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(RESEARCH ARTICLE)

Adsorption characteristics of *Luffa Cylindrica* leaf extract as a corrosion inhibitor of copper in alkaline environment

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Abstract

Copper corrosion inhibition by Luffa cylinderica leaf extract has been studied in 0.01M Sodium hydroxide solution (NaOH) using electrochemical measurement. Inhibition efficiency increase as the inhibitor concentration increases and decrease with temperature while corrosion rate decreases with concentration and increase with rise in temperature. At the optimum concentration (1000mg/L), the highest inhibition efficiency of plant extract was found to be 72.1846% at 303K and 68.1242% at 313K , and the corrosion rate (10-5) was 1.2679 at 303K and 1.4059 at 313K. However, increase in temperature decreased the inhibition efficiency at the temperature range studied. The phenomenon of physical adsorption has been proposed from the activation energy values (3.8933 to 7.9793 kJ mol-1) with Luffa cylinderica leave extract and the heat of adsorption, Qads ranges from (-26.4619 to - 15.3085 kJmol-1) for concentration of inhibitor studied. These values are negative and suggestive of adsorption on metal surface. The surface morphology and the composition of copper metal were also examined using SEM and XRD analysis respectively. The data obtained from this study fits well into the Langmuir adsorption isotherm.

Keywords: Adsorption; *Luffa cylinderica*; Corrosion inhibition; Efficiency; Corrosion rate; Isotherm.

1. Introduction

Increase in industrial activities in economic sectors such as marine, communication, domestic utility productions, electrical and electronics have engaged resourceful use of copper and alloys in respect of its good aesthetic and mechanical qualities.

Copper is a type of material endowed with the ability to establish natural passive surface films in appropriate environmental conditions. This characteristic of copper makes it resistant to corrosion processes in numerous aqueous environments including chemical; however, chemical environments inhabiting chloride ions among others, undermine these qualities and permit corrosion process (1).

According to Patricia *et al*., (2017), these chemicals are capable of removing scales, soil and light rust from metal surfaces (2). Apart from this, they often contained about 1 % organic corrosion inhibitors by volume of the acid such as hydrochloric acid. Synthetic inhibitors have been widely applied to protect metal surfaces against corrosion (3,4).

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However, these inhibitors are toxic, expensive with environmental and safety issues. Alternative sources including natural products, extracts from plants, and other environmental benign organic sources have been widely reported (5).

Many organic compounds have been tested for corrosion inhibition. The functionality of these compounds has been attributed to their electronegative functional groups and presence of π electrons in triple or conjugated double bonds. They also contain nitrogen, sulphur or oxygen atoms in their structures. Their mode of operation is by physical or chemical interactions between their molecules and the metal surfaces (2). Several plant extracts including an extract from *Carica papaya, Rosmarinus officinalis, Damsissa, Murrayakoenigii*, cashew, mango, *Uncaria gambir* and *Ficus sycomorus* have been investigated (6,7,8,9,10,11,12,13,14). According to Helen *et al.* (2014), these plants possess adequate cyclic organic phytochemicals, nitrogen, sulphur and oxygen atoms that are responsible for their inhibition properties (15). The large-scale synthesis of various natural plant extracts is faced with many challenges. The chief of which is the isolation of specific components of the plant extract with inhibitory characteristics. Nevertheless, many natural plant extracts have proven efficient as corrosion inhibitors (16,17,18). To gain insight on the working and applicability of natural extracts for commercial purposes, inhibition mechanism, kinetics and process thermodynamics was investigated on a pilot scale. The extract, after careful concentration under controlled temperature, may however, be added in the right mix to paints of organic solvent base for easy dissolution and dispersion.

Corrosion of metal takes place whenever there is an interaction of two different electrochemical reactions on the material surface. With detail knowledge of these electrochemical processes, potential theory is commonly applied for the prediction of corrosion rate. In many cases, these data are not available which limit the application of mixed potential theory with confidence. Laboratory measurements are therefore made and interpreted in terms of mixed potential theory such as polarization resistance, electrochemical impedance spectroscopy (EIS) and electrochemical (4,19,8). These methods involve the use of advanced instruments with expert advice that is often not available to many researchers. Gravimetric-based method such as weight loss provides integrated mass loss information from corrosion that has occurred over some period of time. Because of simplicity and economic friendliness, gravimetric techniques are commonly used to measure general corrosion rate (14). However, gravimetric-based techniques are unsuitable for continuous field monitoring of corrosion rates.

This present study deployed gravimetric and qualitative techniques to evaluate another ecofriendly material *Luffa cylindrica* Leaf Extract (LCLE), for use as an engineering inhibitor on mild steel (MS) submerged in an alkaline solution. The optimum condition, the kinetics and thermodynamic parameters for maximum corrosion inhibition using *Luffa cylindrica* Leaf Extract (LCLE) were established.

1.1. Statement of the Problem

Corrosion of metals, i.e., the initiation and acceleration of the surface deterioration of metals through an electrochemical reaction with the surrounding intrusive environment, is a global concern because of the economic and environmental impacts. Corrosion is one of the destructive processes affecting the performance of metallic materials in applications of many construction sectors. Corrosion inhibitors are considered the most practical choice among the available corrosion protection techniques due to their effectiveness in terms of functionality and cost. The utilization of green corrosion inhibitors purely acquired from renewable sources is explored, with an in-depth focus on the recent advancements in the use of fruit and vegetable extracts as green corrosion inhibitors (5). Fruits and vegetables are natural sources of various phytochemicals that exhibit key potential in corrosion inhibition.

Corrosion process forms corrosion product and deposits on corroding copper surface which interferes with its electrical, thermal and mechanical qualities and leads to huge economic loss and possible safety concerns (20).

The use of organic compounds containing electronegative functional groups and conjugated πelectron within multiple bonds have been studied and proven to form compact thin films on metal surfaces which enhance the passivation potentials of metals during corrosion processes (21). For this study, *Luffa cylindrical* leaf extract will be used to inhibit the corrosion of copper in an alkaline environment.

Aim and Objectives

The aim of this work is to evaluate the inhibitory adsorption characteristics of *Luffac cylinderical* leaf extract as a corrosion inhibitor of copper in alkaline environment with the following objectives:

- to determine the corrosion inhibition efficiency of the plant on copper.
- to determine the effects of temperature variation on the inhibition process.
- to determine a suitable adsorption for the corrosion inhibition of copper in alkaline environment.

- to examine the surface morphology of copper before and after corrosion inhibition
- studies.

1.2. Scope and Limitation of the Study

This research will be limited in scope to the use of ethanolic leaf extract of *Luffa cylinderica* in an alkaline environment. This work will also be limited to 303, 313 and 323K using gravimetric measurement, UV-visible, Fourier Transform Infrared Spectroscopic (FTIR), X-ray Diffraction (XRD) and Scanning Electron Microscope (SEM) characterization techniques.

1.3. Significance of the Study

This research work is carried out to support the works of many other researchers who are solving the problem of environmental pollution by toxic compounds by using naturally occurring plant source such as; *Luffa cylindrica* Leaf Extract as corrosion inhibitor of copper in alkaline environment.

