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SYNTHESIS OF SOME IMIDAZOLINE DERIVATIVES AND STUDY OF THEIR ANTI-CORROSION EFFICIENCY

IN PARTIAL FULFILLMENT OF THE REQUIREMENTS
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IN
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MUSTAFA SALIH MAHDI

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SYNTHESIS OF SOME IMIDAZOLINE DERIVATIVES AND STUDY OF THEIR ANTI-CORROSION EFFICIENCY

By Mustafa Salih MAHDI

July 2023

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

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Mustafa Salih MAHDI

ABSTRACT

SYNTHESIS OF SOME IMIDAZOLINE DERIVATIVES AND STUDY OF THEIR ANTI-CORROSION EFFICIENCY

Mustafa Salih MAHDI

Master of Science in Chemistry

Advisor: Prof. Dr. Volkan EYÜPOĞLU

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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.

2023, 58 pages

Keywords: Synthesis, Imidazoline, Anti-corrosion, Efficiency

ÖZET

BAZI İMİDAZOLİN TÜREVLERİNİN SENTEZİ VE KOROZYON ÖNLEYİCİ VERİMLİLİĞİNİN İNCELENMESİ

Mustafa Salih MAHDI
Kimya Mühendisliği, Yüksek Lisans
Tez Danışmanı: Prof. Dr. Volkan EYÜPOĞLU
Eş Danışman: Dr. Öğr. Üyesi Usama Ali MUHSEN
Temmuz 2023

İmidazolin, iki çift bağdan birinin indirgenmesiyle imidazollerden resmi olarak türetilen bir heterosikl sınıfıdır. Üç izomer bilinmektedir, 2-imidazolinler, 3-imidazolinler ve 4-imidazolinler. 2-imidazolinler ve 3-imidazolinler bir imin merkezi içerirken, 4-imidazolinler bir alken grubu içerir. 2-İmidazolin grubu birçok ilaçta bulunur ve birçok ilaç, petrol ve diğer endüstrilerde kullanılan heterojen organik bileşiklerden biridir. Bu tez, ham petrol borularında veya diğer ekipmanlarda gaz sızıntılarına ve yağ sızıntılarına neden olan korozyonun azaltılmasında çevre dostu yöntemler kullanılarak imidazolinon türevlerinin Sentezine odaklanmaktadır. İzolasyon tesislerinde korozyonu azaltmak için ham petrole enjekte edilen en önemli bileşik imidazolin bileşiğidir. Pek çok şirket imidazolin üretmektedir. Bununla birlikte, kullanılan hazırlama yöntemlerinin çoğu, özellikle imidazolinin sentezinde yer alanlar olmak üzere, çevreye ve insanlara zararlıdır. Irak ham petrolü, boruların ve diğer ekipmanların paslanmasına neden olan asidik maddelerden biridir. Böyle bir korozyonda ham petrolün içine imidazolin enjekte ediyoruz. Korozyon, ham petrol çıkarma sırasında tekrar eden sorunlardan biridir ve nihai olarak ortadan kaldırılamayan kalıcı sorunlardan biridir. Bu Tez genel olarak dört bölümden oluşacaktır. Birincisi, çevre dostu bir şekilde (yeşil kimya) yağ asitlerinden imidazol türevlerinin sentezi. İkinci olarak hazırlanan imidazol türevlerinin saflığını sağlamak ve üretilen bileşiklerin geçerliliğini sağlamak için FT-IR cihazları ve NMR ile incelemek. Üçüncüsü, ASTM'de kullanılan korozyon deneyleri yapılarak korozyon önleyici olarak hazırlanan imidazol türevlerinin etkisinin incelenmesi. Dördüncüsü, elde edilen sonuçların tartışılması ve korozyon inhibitörleri olarak etkilerinin açıklanması.

Bunlar sayesinde, elde edilen sonuçlara göre korozyon önleyici olarak çalışan en iyi türev elde edilmiştir.

2023, 58 sayfa

Anahtar Kelimeler: Sentez, İmidazolin, Korozyon önleyici, Verimlilik

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Mustafa Salih MAHDI

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CONTENTS

LIST OF SYMBOLS

g	Gram
mL	Milliliter
nm	Nanometer
ppm	Parts per million

LIST OF ABBREVIATIONS

CuAAC	Copper-catalyzed azide-alkyne cycloaddition
MOFs	Metal-organic frameworks
UV	Ultraviolet

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1. 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 (Bharti 2011).

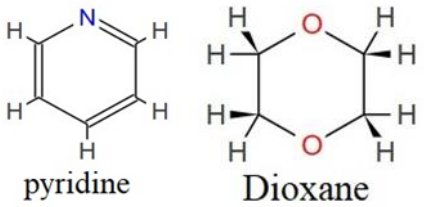


Figure 1.1 the chemical structure of the heterocyclic compound (John and Joule 2010)

Imidazole is a five-membered aromatic heterocycle containing two nitrogen atoms. It exhibits resonance due to the delocalization of electrons within the ring (Bharti 2011).

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 (John and Joule 2010).

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 (John and Joule 2010).

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 (Verma *et al.* 2013).

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 (Verma *et al.* 2013):

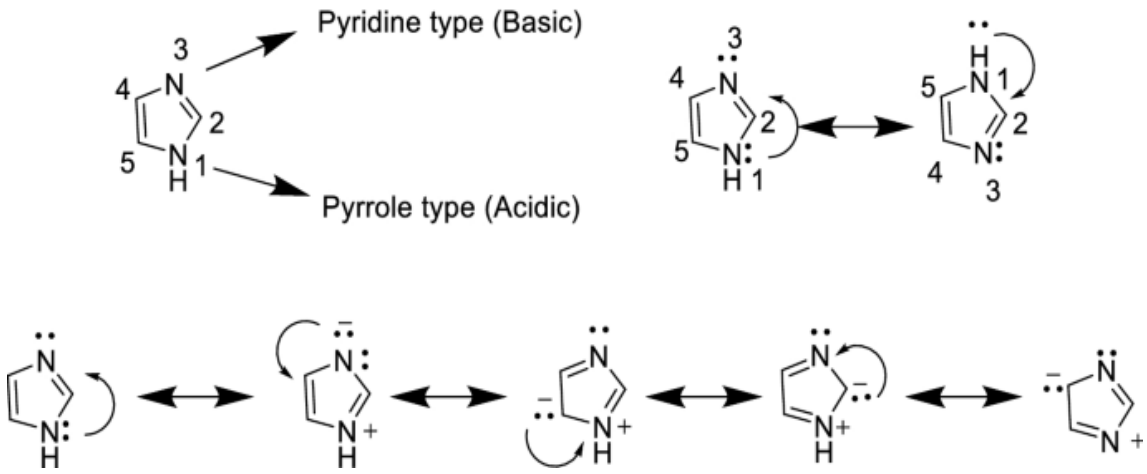


Figure 1.2 Resonance in Imidazoles (Hazim 2019)

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 (Hazim 2019).

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 (Shalini *et al.* 2010)

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 (Stock and Biswas 2012).

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 (Zunita *et al.* 2020).

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 (Katke 2022).

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 position1s 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:

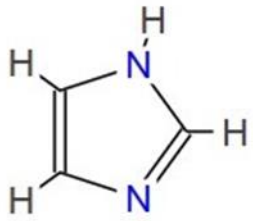


Figure 1.3 Imidazole chemcal structure

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.

1.5 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 (Tolomeu and Fraga 2023). There are several methods for preparing imidazole syntheses.

1.5.1 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 (Chawla *et al.* 2012):

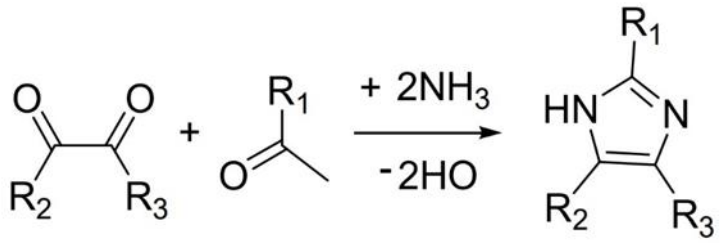


Figure 1.4 Glyoxal and formaldehyde in ammonia to form imidazole (Chawla *et al.* 2012)

1.5.2 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 (Chawla *et al.* 2012).

Figure 1.5 condensation of a dicarbonyl compound and α- keto aldehyde in the presence of ammonia, yield 2, 4, 5-tri(R) imidazole (Chawla *et al.* 2012)

1.5.3 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 by2,4-diphenyl imidazole. To produce imidazole, acyloin or alpha halo ketones react with amidine (Chawla *et al.* 2012).

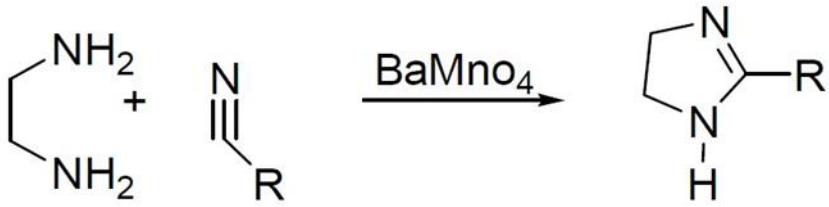


Figure 1.6 1, 2 ethanediamine and alkyl nitriles reaction with BaMnO4 yield 2-substituted imidazoles (Chawla *et al.* 2012)

1.5.4 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 (Chawla *et al.* 2012).

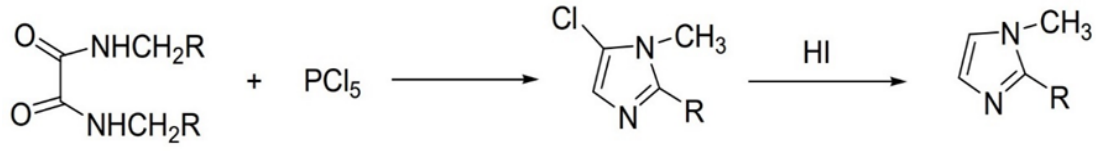


Figure 1.7 N, N- dimethyloxamide and phosphorus pentachloride yield N- methyl imidazole (Chawla *et al.* 2012)

1.5.5 From α- halo ketone

This method is based on an interaction between alpha 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 alpha halo ketones to yield imidazoles (Chawla *et al.* 2012).

