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CHAPTER ONE INTRODUCTION

1.0

1.1 Background to study

It has been estimated that 3-4% of industrial nations GDP was spent on prevention and the maintenance or replacement of product loss or contaminated as result of corrosion (Faiz *et al.*, 2020). Deductively, this amount to ten times what the third world countries spent to fight corrosion. In 2003, it was reported that the direct cost of corrosion of US was about \$41.9 billion (Fayomi *et al.*, 2019) which was about 13 times GDP of Nigeria, Beside this economic significance, the corrosion also represents a large ecological and safety problem, due to possible corrosion induced industrial, civil structure and transportation damages. Therefore, it is of great importance to minimize and control corrosion.

Corrosion is possibly the most important and costly cause of severe operational problems encountered in our daily life and industrial set up such as manufacture, chemical plant, oil and gas production systems. It may occur anywhere in the production system, from the oil/gas well bottom to the final transfer of produced gas or oil to the refinery. Since corrosion is a natural process, it cannot be completely prevented. However, it can be controlled so that the materials and the corresponding equipment can perform their required tasks for a predicted length of time. Corrosion detection, monitoring and control are dominant considerations when seeking maximum equipment lifetime, minimum cost and maximum safety, in many industries. In order to control corrosion, it is necessary first to understand the nature and mechanisms by which it occurs. Among available metals (e.g. stainless steel, aluminum, etc.) that can be used in various industrial applications, mild steels are still the most widely used engineering materials. Because of their low cost and mechanical properties, carbon and low-alloy steels are the workhorse materials in most industries. This include the oil and gas industry, where these materials remain the most used materials for structural members, plates, sheets, tubing and flow lines and transmission pipelines (Kanayo & Joseph, 2015)

Mild steel is an iron alloy with 0.05-0.25 % carbon content. It has excellent properties which attracted its widespread use in numerous fields and industrial application. It found useful as construction material due to its high flexibility and compressive strength as compared to other types of steels. Apart from its high strength and flexibility, mild steel also has other advantages such as lighter, more durable and ductile, and more corrosive resistant as compared to other type of carbon steel (Mohd Ridhwan Adam, 2014) However, mild steel is vulnerable to corrosion especially when exposed to aggressive media due to its thermodynamic instability in such media (Ricky *et al.*, 2021). In the presence of water and oxygen, mild steel tends to form iron oxide as the rust product.

Mild steel is one of the most used metal alloys in industries. The special properties of mild steels make it as a favorite alloy in many fields such as automobile, water treatment, construction etc. However, the long exposures of this metal into the corrosive medium such as acids and moist environment have led to the corrosion problem. As a consequence, the lifetime of mild steels will be less and this problem drives to other serious problem that includes the loss

of life due to accidents. Mild steels are composed essentially of an alloy of iron and a number of trace elements, the main being carbon. This structure provides the carbon steel with good physical properties, for example an excellent ductility, permitting many cold-forming operations. However, this metal has a limited corrosion resistance. This is because of its heterogeneous surface that make it more susceptible to corrosion attacks, particularly in oil and gas production systems, and because it cannot form stable surface passive films, like stainless steels, which would protect the material from further corrosion (Alsabagh *et al.*, 2006).

Corrosion is an electrochemical process that occurs due to the presence of anode and cathode sites on the material's surface, originating from the heterogeneous nature of the bulk material and its surface. The presence of an electrolyte and electrical connection is also essential for electrochemical corrosion to occur. The bulk material and its surface provide the anode, cathode and the electrical connection, while for example, humid air, aqueous solution, etc., provide the electrolyte to complete the corrosion circuit. Corrosion of mild steel do occurs in the presence of dissolve carbon dioxide, hydrogen sulphide and oxygen especially in oil industry. Also during material acidization using acid solution in pickling process, cleaning, in various aqueous electrolyte storage tanks, in boiler and during the removal of scale, rust and corrosion product.(Lebrini *et al.*, 2007).

Elimination of any of the factors that complete the corrosion circuit will automatically stop the corrosion process but in reality this is not possible to fully achieved but there exist ways in which one or more of the factors can be influence so as to reduce the rate at which the corrosion progress .Among the several methods used in corrosion mitigation and prevention which includes selection of high corrosion resistance metals, electroplating, coating and so on, the use of corrosion inhibitors represent the most effective method. A corrosion inhibitor is a substance which when added in small concentration to corrosion media, reduces the metallic corrosion rate effectively (Ogunleye *et al.*, 2019). Inhibitors are categorised based on the prevailing mechanism of actions. The first class of inhibitors works by forming protective oxide films on metals by chemical reaction thereby creating impermeable barrier between it and the corrosion media. The second category constitutes those that inhibit corrosion by selective adsorption on the metal surface in a way that create a barrier that prevents access of corrosive agents to the metal surface. Large numbers of organic compounds have been studied for corrosion inhibition, the synthetic based are highly efficient. However, synthetic based inhibitors are expensive, non-biodegradable and hence, constitute environmental hazards (Ji *et al.*, 2015).

The plant extract provide sources for environmental friendly and biodegradable corrosion inhibitors. The extract from plant contains varieties of actives compounds such alkaloid, tannin, flavonoid, saponin, terpenes and so on which are reported to be effective corrosion inhibitors ((Chapagain *et al.*, 2022) These actives compound can be isolated from the plant material and use individually and synergistically or modify to provide desired corrosion inhibition properties.

1.2 Statement of the Problem

The use of synthetics corrosion inhibitors in the management and control of corrosion of metal in the corrosive environment has become very expensive and increasingly constituting environment hazard due to their non-biodegradable and toxic nature. However, a more sustainable and eco-friendly approach is the use of natural resource such as plant extract in the development of corrosion inhibitor. Plants have been recognized as sources of naturally occurring compounds, some with rather complex molecular structures and having varying of physical, chemical and biological properties (Miralrio, 2020). Most of the compounds extracted from plants are enjoying the use in traditional applications such as pharmaceuticals and biofuels (Buchweishaija 2009). Furthermore, the uses of naturally occurring compounds are of interest, because of their cost effectiveness, abundant availability and more importantly their environmentally acceptability. This study is designed to investigate the corrosion inhibition performance of the isolated phytochemicals in the plant extracts of *Aristolochia ringens* on mild steel corrosion in corrosive environment.

