicrobial, antiviral, and cytotoxic properties. Researchers are continually exploring these marine-derived compounds for potential drug development and medical applications.

Moreover, imidazole derivatives are involved in the regulation of enzyme activity. They serve as essential components in the active sites of various enzymes, facilitating biochemical reactions. These enzymes are involved in processes like DNA repair, cellular respiration, and hormone synthesis.

Furthermore, imidazole-containing peptides and proteins are present in many organisms, contributing to their biological functions. Some proteins possess imidazole moieties within their active sites, allowing them to bind and transport metal ions like copper and iron. This process is critical for various biological processes, including oxygen transport, electron transfer, and enzymatic catalysis.

Overall, natural imidazole products represent a diverse and intriguing group of compounds with significant biological and pharmacological importance. Their presence in various living organisms underscores their evolutionary significance and potential applications in medicine, agriculture, and biotechnology. Researchers continue to study these compounds to unlock their full potential and explore the possibilities they hold for addressing various health and environmental challenges.

1.3 Production Aspect of Imidazole

The production of imidazole involves several synthetic methods and processes, each tailored to produce the compound efficiently and in sufficient quantities for various applications. Imidazole can be synthesized from simple starting materials, and its production is essential for the manufacturing of pharmaceuticals, agrochemicals, and other specialty chemicals. Let's explore the production aspect of imidazole:

Dehydrogenation of Imidazolines: One of the common methods for industrial-scale production of imidazole involves the dehydrogenation of imidazolines. Imidazolines are cyclic compounds with an imidazole ring containing one additional hydrogen atom. By subjecting imidazolines to a dehydrogenation process, typically using a catalyst and elevated temperature, imidazole is formed as a product.

Dehydration of Glyoxal and Ammonia: Another approach to produce imidazole is through the reaction between glyoxal and ammonia. Glyoxal is a dialdehyde compound, and in the presence of ammonia, it undergoes a condensation reaction and subsequent dehydration to yield imidazole.

Ring-Closing Reactions: Imidazole can also be synthesized using ring-closing reactions from appropriate precursor compounds. For example, reacting 1,2-diketones with ammonia or primary amines can lead to the formation of imidazoles through a cyclization process

Hydrolysis of 1,2,3-Triazoles: Another interesting approach involves the hydrolysis of 1,2,3-triazoles to produce imidazoles. 1,2,3-Triazoles can be prepared through a copper-catalyzed azide-alkyne cycloaddition (CuAAC) reaction, commonly known as the "Click" reaction. Subsequent hydrolysis of the triazole ring produces imidazoles.

Bio-based Production: With the growing interest in sustainable and green chemistry practices, researchers have explored biocatalytic routes for imidazole production. Enzymes and microorganisms can be engineered to catalyze specific reactions leading to the synthesis of imidazole and its derivatives.

Use of Imidazole Derivatives: Imidazole derivatives are widely used as building blocks in various chemical syntheses. By introducing specific functional groups to imidazole, it is possible to create a diverse range of compounds with tailored properties and applications.

The choice of the production method depends on factors such as the scale of production, cost considerations, and the desired purity of the final product. Some methods may be more suitable for large-scale industrial production, while others are more commonly used for laboratory-scale synthesis or specific applications.

Imidazole and its derivatives find applications in various industries, including pharmaceuticals (antifungal drugs, antihistamines), agrochemicals (fungicides, herbicides), and as ligands in coordination chemistry. Continuous research and development in this area aim to improve existing synthetic methods, discover new ones, and explore sustainable approaches to imidazole production.

1.4 Chemical Composition of Imidazole

The chemical composition of imidazole is relatively simple, yet it gives rise to a unique and important heterocyclic compound. Imidazole has the molecular formula $C_3H_4N_2$ and consists of a five-membered ring containing two nitrogen atoms and three carbon atoms. Its chemical structure can be represented as follows:

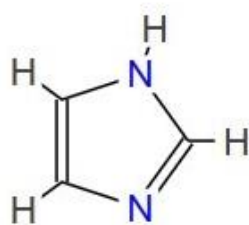


Figure 3 Imidazole chemical structure

Key points about the chemical composition of imidazole: **Ring Structure:** Imidazole is a heterocyclic compound, meaning it contains at least two different elements in its ring structure. The five-membered ring in imidazole is formed by the fusion of two carbon atoms and three nitrogen atoms. The nitrogen atoms are located at positions 1 and 3 of the ring.

Aromatic Nature: Imidazole is an aromatic compound due to its conjugated π -electron system. The two nitrogen atoms and three carbon atoms in the ring form an alternating pattern of single and double bonds, leading to electron delocalization and aromaticity. This characteristic contributes to the stability and reactivity of imidazole.

Basicity: The nitrogen atoms in the imidazole ring are slightly basic. They have lone pairs of electrons, making imidazole a weak base. This property is essential in various biological processes and chemical reactions involving imidazole-containing compounds.

Tautomeric Forms: Imidazole exists in two tautomeric forms, known as the 1H-imidazole and 3H-imidazole. In the 1H-tautomer, the hydrogen atom is attached to the nitrogen at position 1, while in the 3H-tautomer, it is attached to the nitrogen at position 3. The tautomeric equilibrium between these forms is influenced by factors such as solvent and pH.

Derivatives and Substituents: Imidazole serves as a core structural unit in various natural and synthetic compounds. By introducing different functional groups or substituents at positions 2, 4, or 5 of the imidazole ring, a wide range of imidazole derivatives can be created, each with unique properties and applications.

Occurrence in Nature: Imidazole is found naturally in various living organisms and plays critical roles in biological processes. For example, it is present in the amino acid histidine, which is an essential component of many proteins and enzymes. Imidazole-containing compounds also serve as important signaling molecules in the nervous and immune systems.

In summary, the chemical composition of imidazole is characterized by its five-membered ring structure containing two nitrogen atoms and three carbon atoms. This heterocyclic compound's aromatic nature, basicity, and ability to form derivatives make it a fundamental building block in various bioactive molecules and a subject of significant interest in organic chemistry and drug development.⁵

Chapter 2 Imidazole Synthesis

Although there had been discoveries of various derivatives of imidazole in 1840, it was first reported in 1858. The synthesis process of imidazole follows the reaction between formaldehyde in ammonia and glyoxal. This processes a low yield of imidazole but it is still used to form imidazole with C-substitution [19] There are several methods for preparing imidazole syntheses.

2.1 Debus imidazole synthesis: Imidazole was first synthesized by Heinrich Debus in 1858, but various imidazole derivatives had been discovered as early as the 1840s, as shown below: [20]

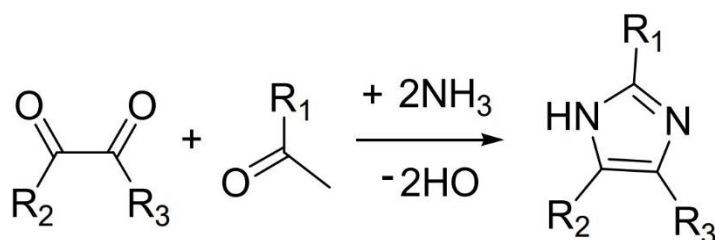


Figure 4 glyoxal and formaldehyde in ammonia to form imidazole (20).

2.2 Radziszewski imidazole synthesis: This method involves the condensation of a dicarbonyl compound, and α - keto aldehyde or α -diketones in the presence of ammonia, yield 2, 3, 5-tri(R) imidazole. [20]

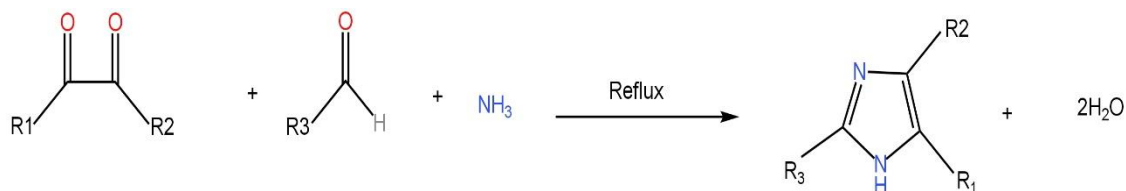


Figure 5 condensation of a dicarbonyl compound and α - keto aldehyde in the presence of ammonia, yield 2, 4, 5-tri(R) imidazole (20)

2.3 Dehydrogenation of Imidazoline: In the presence of sulfur, it was reported that, for converting to imidazole from imidazoline, a milder reagent management was used. Imidazolines are derived from 1, 2 ethane diamine and nitriles after reacting with BaMnO₄ yield 2-substituted imidazole. This reaction includes alpha halo ketones and imidine. For the synthesis of benzamidine and 2, 4- or 2, 5- biphenyl imidazole phenacyl bromide, this method has been successfully applied, based on the process that can be afforded by 2,4- diphenyl imidazole. To produce imidazole, acyloin or alpha halo ketones react with amidine. [20]

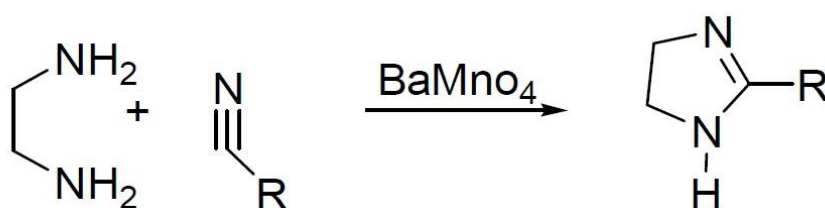


Figure 6 1, 2 ethanediamine and alkyl nitriles reaction with BaMnO₄ yield 2-substituted imidazoles (20)

2.4 Wallach Synthesis: A compound containing chlorine is derived when N, N' - dimethyl oxamide is treated with phosphorus pentachloride, which decreases with hydroiodic acid and provides N- methyl imidazole. N, N' -diethyl oxamide is converted to a chlorine compound under the same condition, which on decrease yields 1- ethyl –2- methyl imidazole. 5- Chloral imidazole is the chlorine compound. [20]

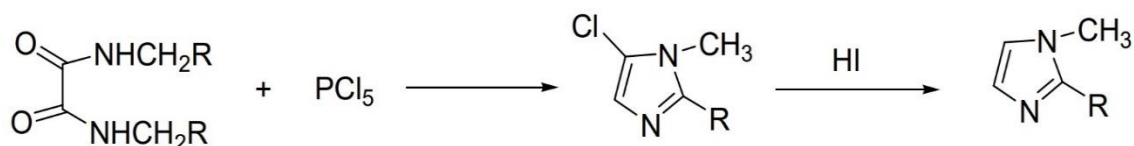


Figure 7 N, N- dimethyloxamide and phosphorus pentachloride yield N- methyl imidazole (20)

2.5 From α - Halo Ketone: This method is based on an interaction between α -halo ketones and imidine. This method has been applied successfully for the synthesis of 2, 4- or 2, 5- biphenyl imidazole. Similarly, acyloin reacts with amidine or α -halo ketones to yield imidazoles [20]

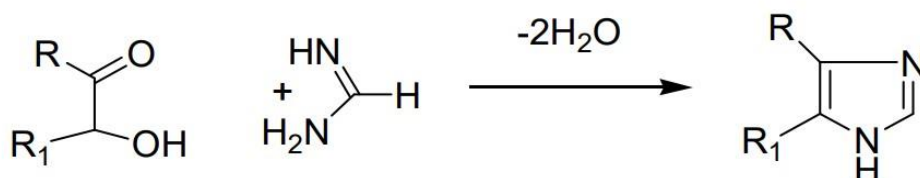


Figure 8 acyloin reacts with amidine to yield imidazoles

2.6 Markwald Synthesis: The preparation of 2- mercapto imidazoles from α -amino ketones or aldehyde and potassium thiocyanate are used for the synthesis of 2-thiol substituted imidazoles. The sulfur can readily be removed by a variety of oxidative methods to give the desired imidazoles [20]