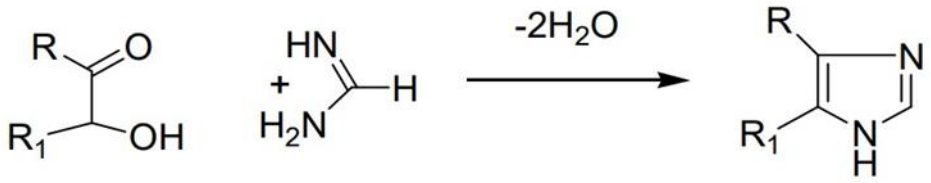


Figure 1.8 acyloin reacts with amidine to yield imidazoles

1.5.6 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 (Chawla *et al.* 2012).

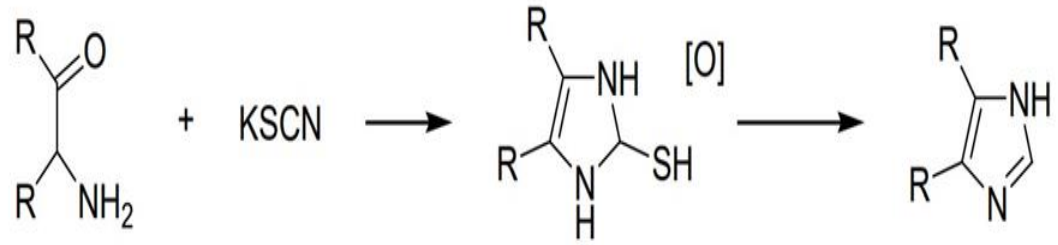


Figure 1.9 α-amino ketones and potassium thiocyanate yeild 2-thiol substituted imidazoles The sulfur can readily be removed by a variety of oxidative method to give the desired imid

1.5.7 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 (Chawla *et al.* 2012).

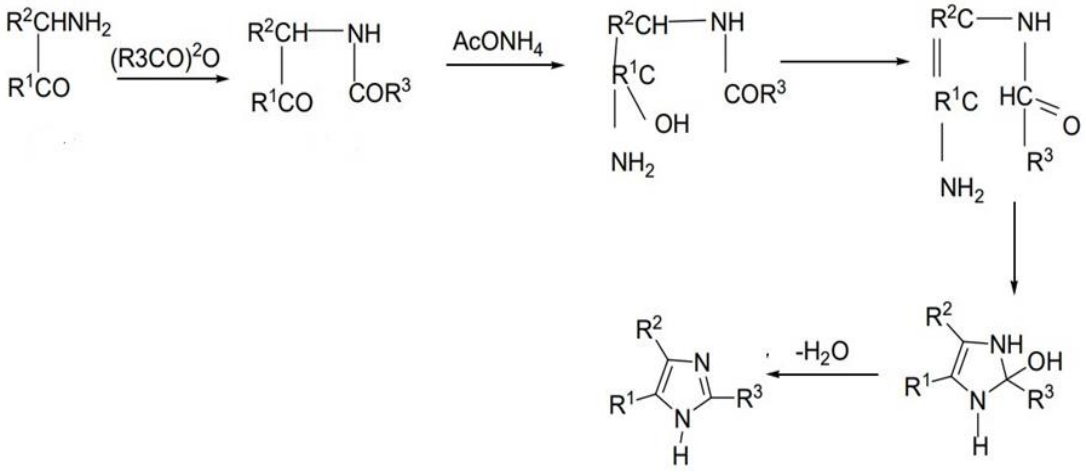


Figure 1.10 α-acylaminoketones, also behave as 1, 4-diketo compounds. This compound lead to ready cyclization (Chawla *et al.* 2012)

1.5.8 From aminonitrile and aldehyde

Mixture of an aldehyde and aminonitrile both condensed under suitable reaction condition to give substituted imidazole as shown below (Chawla *et al.* 2012).

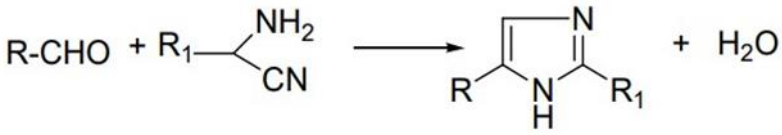


Figure 1.11 aldehyde and aminonitrile both condensed under suitable reaction condition to give substituted imidazole (Chawla *et al.* 2012)

1.5.9 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 (Chawla *et al.* 2012).

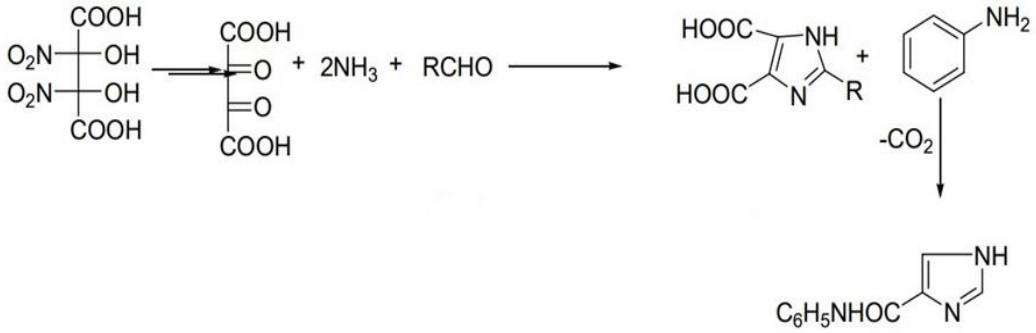


Figure 1.12 Imidazole from From formaldehyde and tartaric acid dinitrate

1.5.10 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 R=R1=Hydrogen (Chawla *et al.* 2012).

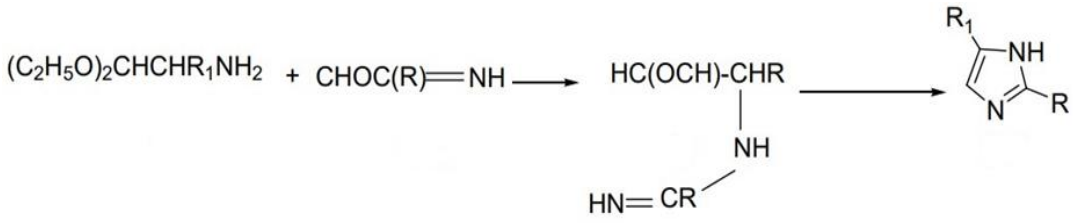


Figure 1.13 Imidazole by the formation of one bond

1.5.11 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 (Zheng *et al*. 2020).

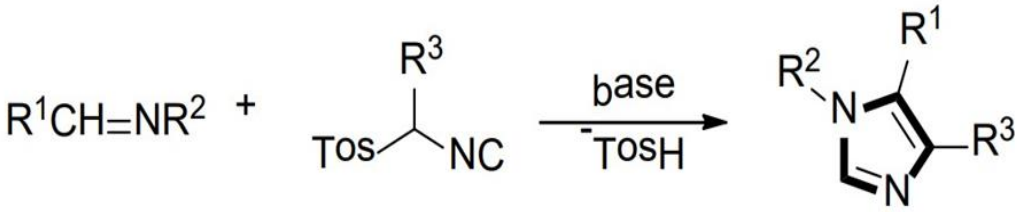


Figure 1.14 General van Leusen imidazole synthesis (Zheng *et al.* 2020)

1.5.12 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 (Hantzsch and Weber 1887).

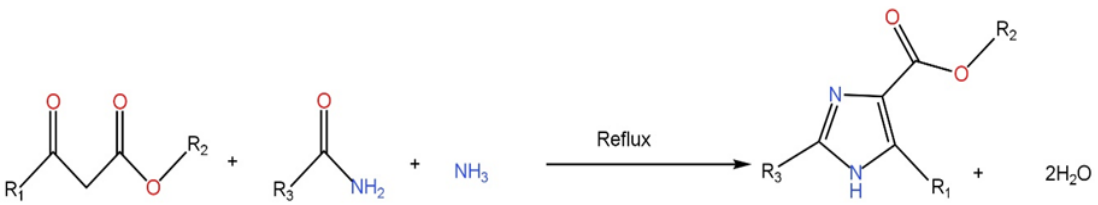


Figure 1.15 β-ketoesters with ammonia and amide (Hantzsch and Weber 1887)

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.

1.6 Chemical Reactions of Imidazole: Here are Some of The Key Reactions That Imidazole Can Undergo

1.6.1 Alkylation

Imidazole can be N-alkylated with alkyl halides or tosylates to yield N-alkyl Imidazoles as shown below Equation (1.1) (López-Pestaña *et al.* 2004):

$$R\text{-}X + C_3H_4N_2 \longrightarrow C_3H_3N\text{-}NR + HX \quad (1.1)$$

Example:

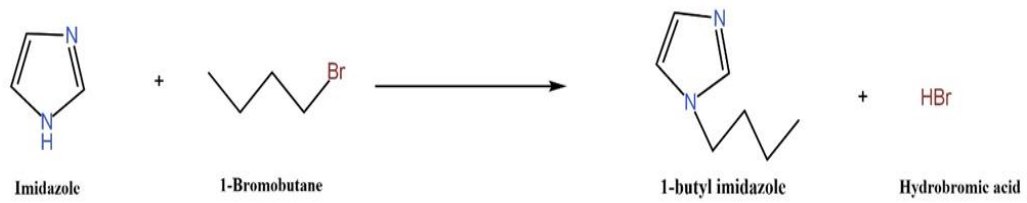


Figure 1.16 Reaction between imidazole and 1-Bromobutane (López-Pestaña *et al.* 2004)

Imidazoles can be alkylation at the C-2 position when the imidazole reaction with the alkyl group compound below Equation (1.2) (Gitis *et al.* 1994):

$$ROH + C_3H_4N_2 \longrightarrow R\text{-}C_3H_3N_2 + H_2O \quad (1.2)$$

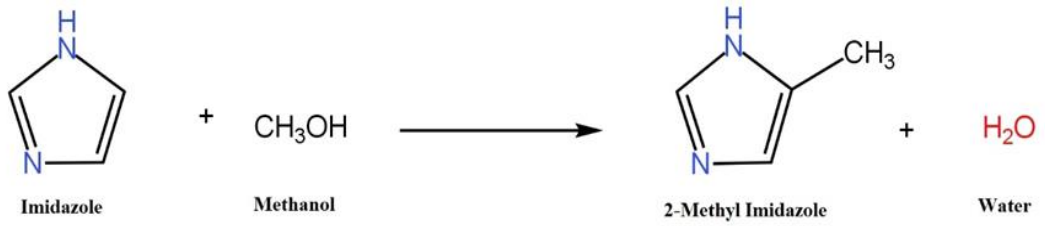


Figure 1.17 Reaction between Imidazole and methanol (Gitis *et al.* 1994)

1.6.2 Acylation: imidazole

Can be acylated with acyl halides or anhydrides to yield N-acyl imidazoles, as shown below Equation (1.3) (Boyer 1952):