1.3 Aim and Objectives

1.3.1 Aim

The aim of the research is to investigate into the corrosion inhibition effect of the plant extracts from *Aristolochia ringens* on mild steel in 1M HCl solution.

1.3.2 The specific Objective

The specific objectives of the research are:

- i. to prepared, isolate and characterized the phytochemical constituents of the corrosion inhibitors from the plant extract of *Aristolochia ringens*
- ii. evaluation of the corrosion characteristics of Akaloid isolated on mild steel in acidic media by gravimetric-based mass loss.

CHAPTER TWO

2.0

LITERATURE REVIEW

Corrosion is metal dissolution caused by interaction with acidic corrosive environments by direct chemical reactions resulting from the formation of noble compounds (Processes-A. Int. J. Electrochem. Sci. 2015, 10, 6120–6134). As described by the International Union of Pure and Applied Chemistry, corrosion is the irreversible reaction of an interlayer (polymer, metal, concrete, wood, or ceramic) with the environment leading to ingestion or dissolution of the environmental component material. Corrosion is an environmental hazard with economic, preservation and safety consequences in many areas such as manufacturing, chemicals, automobiles, mechatronics, metallurgy, and medical applications (Rani, B.; Basu, B.B.J. Green inhibitors for corrosion protection of metals and alloys: An overview. Int. J. Corrosion, 2012).

Different forms of material degradation have been addressed in different environments. There is also a need to create new approaches and methods to counter these hazardous phenomena from the known factors, such as coatings, defensive bushings, cathodic/anodic protection, and sealants. However, the results of studies conducted on anti-corrosion applications in the above engineering fields showed the use of corrosion inhibitors to be the most gentle and easy way to prevent the degradation of metals and alloys in corrosive media (Marzorati, S.; Verotta, L.; Trasatti, S.P. Green corrosion inhibitors from natural sources and biomass wastes Molecules 2018). The corrosion inhibition efficiency of untested organic corrosion inhibitors has been linked to the availability of nitrogen, oxygen, phosphorous and sulfur atoms in organic compounds that have both protective effects and corrosion inhibiting potential. The high-ranking corrosion inhibitors were cited to be green oxygen corrosion inhibitors that display their inhibitory effect *via* hydrolysis or chemical absorption at the mineral solution interface by removing surface water molecules to form a dense barrier film. The incidence of a coordinate covalent bond is subject to the interaction between the lone pair and the electrons available in untested organic corrosion inhibitor molecules with vacant steel orbitals. However, the combined adsorption on the metal surface using the pi-bond formation is outperformed as a final result of the interference of the p electron with the three-dimensional vacant orbital of the iron atom due to the availability of N, O, and S atoms and the double bonds in an organic compound (Karki, N.; Neupane, S.; Chaudhary, Y.; Gupta, D.K.; Yadav, A.P. Berberis aristata: A highly efficient and thermally stable green corrosion inhibitor for mild steel in acidic medium. Anal. Bioanal. Electrochem. 2020).

2.1 Corrosion

Corrosion is the gradual destruction of materials, (usually metals), by chemical reaction with its environment (Ahmed, M.H.O.; Al-Amiery, A.A.; Al-Majedy, Y.K.; Kadhum, A.A.H.; Mohamad, A.B.; Gaaz, T.S. Synthesis and characterization of a novel organic corrosion inhibitor for mild steel in 1 M hydrochloric acid. Results Phys. 2018, 8, 728–733). Rusting, the formation of iron oxides, is a well-known example of electrochemical corrosion. In the most common use of the word, this means electrochemical oxidation of metals in reaction with an oxidant such as oxygen. Rusting, the formation of iron oxides, is a well-known example of electrochemical corrosion. This type of damage typically produces oxide(s) or salt(s) of the original metal (Kesavan, D.; Gopiraman, M.; Sulochana, N. Green inhibitors for corrosion of metals: A review. Chem. Sci. Rev. Lett. 2012). Corrosion is the deterioration or destruction of metals and alloys in the presence of an environment by chemical or electrochemical means. In simple terminology, corrosion processes involve reaction of metals with environmental species. Corrosion is the gradual destruction of materials, (usually metals), by chemical reaction with its environment. This means electrochemical oxidation of metals in reaction with an oxidant such as oxygen.

Rusting, the formation of iron oxides is a well-known example of electrochemical corrosion (Yahaya, L.; Aroyeun, S.; Ogunwolu, S.; Jayeola, C.; Igbinadolor, R. Green and Black Tea Extracts as Corrosion Inhibitor for Mild Steel in Acid Medium. World Appl. Sci. J. 2017). This type of damage typically produces oxide(s) or salt(s) of the original metal. Corrosion can also occur in materials other than metals, such as ceramics or polymers, although in this context, the term degradation is more common.

Corrosion degrades the useful properties of materials and structures including strength, appearance and permeability to liquids and gases(). Many structural alloys corrode merely from exposure to moisture in air, but the process can be strongly affected by exposure to certain substances. Corrosion can be concentrated locally to form a pit or crack, or it can extend across a wide area more or less uniformly corroding the surface. Because corrosion is a diffusion-controlled process, it occurs on exposed surfaces. As a result, methods to reduce the activity of the exposed surface, such as passivation and chromate conversion, can increase a material's corrosion resistance. However, some corrosion mechanisms are less visible and less predictable (Karki, N.; Neupane, S.; Chaudhary, Y.; Gupta, D.K.; Yadav, A.P. Berberis aristata: A highly efficient and thermally stable green corrosion inhibitor for mild steel in acidic medium. Anal. Bioanal. Electrochem. 2020).

2.1.1 Forms of corrosion

The Eight Forms of Corrosion: Uniform attack; galvanic corrosion; crevice corrosion; pitting corrosion; intergranular corrosion; selective leaching; erosion corrosion; stress corrosion cracking including hydrogen embrittlement.