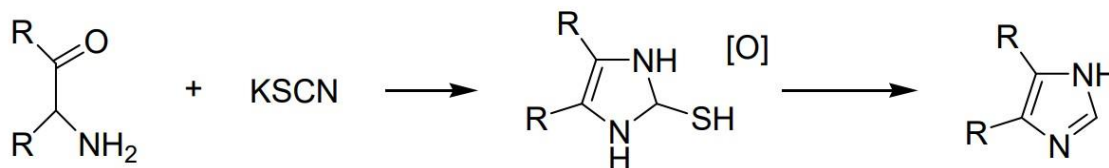


Figure 9 α -amino ketones and potassium thiocyanate yield 2-thiol substituted imidazoles The sulfur can readily be removed by a variety of oxidative method to give the desired imid

2.7 Cyclization of α -Acylaminoketones: α -acylaminoketones, also behave as 1, 4-diketo compounds. This compound lead to ready cyclization, in the presence of anhydride followed by presence of ammonium acetate [20]

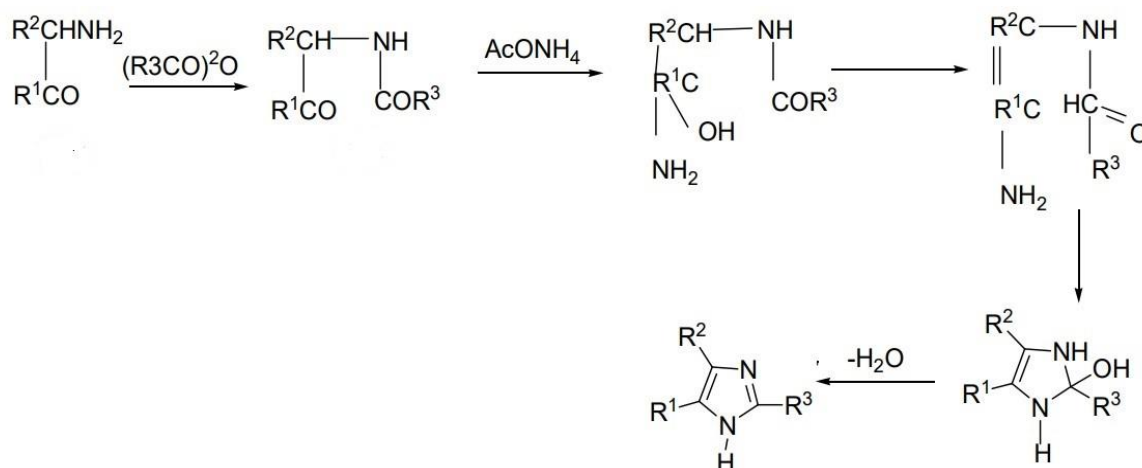


Figure 10 α -acylaminoketones, also behave as 1, 4-diketo compounds. This compound lead to ready cyclization [20]

2.8 From Aminonitrile and Aldehyde: Mixture of an aldehyde and aminonitrile both condensed under suitable reaction condition to give substituted imidazole as shown below [20]

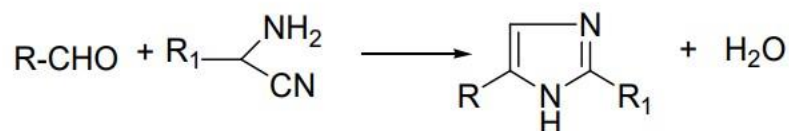


Figure 11 aldehyde and aminonitrile both condensed under suitable reaction condition to give substituted imidazole [20]

2.9 From formaldehyde and tartaric acid dinitrate: Imidazole can best be prepared itself by action of ammonia on a mixture of tartaric acid dinitrate and formaldehyde then heating the dicarboxylic acid with quinoline in the presence of copper to give 2-alkyl substituted 4,5- dicarboxylic acid imidazole further which is reacted with aniline to give 4- substituted benzamide. [20]

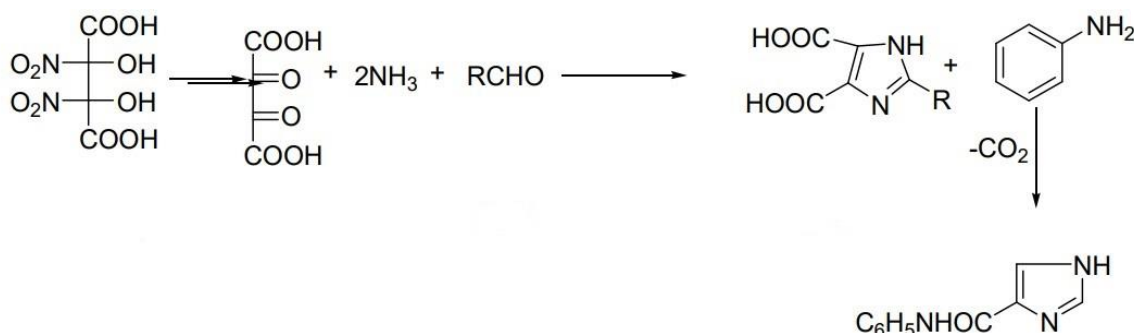


Figure 12 Imidazole from From formaldehyde and tartaric acid dinitrate

2.10 By the formation of one bond: The (1,5) or (3,4) bond can be formed by the reaction of an imidate and an α -aminoaldehyde or α -amino acetal, resulting in the cyclization of an imidine to imidazole. The example below applies to imidazole when $\text{R}=\text{R}_1=\text{Hydrogen}$ [20]

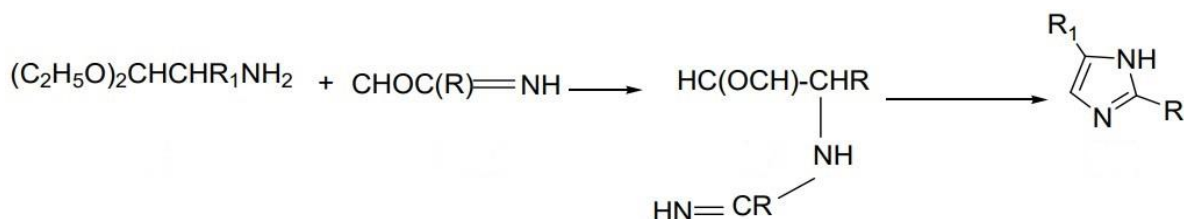


Figure 13 Imidazole by the formation of one bond

2.11 Van Leusen Imidazole Synthesis: The van Leusen reaction based on tosyl methyl isocyanides (TosMICs) is one of the most appropriate strategies to synthesize imidazole-based medicinal molecules, which have been increasingly developed on account of its advantages. [21]

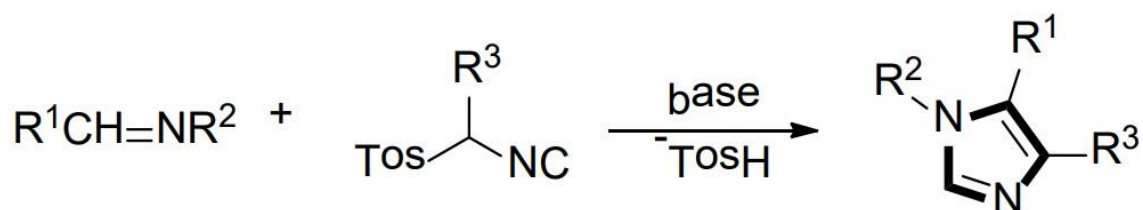


Figure 14 General van Leusen imidazole synthesis (21).

2.12 Hantzsch synthesis: This method involves the condensation of an amide, an amine or ammonia, and a β -keto ester or β -diketone to yield a 2,4,5-trisubstituted imidazole derivative. [22]

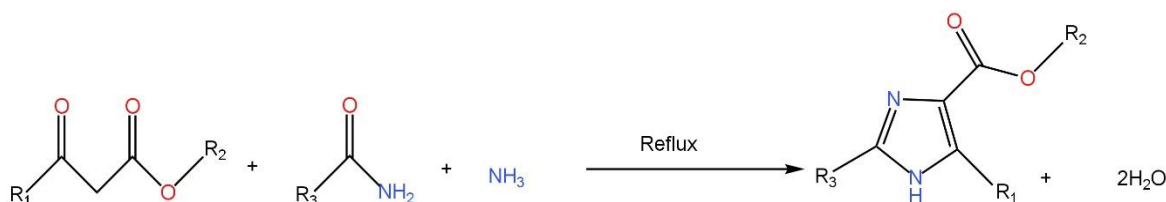


Figure 14 β -ketoesters with ammonia and amide (22).

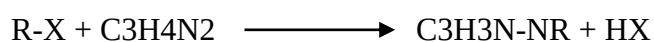
Overall, the choice of method for preparing imidazole derivatives will depend on the starting materials available, the desired product, and the efficiency and selectivity of the reaction.

Chapter 3: Chemical Reactions of Imidazole:

Here are some of the key reactions that imidazole can undergo:

3.1 Alkylation:

3.1.1 Imidazole can be N-alkylated with alkyl halides or tosylates to yield N-alkyl Imidazoles as shown below: [23]



Example:

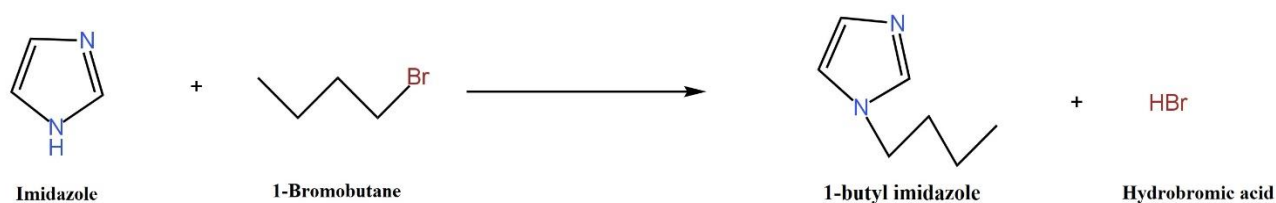


Figure 15 reaction between Imidazole and 1-Bromobutane (23)

3.1.2 Imidazoles can be alkylation at the C-2 position when the imidazole reaction with the alkyl group compound below: [24]

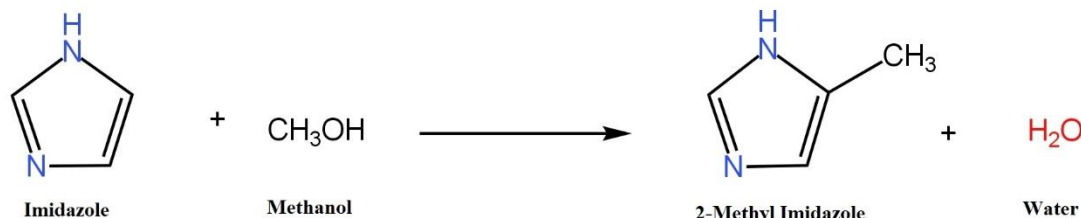
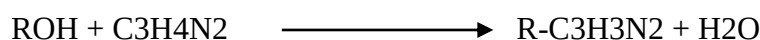
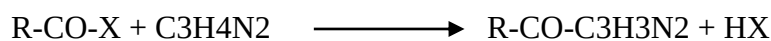


Figure 16 reaction between Imidazole and methanol

3.2 Acylation: Imidazole can be acylated with acyl halides or anhydrides to yield N-acyl imidazoles, as shown below: [25]



Example: isopropenyl acetate reacts with imidazole:

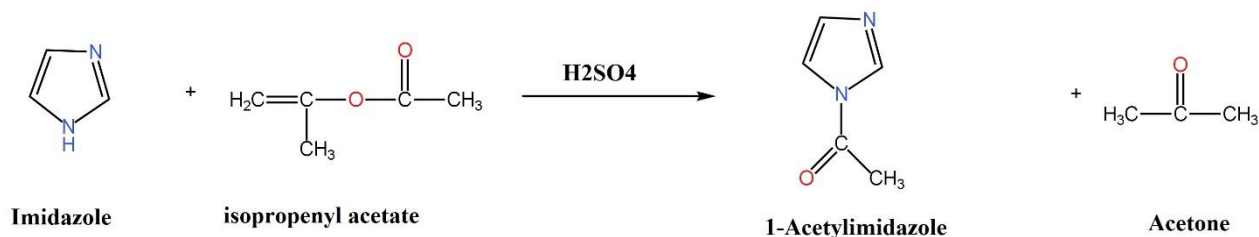


Figure 17 reaction between Imidazole and isopropenyl acetate

3.3 Ring-opening: Imidazole can undergo ring-opening reactions with strong nucleophile

[26] Example: $\text{C}_3\text{H}_4\text{N}_2 \longrightarrow \text{C}_2\text{H}_6\text{N}_2$

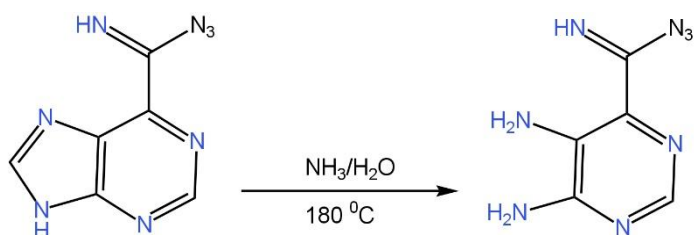


Figure 18 reaction change carbimidoylazide to diaminopyrimidine

3.4 Metal-catalyzed cross-coupling: Imidazole can be used as a coupling partner in palladium-catalyzed cross-coupling reactions with aryl or vinyl halides, as shown below: [27]

Example: $\text{Ar-X} + \text{C}_3\text{H}_4\text{N}_2 \longrightarrow \text{Ar-C}_3\text{H}_3\text{N}_2 + \text{HX}$

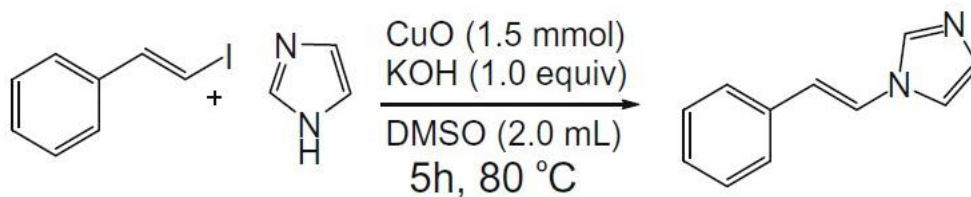


Figure 19 CuO nanoparticles-catalyzed cross-coupling reaction of trans-b-iodostyrene

3.5 Oxidation: Imidazole can be oxidized to yield the corresponding imidazole

2-oxides, as shown below: [28] $\text{C}_3\text{H}_4\text{N}_2 + [\text{O}] \longrightarrow \text{C}_3\text{H}_3\text{N}_2\text{O}$

Example: Imidazole can be oxidized with peracetic acid to yield imidazole-2-oxide:

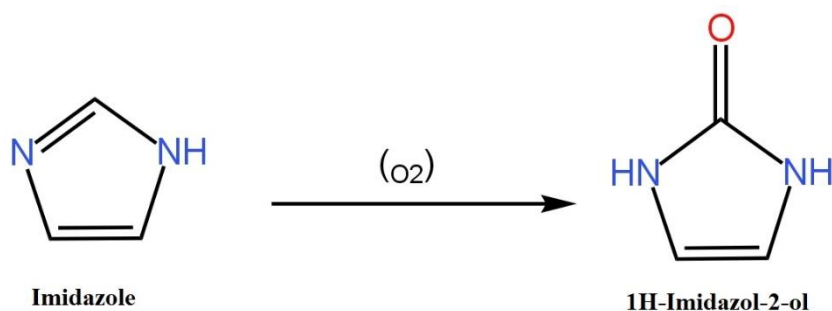
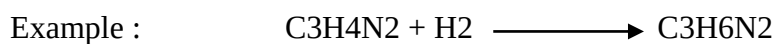


Figure 20 oxidation reaction for imidazole

3.6 Reduction: Imidazole can be reduced to yield 1,2-dihaloimidazole below:



Example: Imidazole can be reduced with hydrogen gas over a palladium catalyst to yield 1,2-dihydroimidazole: [29]

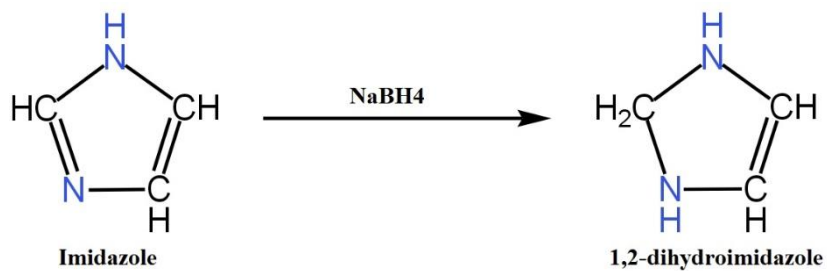
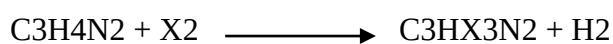


Figure 21 the chemical equation to reduce imidazole

3.7 Halogenated :

3.7.1 Imidazoles react with halogens to yield C-halogen derivatives, as shown below: [28]



Example: Imidazole can be brominated with Br_2

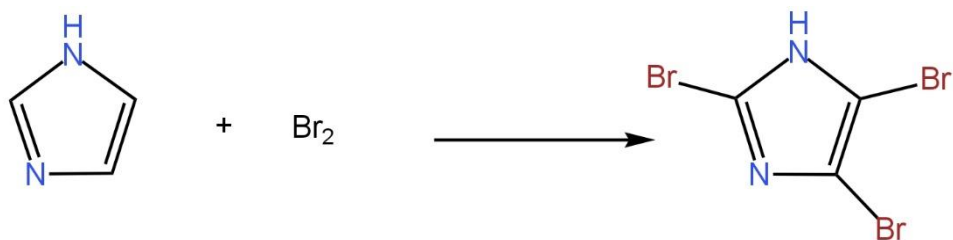


Figure 22 reaction imidazole with brom

3.7.2 N- Reaction of 2, 4, 5-triarylimidazoles (sodium salts) with bromine in dry ether yields N, N'-diimidazolyis. As all the ring carbon atoms are substituted, no rearrangement to C-halo imidazoles can occur as shown below: [28]

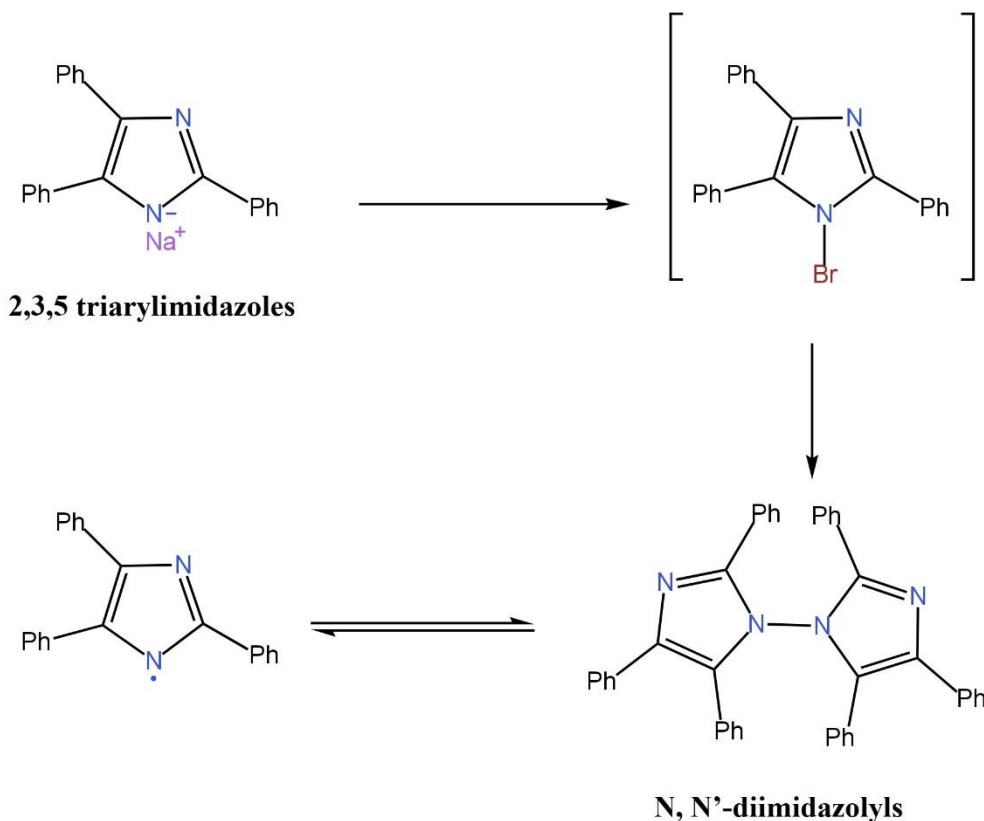


Figure 23 N, N'-diimidazolyis. As all the ring carbon atoms are substituted, no rearrangement to C-halo imidazoles can occur

3.8 Condensation: Imidazole can undergo condensation reactions with aldehydes or ketones to yield imidazole derivatives. [30] These reactions demonstrate the versatility of imidazole and its derivatives, which can be modified and functionalized in a variety of ways to yield compounds with a wide range of properties and applications.

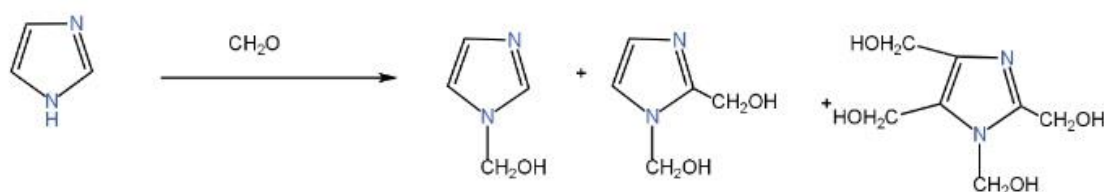


Figure 24 Condensation: Imidazole can undergo condensation reactions

chapter4: Imidazole Derivatives

Imidazole derivatives are compounds that are derived from the parent compound imidazole by modifying its structure through the addition of various functional groups. These derivatives exhibit diverse chemical and biological properties, making them valuable in several applications. Here is a sequential overview of imidazole derivatives and their uses:

4.1 Pharmaceutical Applications:

4.1.1 Antifungal Agents: Imidazole derivatives, such as ketoconazole and fluconazole, are widely used as antifungal drugs as shown below: [9]

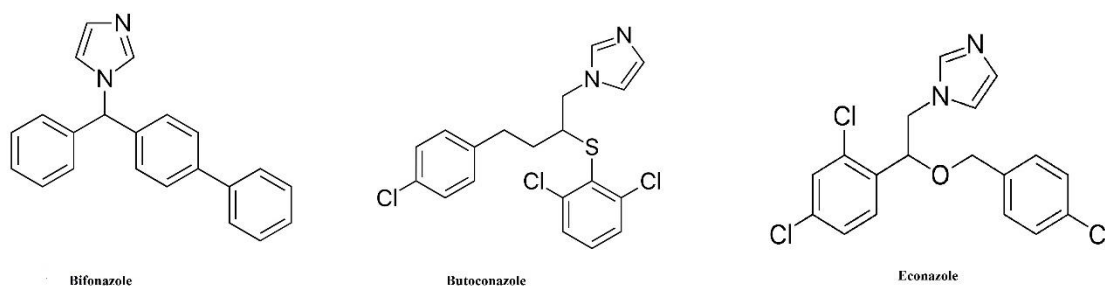


Figure 25 Imidazole antifungal drugs (Bifonazole, butoconazole and econazole) (9)