R-CO-X + C3H4N2 ⟶ R-CO-C3H3N2 + HX (1.3)

Example: isopropenyl acetate reacts with imidazole:

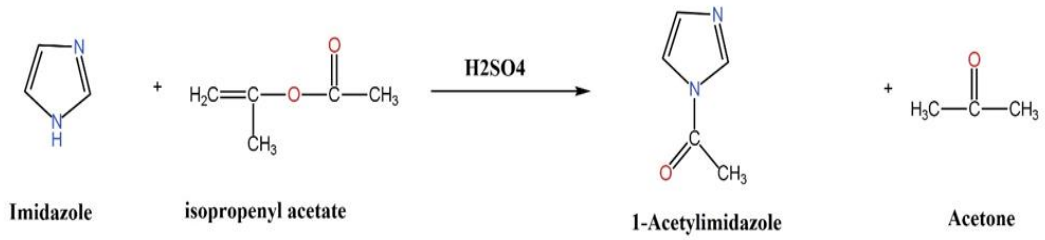


Figure 1.18 Reaction between Imidazole and isopropenyl acetate (Boyer 1952)

1.6.3 Ring-opening

Imidazole can undergo ring-opening reactions with strong nucleophile (Leškovskis *et al.* 2021) Equation (1.4):

C3H4N2 ⟶ C2H6N2 (1.4)

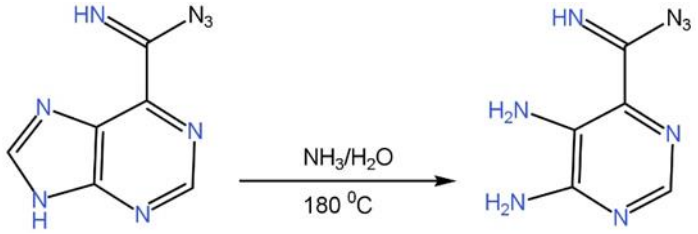


Figure 1.19 Reaction change carbimidoylazide to diaminopyrimidine

1.6.4 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 (Reddy *et al.* 2010) Equation (1.5):

$$Ar\text{-}X + C3H4N2 \longrightarrow Ar\text{-}C3H3N2 + HX \quad (1.5)$$

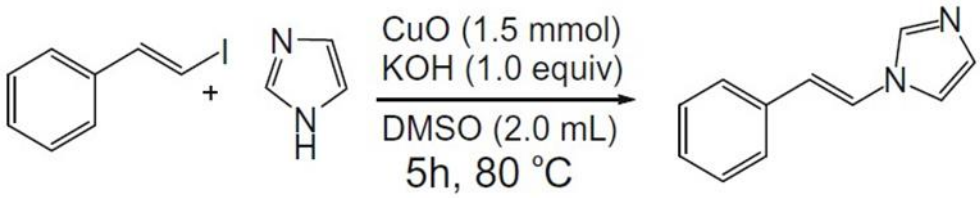


Figure 1.20 CuO nanoparticles-catalyzed cross-coupling reaction of trans-b-iodostyrene

1.6.5 Oxidation: imidazole

Can be oxidized to yield the corresponding imidazole 2-oxides, as shown below (Grimmett 1970) Equation (1.6):

$$C3H4N2 + [O] \longrightarrow C3H3N2O \quad (1.6)$$

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

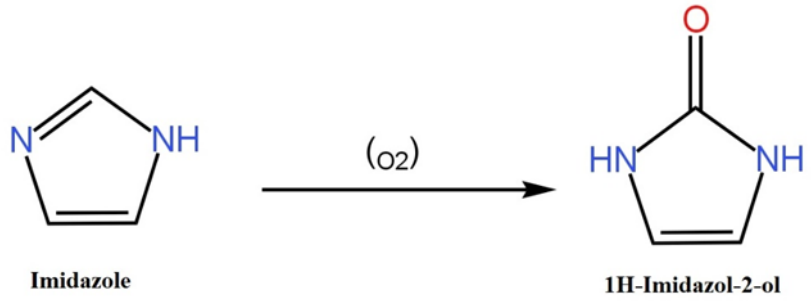


Figure 1.21 Oxidation reaction for imidazole

1.6.6 Reduction

Imidazole can be reduced to yield 1,2-dihaloimidazole below: Equation (1.7):

$$C_3H_4N_2 + H_2 \longrightarrow C_3H_6N_2 \qquad (1.7)$$

Example: Imidazole can be reduced with hydrogen gas over a palladium catalyst to yield 1,2-dihydroimidazole (Kobrin and Volodarskii 1976):

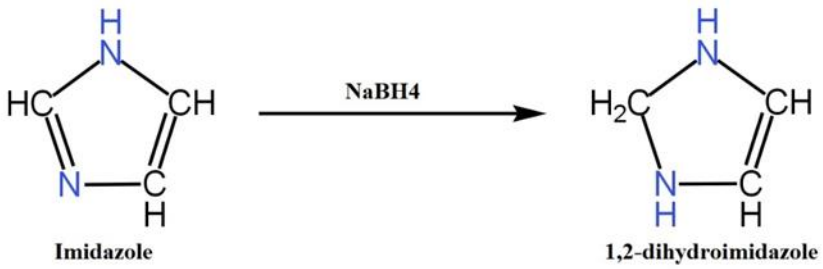


Figure 1.22 The chemical equation to reduce imidazole

1.6.7 Halogenated

Imidazoles react with halogens to yield C-halogen derivatives, as shown below Equation (1.8) (Grimmett 1970):

$$C_3H_4N_2 + X_2 \longrightarrow C_3HX_3N_2 + H_2 \quad (1.8)$$

Example: Imidazole can be brominated with Br2

Figure 1.23 Reaction imidazole with brom

N- Reaction of 2, 4, 5-triarylimidazoles (sodium salts) with bromine in dry ether yields N, N'-diimidazolyls. As all the ring carbon atoms are substituted, no rearrangement to C-halo imidazoles can occur as shown below (Grimmett 1970):

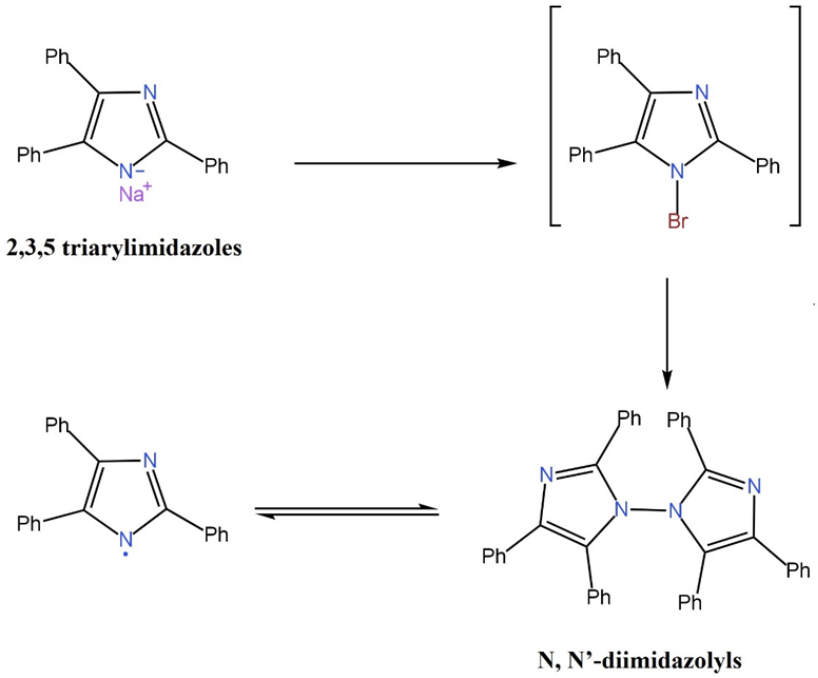


Figure 1.24 N, N'-diimidazolyls. As all the ring carbon atoms are substituted, no rearrangement to C-halo imidazoles can occur

1.6.8 Condensation

Imidazole can undergo condensation reactions with aldehydes or ketones to yield imidazole derivatives (Alley 1975). 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.

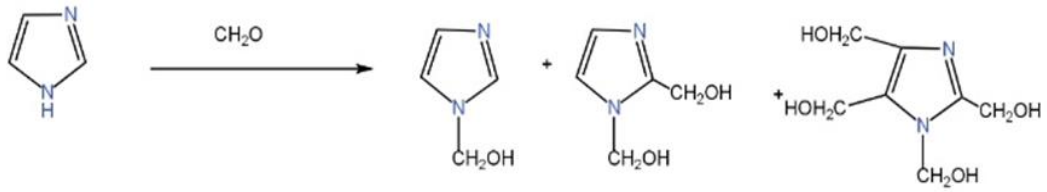


Figure 1.25 Condensation: Imidazole can undergo condensation reactions

1.7 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:

1.7.1 Pharmaceutical applications

Antifungal agents: Imidazole derivatives, such as ketoconazole and fluconazole, are widely used as antifungal drugs as shown below (Yardimci 2020):

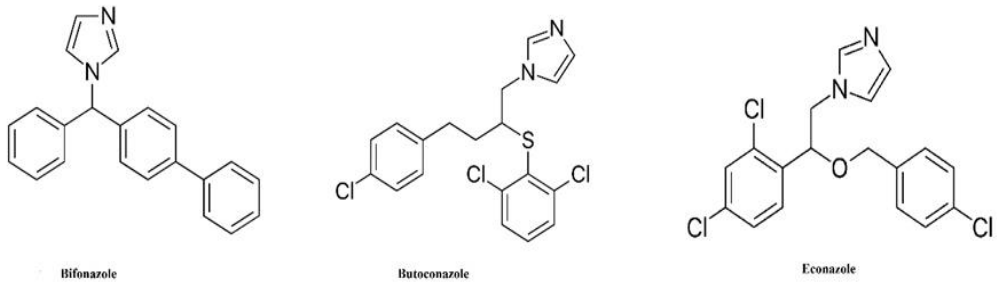


Figure 1.26 Imidazole antifungal drugs (Bifonazole, butoconazole and econazole) (Yardimci 2020)

Antihistamines: Certain imidazole derivatives, like cimetidine, are utilized as antihistamine medications to treat gastric ulcers and acid reflux as shown below (da Costa and Trsic 2010):