1.4. Justification of Study

This research focuses on the corrosion inhibition of Luffa cylindrica Leaf Extract (LCLE) on corrosion of copper in alkaline environment. Success of this work will stimulate more to use plants extract inhibitors than synthetic inhibitors.

2. Literature review

The corrosion of metals is probably one of the most essential aspects of metallurgical sciences, as corrosion-related failures may result in disastrous health and environmental issues (22). According to (Fayomi *et al.,* 2019) coating, cathodic or anodic protection, electroplating, and several other methods are useful approaches for the corrosion protection of metallic structures by isolating the surface from the corrosive environment (23). As an alternative to the abovementioned methodologies, solution chemistry can be adopted to tune down the corrosion process at the metal–solution interface (24). Such actions mostly include the use of corrosion inhibitors, which are additives that slow down the rate at which metal corrodes when added in minute quantities (25). The action of a corrosion inhibitor can be altered or improved via an increase in the extent of adsorption of the inhibitor on the surface of the metal through the mechanism of physisorption or chemisorption or both (26). An increase in the rate of diffusion of the inhibitor (in the bulk solution) to the surface of the metal or an increase in the miscibility of the inhibitor in the solution can be considered (27).

The selection process of corrosion inhibitors has undergone several transformations over the years (28). The development of corrosion inhibitors was focused on the use of inorganic salts or metal oxides, followed by a shift to organics, which has also received wider criticism in recent years due to the presence of unacceptable levels of heavy metals and other toxic compounds, poor solubility (especially in polar electrolytes), and reduced stability under critical conditions (29). The corrosion inhibitors that have been in high demand in recent times are those that meet the conditions shown in Figure 2. (30). In this respect, fruit and vegetable extracts serve as excellent candidates as non-toxic and sustainable corrosion inhibitors. Hence, the current study seeks to evaluate the current advancements, challenges, and opportunities in the field of corrosion inhibition with fruit and vegetable extracts.

Table 1 Characteristics Expected from A Good Corrosion Inhibitors

S/N	Characteristics Expected From A Good Corrosion Inhibitors	Features
1	Economic	It Should Be Less Expensive To Get Or Synthesize
2	Accessibility	It Should Be Easily Accessible And Simple To Apply
3	Environmental Friendliness	It Should Not Be Toxic To The Environment, Either Directly Or Indirectly.
4	No Surface Modification	The Consequences Of Using The Corrosion Inhibitor Should Not Involve Surface Modification Of The Metal.

Considering the above features, significant research effort has been made on the use of organic corrosion inhibitors. Al-Bagawi, (2021) with the advent of green chemistry, the concept of green corrosion inhibition has aroused increasing attention (4). A vast amount of literature exists on the use of natural products (amino acids, polyphenols, and alkaloids), ionic liquids, and plant extracts as green corrosion inhibitors (31).

Plant extract utilization in corrosion inhibition is gaining enormous acceptability and research interest, considering the increasing volume of research articles being published in the field of corrosion inhibition (32). It is certain that there are several known and established corrosion inhibitors that have excellent inhibition efficiency compared to fruit and vegetables extracts, but the major limitation of these other classes of corrosion inhibitors (such as chromate, nitrite, etc.) is their failure to meet the environmental requirements (33). Various studies have been carried out on plant extracts as green corrosion inhibitors during the last decade. The basic techniques for the analysis of corrosion inhibition are explored. The challenges and prospects for the widespread use of fruit and vegetable extracts as corrosion inhibitors are discussed. Four equations are basically needed to explain the chemistry involved in foul removal. Three of those equations represent cathodic processes and anodic processes [i.e., the dissolution of tubular material

$$Fe_2O_3 + 4Cl^- + 6H^+ + 2e \rightarrow 2\ FeCl_2\ (aq) + 3H_2O \ \ldots\ldots \text{Eqn 1}$$

$$Fe_3O_4 + 6Cl^- + 8H^+ + 2e \rightarrow 3FeCl_2\ (aq) + 4H_2O \ \ldots\ldots \text{Eqn 2}$$

$$2H + 2e^- \rightarrow H_2 \ \ldots\ldots \text{Eqn 3}$$

$$Fe + 2Cl \rightarrow FeCl_2\ (aq) + 2\ e^- \ldots\ldots \text{Eqn 4}$$

(Adindu *et al.,* 2017) studied *Ocimum gratissimum* leaves extract as an organic green inhibitor on the corrosion by adopted certain investigation tools (gravimetric, electrochemical, FTIR, and scanning electron microscopy) to investigate the inhibition of the acidic corrosion (0.5 M H_2SO_4 and 1 M HCl) of mild steel by *Ocimum gratissimum* leaf extract (34). They obtained inhibition efficiency values of 92.4 and 71.1% for the metal in HCl and H_2SO_4, respectively. From the FTIR spectra of the inhibitor and corrosion products, the functional groups responsible for the adsorption of the inhibitor were deduced, while the scanning electron micrograph revealed the smooth surfaces of the metals, the corrosion of which was inhibited.

Ashish and Quraishi, 2010 reported that adsorption isotherms explain the degree of interaction of molecules of various inhibitors with the metal surface. Since corrosion inhibition using organic inhibitor occurs with the development of protective films caused by the adsorbed extract molecules on metal surfaces (10). Isotherm equations were used to confirm that the inhibition mechanism is truly adsorption. It is also used to establish the closest equation that relates the concentration of inhibitors to the adsorbed concentration at saturation, the empirical equations such as exponential, hyperbolic, logarithmic and power are difficult to associate with the given mechanisms of the adsorption process. The Freundlich, Langmuir, and Temkin isotherms apart from their simplicity are easy to apply to derive complete information from their parameters to characterize the corrosion inhibition system.

Okewale *et al*., 2020, provided a brief report on the effectiveness of a pumpkin pod extract as a corrosion inhibitor and provided a statistical relationship between the inhibition efficiency and factors that affect it, such as the time, concentration, and others (35). A detailed study to elucidate the mechanism of inhibition was not reported in their work. Pumpkin seeds have been employed by Radi *et al*., 2021 to inhibit the corrosion of aluminum in saline solution (NaCl), with a maximum recorded efficiency of 95% at the inhibitor concentration of 1 g/L. Atomic force microscopy, scanning electron microscopy, and X-ray diffraction studies have revealed information on the protection of the surfaces of metals when corrosion was inhibited compared to metals in blank solution. Electrochemical methods employed for such studies indicated that the extracts acted as cathodic inhibitors, while the results of curve fittings to adsorption isotherms indicated the Langmuir isotherm as the best-fitting isotherm (36).