4.1.2 Antihistamines: Certain imidazole derivatives, like cimetidine, are utilized as antihistamine medications to treat gastric ulcers and acid reflux as shown below: [10]

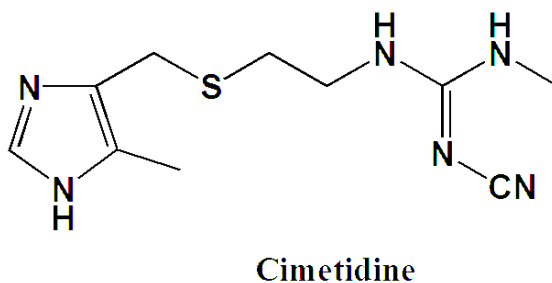


Figure 26 Antihistamines cimetidine (10)

4.1.3 Anti-inflammatory Agents: Imidazole derivatives, including nimesulide, possess anti-inflammatory properties and are employed in the treatment of pain and inflammation as shown below: [11]

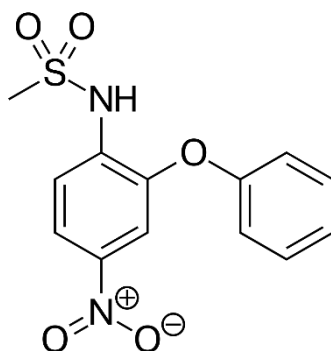


Figure 27 Anti-inflamImidazole nimesulide (11)

4.2 Coordination Chemistry: Ligands: Imidazole derivatives often act as ligands in coordination complexes with transition metals. These complexes find applications in catalysis, material science, and medicinal chemistry as shown below: [12]

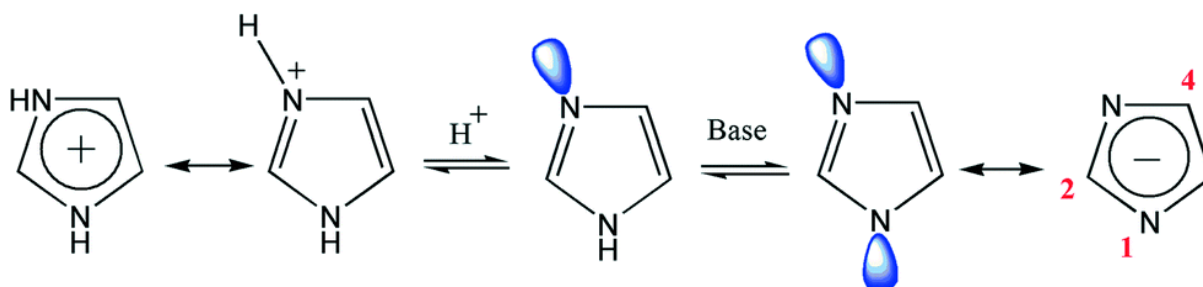


Figure 28 Schematic of the protonation and deprotonation of imidazole (12)

4.3 Corrosion Inhibitors: Imidazole derivatives have corrosion-inhibiting properties, making them useful in coatings, metal treatments, and corrosion prevention in various industries as shown below: [13]

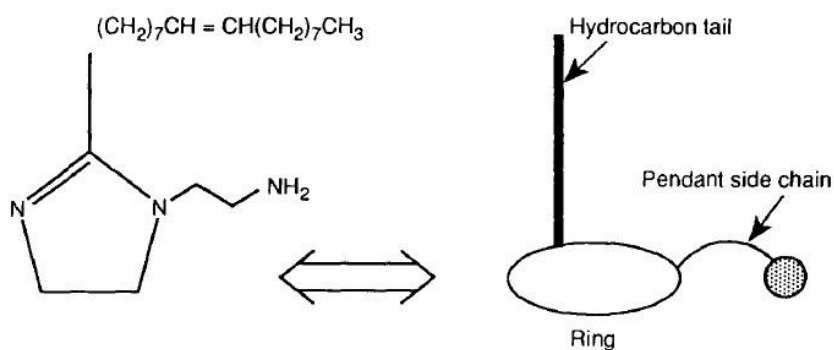


Figure 29 Representation of oleic imidazoline (13)

4.4 Organic Synthesis: Imidazole derivatives serve as valuable building blocks in organic synthesis. They participate in diverse reactions, such as nucleophilic substitutions, electrophilic additions, and cyclizations, allowing for the synthesis of complex organic compounds as shown below: [14]

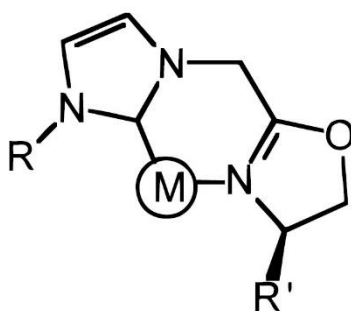


Figure 30 General formula of oxazoline/imidazoline-2-ylidene complexes (14)

4.5 Agricultural Applications: Fungicides and Herbicides:

Certain imidazole derivatives are employed as fungicides and herbicides in agriculture to protect crops from fungal infections and weed growth as shown below: [15]

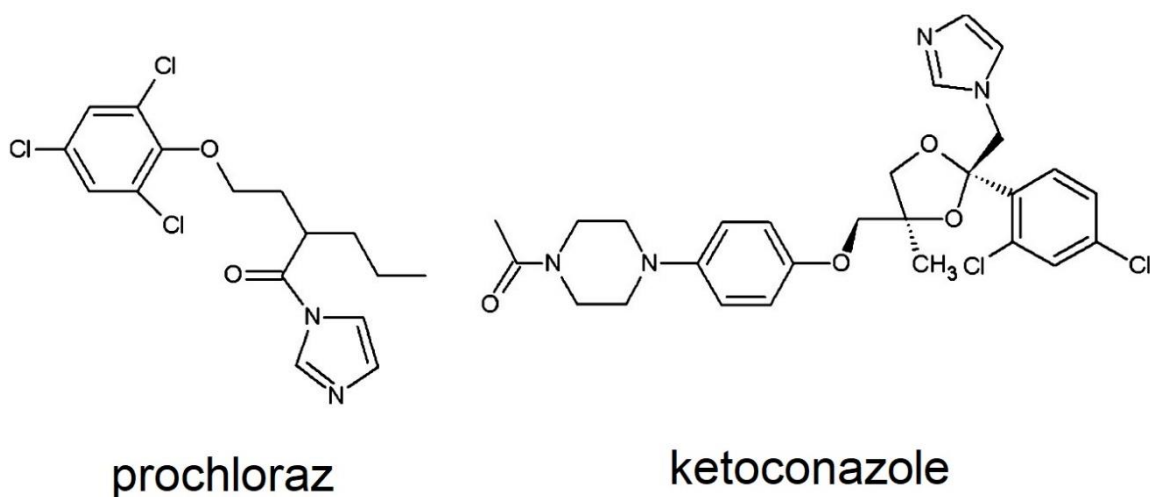


Figure 31 Chemical structure of two imidazole fungicides: prochloraz to the Left and ketoconazole to the Right (15).

4.6 Biological Activities:

4.6.1 Enzyme Inhibitors: Imidazole derivatives can act as enzyme inhibitors, targeting specific enzymes involved in disease processes as shown below: [16]

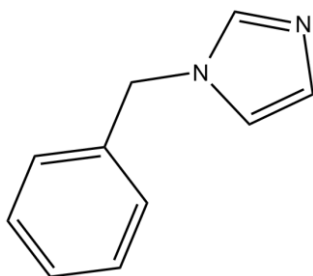


Figure 32 1-Benzyl imidazole (16)

4.6.2 Bioactive Compounds: Some imidazole derivatives exhibit bioactivity, including antimicrobial, antiviral, and anticancer properties. [17]

Example: 5,6-dichloro-1-cyclopentyl-1h benzo imidazole has anti-proliferative activity by inhibiting cyclin-dependent kinase 6 (CDK6) and inducing apoptosis on myeloid cell leukaemia 1 (Mcl-1) protein as shown below:

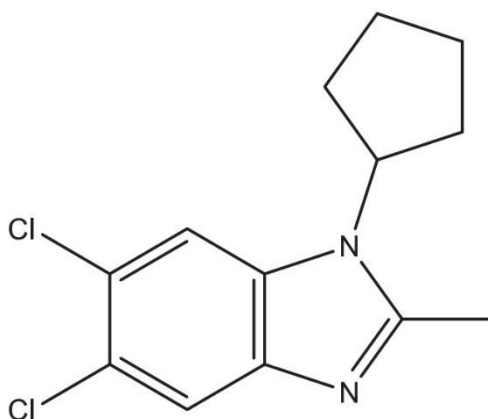


Figure 33 5,6-dichloro-1-cyclopentyl-1h benzo imidazole

Overall, imidazole derivatives find extensive applications in pharmaceuticals, coordination chemistry, corrosion inhibition, organic synthesis, agriculture, and various biological activities. Their diverse properties and functional groups make them versatile compounds with significant importance in several scientific and industrial fields.

4.3 Imidazole as a corrosion inhibitor :

Corrosion, the deterioration of materials due to chemical or electrochemical reactions with their environment, poses a significant challenge in various industries, including manufacturing, infrastructure, and transportation. It leads to substantial economic losses, safety concerns, and environmental impact. To combat corrosion, the development of effective corrosion inhibitors has gained considerable attention.

Imidazoline derivatives have emerged as promising corrosion inhibitors due to their remarkable inhibitory properties and versatility in chemical structure. These compounds exhibit the ability to form a protective film on metal surfaces, thereby reducing corrosion rates and extending the lifespan of metallic materials. Moreover, their synthesis can be tailored to obtain derivatives with specific properties, enhancing their applicability in diverse corrosive environments. [18]

Imidazoline derivatives act as mixed-type corrosion inhibitors, which inhibits both cathodic and anodic processes by being adsorbed on the electrode surface according to the adsorption isotherms. With a slight positive shift in corrosion potential. The inhibitor adsorbed on the surface of the electrode affects the kinetic processes of the cathodic and anodic reactions and increases the activation energy of the reaction. Both effective dynamic polarization and EIS measurements reveal this. The inhibition efficiency increases with increasing immersion time. [18]

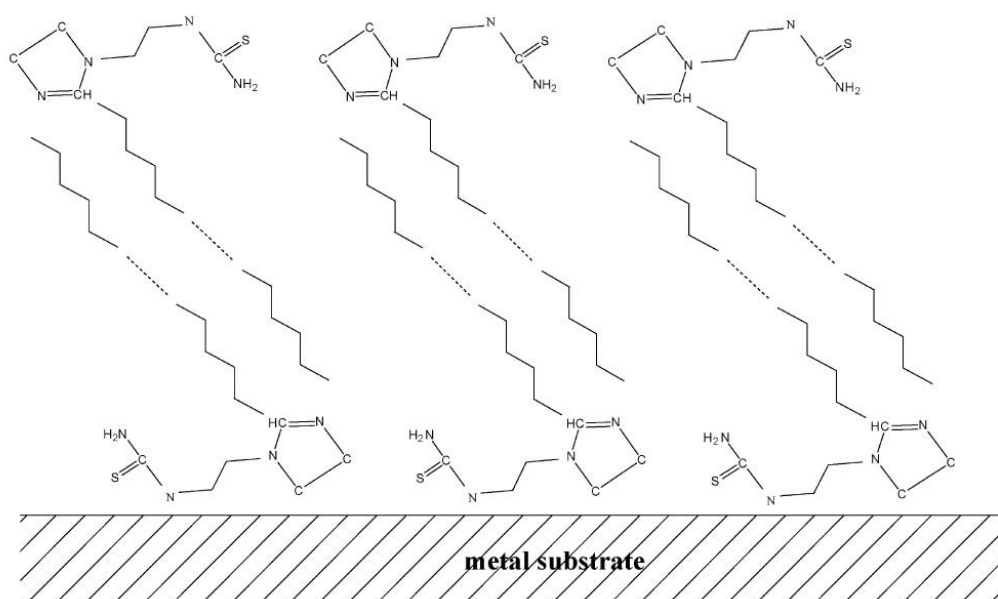


Figure 34 Bilayer schematic model for imidazoline derivative adsorption on metal surface (18).