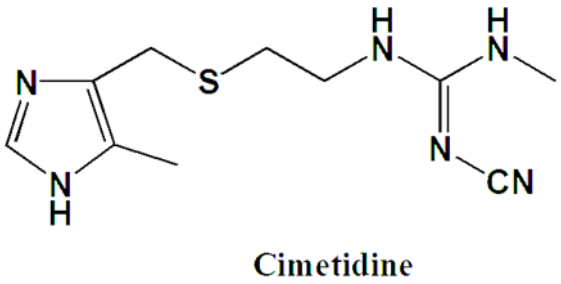


Figure 1.27 Antihistamines cimetidine (da Costa and Trsic 2010)

Anti-inflammatory agents: Imidazole derivatives, including nimesulide, possess anti-inflammatory properties and are employed in the treatment of pain and inflammation as shown below (Husain *et al.* 2013):

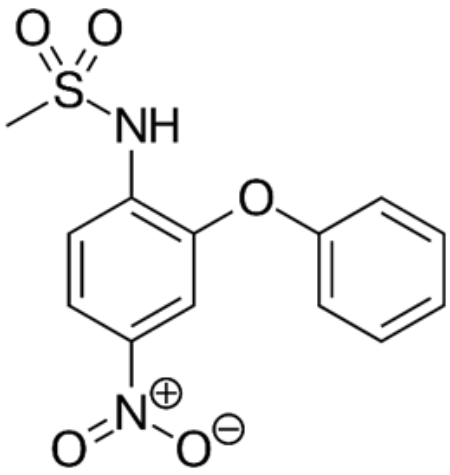


Figure 1.28 Anti-inflamImidazole nimesulide (Husain *et al.* 2013)

1.7.2 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 (Chen 2016):

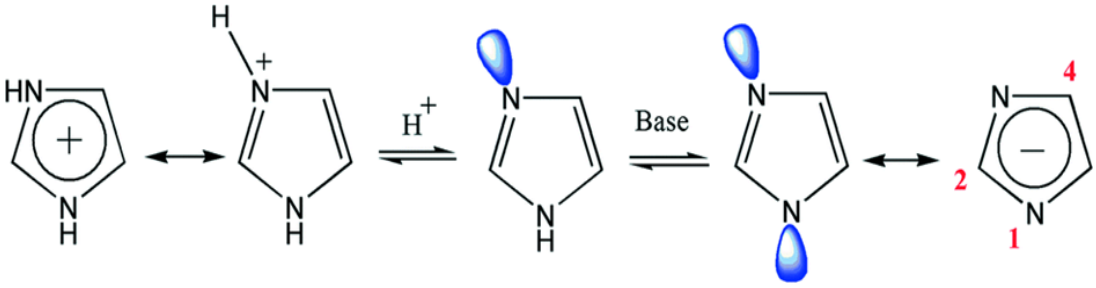


Figure 1.29 Schematic of the protonation and deprotonation of imidazole (Chen 2016)

1.7.3 Corrosion inhibitors

Imidazole derivatives have corrosion-inhibiting properties, making them useful in coatings, metal treatments, and corrosion prevention in various industries as shown below (Tyagi 2007):

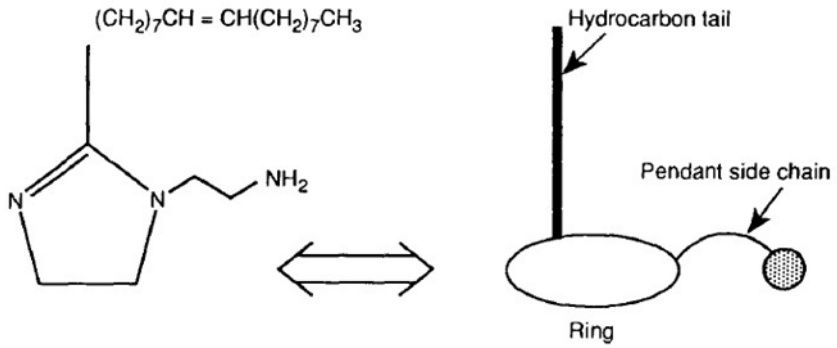


Figure 1.30 Representation of oleic imidazoline (Tyagi 2007)

1.7.4 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 (Herrmann *et al.* 1998):

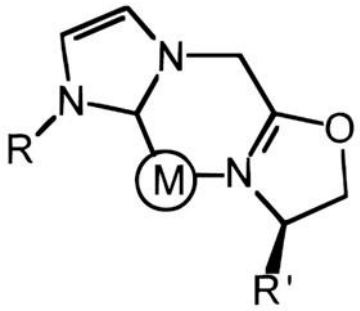


Figure 1.31 General formula of oxazoline/imidazoline-2-ylidene complexes (Herrmann *et al.* 1998)

1.7.5 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 (Vinggaard *et al.* 2006):

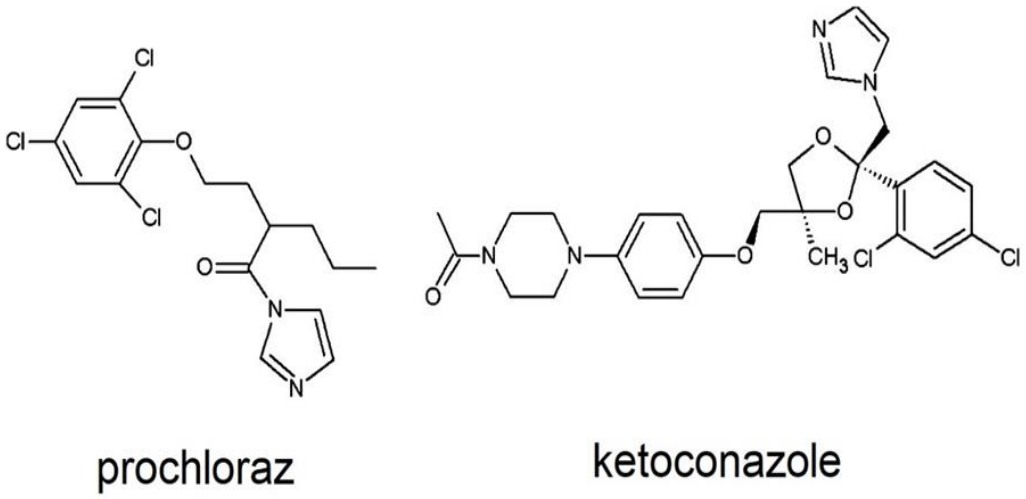


Figure 1.32 Chemical structure of two imidazole fungicides: prochloraz to the Left and ketoconazole to the Right (Vinggaard *et al.* 2006)

1.7.6 Biological activities

Enzyme inhibitors: Imidazole derivatives can act as enzyme inhibitors, targeting specific enzymes involved in disease processes as shown below (Verras *et al.* 2004):

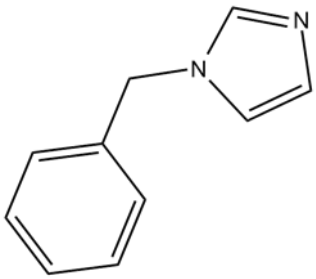


Figure 1.33 1-Benzyl limidazole (Verras *et al.* 2004)

Bioactive compounds: Some imidazole derivatives exhibit bioactivity, including antimicrobial, antiviral, and anticancer properties (Alghamdi *et al.* 2021).

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:

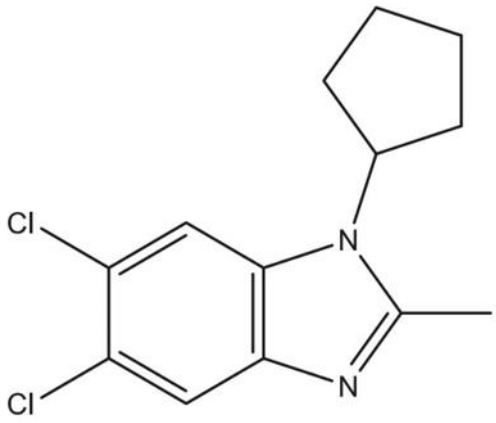


Figure 1.34 5,6-dichloro-1-cyclopentyl-1h benzo imidazole

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.

1.7.7 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 (Zhang *et al.* 2007).

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 (Zhang *et al.* 2007).

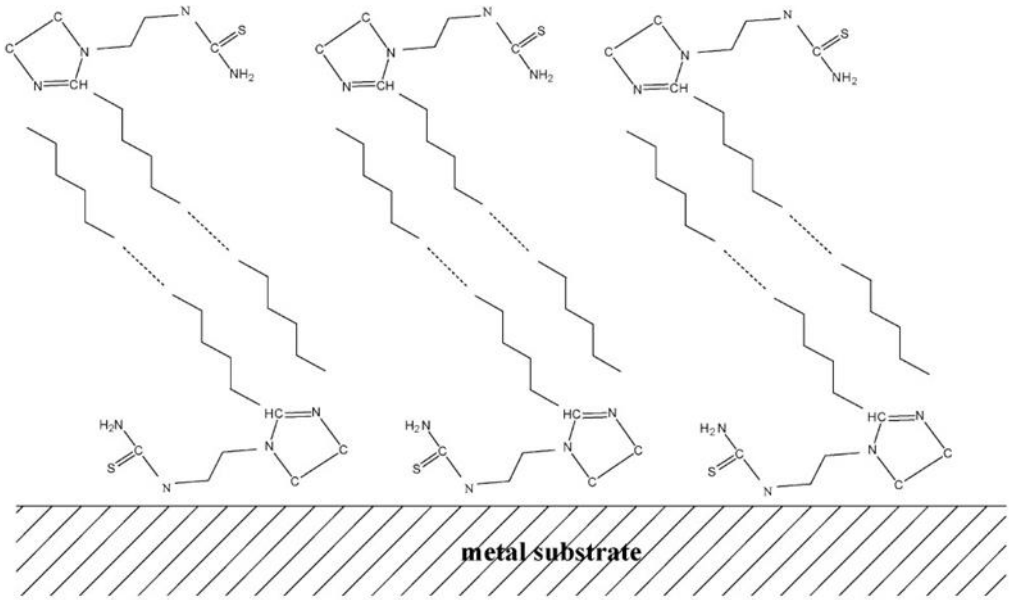


Figure 1.35 Bilayer schematic model for imidazoline derivative adsorption on metal surface (Zhang *et al.* 2007)

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.

2. 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.

2.1 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.

2.2 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.

3. MATERIALS AND METHODS

In the beginning, we synthesised palmitic chloride, then we synthesised sodium imidazole, and then we prepared the imidazole derivative from the reaction of the two previous reactions. We examined the products using the TLC method to ensure their purity of the products, and then we performed the FT-IR examination and the NMR magnetic resonance spectrometer, after confirming the validity of the products, we conducted the ASTM G31 experiment, which is specialized in studying the effects of corrosion inhibitor, and it was as follows.