(Nwachukwu *et al.,* 2021) adopted different blends of guava and pumpkin leaves to inhibit the corrosion of mild steel in hydrochloric acid and found an optimum inhibition efficiency of 95%. They reported that the combined extracts contained various phytochemicals and that the inhibition efficiency was dependent on the period of contact, the concentration of the inhibitor, and the temperature (37). Based on the application of response surface analysis, the authors concluded that the experimental inhibition efficiency was very close to the proposed theoretical value. However, due to the openness of the corrosion inhibition mechanism, this projection may fail. The work was based on weight loss only and did not use other established methods of analysis. The data sample size might not have been close to the expected population data, in addition to other interactions. Despite this, it is evident from the results obtained that the leaves of this plant were a major contributor to the corrosion inhibition process. Anaee, (2015) reported good inhibition efficiency (range = 72.4 to 75.7%) against the corrosion of carbon steel in a petroleum environment between 323 and 353 K. The results were obtained using different investigative methods, namely galvanostatic polarization, open circuit potential measurement, optical microscopy, and Fourier transform infrared spectroscopy techniques. Increases in the quantity of the extract led to increased efficiency. Potentiodynamic polarization plots indicated the inhibitor to be a mixed-type inhibitor. The adsorption fitted the Langmuir isotherm and was consistent with the physisorption mode (38). (Mailbulangu *et al.,* 2017) also acknowledged the inhibitory role of pumpkin leaf toward the corrosion of copper in an acidic medium. They observed characteristics that are typical of adsorption inhibitors with physisorption mechanisms (39).

The above review showed that the extract's inhibition efficiency is certainly a function of the phytochemical constituents of the leaf.

2.1. Forms of general corrosion

2.1.1. Atmospheric Corrosion

This is the corrosive attack on metals by the pollutant components of air. It is a common occurrence in ferrous metals on which no inhibitors or protective coats have been applied. In this case, a uniform rust layer is formed over the entire surface that has been exposed to air (40).

2.1.2. Galvanic Corrosion

This is the type of corrosion that occurs between two dissimilar metals that are in electrical contact with one another in the presence of an electrolyte. They are referred to as a galvanic couple. One of the metals, the anode, is corroded preferentially while corrosion in the other metal, the cathode, is slowed. When the same metals are put separately into the same kind of electrolyte, they corrode at their own specific rates. It is an electrochemical process and the potential difference between the two metals is the force that drives this type of corrosion. Galvanic corrosion can also be referred to as bimetallic corrosion. It is of great concern to the marine industry as well as industries which have pipes or metals in contact with salt water. Environmental variables such as temperature, salinity and humidity as well as the size of the anode in relation to the cathode and the specific types of dissimilar metals are all factors that affect galvanic corrosion (41).

2.1.3. High Temperature Corrosion

This is the corrosive attack by molten salt and metals, solids and gases that generally occurs at temperatures above 400. This type of corrosion is found in places dealing with very high temperatures such as furnaces, gas turbines and diesel engines as well as other machinery that interact with hot gas containing some corrosive contaminants. Some forms of high temperature corrosion include oxidation, carburization, sulphidation, deposit corrosion and flue gas corrosion. It is possible to take advantage of some of the products of high temperature corrosion. For instance, oxides formed on stainless steels can act as a protective barrier that prevents further atmospheric attack. This is advantageous because the material can then be used for much longer in height as well as room temperatures. These kinds of products of high temperature corrosion can help reduce the warring away of materials when they slide over each other at high temperatures (42).

2.2. Forms of localized corrosion

2.2.1. Crevice Corrosion

Crevice corrosion refers to the type of corrosion that occurs in discrete places where two metal parts are joined together, unintentionally forming a crevice. When the crevice is of a favourable size, a difference in oxygen concentration between the outer part (which has more oxygen) and the inner part (which has less oxygen) results in the formation of a cathode and anode respectively at each site. Corrosion occurs at the anode, the inner part of the crevice consequently increasing the size of the crevice. This type of corrosion is particularly insidious because it usually occurs at sites that are hidden from sight and can remain undetected till it causes total failure or breakdown of the machine component. It is a recurring problem in situations where materials are in some form of mechanical connection.

Examples of sites where this type of corrosion occurs easily are crevices made by welds that are incompletely fused, gasketed joints, O-rings and riveted seams. Crevice corrosion can be affected by the geometrical features of the surface for example, how rough it is; the conditions of the environment such the oxygen concentration or the pH; the composition of the alloy and finally, whether the two materials joined are of the same metal or not (43).

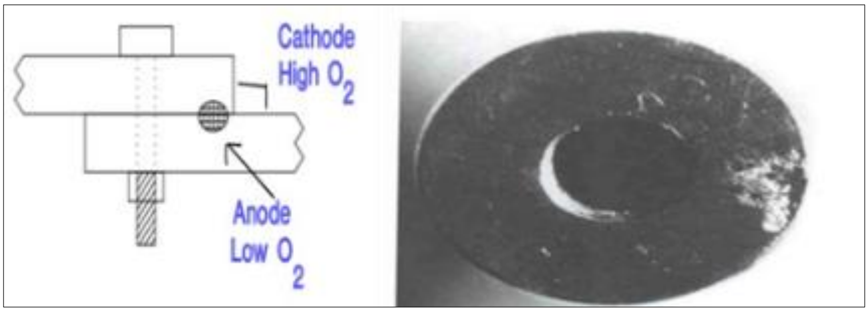

Figure 1 Crevice corrosion of stainless steel

2.2.2. *Filiform Corrosion*

Filiform corrosion is the type of corrosion that appears under thin organic films that are used to coat metals. The films are usually about 0.1mm thick. This form of corrosion appears as worm-like threads or filaments coming from one or multiple sources scattered in many different directions. It can also be referred to as under film corrosion or filamentary corrosion. The worm-like threads are lines of the products formed from the corrosion of the metals underneath the coating. The force that drives filiform corrosion is a differential oxygen concentration between the anode which is the head growing under the paint and the cathode which is at the tail end. Filiform corrosion usually follows the marks left behind by the grinding or polishing of the metal and it is unaffected by the metallurgy of the material. This type of corrosion can be found where organic coatings are used on beverage cans, gaskets, flanges and even aircraft structures (44).

2.2.3. *Pitting Corrosion*

Pitting corrosion is the type of corrosion that appears as holes or cavities in the metal. It is a very localized form of corrosion because it does not spread across a large area but is confined to specific often hidden spots. It is regarded as more dangerous than uniform corrosion because its detection or prediction is quite difficult. As such, it is also very difficult to design against it. Its driving force is usually when a small area of the material stops being passive, thus creating a suitable anode. The surrounding area then becomes a cathode resulting in a much-localized form of galvanic corrosion. It is a very menacing type of corrosion because a small occurrence of pitting that has not resulted in great metal loss can still result in the total failure of an entire engineering system. Important to the discussion of pitting corrosion is the pitting potential. It refers to the potential beyond which it is suitable for pitting corrosion to begin. Knowledge of the pitting potential is useful in predicting the tendency of a material to undergo pitting corrosion (44).