2.1.1 General (Uniform) attack:

This is the most common form of corrosion. It occurs when a chemical or electrochemical attack occurs over a large area in a uniform manner. This is often referred to as a general wall loss or thinning. Uniform attack is the greatest destruction of metal on a tonnage basis. It can be prevented by using: proper materials, including coatings, inhibitors or cathodic protection.

2.1.2 Crevice corrosion

Is also known as Two-Metal Corrosion. A potential difference usually exists between two dissimilar metals when they are immersed in a corrosive or conductive solution. If these metals are placed in contact (or otherwise electrically connected), this potential difference produces electron flow between them. This occurs through the electrochemical cell. It requires electron flow, and is characterized by the presence of an anode (negative), cathode (positive) and an electrolyte. Most corrosion occurs at the anode, although some corrosion will occur at the cathode. Depending on the cell configuration, the corrosion may be localized or uniform. Crevice corrosion is a highly localized attack occurring in a crevice or an otherwise shielded area when a material is exposed to a stagnant corrosive media. Common locations for crevice corrosion are:

- (a) Crevices(such as under bolt or rivet heads)
- (b). Gasket surfaces
- (c) Holes
- (d). Lap joints
- (e.) Surface deposits

It is sometimes called deposit or gasket corrosion.

2.1.3: Galvanic corrosion

Is an electrochemical process in which one metal corrodes preferentially to another when both metals are in electrical contact in the presence of an electrolyte.

2.1.4: Pitting corrosion

Is a form of extremely localized attack that results in holes in the metal. A highly localized corrosion attack that results in holes is referred to as pitting. Pits may be isolated or localized and of virtually any configuration. They occur at defects or imperfections in a protective or passive film. It is a form of corrosion difficult to predict by laboratory test.

2.1.5: Intergranular corrosion:

Is a common form of attack on alloys but occurs rarely in pure metals. The attack is usually caused by impurities at the grain boundaries, enrichment of one of the alloying elements or depletion of one of these elements in the grain- boundary areas. All materials, with the exception of amorphous materials (such as plastic), are composed of grains and grain boundaries. Intergranular corrosion occurs when the grain boundaries are attacked in preference to the material matrix. The only difference between this and uniform corrosion is that the grains remain undamaged. Metallographic examination is usually the only way to identify this corrosion mechanism.

2.1.6: Selective leaching

It is also known as dealloying: This is the removal of one element from a solid alloy by corrosion processes. The most common example is the selective removal of zinc in brass alloys (dezincification). Most materials are made up of a combination of several elements. Dealloying occurs when one of the elements is removed from the metal matrix, leaving an altered residual structure. It is commonly identifiable by a color change or a drastic change in mechanical strength.

2.1.7: Erosion corrosion

Is used to describe the increased rate of attack caused by a combination of erosion and corrosion. If a fluid stream contains suspended particles, or where there is high velocity or turbulence, erosion will tend to remove the products of corrosion and any protective film and the rate of attack will be markedly increased. For example: plastics inserts are used to prevent erosion–corrosion at the inlet to heat-exchanger tubes.

2.1.8: Stress corrosion cracking including hydrogen embrittlement

Stress corrosion cracking refers to cracking caused by the simultaneous presence of tensile stress and a specific corrosive medium. Many investigators have classified all cracking failures occurring in corrosive medium as stress corrosion cracking, including hydrogen embrittlement. However, these two types of cracking failures respond differently to environmental variables. To illustrate, cathodic protection is an effective method for preventing stress-corrosion cracking whereas it rapidly accelerates hydrogen–embrittlement effects. Hence, the importance of considering stress- corrosion cracking and hydrogen embrittlement as separate phenomena is obvious.

2.2 Alkaloids

Alkaloids are naturally occurring organic substances, predominantly found in plant sources including marine algae and rarely in animals (e.g. in the toxic secretions of fire ants, ladybugs and toads). They occur mostly in seed-bearing plants mainly in berries, bark, fruits, roots and leaves. Alkaloids often contain at least one nitrogen atom in heterocyclic ring. These are basic in nature and so referred the term alkaloid (alkali-like). Alkaloids possess remarkable physiological action on human and other animals.

These are the active components of numerous medicinal plants or plant-derived drugs. Their structural diversity and different physiological activities are unique to any other group of natural products. Many drugs used by man for both medical and non medical purposes are produced in nature in the form of alkaloids e.g. atropine, strychnine, caffeine, nicotine, morphine, codeine, cocaine etc. Naturally occurring receptors for many alkaloids have also been identified in human and other animals, suggesting an evolutionary role for the alkaloids in physiological processes. Alkaloids are relatively stable compounds that accumulate as end products of different biosynthetic pathways, mostly starting from common amino acids such as lysine, ornithine, tyrosine, tryptophan, and others.

These substances are usually colourless but several coloured alkaloids are also reported e.g. berberine is yellow, sanguinarine salt is copper-red and betanidin is red (Kokate et al., 2005). These are crystalline solids, having ring structure, definite melting points and bitter in taste. In plants they may exist in free state, in the form of salt or as N-oxides, rarely found in the form of glycosides (Biswas and Sharia, 1978; Tanahashi et al., 2000; Kashiwaba et al., 2000). In addition to the elements carbon, hydrogen and nitrogen, most alkaloids contain oxygen. A few such as coniine from hemlock and nicotine from tobacco are oxygen free. The free bases are sparingly soluble in water but readily soluble in organic solvents, however with their salts, the reverse is often the case, e.g. strychnine hydrochloride is more soluble in water than in organic solvents. Most of the alkaloids are optically active, generally due to the presence of tertiary nitrogen in their structures. This results the various isomeric forms having different physical, chemical and pharmacological properties e.g. (+)-tubocurarine isolated from *Chondrodendron tomentosum* (Bisset, 1992), have muscle relaxant activity, whereas its leave or isomer is less active.

2.2.1 Classification of alkaloids

2.2.1.1 Taxonomical classification:

This classification is based on the distribution of alkaloids in various plant families, like solanaceous or papilionaceous alkaloids. Sometimes they are grouped as per the name of grouped genus in which they occur, e.g. ephedra, cinchona, etc.