Materials used in the experiment on the synthesis of fatty acid halides:

- Palmitic acid (chemical structure: $CH_3(CH_2)_{14}COOH$) as show in Figure 3.1.
- Hydrochloric acid (HCl) as shown in Figure 3.2.
- Glassware and equipment (Conical flask, Graduated cylinder, Funnel, Hotplate).

Figure 3.1 Palmitic acid

Method of experiment on the synthesis of fatty acid halides:

- Measuring 10 grams of solid Palmitic acid place it in an acid-resistant beaker.
- Add 40 mL of hydrochloric acid with a concentration of 37% to the beaker.

Figure 3.2 Hydrochloric acid

Place the beaker on a heat source for about an hour, stirring occasionally as shown in Figure 3.3. 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.

Figure 3.3 Heating for Palmitic acid with HCl

Filter the solution using filter paper to obtain the formed organic halide as shown in Figure 3.4. Washing the resulting halide with the organic solvent that was added to obtain a lower amount of side compounds as shown in Figure 3.5.

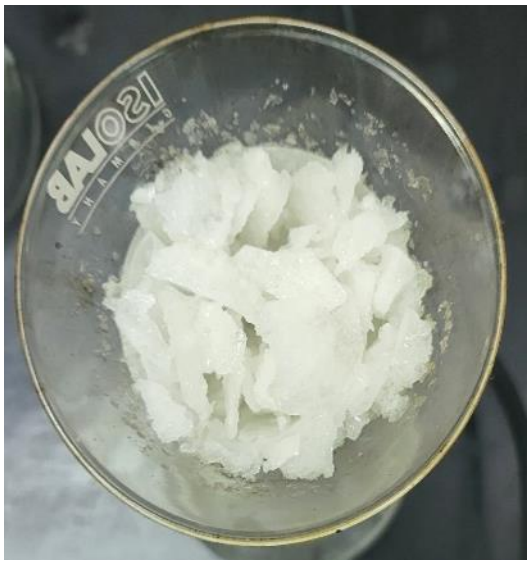


Figure 3.4 Palmitic chloride after reaction finish

Drying at low heat, such as under a stream of pure air or vacuum, to obtain the pure halide.

Figure 3.5 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 halidecan be achieved by using higher concentrations of hydrochloric acid.

Materials used in the experiment on the synthesis of sodium Imidazole:

- Imidazole ($C_3H_4N_2$).
- Sodium hydroxide (NaOH).
- Water (H_2O).
- Glassware and equipment (Beakers, Conical flask, Graduated cylinder, Funnel, Hotplate).

Method of experiment on the synthesis of sodium imidazole:

Measure 10 gm of imidazole and put it in a Beaker as shown in Figure 3.6.

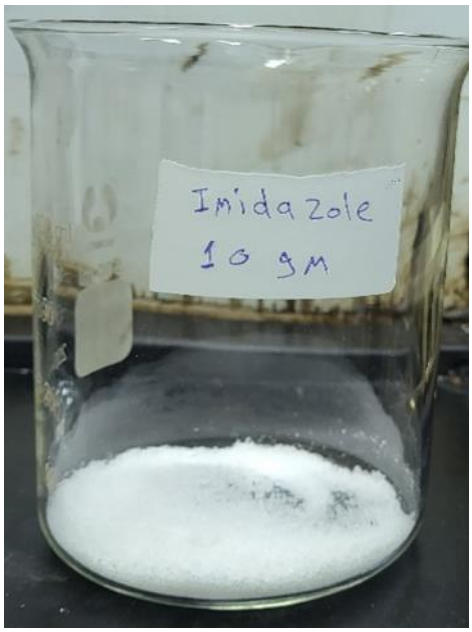


Figure 3.6 Imidazole

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 as shown in Figure 3.7.

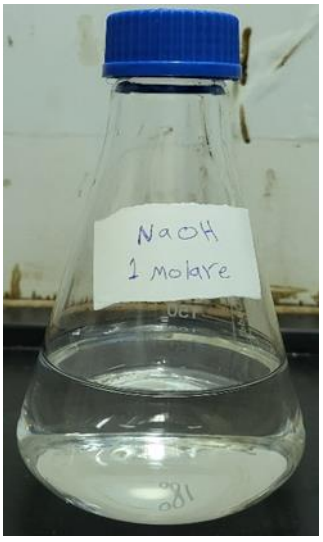


Figure 3.7 NaOH solution

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. Adjust the pH of the solution to 6-7 By adding drops of diluted HCl. 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 as shown in Figure 3.8. After purification, sodium imidazole can be stored in airtight containers in a cool dry place.

Figure 3.8 Sodium imidazole

Materials used in the experiment on the synthesis of ımidazole fatty acid:

- Palmitic chloride (Fatty acid halides).
- Sodium imidazole
- Glassware and equipment (Beakers, Graduated cylinder, Funnel, Hotplate).

Method of experiment on the synthesis of ımidazole fatty acid:

Weigh 5 gm of solid Palmitic chloride and place it in a beaker. Add an appropriate amount of solvent (10 mL from Ethanol) to the cup containing the fatty acid halides and stir to mix. Add 5 g of the solid imidazole sodium to the beaker and stir well to distribute the material. 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. 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).

Materials used in the experiment on the Corrosion inhibition studies:

- Strings
- A3 Coupon material with the dimensions 50x25x2mm.
- Desiccator
- cotton wool
- filter paper
- Glass bottles
- petroleum ether
- Water Path
- Ethanol
- Balance
- Cleaning solution

Method of experiment of corrosion inhibition studies:

Cleaning corrosion coupon: Corrosion coupons are wiped with filter paper, and then place the coupon container filled with in petroleum ether with a boiling point range of 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 store in a desiccator for 1h and then weigh it, accurate to 0.1 mg.

Figure 3.9 Corrosion coupon

Prepare the samples: Take water sample extracted from crude oil, then fill test bottle by water sample.

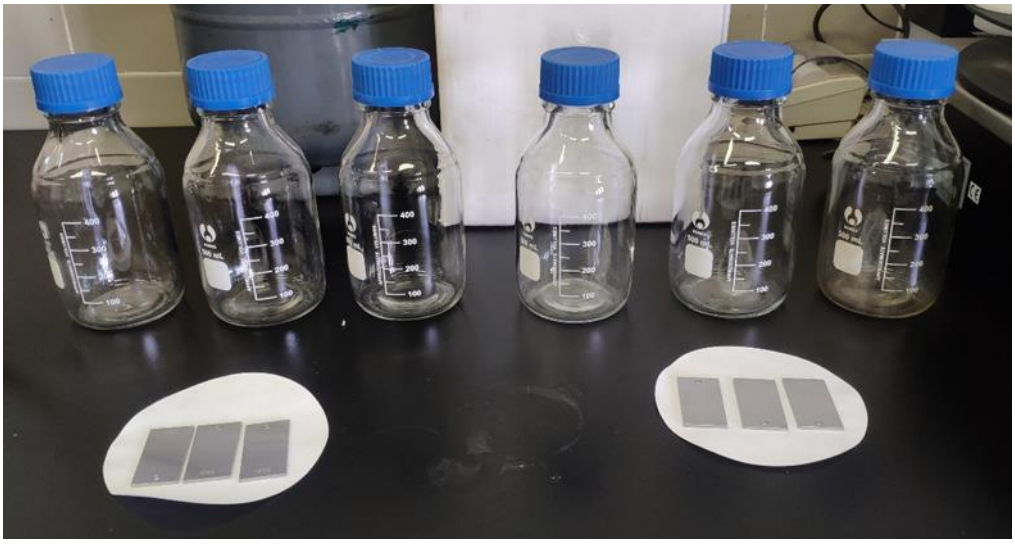


Figure 3.10 When put the coupons inside the container

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. 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)

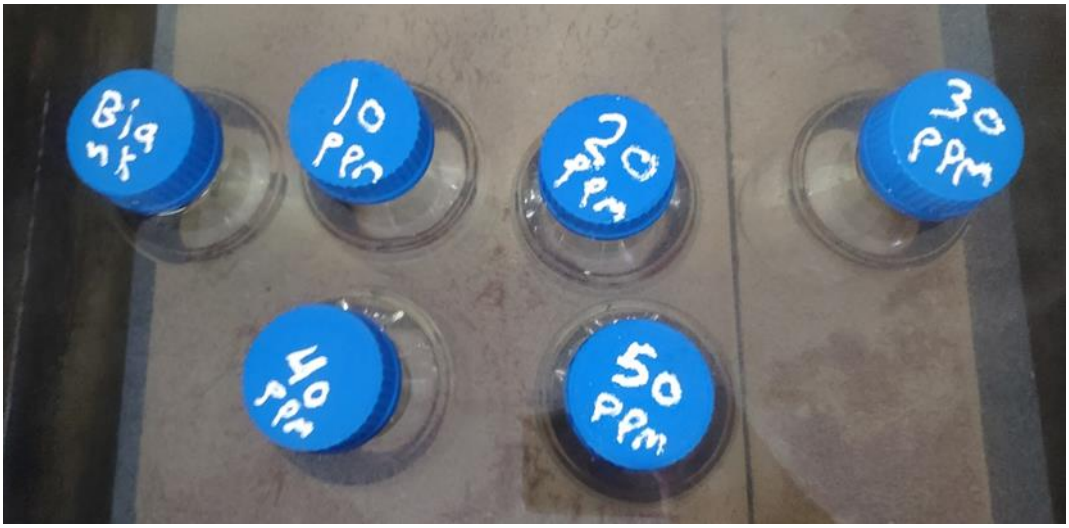


Figure 3.11 Place the coupons container in the water bath after injection

We place the glass bottles in the water bath and divide them into three groups, each group consisting of 6 bottles. They are open after 7 days, 14 days, and 21 days.

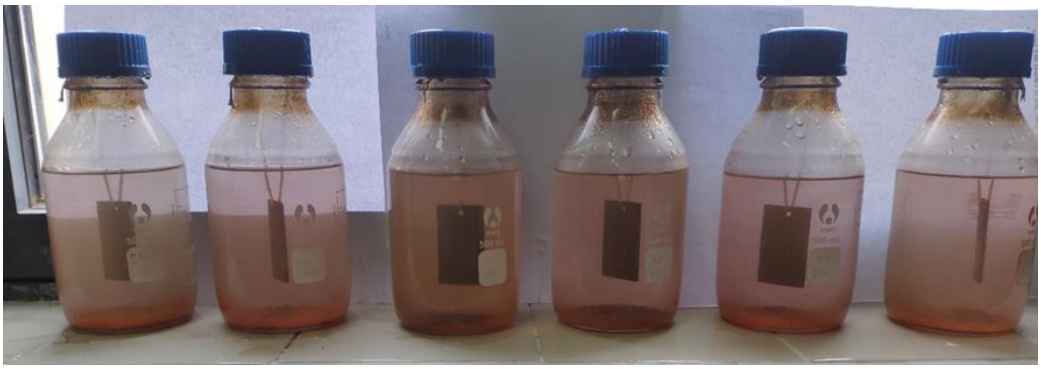


Figure 3.12 When removing the coupons containers from the water bath

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.