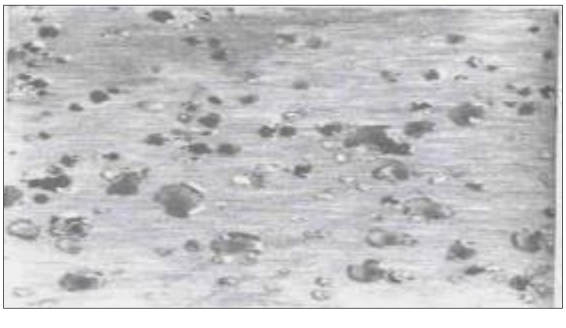

Figure 2 Localized pitting of stainless steel

2.2.4. *Localized Biological Corrosion*

Localized biological corrosion which can also be referred to as microbiologically induced corrosion is the type of corrosion brought about by the metabolic activities of some microorganisms. They are usually chemoautotrophs; these microorganisms can alter the chemical environment on the surface of a metal. As a result of this, the electrochemical processes taking place on the metal are affected, giving rise to a change in the rate of corrosion on the metal. The activities of microorganisms can affect both the type and rates of electrochemical processes occurring on the metal resulting in a large variety of outcomes. It can give rise to very severe localized corrosion, and it can also cause a significant reduction in the rate at which the metal corrodes. It can result in crevice corrosion, pitting corrosion, selective de-alloying, acceleration of the erosion rate and enhancement of galvanic corrosion. Some microorganisms can carry out their activities in the absence of oxygen (Anaerobic) giving rise to products like hydrogen sulfide resulting in sulfide stress cracking. They can also carry out their activities in the presence of oxygen (Aerobic), directly oxidizing iron into oxides and hydroxides. Microorganism is ever present in fluid systems so as a result, this kind of corrosion is common in marine environments (45).

2.3. Forms of metallurgically influenced corrosion

2.3.1. Inter-granular Corrosion

Inter-granular corrosion is the type of corrosion characterized by corrosion of the grain boundaries or areas that are close to the grain boundaries with little or next to no corrosion occurring on the grains. It is usually the result of precipitation or impurities present at grain resistance is depleted. These factors cause the grain boundary to be anodic to the remainder of the surface resulting in an increased corrosion rate of just the grain boundary and nearby areas. The occurrence of inter-granular corrosion is greatly influenced by the properties and microstructure of the metal. This type of corrosion moves along the grain boundary in a narrow path. In severe cases of this type of corrosion, the total breakdown of the grain boundary can lead to entire grains of metal being dislodged causing failure of machine systems (46).

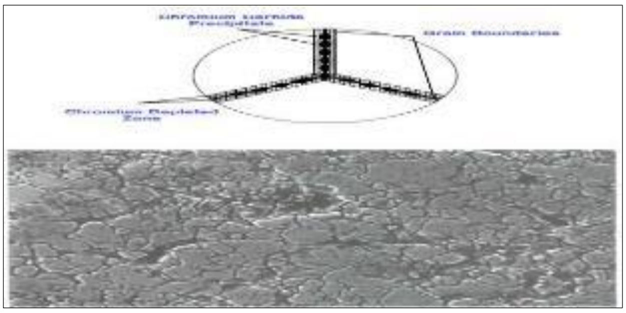

Figure 3 Inter-granular attack

2.3.2. De-alloying Corrosion

De-alloying corrosion also known as Selective Leaching corrosion refers to the type of corrosion in which one component of an alloy which is anodic to the remainder of the alloy components is corroded preferentially. In this type of corrosion, the more reactive metal in an alloy experience accelerated galvanic corrosion with respect to the more noble metal. Kinds of alloys most susceptible to this form of corrosion are those which contain metals which are far apart in the galvanic series. A good example of this is brass. Brass contains copper and zinc. Zinc is more reactive than copper and as such, zinc is the anode in the alloy.

Copper is nobler and is the cathode in the alloy.

Figure 4 De-alloying of brass

As a result of this, the zinc is corroded selectively, and the copper is enriched causing the colour of the brass to turn from yellow to red. This form of dealloying corrosion is referred to as dezincification. Other forms of dealloying corrosion are graphitization, destannification, decobaltification and so on (23).

2.4. Forms of mechanically assisted degradation

2.4.1. Erosion Corrosion

Erosion corrosion, also referred to as impingement corrosion is a cyclic process that involves a combination of erosion as well as corrosion that leads to a faster deterioration of a metal. This type of corrosion occurs because of the motion of fluids carrying abrasives on metals. These abrasives wear away and eventually deplete the passive layer, exposing the metal to corrosive attack. Even if a passive layer is formed again, the fluid in motion will continue to wear it away until the bare metal is exposed again to corrosive attack. The rate of erosion corrosion is greatly affected by the number of abrasive particles contained in the fluid as well as the rate of flow of the fluid. Almost all equipment types which encounter fluids in motion are susceptible to this type of corrosion. Examples of such equipment include inlet pipes, blades of steam turbines, steam condenser tubes, pump impellers and so on.

Figure 5 Erosion corrosion

2.4.2. Fretting Corrosion

Fretting corrosion is the type of corrosion which occurs between two metals in close contact which undergo small but continuous relative motion. The small relative motion can be caused by vibration or some other similar force. This motion causes the wearing away of the surfaces in contact, creating debris that remain at the point of contact. This debris as well as the exposed surfaces are then corroded. The products of corrosion formed from the debris are usually harder than its source material and can act as abrasive, further worsening the amount of fretting corrosion. This kind of corrosion shows up as grooves which are lined with corrosion products. The severity of fretting corrosion is affected by factors such as the amplitude and frequency of the relative motion, the temperature and corrosiveness of the surroundings as well as the contact load between the two materials. This kind of corrosion is commonly found at surfaces like the bolted tie plates on rails or in rolling contact bearings (47).

2.4.3. Cavitation and Water Drop Impingement

This is the type of material degradation that occurs when vapour bubbles are formed and collapse immediately on a metal surface. The formation and collapse happen when the operational pressure of a fluid falls below the vapor pressure. This can occur in a very explosive manner, with the collapsing bubbles producing pressures of up to 60,000psi. This extreme amount of pressure can result in plastic deformation in metals. Furthermore, this process can result in the formation of an airlock. This will hamper the flow of any incoming fluid, further worsening the problem. The surface damage that occurs because of cavitation closely resembles that of pitting corrosion. This type of corrosion is found in pump impellers, hydraulic turbines, ship propellers (47).

2.4.4. Corrosion Fatigue

This refers to the material degradation that occurs when metals undergo repeated stress while being in a corrosive environment. It must be separated from the concept of fatigue because fatigue is restricted to when there is no corrosive environment (As is the case in dry inert gas or if the metal is surrounded by a vacuum). Corrosion fatigue is like stress corrosion cracking and can be considered as environmentally assisted cracking. However, the effects of environmentally assisted cracking are more narrowed down unlike corrosion fatigue which is generalized. Most structures undergo repeated stress and have a life span regardless but when a metal is in a corrosive environment, its life span is greatly reduced.

Corrosion fatigue is a common occurrence in acid solutions and saltwater (48).