2.2.1.2 Biosynthetic classification:

This method gives significance to the precursor from which the alkaloids are biosynthesized in the plant. Hence the variety of alkaloids with different taxonomic distribution and physiological activities can be brought under same group, if they are derived from same precursor, e.g. all indole alkaloids from tryptophan are grouped together. Alkaloids derived from amino acid precursor are grouped in same class such as ornithine, lysine, tyrosine, phenylalanine, tryptophan, etc.

2.2.1.3 Pharmacological classification:

This classification is based on the physiological action or biological activity of alkaloids on animals like CNS stimulants or depressants, sympathomimetics, analgesics, purgatives, etc. This method does not take account of chemical nature of alkaloids. Within the same chemical structure the alkaloids can exhibit more than one physiological action e.g. morphine is narcotic-analgesic, while quinidine is cardiac depressant.

2.2.1.4 Chemical classification:

This classification is most accepted way to specify the alkaloids. The alkaloids are categorized into three divisions.

- a. True alkaloids: These have heterocyclic ring with nitrogen and derived from amino acids.
- b. Proto alkaloids: These do not have heterocyclic ring with nitrogen and derive from amino acids, e.g. colchicine.
- c. Pseudo alkaloids: These have heterocyclic ring with nitrogen and derived from terpenoids or purines but not derived from amino acids.

2.3. Mild Steel

Mild steel, which contains small percentage of carbon and is strong and easily worked but not readily tempered or hardened (Salami, L., Wewe, T.O.Y., Akinyemi, O.P. And Patinvoh, R.J. "A Study Of The Corrosion Inhibitor Of Mild Steel In Sulphuric Acid Using Musa Sapientum Peels Extract" Fourth Edition 2008). Due to its surroundings it tends to corrode, and is affected by numbers of factors such as, metal composition, temperature, presence of gases like sulfur dioxide, hydrogen chloride, chlorine and other corrosive gases, for structure. Few studies have been conducted on the consistently available corrosion inhibitor potentials of some plant extract, which may be known as "green inhibitor" (Ramananda S. Mayanglambam*, Vivek Sharma, Gurmeet Singh, "Musa Paradisiaca Extract as a Green Inhibitor for Corrosion of Mild Steel in 0.5 M Sulphuric Acid Solution", Department of Chemistry, University of Delhi, 2011). In the chemical environment, to prevent the corrosion rate of metal by using the green corrosion inhibitor. The adsorption of inhibitor can be enhanced by the presence of hetero atoms such as N, O, P and S. Corrosion processes develop fast after disruption of the protective barrier and are accompanied by a number of reactions that change the composition and properties of both the metal surface and the local environment, for example, formation of oxides, diffusion of metal into coating matrix, local pH. change and electrochemical potential (Int. J. Electrochem. Sci., "Corrosion Inhibition of Mild Steel in 1M HCl Using Aqueous Extract of Eggplant Peel", American University of Sharjah, Department of Chemical Engineering, 2011). The study of corrosion of mild steel is a matter of tremendous theoretical and practical concern and as such has received a considerable amount of interest. Acid solutions, widely used in industrial acid cleaning, acid descaling, acid pickling and oil well acidizing, require the use of corrosion inhibitors in order to restrain their corrosion attack on metallic materials (Nnabuk O. Eddy1, Paul Ameh1, Casmir E. Gimba1, Eno E. Ebenso2- "Chemical Information from GCMS of Ficus Platyphylla Gum and its Corrosion Inhibition Potential for Mild Steel in 0.1 M HCl"- June 2012).

Mild steel possesses excellent ductility because of carbon content, which is essential for the deep drawing of sheet but its strength is low. As the percentage is increased (0.2%) the strength of steel rises into range required. Reduction of carbon percentage and addition of small amount of other alloying elements can achieve this, mild steel possesses the desirable properties as good tensile strength (B. E. Amitha Rani and Bharathi Bai J. Basu "Green Inhibitors for Corrosion Protection of Metals and Alloys An Overview," Surface Engineering Division, CSIR-National Aerospace Laboratories, Bangalore, 560037, India, 2012). With the above properties along with relative cheapness has made mild steel a desirable material for the use in industry for many mechanical and structural engineering purposes of bridge work, reactors, boiler plates, parts of various components and engines. With the help of certain materials, including eco-friendly extract of a number of organic compounds have been used as potent corrosion inhibitors (Salami, L., Wewe, T.O.Y., Akinyemi, O.P. And Patinvoh, R.J. "A Study Of The Corrosion Inhibitor Of Mild Steel In Sulphuric Acid Using Musa Sapientum Peels Extract" Fourth Edition, 2008).

2.4 Aristolochia Rigens

Aristolochia ringens. is a perennial plant in the Aristolochiaceae family. In the south-western Nigeria (Yoruba), the plant is commonly known as 'Akogun'. It is an aromatic liane, scrambler, a climbing shrub or rhizome. The plant contains alkaloids and aristolochic acids. The plant is used locally in the treatment of wounds, dysentery, throat infections and skin problems. The antimicrobial potential and phytochemical composition of *A. ringens* root and bark have been investigated. In addition, the antidiabetic, antitrypanosomal and anticancer activities of the plant have been reported (Maberley, 2013). *A. ringens* is used to treat various ailments such as wounds, dysentery, throat infections and skin problems of which are linked to microbial infestations. In Asian countries, especially India, over 2,500 plants have been studied to have provided alternative medicine and curative properties to the available synthetic drugs (Sarmiento et al., 2011; Thirumal et al., 2012). Extracts from *Aristolochia* sp, especially, phytochemicals and essential oil have been receiving earnest in-vitro investigations for their numerous activities.