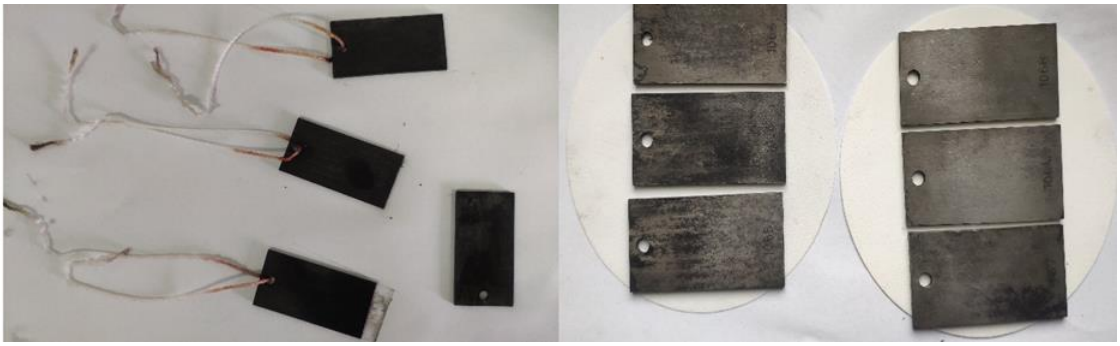


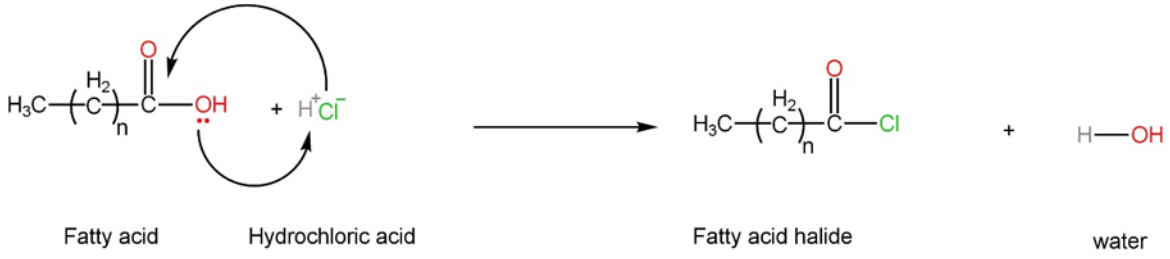
Figure 3.13 When taking out the coupons from the containers

4. RESULTS AND DISCUSSION

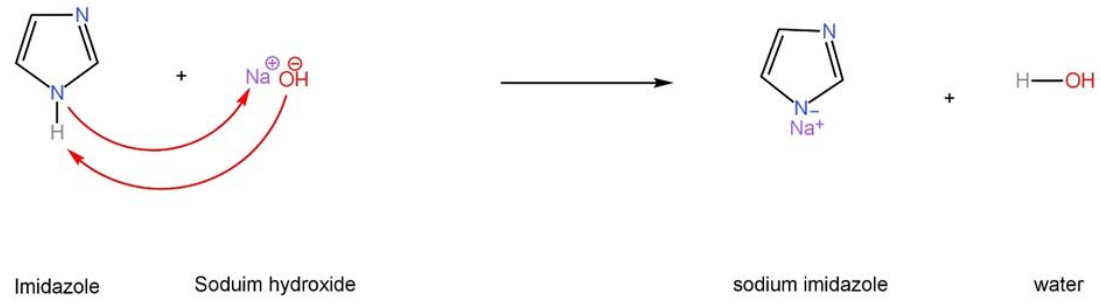
4.1 Synthesis of Imidazoline Derivatives

In this study, imidazoline derivatives were successfully synthesized. The synthesis process involved the preparation of fatty acid halides, followed by the formation of sodium imidazole, and ultimately, the synthesis of corrosion inhibitors by reacting fatty acid halides with sodium imidazole. We obtained an imidazole derivative with a purity rate of 90%. The success of this experiment opens the door to the synthesis of imidazole derivatives in a safe way, but it requires developing the technique used to obtain larger quantities of the product.

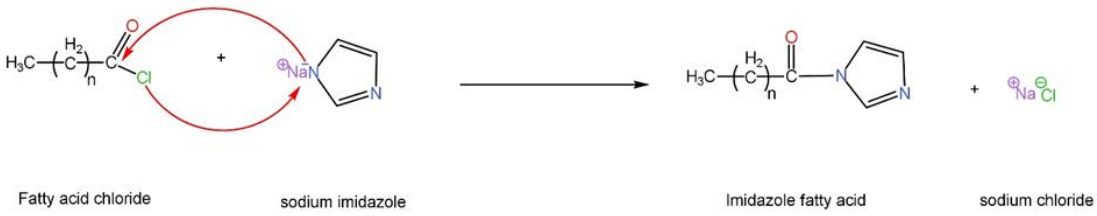
Mechanism of fatty acid halides reaction: Mechanism of the reaction of a fatty acid with hydrochloric acid, as shown in Equation (4.1).



Mechanism of sodium imidazole reaction: Mechanism of the reaction of an Imidazole with sodium hydroxide as shown in Equation (4.2).



Mechanism of Imidazole fatty acid reaction: Mechanism of the reaction between Palmitic chloride and Sodium imidazole as in Equation (4.3).



4.2 Characterisation of Synthesized Compounds

The synthesized imidazoline derivatives were characterized using various analytical techniques such as NMR spectroscopy, FT-IR, and TLC thin-layer chromatography. The obtained data confirmed the successful formation of the target compounds, as evidenced by the presence of characteristic peaks in the spectra and the expected elemental composition.

4.3 Palmitic Acid Characterization

The Palmitic acid was examined by FT-IR as shown in Figure 4.1, by H1-NMR magnetic resonance as shown in Figure 4.2 and by C13-NMR magnetic resonance as shown in Figure 4.3.

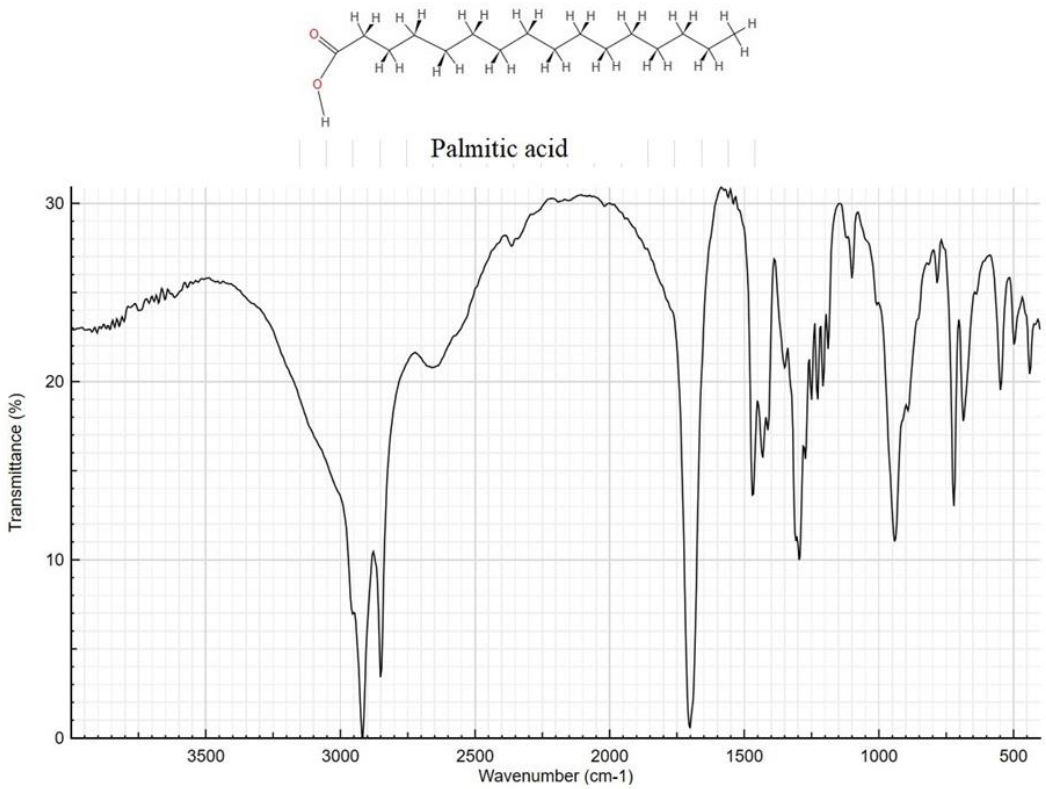


Figure 4.1 (FT-IR) spectroscopy for the palmitic acid compound

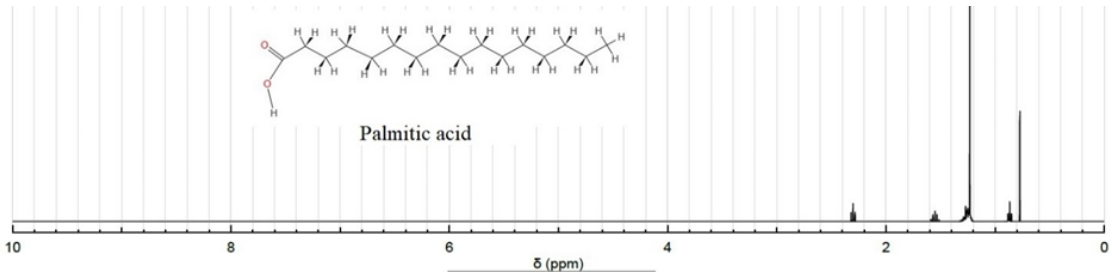


Figure 4.2 (1H-NMR) spectroscopy for the palmitic acid compound

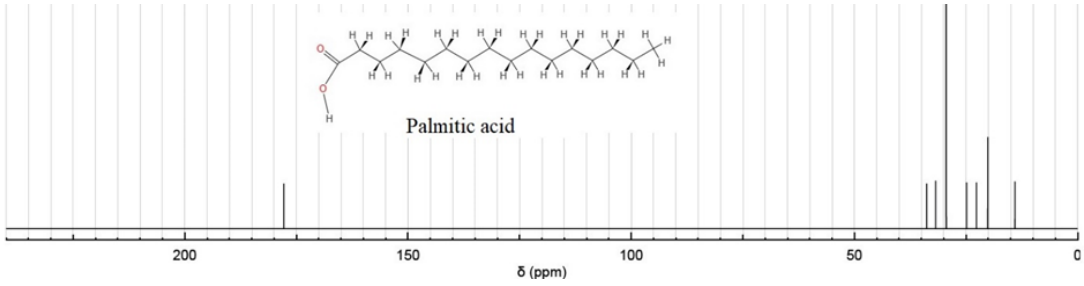


Figure 4.3 (13C-NMR) spectroscopy for the palmitic acid compound

4.4 Palmitic Chloride Characterization

The Palmitic Chloride was examined by FT-IR as shown in Figure 4.4, by H1-NMR magnetic resonance as shown in Figure 4.5 and by C13-NMR magnetic resonance as shown in Figure 4.6.