The main objective of this thesis is to investigate the synthesis of imidazoline derivatives that are environmentally friendly and do not consume much energy when synthesized. and evaluate its corrosion resistance efficiency. Through a systematic study, we aim to contribute to the understanding of the structure-activity relationships of these compounds, paving the way for the development of new and effective corrosion inhibitors.

In this research, we will employ a combination of established synthetic methodologies and innovative approaches to synthesize a series of imidazoline derivatives. Careful characterization of the synthesized compounds will be conducted using techniques such as nuclear magnetic resonance (NMR) spectroscopy, mass spectrometry, and infrared spectroscopy to confirm their structures and purities.

Furthermore, the anti-corrosion efficiencies of the synthesized imidazoline derivatives will be evaluated systematically using corrosion test methods, as found in the ASTM Laboratory Test Corrosion Inhibitors. These technologies will provide insights into the inhibitory performance, wear rates, and mechanisms of composites synthesized in different corrosion environments.

The findings of this research will contribute to the development of corrosion mitigation strategies by expanding the knowledge of imidazoline derivatives as effective corrosion inhibitors. The results may find applications in industries such as oil and gas, marine, and automotive, where corrosion protection is crucial for maintaining structural integrity and ensuring operational safety.

Chapter 5 LITERATURE REVIEW

Imidazole derivatives have been widely studied as potential corrosion inhibitors due to their unique chemical properties and their ability to interact with metal surfaces. In this literature review, we will discuss the preparation of imidazole derivatives and their use as corrosion inhibitors.

Preparation of Imidazole Derivatives

There are several methods for preparing imidazole derivatives, including the Debus-Radziszewski reaction, the Radziszewski reaction, and the Knorr synthesis. The Debus-Radziszewski reaction involves the condensation of glyoxal with a primary amine in the presence of ammonium chloride to yield an imidazole derivative. The Radziszewski reaction involves the condensation of a diamine with an aldehyde or a ketone in the presence of ammonium chloride to yield an imidazole derivative. The Knorr synthesis involves the cyclization of a 1,2-diketone with an amine or ammonia to yield an imidazole derivative.

Studies on Corrosion Inhibition Several studies have investigated the use of imidazole derivatives as corrosion inhibitors for various metals, including iron, copper, and aluminum.

For example, Zhang et al. (2020) synthesized a series of imidazole derivatives and evaluated their corrosion inhibition efficiency for copper in 0.5 M HCl solution. They found that the imidazole derivatives exhibited good corrosion inhibition properties, with inhibition efficiencies ranging from 50% to 94%.

In another study, Li et al. (2019) synthesized a series of imidazole derivatives and evaluated their corrosion inhibition efficiency for carbon steel in a 3.5 wt% NaCl solution. They found that the imidazole derivatives exhibited good corrosion inhibition properties, with inhibition efficiencies ranging from 80% to 95%.

In addition to experimental studies, several computational studies have also been conducted to investigate the mechanism of corrosion inhibition by imidazole derivatives.

For example, Liu et al. (2017) conducted a computational study to investigate the interaction between imidazole derivatives and iron surfaces. They found that the imidazole derivatives could form stable adsorption layers on the iron surface, inhibiting the corrosion process.

Conclusion

In conclusion, imidazole derivatives have been shown to be effective corrosion inhibitors for various metals. The preparation of imidazole derivatives can be achieved through several methods, including the Debus-Radziszewski reaction, the Radziszewski reaction, and the Knorr synthesis.

Several experimental and computational studies have demonstrated the potential of imidazole derivatives as corrosion inhibitors, highlighting their unique chemical properties and their ability to interact with metal surfaces. Further research is needed to optimize the synthesis of imidazole derivatives and to investigate their potential for corrosion inhibition in different environments and applications.

Chapter 6 : MATERIALS AND METHODS:

6.1 Preparation of fatty acid halides:

6.1.1 Materials:

- 1- Fatty acids (Palmitic Acid)
- 2- Hydrochloric acid (HCl)
- 3- Ethanol ($\text{CH}_3\text{CH}_2\text{OH}$)

6.1.2 Methods:

- 1- Measuring 10 grams of solid Palmitic acid place it in an acid-resistant beaker.
- 2- Add 40 mL of hydrochloric acid with a concentration of 37% to the beaker.
- 3- Place the beaker on a heat source for about an hour, stirring occasionally.



Figure 35 Palmitic acid

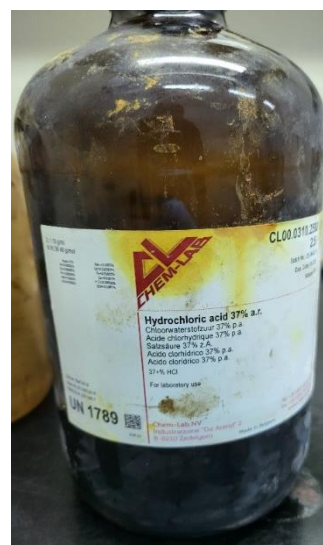


Figure 36 Hydrochloric acid

- 4- After the reaction is complete, allow the solution to cool completely. Adding an organic solvent such as acetone or ethanol to the cooled solution until it reaches a volume of 100 mL.
- 5- Filter the solution using filter paper to obtain the formed organic halide. Washing the resulting halide with the organic solvent that was added to obtain a lower amount of side compounds.
- 6- Drying at low heat, such as under a stream of pure air or vacuum, to obtain the pure halide.



Figure 37 Heating for Imidazole with HCl



Figure 38 ethanol

To achieve a conversion rate of 90%, the reaction rate and temperature can be adjusted. Generally, the reaction rate increases with an increase in temperature, but excessive temperature should be avoided to prevent the formation of undesirable side compounds. Additionally, increasing the conversion of palmitic acid to the halide can be achieved by using higher concentrations of hydrochloric acid.

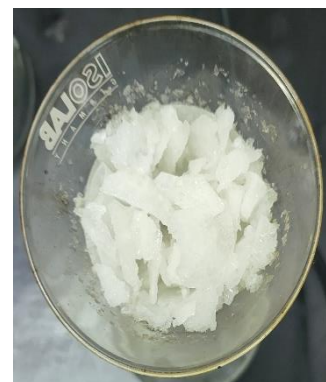


Figure 39 palmitic chloride after reaction finish

6.2 Prepare imidazole sodium

6.2.1 Materials:

1. Imidazole ($C_3H_4N_2$)
2. Sodium hydroxide (NaOH)
3. Water (H_2O)

6.2.2 Method :

- 1- Measure 10 gm of imidazole and put it in an Erlenmeyer flask.
- 2- Prepare a 1-10 M NaOH solution and add 20 ml of a 10-1 M NaOH solution to the conical flask containing imidazole. Close the conical flask and place it in a water bath heated to 70-80 °C for 60 minutes.

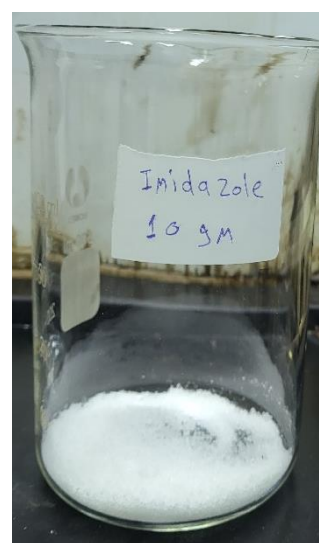


Figure 40 Imidazole

- 3- After the time is up, remove the conical flask from the water bath and let it cool to room temperature. add 20 mL of distilled water to the conical flask.
- 4- Adjust the pH of the solution to 6-7 By adding drops of diluted HCl

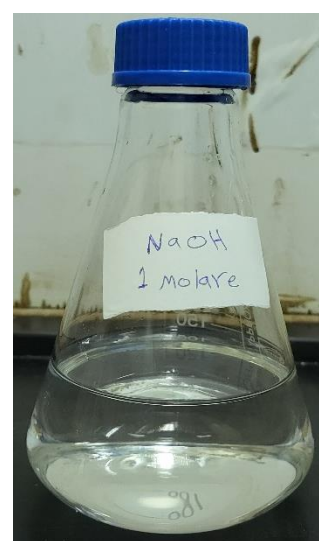


Figure 41 NaOH Solution

- 5- Purify the solution by evaporating the solution to get rid of the water and obtaining sodium imidazole salt only this is done by heating it on a heat source at a low temperature.
- 6- After purification, sodium imidazole can be stored in airtight containers in a cool dry place.



Figure 42 Sodium Imidazole

6.3 Synthesis of Imidazolium Derivatives with Fatty Acid

6.3.1 Materials:

- 1- Fatty acid halides
- 2- Sodium imidazole

6.3.2 Method:

- 1- Weigh 5 gm of solid fatty acid and place it in a beaker
- 2- Add an appropriate amount of solvent (10ml from Ethanol) to the cup containing the fatty acid halides and stir to mix.
- 3- Add 5 gm of the solid imidazole sodium to the beaker and stir well to distribute the material.
- 4- Place the beaker on the heat source and leave it for one to two hours. a reaction takes place between the substances in the beaker, in which imidazole palmitic acid is formed.
- 5- - After the reaction is complete, the beaker is cooled and filtered to obtain the final product. The product can be purified using appropriate filtration and washing techniques (eg ethanol).

6.4 Mechanism of reaction:

- 1- Mechanism of the reaction of a fatty acid with hydrochloric acid, as shown in Figure (12)

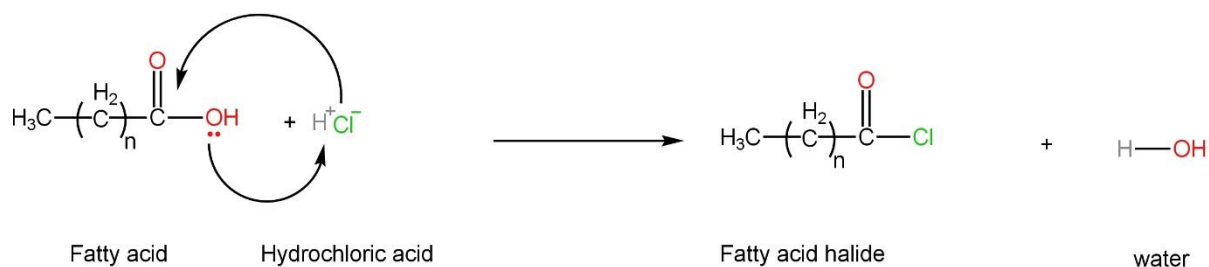


Figure (17)

- 2- Mechanism of the reaction of imidazole with sodium hydroxide as in Figure (13)