Among the documented activities traced to such phytochemical properties of *A. ringens* are antimicrobial, anti-inflammatory, anti-venom, antipyretic, antiseptic, abortifacient, emmenagogues, storage stability (preservative), foaming (lather), curative, taste, flavours and aroma on one hand and potent nephrotoxic, anti fertility and antispermaticogenic on the other (AshokKumar *et al.*, 2010). In recent years, the traditional application of natural compounds of plant origin has been receiving a lot of attention as an alternative source of remedy for the treatment of diseases coupled with the belief of their better safety nature and of less or non toxicity (Kumar *et al.*, 2011). The search for new drugs has led to the increase in laboratory (in-vitro) research into herbal medicine to establish their acclaimed efficacy and their therapeutic applications. (Sinha and Choudhury, 2010)

2.5 Inhibitive Effect on Corrosion

During the acidizing procedure a number of organic compounds serve as CIs for steels, including acetylenic alcohols, aldehyde-containing aromatic compounds, imminium salts, nitriles, triazoles, thiocyanates, pyridine and its derivatives or salts, quinoline derivatives, thiourea derivatives, quaternary salts, thiosemicarbazide, quinoline derivatives, thiourea derivatives, thiosemicarbazide derivatives, nitriles, imminium-based salts, and nitrogen heterocycles (D.A. Williams, P.K. Holifield, J.R. Looney and L.A. McDougall, Method of Inhibiting Corrosion in Acidizing Wells, in: US Patent 5,200,096, Exxon Chemicals Patents, Inc., Linden, N.J., 2003). Nitrogen and acetylenic alcohol molecules appear to form a film on metal surfaces and to be able to delay the metal breakdown (anodic response) and production of hydrogen (a cathodic reaction). Propargyl alcohol is soluble in acidic liquids, however, with an ever larger length of carbon chain, the solubility of other acetylenic alcohols reduces (E. Barmatov, J. Geddes, T. Hughes and M. Nagl, Research on corrosion inhibitors for acid stimulation, NACE Int., 2012). In conjunction with quaternary ammonium surfactants, the solubility of these acetylenic alcohols can be improved. The industrial supply and cost efficiency of acetylenic alcohols was commonly used. The normal CI for acidification is generally propargyl alcohol, with the synergistic influence of other compounds often substantial. Propargyl alcohol, its salts, cinnamaldehyde, and aromatic compounds such as quaternary pyridinium chloride are the most widely encountered CIs in the natural resource industry. (Tajkarimi *et al.*, 2010) In 1984 an outline of the use of CIs for acid media was published by Schmitt. Natural products such as plant extracts, coumarins, amino acids, and natural polymers were published to be effective inhibitors for mild steel corrosion (M.A. 2001).

Green-based inhibitors that are nontoxic in nature, such as plant extracts, are in higher demand compared to commercial inhibitors (S. Zakvi and G. Mehta, Acid corrosion of mild steel and its inhibition by *Swertia angustifolia* – study by electrochemical techniques, Trans. SAEST, 2008). This is because plant extracts are green and sustainable materials due to their natural and biological properties and can inhibit metals and alloys from corroding. The leaf, out of all parts of the plant, has the utmost preference for its abundance of phytochemicals (active components) produced through synthesis, that act similarly to commercial inhibitors (Abhijit and Jitendra, 2011). It is also vital to acknowledge that the extract of other parts of a plant such as root, bark, flower, fruit, wood, seed and peel have contributed to the inhibition efficiency (M. Quraishi, I. Farooqi and P. Saini, Investigation of some green compounds as corrosion and scale inhibitors for cooling systems, Corrosion, 2004). Furthermore, phytochemical synthesis consumes carbon dioxide, which is known as the highly poisonous greenhouse gas, to use in photosynthesis, contributing to the green chemistry theory as well (P. Sakthivel, P. Nirmala, S. Umamaheswari, A. Antony, G. Kalaignan, A. Gopalan and T. Vasudevan, Corrosion inhibition of mild steel by extracts of *Pongamia glabra* and *Annona squamosa* in acidic media, Bull. Electrochem., 2004).

Green-based corrosion inhibitors can be divided into two classes: organic and inorganic. The organic class of green-based corrosion inhibitors consists of synthetic substances that are nontoxic for the environment. Flavonoids, alkaloids, and byproducts of plants are examples of organic inhibitors. The inorganic class of inhibitors are vastly utilized in aqueous systems due to their high productivity. Chromates are toxic in nature so the employment of this inorganic inhibitor for industrial use is limited. Concerning this issue, lanthanide salts were studied as an eco-friendly inhibitor substitute (S. Verma and G. Mehta, Effect of acid extracts of *Acacia arabica* on acid corrosion of mild steel, Bull. Electrochem., 2003).

Coumarins act as corrosion inhibitors due to the presence of an aromatic system and heteroatoms, that are adsorbed on mild steel surface *via* ion pairs of electrons on heteroatoms. This induces superior adsorption on the inhibitor molecules and excellent inhibition efficiency in decreasing the corrosion rate. Physisorption and/or chemisorption are the mechanisms by which inhibitors are adsorbed on the surface of mild steel and form a protective film barrier from acidic solutions. There are certain ways to protect a metal from corrosion such as coating, alloying, cathodic protection, and anodic protection. Moreover, laser treatment of metal surface has recently been used for this purpose as a way to improve the properties of metals like roughness, hardness, resistance to corrosion, *etc* (F. Zucchi and I. Omar, Plant extracts as corrosion inhibitors of mild steel in HCl solutions, Surf. Technol., 2005).

Corrosion inhibitors are of considerable practical importance, as they are extensively

employed in reducing metal waste during production and in minimizing the risk of material failure, both of which can result in a sudden shut-down of industrial processes, which in turn leads to added costs. It is also important to use corrosion inhibitors to prevent metal dissolution and minimize acid consumption. There are two kinds of corrosion interaction according to the nature of corrosive environments: wet and dry corrosion. These types of corrosion can be classified into: general corrosion; pitting corrosion; crevice corrosion; intergranular corrosion. Corrosion inhibitors are generally used in acid *Int. J. Corros. Scale Inhib.*, 2021. The process of selecting an appropriate corrosion inhibitor is one of the most important issues to consider when working in an acid environment, and therefore the use of organic compounds is of great importance. The efficiency of organic compounds in corrosive solutions is attributed to several factors, the most important of which are the presence of functional groups and electron pairs in the phosphorus, sulfur, oxygen and nitrogen atoms, in addition to the double and triple bonds, as well as aromatic rings. Small amounts of corrosion inhibitors have the ability to block the corrosion sites and improve the adsorption process on the mild steel surface, thus protecting the mild steel surface and increasing its life time. The degree of corrosion inhibition due to absorption depends on the geometry of the particles and their size in addition to the vacuum impediment, and the thermal stability of the inhibitor molecules. Thus, inhibitor molecules improve mild steel resistance to corrosive solutions by adsorbing on the metal surface and forming a barrier that blocks the mild steel active sites. Inhibitor adsorption on mild steel is affected by the nature of the mild steel, type of electrolyte and molecular structure of the inhibitor (*Int. J. Corros. Scale Inhib.*, 2021).