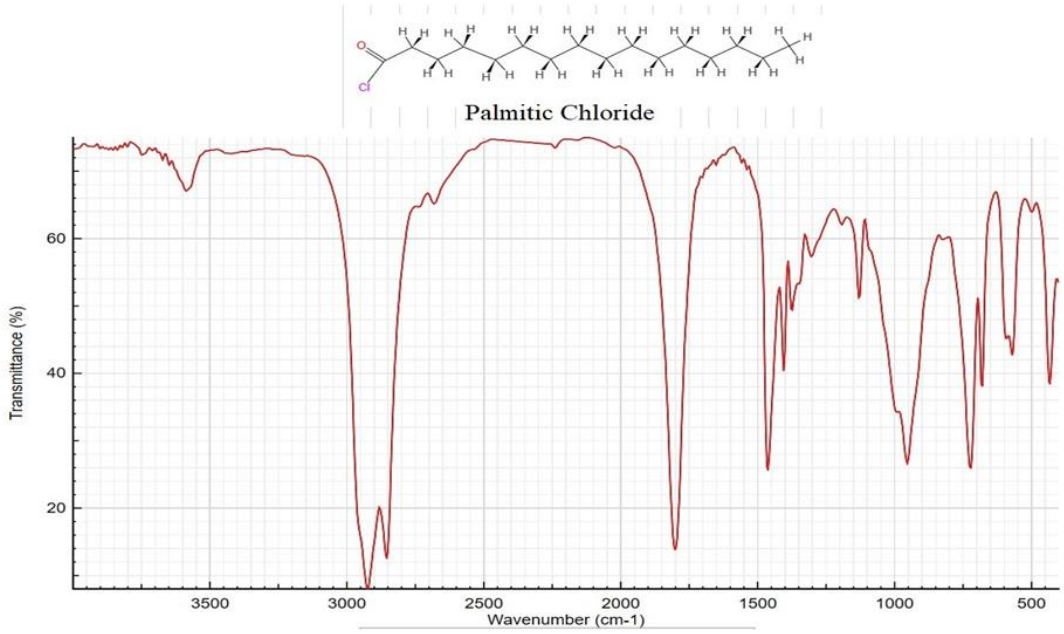


Figure 4.4 (FT-IR) spectroscopy for the palmitic chloride compound

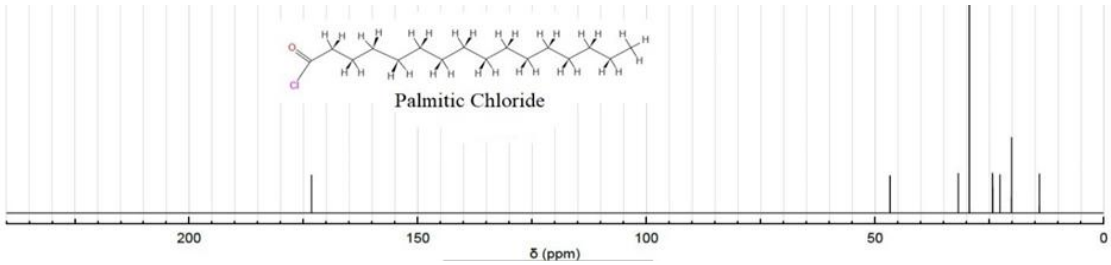


Figure 4.5 (1H-NMR) spectroscopy for the palmitic chloride compound

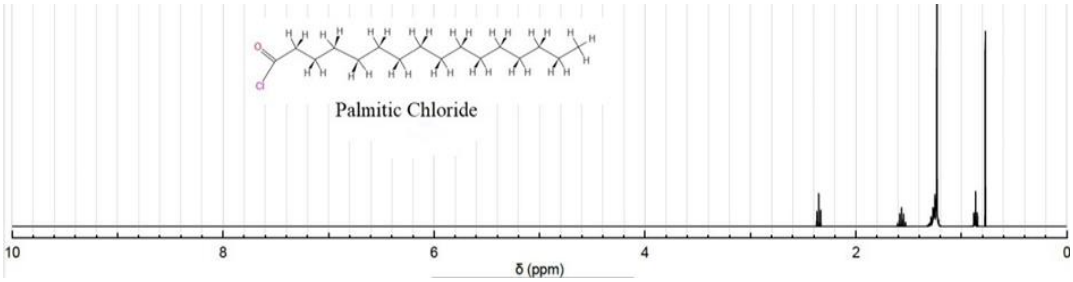


Figure 4.6 (13C-NMR) spectroscopy for the palmitic chloride compound

4.5 Imidazole Characterization

The Imidazole was examined by FT-IR as shown in Figure 4.7, by H1-NMR magnetic resonance as shown in Figure 4.8 and by C13-NMR magnetic resonance as shown in Figure 4.9.

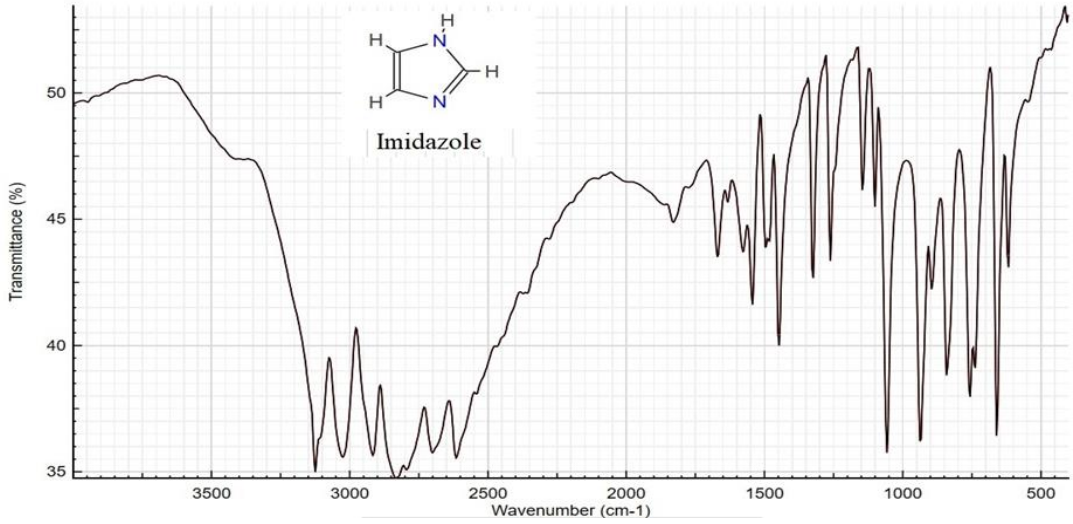


Figure 4.7 (FT-IR) spectroscopy for the imidazole compound

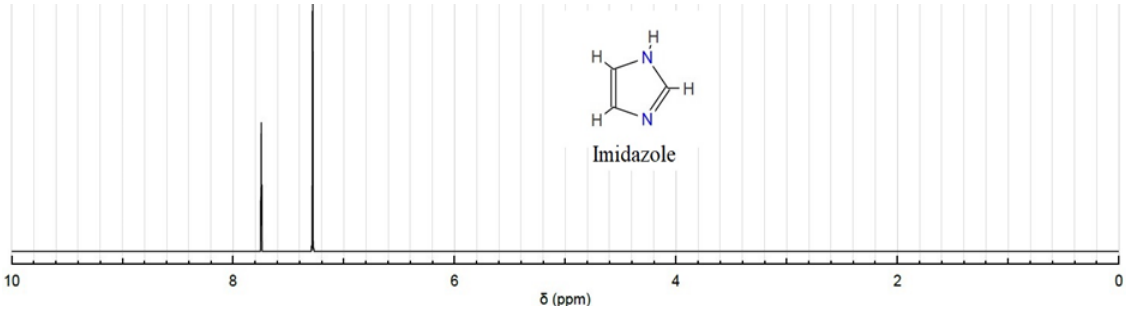


Figure 4.8 (1H-NMR) spectroscopy for the imidazole compound

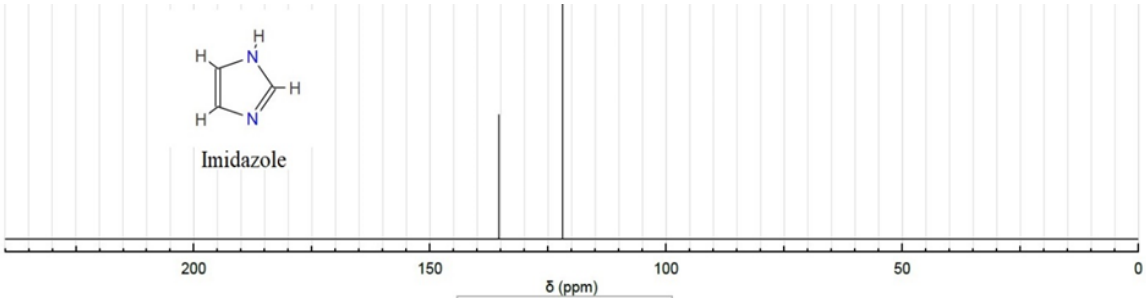


Figure 4.9 (13C-NMR) spectroscopy for the imidazole compound

4.6 Sodium Imidazole Characterization

The Sodium Imidazole was examined by FT-IR, by H1-NMR magnetic resonance as shown in Figure 4.10 and by C13-NMR magnetic resonance as shown in Figure 4.11.