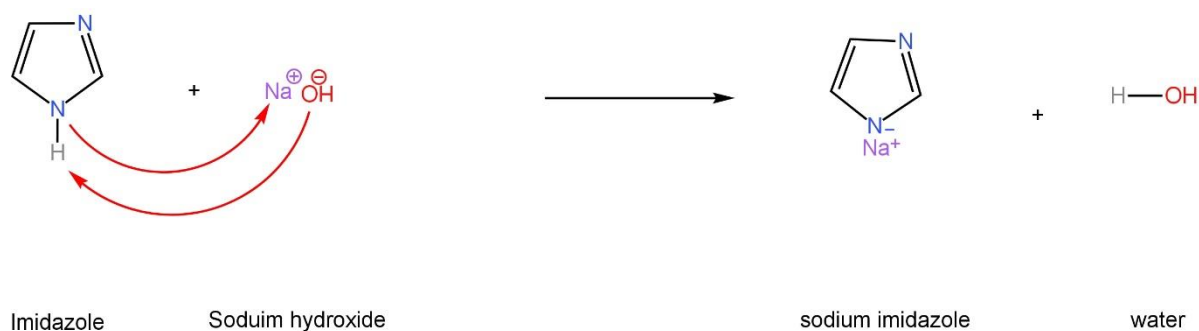
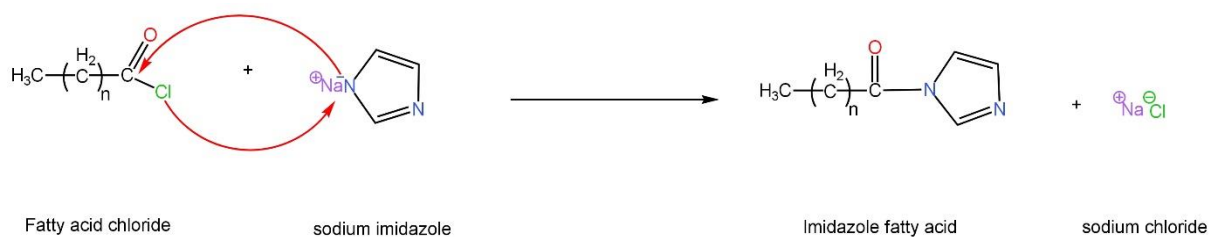


Figure (18)

- 3- Mechanism of the reaction between Fatty acid chloride and Sodium imidazole as in Figure (14)



Figure(19)

6.5 Characterization:

To further investigate the chemical structure and properties of the synthesized imidazole derivatives, NMR (magnetic resonance) and FT-IR (infrared spectroscopy) were used. NMR is a powerful analytical technique that provides valuable insights into the structural features and composition of organic compounds. By analyzing the NMR and FT-IR data, we were able to confirm the successful formation of the desired derivatives and assess the potential for corrosion inhibition based on their structural properties.

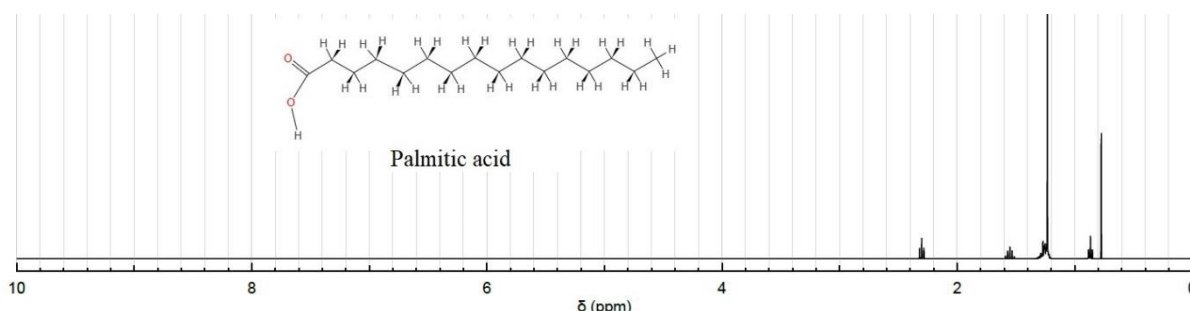


Figure 44 (¹H-NMR) spectroscopy for the palmitic acid compound

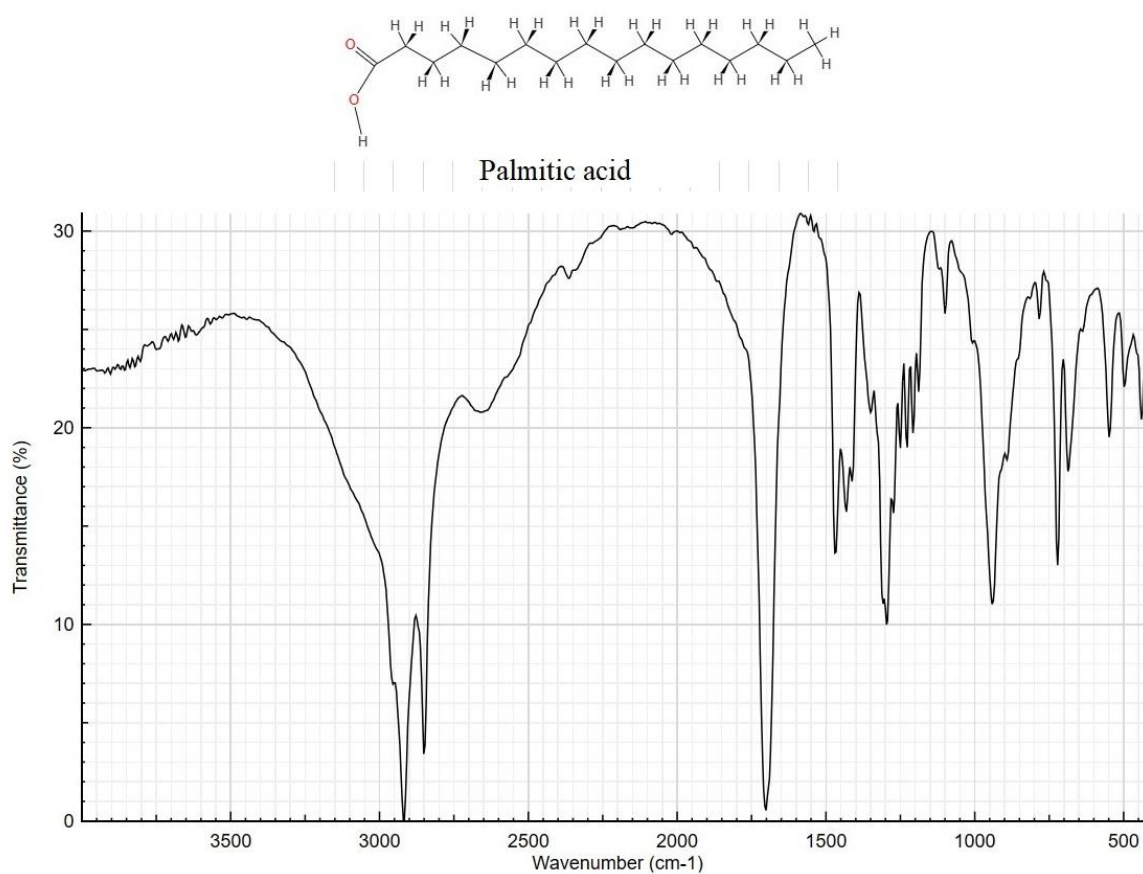
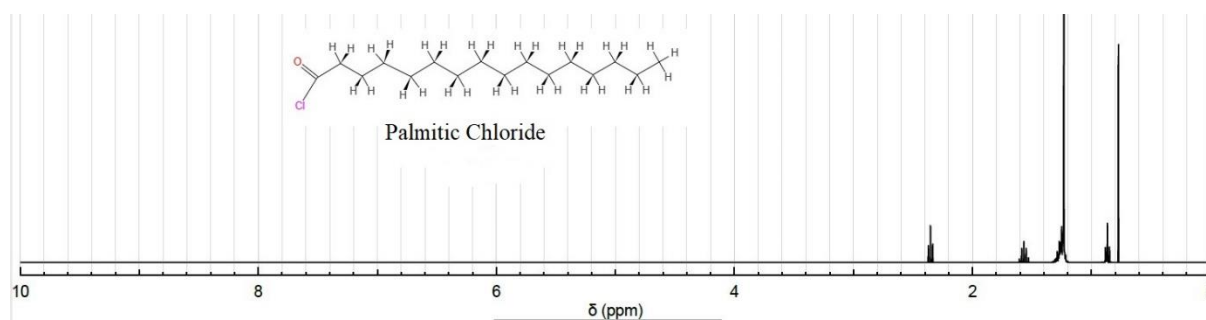
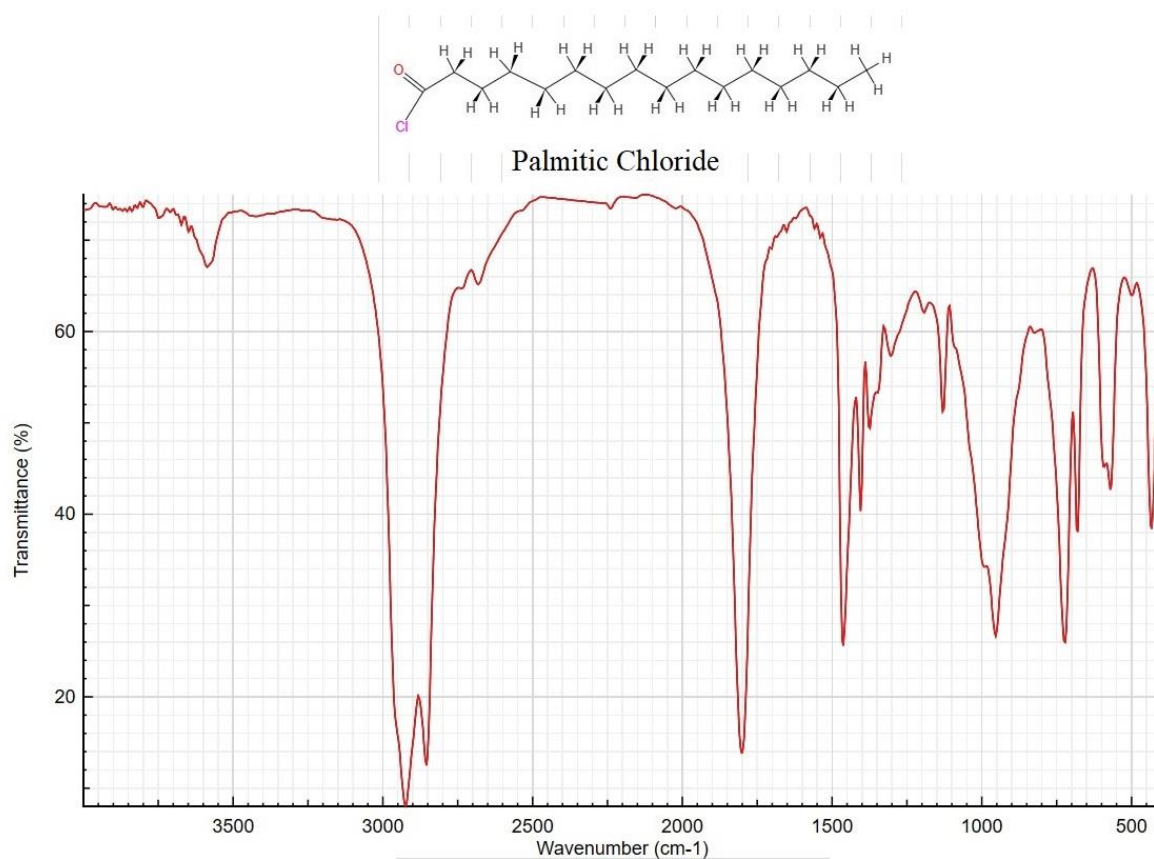
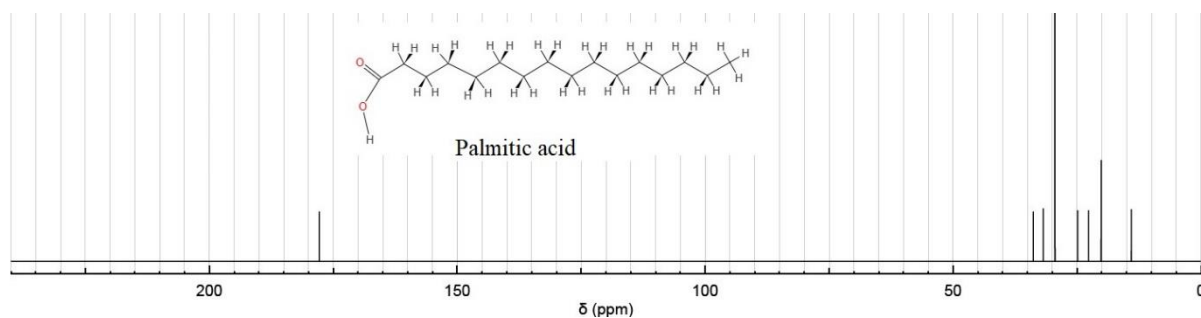