2.6 Importance of Green Inhibitors

The term corrosion is destruction of metal or alloy; they deteriorate slowly by the action of atmospheric gases, moisture and other chemicals. The rate at which it can propagate with a view of improving the lifetime of metallic and alloy materials. A corrosion inhibitor is a substance which, when added in small concentration to an environment, effectively reduces the corrosion rate of a metal exposed to that environment. To overcome those destruction of metal, different plant extract can be used as corrosion inhibitors, which is commonly known as green corrosion inhibitors. Plants are sources of naturally occurring compounds. Naturally occurring compounds are mostly used because they are environmentally acceptable, cost effective and have abundant availability. Some with complex molecular structures and having different chemical, biological and physical properties. By adding inhibitors in low concentrations to corrosive media is to delay the reaction between the metal and the corrosive species in the medium.

The properties that must be met by corrosion inhibitor as:

1. Capability of reducing corrosion rate
2. The active principle of the corrosion inhibitor must be in contact with the metal
3. Must not have side effects.

CHAPTER THREE

3.0 MATERIAL AND METHOD

3.1 Material

Aristolochia ringens root, Soxhlet extractor, batch distiller, weighing balance, test tubes, heating bath, spatula, methanol, ethanol, distilled water.

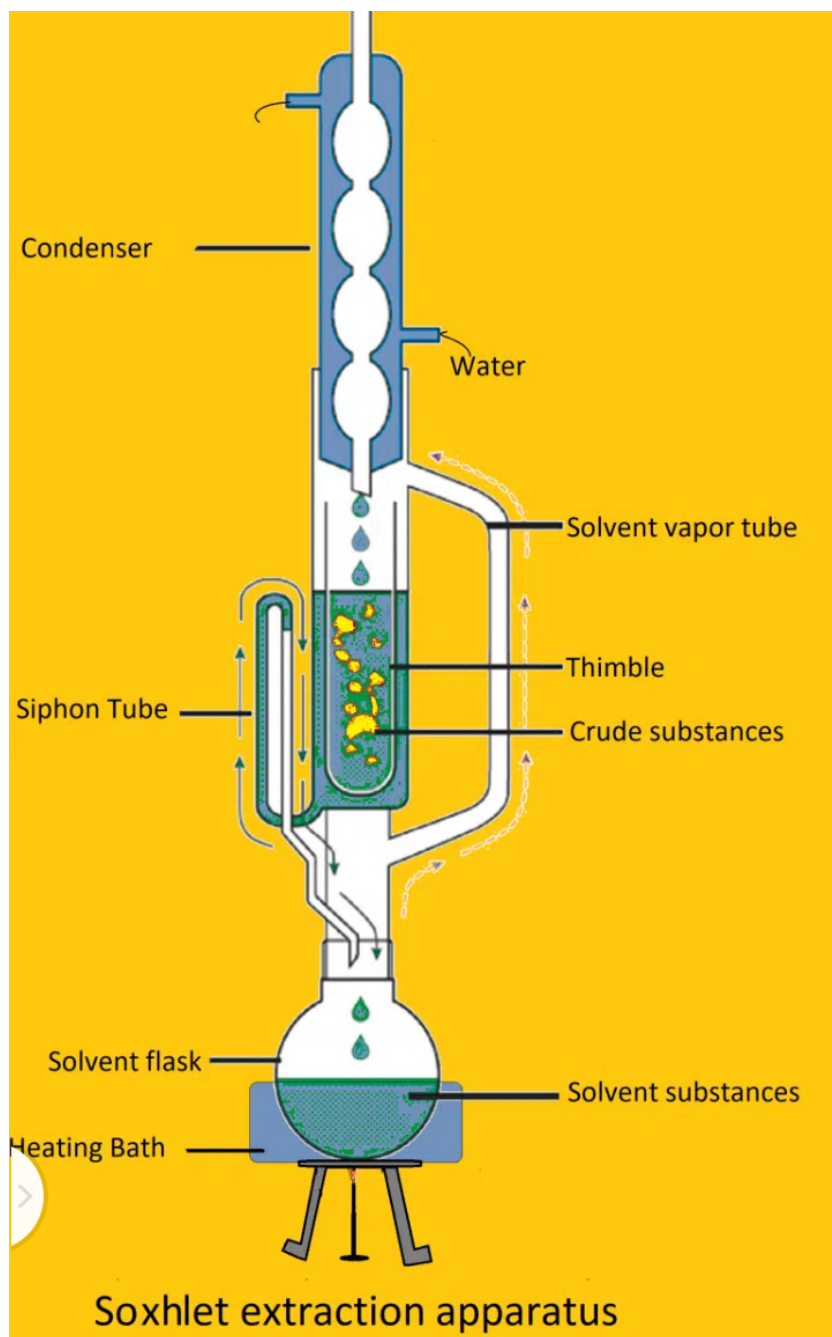
3.1.1 Collection and preparation of plant material

The plant materials used for the work were the root of *Aristolochia ringens* (Ako-igun in Yoruba). The materials were purchased at Ipata market Ilorin, Kwara state North west Nigeria. They were thoroughly washed with sterile distilled water and air dried before milling into powder for antimicrobial in-vitro analysis.