2.5. Forms of environmentally induced cracking

2.5.1. Stress Corrosion Cracking

This is the type of corrosion characterized by the appearance of cracks on a metal because of stress (Mostly static or tensile) and a corrosive environment acting simultaneously on the metal for extended periods of time. The stress resulting in this type of corrosion may come about as stresses applied directly or as residual stresses. Residual stresses may be the effect of processes like machining, grinding, heat treatment and cold deformation. The effect of this type of corrosion may appear as dry cracks in the microstructure of the metal or a general reduction of the fatigue threshold of that metal. It is a chemically specific form of corrosion. This is because it is prevalent in metal alloys rather than in pure metals. Furthermore, only some specific alloys of metals can undergo stress corrosion cracking and only when they are exposed to a limited range of chemical environments. The chemical environment capable of causing stress corrosion cracking in an alloy is usually mildly corrosive to that alloy. As a result of this, it is possible for the metals to maintain a lustrous appearance while it is filled with numerous microscopic cracks. As such it often undetected before the total breakdown of the machine or structure is affected. This type of corrosion is that observed in the cracking of brass cartridge cases of rifles when ammonia is in the environment as well as that observed in riveted boilers made of steel used in steam driven locomotives (23).

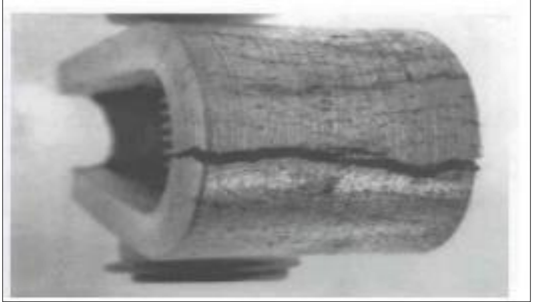

Figure 6 Stress corrosion cracking

2.5.2. Hydrogen Embrittlement

Hydrogen embrittlement is the type of corrosion that is characterized by a loss of strength as well as the ductility of a metal because of the presence of atomic hydrogen in the lattice of the metal. Hydrogen can find its way into a metal lattice through many ways but the most common of these is the exposure of metal alloys during early metallurgical processes such as chemical cleaning, cathodic protection, casting, pickling and various welding operations. This is because hydrogen enters the alloy more easily in its molten form than when it has cooled and solidified. The entry of hydrogen into the metal lowers its ductility and the amount of load it can bear before failure. It can induce cracking and brittle failure at stresses which are far below the normal yield stresses of the affected metals. It is important to note that some metals are more prone to this form of corrosion such as nickel, iron and titanium while others are much less prone for example, aluminum and copper (47).

2.5.3. Metal Ion Concentration Cell Corrosion

Concentration cell corrosion is the type of corrosion that occurs when a metal surface is in contact with an electrolytic solution at different points with varying concentration at each point. This corrosion occurs because the same metal in different concentrations of the same electrolytic solution will possess varying electrochemical properties. Metal ion concentration corrosion takes place at areas where there are two surfaces in contact at a joint in the presence of water. This is because when water is present, a relatively high concentration area of metal ions will be under the two surfaces in contact while a relatively low concentration area of metal ions be near the crevice made by the two surfaces in contact. This difference in concentration will cause an electrical potential between the areas. The low concentration area will be protected from corrosion as it will be cathodic while the high concentration area will experience accelerated corrosion as it will be anodic. This type of corrosion can be observed in pipelines passing through swampy areas that are close to dry areas or that passing through different type of soils (49).

2.5.4. Soil Corrosion

This is the type of corrosion that affects concrete, or metals located deep in the soil or bedrock. The soil is made up of numerous chemical constituents. These constituents can react with metals and even other construction materials such as concrete. The phenomenon is complex in nature, involving numerous variables. However, the rate at which soil corrodes materials varies significantly depending on the location. For example, cast iron pipes used for the transport of water may be used in the highly corrosive Southern California soil for just 20 years before experiencing failure while the same pipe can be used for the same purpose in the less corrosive New England soil for up to 50 years before experiencing failure. Some properties of soil can be used to predict the tendency of a soil to corrode ferrous metals such as the pH, its electrical resistivity as well as the chloride content of the soil (50).

2.6. Effects of corrosion

Corrosion is an omnipresent challenge that cuts across every facet of life. Some major effects include:

- Safety Effects
- Health Effects
- Technological Effects
- Cultural Effects
- Economic effects

2.6.1. Safety Effects

Corrosion is a problem that affects important structures and devices. It wears away important components of these structures, reducing their integrity. If these structures are to undergo total failure because of corrosion, it can lead to severe injuries and even a great loss of lives. This is because these structures are constantly in use by several people at a time. Example of some of these structures and devices are bridges, aircrafts, pipelines, automobiles and so on (51).

2.6.2. Health Effects

Medical advancements have reached the level at which metal prosthetic devices can be inserted into the body for various uses. These prosthetic devices may be hearing aids, pacemakers, plates, pins, hip joints and so on. Even though newer alloys and more sophisticated methods of implantation have been devised, corrosion continues to pose a serious problem. It can result in the fracture of prosthetic devices which are involved in bearing weight as in hip joints. Products of corrosion can also cause inflammation of the tissue surrounding the implants from which they are generated. Corrosion can even degrade the electrical connection contained in delicate devices such as pacemakers (43).

2.6.3. Technological Effects

Technological advancements in virtually every facet of life are hampered by the problem of corrosion. This is technology aimed at producing devices with materials that are capable of operation in environments with more hostile conditions such as stronger pressures, hotter temperatures and more highly corrosive environments. A prominent example of this issue is that faced when designing futuristic oil drills for sea and land. It involves surmounting several forms of corrosion such as soil corrosion, microbiological induced corrosion as well as sulfide stress corrosion among others. These hurdles hamper the development of economically viable systems (52).

2.6.4. Cultural Effects

All over the world, there are monuments erected for symbolic purposes. They may be in the form of statues, busts, stone symbols and so on. They are usually left out in the open where the environment if free to act upon it without ceasing. Materials with even the highest amount of protective coating are bound to corrode eventually upon daily action of the sun, rain and other atmospheric elements especially with the increased rates of pollution over the years. Even when these cultural treasures are stored in museums, they must undergo continuous restoration in order to remove traces of corrosion (53).

2.6.5. Economic Effects

According to the most recent international study carried out by the National Association of Corrosion Engineers in 2013, the global cost of corrosion is alarming indeed. It was estimated that the global cost of corrosion is USD2.5 trillion. That value represented up to 3.4% of the entire global GDP as of 2013. It was pointed out however, that by employing the corrosion control techniques available, savings in the range of 15% - 35% of that value could be made. That amounts to between USD375 billion and USD875 billion annually. This goes to show that a significant amount of funds goes to the combating and mitigation of corrosion (54).

2.7. Copper metal

Copper is a chemical element; it has symbol Cu (from Latin: cuprum) and atomic number 29. It is a soft, malleable, and ductile metal with very high thermal and electrical conductivity. A freshly exposed surface of pure copper has a pinkish-orange color. Copper is used as a conductor of heat and electricity, as a building material, and as a constituent of various metal alloys, such as sterling silver used in jewelry, cupronickel used to make marine hardware and coins, and constantan used in strain gauges and thermocouples for temperature measurement.