Figure 43 (FT-IR) spectroscopy for the palmitic acid compound



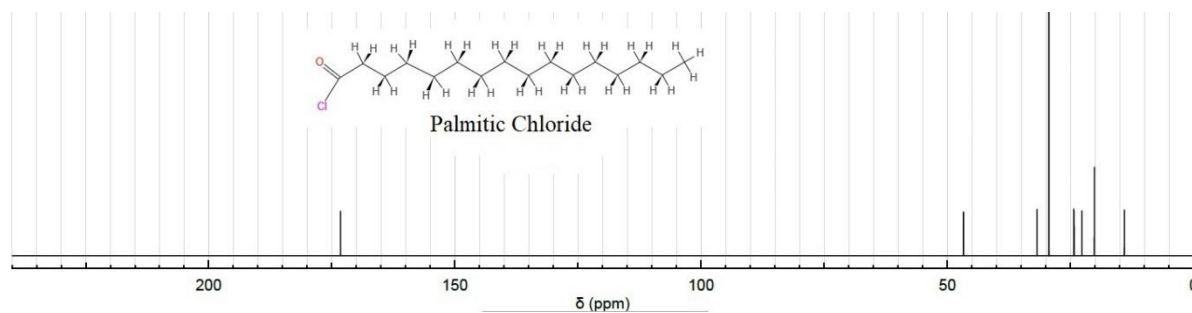


Figure 48) (^{13}C -NMR) spectroscopy for the palmitic chloride compound

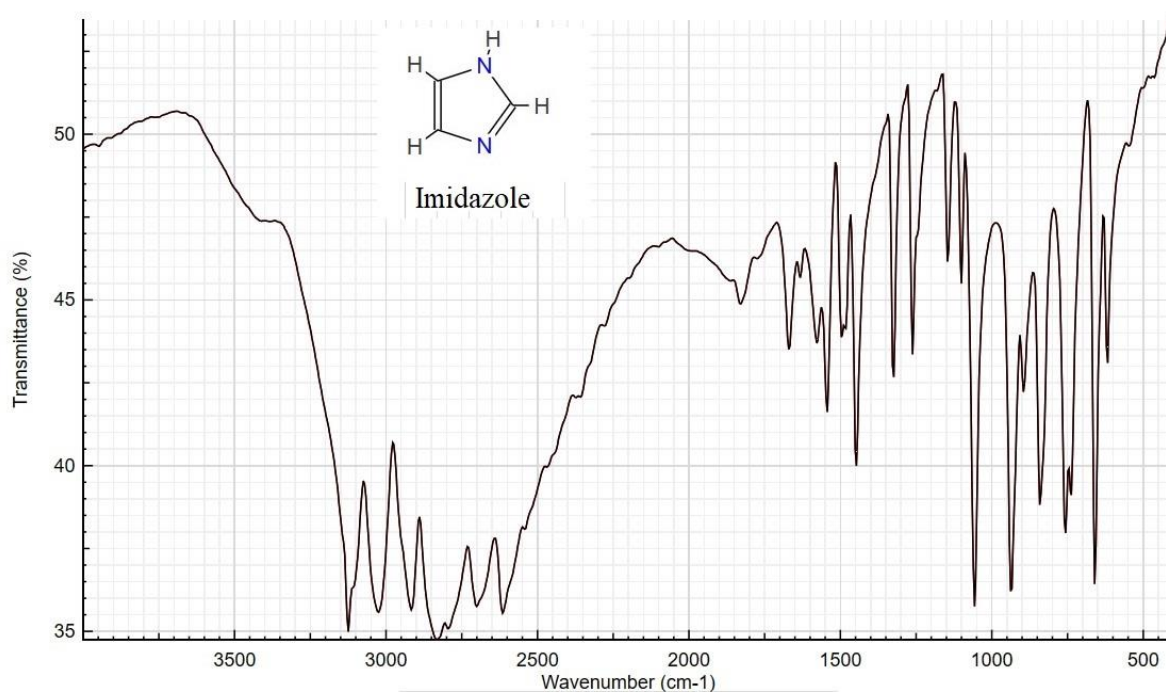


Figure 49 (FT-IR) spectroscopy for the imidazole compound

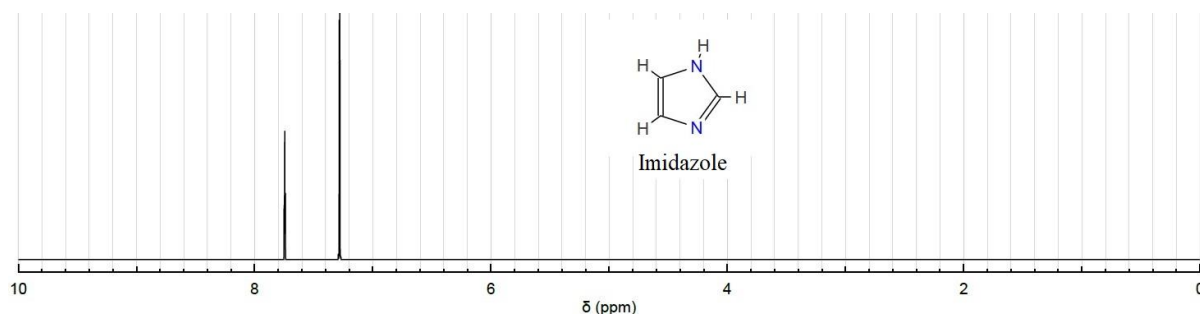


Figure 50 (^1H -NMR) spectroscopy for the imidazole compound

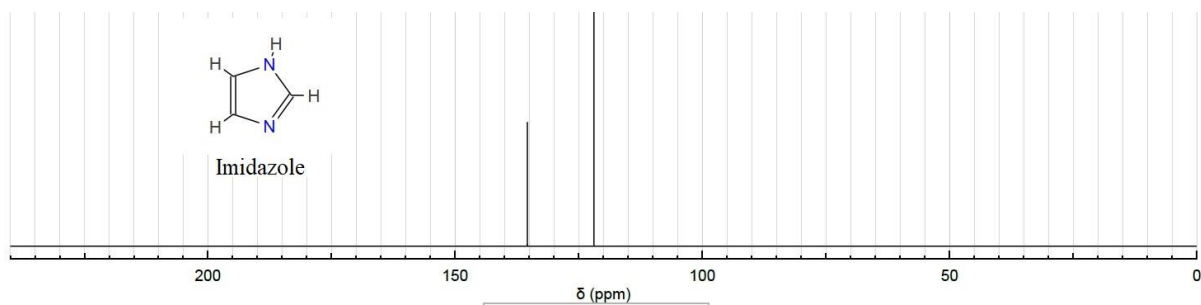


Figure 51 (^{13}C -NMR) spectroscopy for the imidazole compound

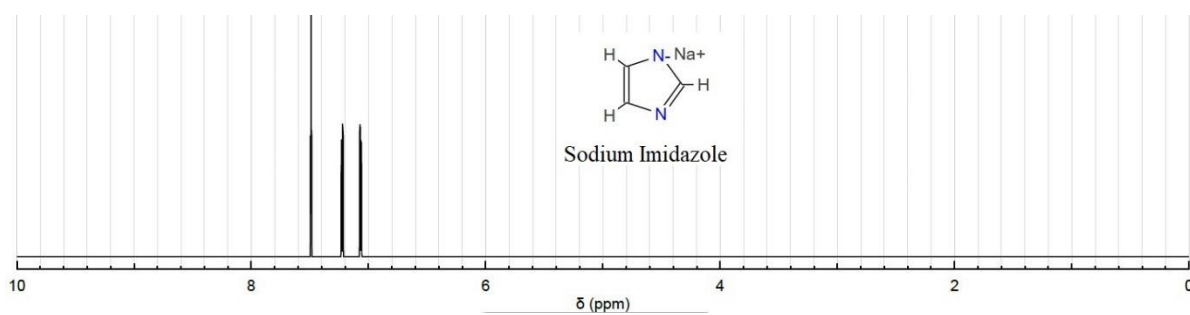


Figure 52 (^1H -NMR) spectroscopy for the sodium imidazole compound

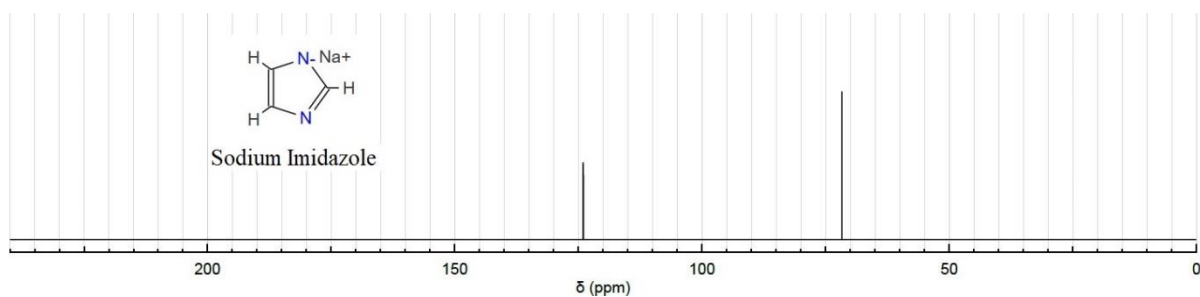


Figure 53 (^{13}C -NMR) spectroscopy for the sodium imidazole compound

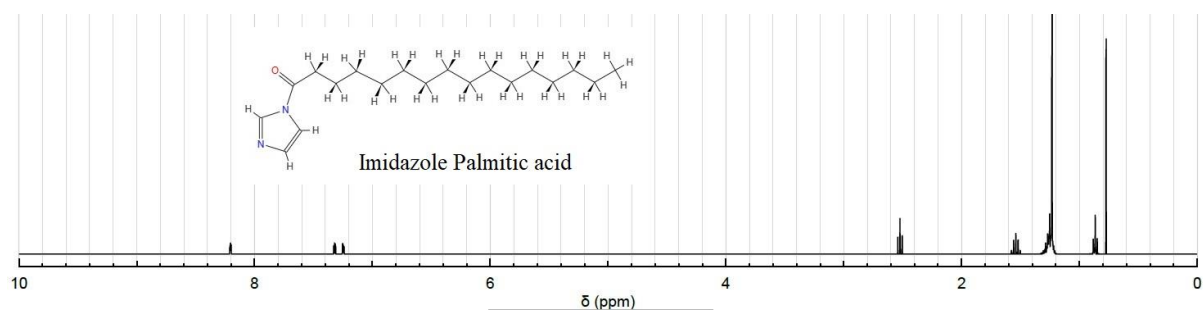


Figure 54 (^1H -NMR) spectroscopy for the imidazole palmitic acid compound

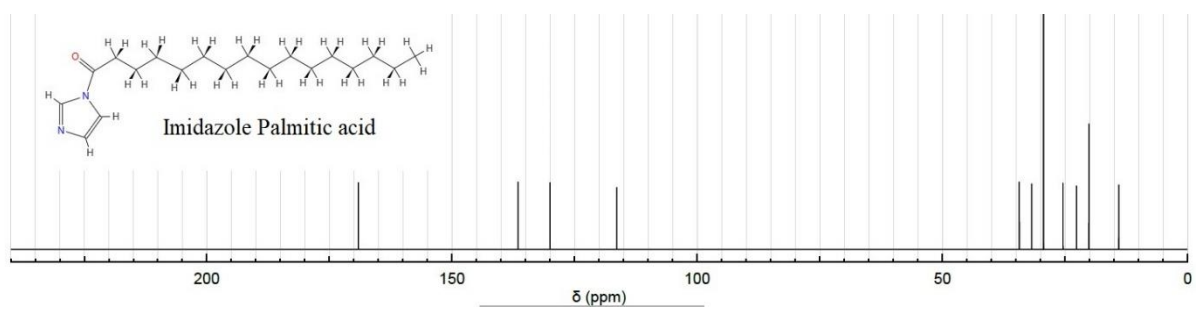


Figure 55 (^{13}C -NMR) spectroscopy for the imidazole palmitic acid compound

6.6 Corrosion inhibition studies:

The synthesized imidazole derivatives were tested for their corrosion inhibition properties using the ASTM G31 standard guide for laboratory immersion corrosion testing of metals

6.6.1 Test Conditions

- Temperature: 50°C, Duration: 14 days.
- Test medium: A3 material with the dimension 50x25x2mm.