3.2 Plant Extraction (Soxhlet Extraction)

Fifty grams (50g) of powdered root of *A. ringens* was measured into a white handkerchief which is then kept in glass cylinder. The cylinder is provided with a siphon tube and an inlet tube. A water condenser is attached to the cylinder at the top. The entire assembly is fitted into the neck of a round bottom flask containing 500ml of each of the two solvents: Methanol and Ethylacetate. The flask is then heated in a water bath. The solvent vapours reach the cylinder through the inlet tube and condense on passing upward into the condenser. The condensed solvent comes in contact with the crude organic substance and dissolves it. As soon as the solution reaches the top end of the siphon. In this way, a continuous supply of solvent vapours is maintained in the

cylinder, and the dissolved organic compound flows back into the flask. The heating is then stopped, the solution in the flask is distilled to recover the solvent and the organic compound separately. The solute (*Aristolochia*) was collected and weighed then kept. A dark green slurry of alkaloids in the organic layer was collected. A small amount of HCl was added to the alkaloid fraction to remove traces of water. The solution was then filtered. The organic layer thus obtained was separated, concentrated by using a rotatory evaporator under reduced pressure, and evaporated (below 40 °C) up to dryness to obtain alkaloids of *A. ringens*. The presence of alkaloids was ensured by Mayer's test.



3.3. Extraction of Alkaloids

Extraction of alkaloid extracts of *Aristolochia ringens* possible with the use of diethyl ether, 0.5 M HCl acid and ammonia. A 1000 ml separating funnel was used to host 50 g of crude ethanol extract of *Aristolochia ringens* and 250 ml of 0.5 M HCl and diethyl ether each. The funnel was stoppered properly and the content shook to attain homogeneity. The mixture was allowed to stand for 3 hours after which the tailing content was separated and the top portion basified with ammonia and 250 ml of diethyl ether added for separation and kept for another 3 hours. The mixture was partitioned in the separating funnel and the tailing content was the collected and evaporated over a water bath. The remaining content after evaporation was the alkaloids.

3.4 Tests for Alkaloids

A chemical test was carried out for the alkaloid test. For this, a small amount of extract

was treated with Mayer's (HgCl₂ + KI) and Dragendorff's reagents.

3.5 Resources Mild and corrosive media

Steel sheets composed of 0.18% carbon, 0.68% manganese, 0.03% sulfur, 0.04% phosphorus, with the remainder being iron were utilized. Acidic medium was simulated by using 36 w/w % HCl (S.G =1.18 gdm⁻³), which was serially diluted with distilled water to obtain the desired 1MHCl solution. Similarly, the alkaline and saline environment was obtained from using 1M NaOH solution and 1M NaCl solution, respectively. To prepare 1M NaOH solution, 40 g of sodium hydroxide NaOH pellets were dissolved in 250 ml of distilled water and made up the solution to 1 litre. 1M NaCl solution was prepared by measuring and placing 58.4 g of NaCl salt in 1 1-litre volumetric flask, and the flask was filled with distilled water to the graduation mark.

3.6 Weight Loss

Weight Loss Measurement Method

The weight of each MS coupon before and after immersion in acid and inhibitor solution was measured using an analytical electronic balance (Ohaus Corporation, Parsippany, NJ, USA and Model: E1RR80). Measurements were carried out for each immersion experiment in inhibitor solution of different concentrations (200, 400, 600, 800, and 1000 ppm) as well as in different intervals of time (0.5, 3, 6, 9, and 24 h). Similarly, the temperature effect was studied by immersing the sample in 600 ppm inhibitor solution at 25, 35, 45, and 55 °C for 6 h.

The corrosion rate (CR) and inhibition efficiency (IE%) were calculated using the formula:

$$\text{Corrosion rate (CR) in mm/yr} = \frac{K \times W}{A \times T \times D} \quad (1)$$

$$\text{Inhibition efficiency (IE \%)} = \frac{W_a - W_p}{W_a} \times 100 \quad (2)$$

Where, K = 87,600, W = weight loss in gram, A = total surface area of coupons in cm²,

T = time of immersion in h,

D = density of mild steel in g/cm³

W_a = weight loss in the absence of inhibitors

W_p = weight loss in the presence of inhibitors

CHAPTER FOUR

4.0 RESULT AND DISCUSSION

4.1 Qualitative Chemical Test

Extracted alkaloids were confirmed by qualitative chemical tests. A qualitative test for confirmation of the presence of alkaloids was performed by Mayer's and Dragendorff's test methods. The detailed observations are given in Table 4.1. The possible chemical reaction with the reagent used is given regarding the dendroxine molecule in Figure 4.1 (Li, 2019)

Table 4.1:. Phytochemical screening of the extract solution.

| S.N. | Experiment | Observation | Inference |
|------|--------------------|---|-------------------|
| 1. | Mayer's Test | The appearance of an orange precipitate | Alkaloid Presence |
| 2. | Dragendorff's Test | The appearance of orange-red color. | Alkaloid Presence |