Copper, $_{29}$Cu

Copper

Appearance	Red-orange metallic luster

Figure 7 Copper (Cu)

Copper is one of the few metals that can occur in nature in a directly usable metallic form (native metals). This led to very early human use in several regions, from c. 8000 BC. Thousands of years later, it was the first metal to be smelted from sulfide ores, c. 5000 BC; the first metal to be cast into a shape in a mold, c. 4000 BC; and the first metal to be purposely alloyed with another metal, tin, to create bronze, c. 3500 BC (55). In the Roman era, copper was mined principally on Cyprus, the origin of the name of the metal, from aes cyprium (metal of Cyprus), later corrupted to cuprum (Latin). Copper (Old English) and copper were derived from this, the later spelling first used around 1530 (56).

Commonly encountered compounds are copper (II) salts, which often impart blue or green colors to such minerals as azurite, malachite, and turquoise, and have been used widely and historically as pigments. Copper used in buildings, usually for roofing, oxidizes to form a green patina of compounds called verdigris. Copper is sometimes used in decorative art, both in its elemental metal form and in compounds as pigments. Copper compounds are used as bacteriostatic agents, fungicides, and wood preservatives. Copper is essential to all living organisms as a trace dietary mineral because it is a key constituent of the respiratory enzyme complex cytochrome c oxidase. In mollusks and crustaceans, copper is a constituent of the blood pigment hemocyanin, replaced by the iron-complexed hemoglobin in fish and other vertebrates. In humans, copper is found mainly in the liver, muscle, and bone (57).

2.7.1. Occurrence

Copper is produced in massive stars (58), and is present in the Earth's crust in a proportion of about 50 parts per million (ppm) (59). In nature, copper occurs in a variety of minerals, including native copper, copper sulfides such as chalcopyrite, bornite, digenite, covellite, and chalcocite, copper sulfosalts such as tetrahedrite-tennantite, and enargite, copper carbonates such as azurite and malachite, and as copper(I) or copper(II) oxides such as cuprite and tenorite, respectively (60). The largest mass of elemental copper discovered weighed 420 tonnes and was found in 1857 on the Keweenaw Peninsula in Michigan, US (59). Native copper is a polycrystal, with the largest single crystal ever described measuring 4.4 × 3.2 × 3.2 cm. Copper is the 25th most abundant element in Earth's crust, representing 50 ppm compared with 75 ppm for zinc, and 14 ppm for lead (59).

Typical background concentrations of copper do not exceed 1 ng/m3 in the atmosphere; 150 mg/kg in soil; 30 mg/kg in vegetation; 2 µg/L in freshwater and 0.5 µg/L in seawater (61).

2.7.2. Characteristics of Copper

Physical Properties

Copper, silver, and gold are in group 11 of the periodic table; these three metals have one orbital electron on top of a filled d-electron shell and are characterized by high ductility, and electrical and thermal conductivity. The filled d-shells in these elements contribute little to interatomic interactions, which are dominated by the s-electrons through metallic bonds. Unlike metals with incomplete d-shells, metallic bonds in copper lack a covalent character and are relatively weak. This observation explains the low hardness and high ductility of single crystals of copper (62). At the macroscopic scale, introduction of extended defects to the crystal lattice, such as grain boundaries, hinders flow of the material under applied stress, thereby increasing its hardness. For this reason, copper is usually supplied in a fine-grained polycrystalline form, which has greater strength than mono-crystalline forms (63).

The softness of copper partly explains its high electrical conductivity (59.6×106 S/m) and high thermal conductivity, second highest (second only to silver) among pure metals at room temperature (60). This is because the resistivity to electron transport in metals at room temperature originates primarily from scattering of electrons on thermal vibrations of the lattice, which are relatively weak in a soft metal (62). According to Resistance Welding Manufacturing Alliance (2003) the maximum permissible current density of copper in open air is approximately 3.1×106 A/m2, above which it begins to heat excessively (64).

Copper is one of a few metallic elements with a natural color other than gray or silver. Pure copper is orange-red and acquires a reddish tarnish when exposed to air. This is due to the low plasma frequency of the metal, which lies in the red part of the visible spectrum, causing it to absorb the higher-frequency green and blue colors (65).

Chemical Properties

Copper does not react with water, but it does slowly react with atmospheric oxygen to form a layer of brown-black copper oxide which, unlike the rust that forms on iron in moist air, protects the underlying metal from further corrosion (passivation). A green layer of verdigris (copper carbonate) can often be seen on old copper structures, such as the roofing of many older buildings (66). Copper tarnishes when exposed to some sulfur compounds, with which it reacts to form various copper sulfides (67).

2.8. Corrosive environments for copper

Corrosive environments for copper and the corrosion inhibitors used for protection of copper in this study will be reviewed based on the corrosive environments (acidic, basic and neutral environments).

2.8.1. Copper in NaCl Solutions

(Qiang *et al*., 2015) evaluated the corrosion inhibition efficiency of 5-nitroindazole for corrosion of copper in NaCl solution. They reported that this compound acts as a cathodic corrosion inhibitor and its efficiency increases with concentration (68). Moreover, the maximum corrosion inhibition efficiency of 99% is obtained in 0.4 mM of corrosion inhibitor concentration.

2.8.2. Copper in Alkaline Environments

About the effect of corrosion inhibitors on the corrosion of copper in alkaline environments, many types of research have been done similar to acidic and neutral environments. Subramanian and Lakshminarayanan (2002) investigated the adsorption and corrosion inhibition behavior of some compounds such as imidazole (IMD), mercaptobenzimidazole (MBIMD), benzimidazole (BIMD), Mercaptobenzothiazol (MBT) and BTA on corrosion of copper in NaOH solution (69). Referring to following Langmuir adsorption isotherm, the corrosion inhibition efficiency of mentioned compounds increases in the following order: BTA = IMD < BIMD = MBIMD < MBT. The molecular structure of mentioned azoles is shown below in Fig. 2.9