6.6.2 Test Procedure

- 1- Cleaning corrosion coupon: Corrosion coupon are wiped with filter paper, and then place the coupon container filed with in petroleum ether with boiling point range 60-90°C, then remove the oil on the coupon surface with cotton wool, soak it in anhydrous ethanol for about 5min, further degrease and dehydrate. Remove the corrosion coupon with filter paper and stored in a desiccator for 1h and then weigh it, accurate to 0.1mg.



Figure 56 corrosion coupon

- 2- Prepare the samples: Take water sample extracted from crude oil, then fill test bottle by water sample to mark line.

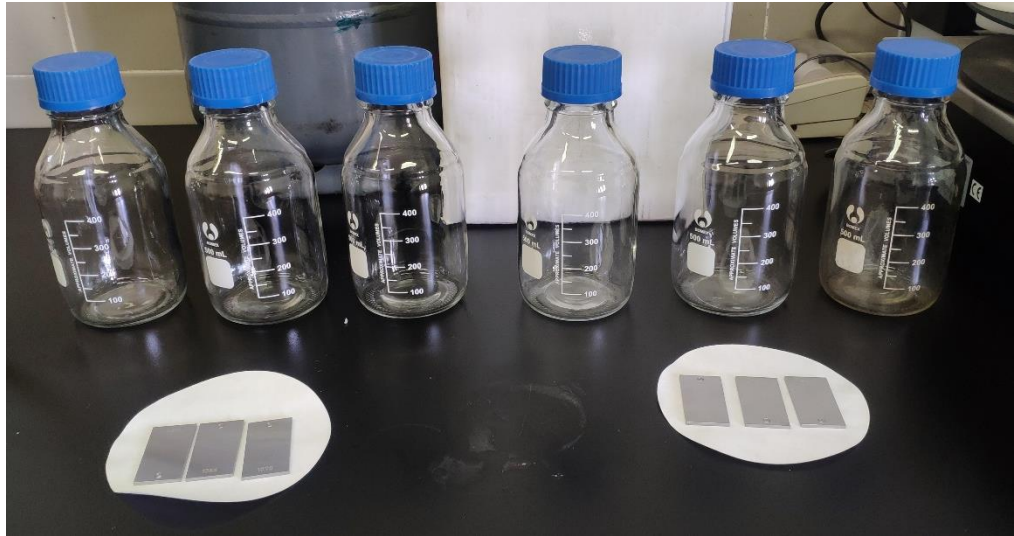


Figure 57 When Put the coupons inside the container

- 3- Suspension corrosion coupon: The test corrosion coupons were not allowed to contact with the container wall, the space between the corrosion coupons should be above 1cm, the upper end of the corrosion coupon should be at least 3cm above the fluid surface, and tighten the lid.
- 4- inject the corrosion inhibitor that we prepared into each of the containers, with specific concentrations as follows:
(10 ppm - 20 ppm - 30 ppm - 40 ppm - 50 ppm)



Figure 58 Place the coupons container in the water bath after injection

- 5- Leave the coupon container in water bath: Put the wide mouth bottles in water bath at 50°C and keep them for 14 days.

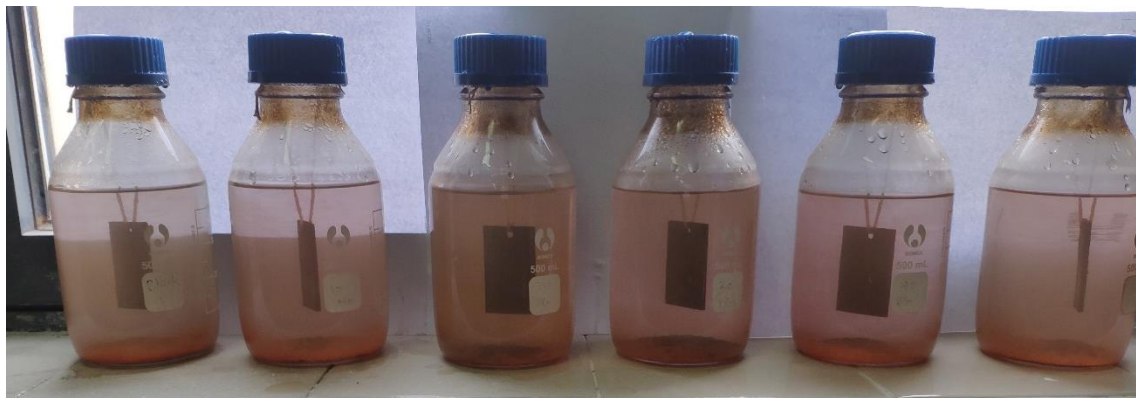


Figure 59 when removing the coupons containers from the water bath

- 6- Observation records: Observe and record surface corrosion and corrosion product adhesion situation, and immediately rinse off the test fluid with water and dry it with filter paper. According to the experimental steps cleaning the corrosion coupons, then stored in a desiccator for 1h and then weigh it. Then record the data.

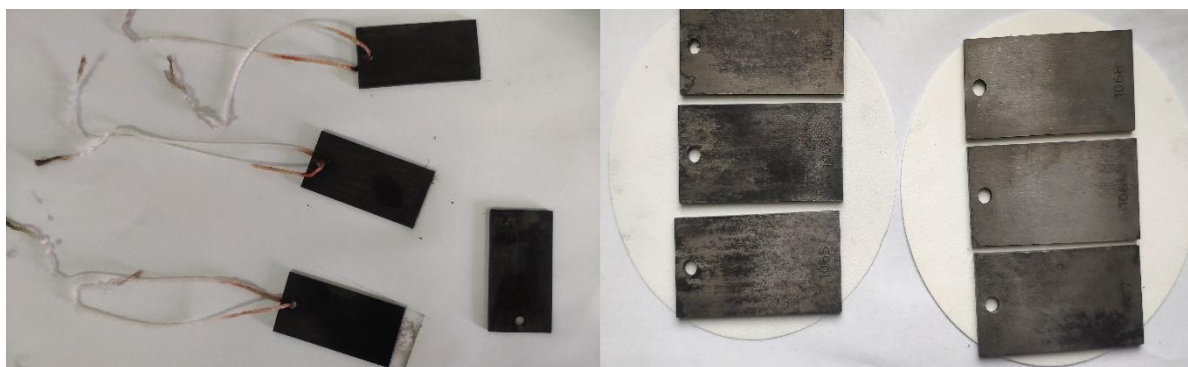


Figure 60 When taking out the coupons from the containers

6.6.3 Test Results

Take a water sample from water extracted from crude oil, according to the Evaluation method for the behaviour of corrosion inhibitor for produced water of an oilfield,(SY/T 5273-2000 standard), the test results see as below table:

Table 1 Corrosion rate

Test bottle No.	Type of Injected Chemical	Injected Dosage in ppm	Coupon ID	Initial Coupon Weight (g)	Final Coupon Weight (g)	Loss of weight (g)	Corrosion rate (mm/a)
1	Blank	0.0	1064	19.6364	19.6003	0.0361	0.0862
2	Imidazole Palmitic acid	10.0	1065	19.3793	19.3528	0.0265	0.0633
3		20.0	1066	19.3947	19.3724	0.0223	0.0532
4		30.0	1067	19.5146	19.4917	0.0229	0.0547
5		40.0	1068	19.3313	19.3102	0.0211	0.0504
6		50.0	1070	19.4021	19.3823	0.0198	0.0473

$$\text{Corrosion Rate (mm/a)} = (8.76 \times 10000 \times \Delta M / S \times \rho \times h) \times 2$$

ΔM = Loss of Weight

S = Surface Area = $(50\text{mm} \times 25\text{mm} \times 2\text{mm}) \times 2 = 28\text{cm}^2$

ρ = Coupon Density = 7.8 g/cm^3

h = Time of test = $7\text{days} \times 24 = 168 \text{ h}$

h = Time of test = $14\text{days} \times 24 = 336 \text{ h}$

h = Time of test = $21\text{days} \times 24 = 504 \text{ h}$

h = Time of test = $28\text{days} \times 24 = 672 \text{ h}$

Chapter 7: Discussion and Conclusion :

The successful synthesis of imidazoline derivatives through a non-harmful method is a noteworthy accomplishment. It addresses the environmental concerns associated with conventional methods and opens new possibilities for green and sustainable corrosion inhibitors. Furthermore, the detailed characterization using NMR and IR ensures the reliability of the obtained compounds, boosting confidence in their anti-corrosion evaluation.

The experimental data revealed promising outcomes, indicating that the imidazole derivatives synthesized through the non-harmful approach exhibited notable corrosion inhibition properties. The results obtained from the ASTM G31 experiments were documented and presented in a table format. Comparative analysis with traditional corrosion inhibitors and commercial products further reinforced the potential of the eco-friendly imidazole derivatives as corrosion inhibitors.

Conclusion: In conclusion, this research highlights the significance of employing environmentally friendly methods for the synthesis of imidazole derivatives as corrosion inhibitors. The non-harmful approach presented in this study not only ensures the safety of researchers and the environment but also offers a feasible route for large-scale industrial production. The MRI characterization provided valuable insights into the structural features of the synthesized derivatives, adding to their potential as effective corrosion inhibitors. The positive results from the ASTM G31 corrosion inhibition experiments further support the practical application of these derivatives in industrial corrosion protection.

It is important to acknowledge that further studies may be required to explore the long-term stability and performance of these imidazoline derivatives under different environmental conditions. Additionally, investigating the mechanism of inhibition and exploring potential synergistic effects with other additives could enhance the overall efficiency and applicability of these compounds in practical settings.

Future Implications: The success of this research opens up avenues for further exploration in the field of eco-friendly corrosion inhibitors. Future studies could focus on optimizing the

synthesis process, investigating the mechanism of inhibition, and exploring the effectiveness of these imidazole derivatives in real-world scenarios. Additionally, the economic feasibility and potential commercialization of these eco-friendly inhibitors could be evaluated to facilitate their integration into various industries, promoting sustainable and environmentally conscious practices.

Overall, this research provides valuable contributions to the field of corrosion inhibition and organic synthesis, offering a greener approach to the preparation of imidazole derivatives and highlighting their potential as effective and environmentally friendly corrosion inhibitors.

And, this research contributes to the growing body of knowledge concerning green synthesis methods and their applications in the field of corrosion science. The development of efficient and sustainable corrosion inhibitors is a vital step in promoting environmentally friendly practices in industries reliant on metallic materials.

Chapter 8 References

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- **APPENDICES**

APPENDIX 1.

APPENDIX 2.

APPENDIX 1.

APPENDIX 2.

• CURRICULUM VITAE

Personal Information

Name and Surname: Name SURNAME

Education

MSc	Çankırı Karatekin University Graduate School of Natural and Applied Sciences Department of Chemical Engineering	2023-Present
Undergraduate	Çankırı Karatekin University Faculty of Engineering Department of Chemical Engineering	2021-2023

Work Experience

Year	Institution	Position
2023-Present	Çankırı Karatekin University, Department of Chemistry	Research Asst.

Academic Activities

- (If any, a paper, article, chapter or book should be written.)