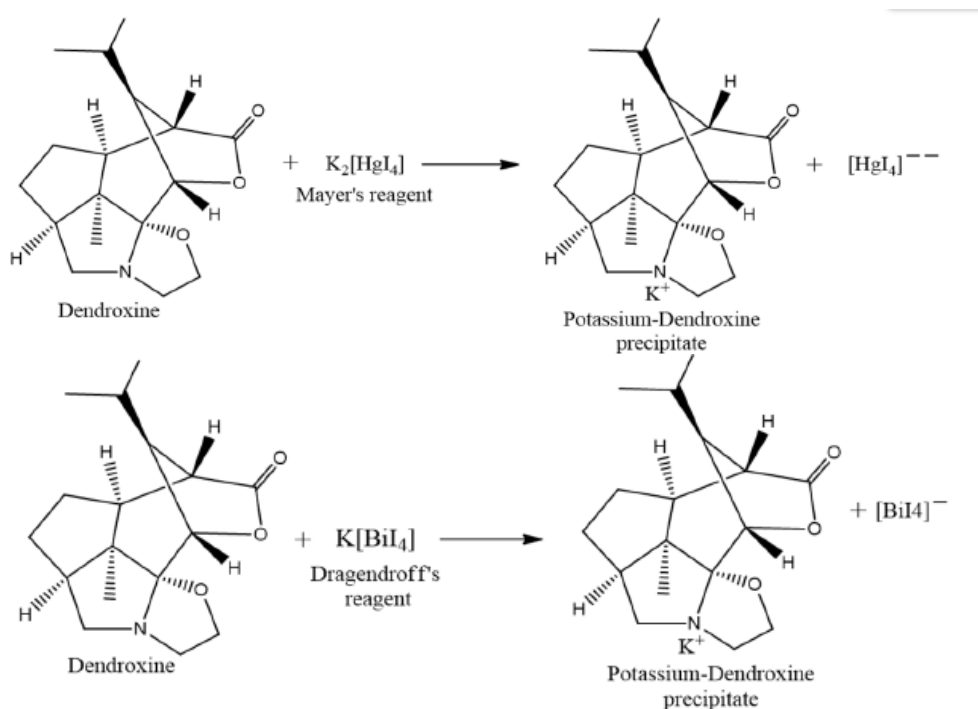


Figure 4.1: Possible chemical reaction involved in the chemical test of alkaloid

4.2 Effect of Immersion Time

Weight loss of MS and inhibition efficiency of alkaloids extracted from *Aristolochia ringens* root at various time intervals are observed and represented in Table 4.1, Figure 4.2a and . Figure 4.2b respectively. Initially, the weight loss of MS in acid and the presence of the inhibitor is slightly different; however, later on, the weight loss of MS samples in presence of an inhibitor is significantly decreased. This shows that initially the protective layer of the inhibitor is not completely formed, but later the formation of the protective layer is completed, hence weight loss is minimized. Due to a longer period of exposure to MS in acidic medium desorption can also happen, which results in slight weight loss even in presence of an inhibitor

Table 4.2 Immersion Time with Inhibition Efficiency

| H | Immersion time Weight loss in g/cm ² | | | I E % |
|-----|---|----------|--|-------|
| | HCl | Alkaloid | | |
| 0 | 0 | 0 | | 0 |
| 0.5 | 0.04 | 0.02 | | 55 |
| 3 | 0.112 | 0.025 | | 77.68 |
| 6 | 0.362 | 0.032 | | 91.18 |
| 9 | 0.550 | 0.96 | | 82.54 |
| 24 | 1.35 | .0.2 | | 84 |

Figure 4.2 (a) Variation of weight loss of MS samples in acid and inhibitor solution at fixed Intervals of time

Figure 4.2 (b) Inhibition efficiency of alkaloid solution at different intervals of time.

The 1000 ppm inhibitor solution was used in this experiment. This concentration seems sufficient for the formation of a protective layer on the MS surface. Initially, there is very small weight loss; however, at a longer period of immersion time, the weight loss has increased. This may be due to the presence of defects on the inhibitor layer on the MS surface. This type of defect may arise due to orientation, size, and interaction among the inhibitor molecules. Even increasing in weight loss, the inhibition efficiency is constant; this is because the exposure of MS surface to the aggressive environment in the presence of inhibitor solution is only from the point of the defect however in absence of inhibitor all the surface area is available for the aggressive environment, and this is much more pronounced than in the presence of an inhibitor.

4. 3 Effect of Concentration

Immersion tests have been carried out in 200, 400, 600, 800, and 1000 ppm alkaloid solutions for 6 h to study the effect of inhibitor concentration. The observation showed that at low inhibitor concentration, high weight loss and low efficiency indicate a high corrosion rate. Here, the rate of corrosion decreases by increasing the alkaloid concentration. The maximum efficiency observed is 85.33% in 1000 ppm solution as in Figure 4.3.

The surface of MS dipped in the acid solution only at 0.5 h and 3 h immersion study reveals that there are large pits/grooves on the surface due to dissolution of MS surface by acid molecules. The formation of pits/grooves on the surface is higher in the 3 h immersion sample than in the 1 h immersed sample; however, inhibitor molecules are visible on the MS surfaces which are immersed in the presence of inhibitor. The surface of MS is very smooth for 1 h immersed sample than that of 3 h immersed sample; however, in both cases, inhibitors molecules are adsorbed on the surface, thus reducing the corrosion.

Figure 4.3 The effect of Alkaloid concentration on Inhibition Efficiency

Figure 4.4 The effect of Alkaloid concentration at various Temperature on Inhibition Efficiency

From the Figure 4.4, Series 1:= 25^oC, Series 2 = 35 ^oC, Series = 45^oC, Series = 55^oC

4.4. Temperature Effect

The inhibition efficiency of the alkaloid inhibitor greatly depends on temperature. This effect was studied by taking reference to 600 ppm inhibitor solution at different temperatures. It is found that inhibition efficiency is almost constant at 25^oC and 35 ^oC but with the rise in temperature above it, the efficiency decreases, it may be due to desorption of inhibitor molecule from the metal surface, Figure 4.4. This gives the key idea that there is physical adsorption between the inhibitor molecule and the MS surface.

4.5. Adsorption Isotherm

Based on the value of surface coverage and the inhibitor concentration used, various adsorption isotherms have been tested. For reference, the molecular mass of the dendroxine molecule has been used; however, this only is not responsible for the inhibition. Langmuir isotherm has been tested by plotting C/θ versus C (mol/L) from Equation (5) and shown in Figure 11a. The correlation coefficient (R^2) value is equal to 0.994, indicating that the adsorption of inhibitor on the MS surface strongly obeys Langmuir adsorption isotherm and a monolayer of inhibitor formed on the MS surface before multilayer formation. From the value of intercept, the adsorption constant has been calculated and is used for the calculation of the free energy of the adsorption. The free energy of the adsorption has been found at 27.33 kJ/mol. This value is higher than the value of physical adsorption (<20 kJ/mol) and lower than chemical adsorption (>40 kJ/mol) indicating physical dominated chemical adsorption of inhibitor on MS surface.

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**CHAPTER FIVE
CONCLUSION**

5.0

Alkaloids have been extracted successfully from *Aristolochia ringens* root by the solvent extraction process. Qualitative chemical test (Mayer and Dragendorff's method) supports, shows the presence of N-H, O-H, N-O, -C=C- groups in the structure of alkaloid. It has been found that alkaloid inhibition increases with an increase in its concentration. Inhibitor protects mild steel from an aggressive environment by the formation of a protective layer on the surface through the adsorption process. Adsorption of the alkaloid (inhibitor) on the metal surface depends on the contact time, concentration and temperature. Due to this fact, inhibition efficiency is greatly influenced by temperature, concentration, and contact time. The inhibition efficiency of inhibitor by weight loss method is found to be 85.33% for 1000 ppm concentration

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