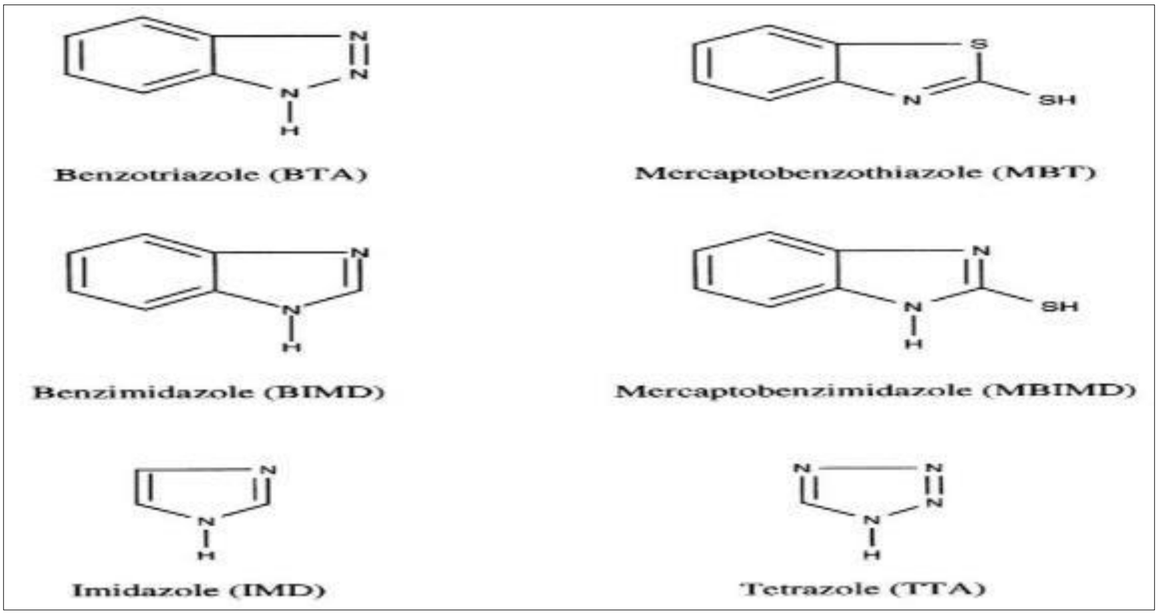

Figure 8 The structure of azoles (69)

In this regard, the corrosion inhibition of copper corrosion with azole compounds in borate buffers at various pH values (pH = 6.4, 8.4 and 10.4) has been investigated. It is reported that azole corrosion inhibitors act as mix-type corrosion inhibitors and reduce both anodic and cathodic reactions. The corrosion inhibition efficiency increases with pH value and concentration of corrosion inhibitors and the corrosion inhibition efficiency decrease in the following order: MBT > TDA > BIMD = MBIMD > BTA (70). In addition to azole compounds, the corrosion inhibition effect of cysteine for copper in Na2SO4 solution in pH = 9 has been investigated. The presence of corrosion inhibitor results in decreasing corrosion rate due to adsorption of Cu (I)ads and Cu (II)ads (at higher anodic potential) on copper surface and formation of Cu(I)-cys protective film.

2.9. Corrosion inhibitors

An inhibitor is the name given to a chemical substance or combination of various chemical substances that are added in low concentrations to a corrosive environment with the aim of significantly reducing or totally preventing the occurrence of corrosion while having little or no effect on the original process being carried out in the environment. Inhibitors act in two ways. The first is through the formation of an adsorbed layer on the metal, preventing the metal from meeting corrosive agents. The other is through the retardation of either the cathodic or anodic corrosion reactions. There are even some inhibitors used in practice that are capable of retarding both reactions (71). Inhibitors are most effective in closed systems that have adequate circulation. This ensures the stability of the required inhibitor concentration. Examples of such systems include those used for acid pickling, refining of oil and water recirculation systems. One of the most popular applications of inhibitors is the use of antifreeze or engine coolant for radiators of automobiles (72). Broadly speaking, corrosions can either be organic or inorganic, are classified based on three major criteria:

- The environment
- The mechanism
- The mode of protection

The efficiency of an inhibitor can be expressed by a measure of this improvement:

$$\text{Inhibitor efficiency (\%)} = 100\ \text{x}\,\frac{\text{CR}_{\textbf{un-inhibited}}\text{CR}_{\textbf{inhibited}}}{\text{CR}_{\textbf{un-inhibited}}}$$

Where,

$\text{CR}_{\text{un-inhibited}}$ = corrosion rate of the un-inhibited system $\text{CR}_{\text{inhibited}}$ = corrosion rate of the inhibited system.

2.9.1. Types of Corrosion Inhibitors According to Environment

Based on the environment in which the inhibitors are applied, they are classified into the following:

2.9.2. Acidic Inhibitors

These are inhibitors which have been found to be most effective in reducing corrosion in acidic corrosive media. They combat corrosion by lowering the rate at which electropositive ions are deposited on the surface of the metal. This results in the reduction of the voltage that occurs as a result of the depolarization reaction at the cathode (73). As_2O_3 and Sb_2O_3 have been found to be effective acidic inhibitors. Recent studies also show that inhibition of corrosion in iron in acidic media can be achieved by adding heavy metal ions such as Mn_2^+ and Cd_2^+ (74).

2.9.3. Alkaline Inhibitors

These are inhibitors which have been found to be most effective for the reduction of corrosion in alkaline media. They are effective because they are able to form metal complexes on the surfaces of metals in alkaline solutions. They are most often used to protect metals that form both acidic and basic oxides. These types of metals are the most prone to corrosion in alkaline media.

Example of such inhibitors include naphthol, thiourea and substituted phenols (23).

2.9.4. Neutral Inhibitors

Corrosive media that are neutral or near neutral in nature require inhibitors different to those that are acidic and alkaline. This is because the mechanism required to retard corrosion is different (75). Neutral inhibitors retard the reduction of oxygen that occurs at the cathodic sites. As a result, the surface of the metal is protected from corrosion. Chelating inhibitors is an example of inhibitors that are efficient in neutral solutions (76).

2.10. Types of inhibitors according to mechanism

Based on the mechanism of inhibition, which is the electrode process which they act upon, corrosion inhibitors are divided into the following:

2.10.1. Anodic Inhibitors

This is the type of inhibitor that retards corrosion by increasing the polarization of the anodic region. This shifts the corrosive potential towards the cathodic region. The increased anodic polarity is because of the formation of a protective barrier on the anodic region (77). As a result, the reactions that cause corrosion are significantly slowed down. Anodic inhibitors are also referred to as passivation inhibitors. The most common anodic inhibitors are ions of transition elements which have an abundant content of oxygen. Examples include tungstate's, chromates, phosphates, orthophosphates, silicates and so on. Although anodic inhibitors can be effective, they can also be dangerous. If the concentration of the inhibitor in the corrosive environment falls below the required level, areas of the surface of the metal will remain unprotected. This can result in the stimulation of pitting corrosion (72). Anodic inhibitors are of two types:

- **Non-Oxidizing ions:** This type of anodic inhibitor requires the presence of oxygen to function properly. Examples include molybdates, tungstates and phosphates.
- **Oxidizing ions:** This type of anodic inhibitors functions properly in the absence of oxygen.

Examples include Nitrates, nitrites and chromates.

2.10.2. Cathodic Inhibitors

This type of corrosion inhibitor can reduce corrosion by retarding the reduction reaction that occurs at the cathodic sites in a corrosive environment. The ions of the inhibitors form precipitates in the cathodic regions. This results in a shift in the corrosive potential from the cathodic region to the anodic region (78).The inhibition action occurs through 3 means:

- **Cathodic Precipitation:** The se inhibitors form precipitates in the cathodic region which serves as a protective layer for the surface of the metal. Examples include magnesium and calcium.
- **Oxygen Scavenging:** These inhibitors react with environmental oxygen, forming various products and reducing corrosion in the process. Examples include Sb_3^+ and As_3^+.