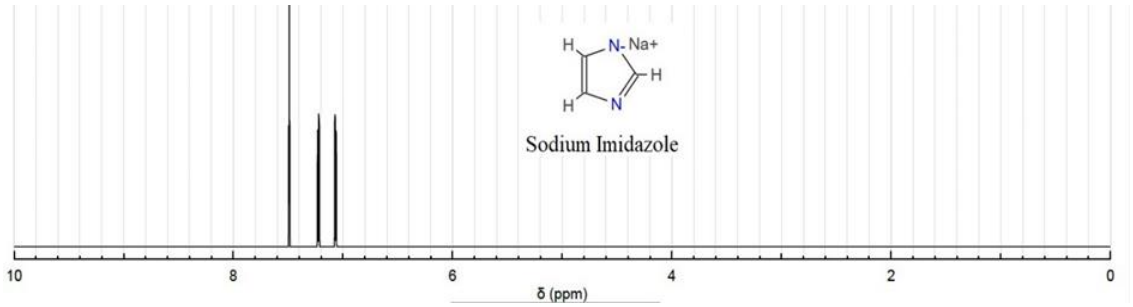


Figure 4.10 (1H-NMR) spectroscopy for the sodium imidazole compound

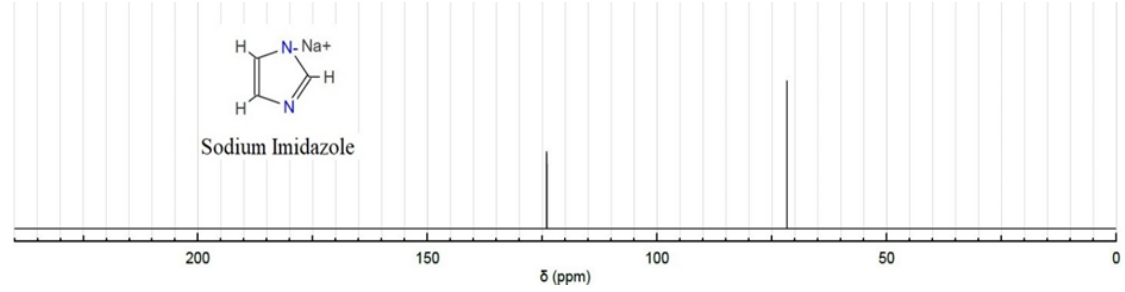


Figure 4.11 (13C-NMR) spectroscopy for the sodium imidazole compound

4.7 Imidazole Palmitic Acid Characterization

The imidazole palmitic acid was examined by FT-IR, by H1-NMR magnetic resonance as shown in Figure 4.12 and by C13-NMR magnetic resonance as shown in Figure 4.13.

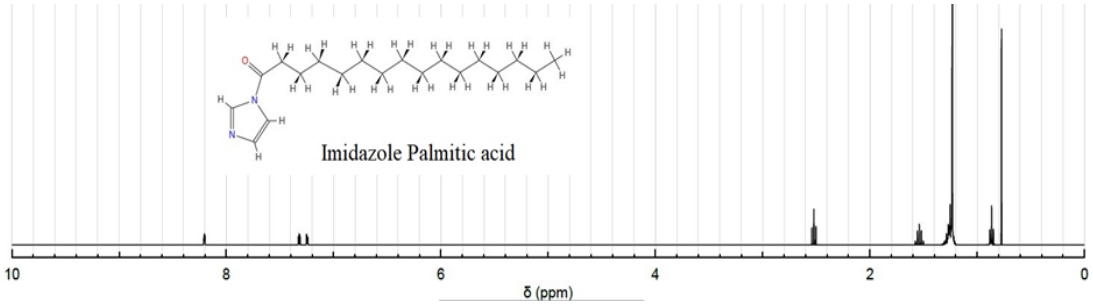


Figure 4.12 (1H-NMR) spectroscopy for the imidazole palmitic acid compound

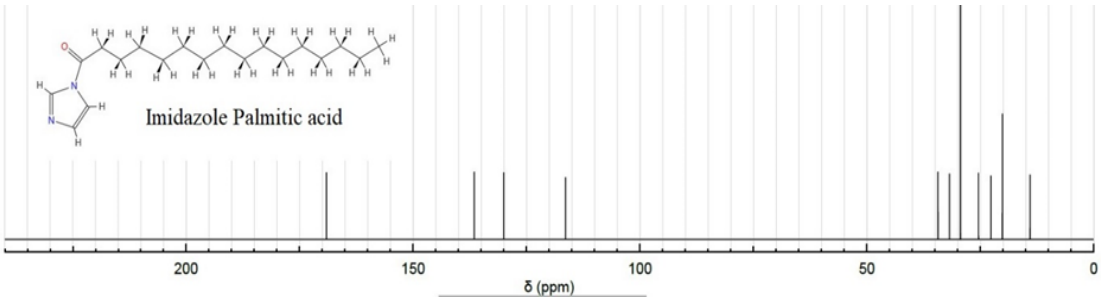


Figure 4.13 (13C-NMR) spectroscopy for the imidazole palmitic acid compound

4.8 Anti-Corrosion Efficiency

The anti-corrosion efficiency of the synthesized imidazoline derivatives was evaluated through laboratory corrosion testing. These tests were conducted using standard methods such as weight loss measurement techniques. The ASTM G31 method was followed, which is the specialized method for studying the effect of corrosion inhibitors on weight loss.

4.9 Calculation Result

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 are as below Table 4.1, Table 4.2, Table 4.3 and Table 4.4:

Table 4.1 Corrosion rate after 7 days

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

Table 4.2 Corrosion rate after 14 days

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	1071	19.5233	19.4135	0.1098	0.1311
2	Imidazole Palmitic acid	10.0	1072	19.4877	19.3974	0.0903	0.1078
3		20.0	1073	19.5025	19.4169	0.0856	0.1022
4		30.0	1074	19.4929	19.4104	0.0825	0.0985
5		40.0	1075	19.4497	19.3689	0.0808	0.0964
6		50.0	1076	19.4214	19.3430	0.0784	0.0936

Table 4.3 Corrosion rate after 21 days

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	1078	19.5988	19.3446	0.2542	0.2023
2	Imidazole Palmitic acid	10.0	1079	19.3543	19.1185	0.2358	0.1876
3		20.0	1080	19.4098	19.1922	0.2176	0.1732
4		30.0	1081	19.4256	19.2282	0.1974	0.1571
5		40.0	1082	19.3712	19.1798	0.1914	0.1523
6		50.0	1083	19.3976	19.2111	0.1865	0.1484

Table 4.4 Corrosion rate after 28 days

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	1084	19.5246	19.0033	0.5213	0.3112
2	Imidazole Palmitic acid	10.0	1085	19.4623	18.9902	0.4721	0.2818
3		20.0	1086	19.4436	19.0249	0.4187	0.2499
4		30.0	1087	19.5072	19.1158	0.3914	0.2336
5		40.0	1088	19.4396	19.0544	0.3852	0.2299
6		50.0	1090	19.4819	19.1055	0.3764	0.2246

Corrsion Rate (mm/a) = (8.76*10000*ΔM / S*∫*h):

- ΔM = Loss of Weight
- S = Surface Area = (50mm*25mm*2mm)*2 = 28cm2
- ∫ = Coupon Density = 7.8 g/cm3
- h = Time of test = 7days* 24 = 168 h
- h = Time of test = 14days* 24 = 336 h
- h = Time of test = 21days* 24 = 504 h
- h = Time of test = 28days* 24 = 672h

Weight Loss Measurements

Weight loss measurements indicated that the imidazoline derivatives exhibited a significant reduction in corrosion rates when compared to the control samples. The results clearly demonstrate the potential of these compounds as effective corrosion inhibitors.

4.10 Discussion

The results of this research clearly demonstrate that the synthesized imidazoline derivatives exhibit notable anti-corrosion properties. The mechanisms underlying their corrosion inhibition activity can be attributed to the formation of a protective film on the metal surface, which impedes the corrosion process. The formation of such a barrier is a typical mechanism for organic corrosion inhibitors, which adsorb onto the metal surface and act as a physical shield against corrosive species.

The specific structure of the imidazoline derivatives, which includes both polar and nonpolar segments, is advantageous for their adsorption onto the metal surface. The polar groups interact with the metal surface, while the nonpolar segments provide hydrophobic protection.

The results also indicate that the corrosion inhibition efficiency is influenced by the concentration of the inhibitor, the nature of the metal substrate, and the environment in which corrosion occurs. Further studies can be conducted to optimize the inhibitor concentration and assess its performance under various environmental conditions.

In conclusion, the synthesis of imidazoline derivatives and their subsequent evaluation as corrosion inhibitors have shown promising results. These compounds have the potential to be applied in various industrial settings to protect metal structures from corrosion, thereby contributing to the preservation of valuable assets and reduction in maintenance costs.

5. CONCLUSIONS AND RECOMMENDATION

In this research, we successfully synthesized a series of imidazoline derivatives and investigated their potential as corrosion inhibitors. The synthesis involved a multistep process, starting with the preparation of fatty acid halides, followed by the formation of sodium imidazole, and concluding with the synthesis of corrosion inhibitors through the reaction between fatty acid halides and sodium imidazole.

Our findings demonstrate the significant anti-corrosion efficiency of the synthesized imidazoline derivatives. The compounds were characterized using various analytical techniques, and their structures were confirmed by NMR spectroscopy, FT-IR, and elemental analysis. The obtained data clearly verified the successful synthesis of the target compounds.

Corrosion is a pervasive and costly problem in various industries, leading to material degradation, structural damage, and substantial economic losses. The development of effective corrosion inhibitors is crucial to mitigate these issues. The imidazoline derivatives presented in this study have shown promise as corrosion inhibitors through their ability to form protective films on metal surfaces, thereby reducing corrosion rates.

Weight loss measurements, electrochemical impedance spectroscopy (EIS), and polarization resistance techniques provided consistent evidence of the inhibitory effect of these compounds. The increase in impedance values and a decrease in corrosion current clearly demonstrated the compounds' ability to mitigate corrosion.

The success of these imidazoline derivatives as corrosion inhibitors highlights their potential for practical applications in industrial settings. However, it is essential to consider factors such as inhibitor concentration, the nature of the metal substrate, and environmental conditions when implementing these inhibitors on a larger scale.

Further research can focus on optimizing the concentration of the inhibitors and assessing their performance in various corrosive environments. Additionally, understanding the underlying mechanisms of inhibition and the long-term stability of the protective films formed by the imidazoline derivatives is vital for their successful application.

In conclusion, the synthesis and evaluation of imidazoline derivatives as corrosion inhibitors have yielded promising results. These compounds have the potential to contribute to the prevention of corrosion-related issues, thereby extending the lifespan of metal structures, reducing maintenance costs, and preserving valuable assets in various industries. The study opens the door to further research in this area, aiming to enhance the effectiveness and practicality of these inhibitors for real-world applications.

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.

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