- **Cathodic Poisoning:** These inhibitors hinder the hydrogen recombination reaction that occurs at the cathode, reducing corrosion in the process. However, these inhibitors have the disadvantage of making the metal more susceptible to hydrogen induced cracking. Examples include arsenic, antimony and bismuth.

2.10.3. Mixed Inhibitors

This type of corrosion inhibitor has the unique property of being able to hamper the reactions that occur at both the cathodic and anodic regions. They can make protective films formed by the deposition of precipitates on the metal. This blocks the reactions at both the cathodic and the anodic indirectly, reducing the rate of corrosion (72).

2.11. Types of inhibitors according to mode of protection

Based on the mode of protection carried out by corrosion inhibitors, they are divided into the following:

2.11.1. Adsorption Inhibitors

Adsorption inhibitors are the most used type of corrosion inhibitors. They are generally organic, and they work by getting absorbed onto the surface of the metal. This forms a protective layer over the entire surface, including both cathodic and anodic regions. They are known to affect both regions equally but that may not be the case in all situations. A common use of adsorption inhibitors is in the removal of black mill scale that is formed in pickling processes. Adsorption inhibitors are mostly compounds which have lone pairs of electrons. For example, Oxygen in aldehydes, nitrogen in amines (79).

2.11.2. Chemical Passivators

Chemical passivators are substances which possess a small over potential and a high equilibrium potential (also known as electrode potential). Their specific chemical properties enable them to reduce corrosion rate by attaining passivity (43). Some applications of this type of corrosion inhibition are the use of Zinc Molybdate in paints and the use of chromates in cooling waters.

2.11.3. Film Forming Inhibitors

Film forming inhibitors reduce the rate of corrosion in metals with the formation of a protective barrier on the surface of the metal. They differ from adsorption inhibitors due to the fact that the protective layer formed by film forming inhibitors are made of materials other than the inhibitors themselves. The materials formed are usually specific to either the cathode or the anode. For example, Salts of Zinc and Calcium are film forming specific to cathodes while benzoate is film forming specific to anodes (80).

2.11.4. Vapour Phase Inhibitors

Although vapour phase inhibitors are usually substances of low vapour pressure, the vapour pressure is significant. This vapour pressure enables them to carry out their function well. Vapour phase inhibitors are most used in spaces that are closed. For example, in a parcel when it is being shipped or stored. They are also referred to as volatile inhibitors. They absorb onto the surface of the metal and are hydrolyzed by the moisture. Protective ions are then released, reducing corrosion in the process. Examples of vapour phase inhibitors include benzotriazole used for inhibiting corrosion of copper and phenyl thiourea used in protecting brass (23).

2.12. Luffa Cylindrica Plant

The *Luffa cylindrica* plant is a member of the cucurbitaceous family with smooth and rounded shaped fruits. It is called sponge gourd, vegetable sponge, bath sponge or dishcloth gourd (81). Luffa plants grow by climbing on other physical solid materials.

Typical leaves of a matured *Luffa cylindrica* plant are shown below.

Herbal medicine is still the mainstay of about 75-80% of the world population, mainly in developing countries, for primary health care because of better cultural acceptability, better compatibility with the human body and lesser side effects. The chemical constituents present in the herbal medicine or plant are a part of the physiological functions of living flora and hence they are believed to have better compatibility with human body. Natural products from plants are a rich resource used for centuries to cure various ailments. The use of bioactive plant-derived compounds is on the rise, because the main preoccupation with the use of synthetic drugs is the side effects which can be even more dangerous than the diseases they claim to cure. In contrast, plant derived medicines are based upon the premise that they contain natural substances that can promote health and alleviate illness and proved to be safe, better patient tolerance, relatively less expensive and globally competitive. So, in respect of the healing power of plants and a return to natural remedies is an absolute requirement of our time (82).

Figure 9 *Luffa cylindrica* plant

Luffa [*Luffa cylindrica* (L.) syn *Luffa aegyptiaca* Mill] commonly called sponge gourd, loofa, vegetable sponge, bath sponge or dish cloth gourd, is a member of cucurbitaceouse family. The fruits of *Luffa cylindrica* are smooth and cylindrical shaped. One mature Luffa sponge will produce at least 30 seeds. Some will produce many more. *Luffa cylindrica* has alternate and palmate leaves comprising petiole. The leaf is 13 and 30 cm in length and width respectively and has an acute-end lobe. It is hairless and has serrated edges. The flower of *Luffa cylindrica* is yellow and blooms in August-September. *Luffa cylindrica* is monoecious and the inflorescence of the male flower is a raceme, and one female flower exists. Its fruit, a gourd, is green and has a large cylindrical shape and grows climbing on other physical solid materials (83).

Taxonomy (84) Loofa sponge is a lignocellulosic material composed mainly of cellulose, hemicelluloses and lignin. The fibers are composed of 60% cellulose, 30% hemicellulose and 10% lignin (85).

A study on L. cylindrica was carried out where different chemical treatments were conducted on the fibers (L. cylindrica) with aqueous solutions of NaOH 2%, or methacrylamide (1 - 3%) at distinct treatment times. L. cylindrica was characterized via chemical analysis and analytic techniques such as FTIR, XPS/ESCA, X-Ray, TGA and SEM. Methacrylamide 3% treatment for all times (60, 120 or 180 min) severely damaged the fibers. NaOH, on the other hand, showed the same beneficial effect regarding enhancement of surface area and thermal stability together with similar levels of lignin and hemicellulose extraction, without causing exaggerated harm to fiber integrity (86).

Luffa cylindrica Linnage.

- Kingdom : Plantae
- Division: Mangoliophyta
- Class: Mangoliosida
- Order: Cucurbitales
- Family : Cucurbitaceae
- Genus : Luffa
- Specie : Cylindrica

Vernacular Names

- Hindi : Ghiatarui
- Sanskrit : Rajakoshataki
- Bengali : Dhundul
- Tamil : Pikku
- Telungu : Guttibira
- Bombay state : Ghosali
- Malayalam : Tureippirku

2.12.1. Geographical source

Luffa cylindrica is a sub-tropical plant, which requires warm summer temperatures and long frost-free growing season when grown in temperate regions. It is an annual climbing plant which produces fruit containing fibrous vascular system. It is summer season vegetable. It is difficult to assign with accuracy the indigenous areas of luffa species. They have a long history of cultivation in the tropical countries of Asia and Africa. Indo-Burma is reported to be the center of diversity for sponge gourd. The main commercial production countries are China, Korea, India, Japan and Central America (83).

2.12.2. Phytochemistry

- The fruit contains triterpenoid saponins: lucyosides A, B, C, D, E, F, G, H, I, J, K, L, M (87) ginsenosides Re, Rg1, etc.
- The leaf contains triterpenoid saponins: lucyin A, lucyosides G, N, O, P, Q, R (88).
- 21β- hydroxyoleanoic acid, 3-O-β-D glucopyranosyl- maslinic acid (89) ginsenosides Re, Rg1; flavonoids:apigenin (90).
- The seed contains polypeptides: luffins P1, S (91) (Li *et al.,* 2003), luffacylin (92).

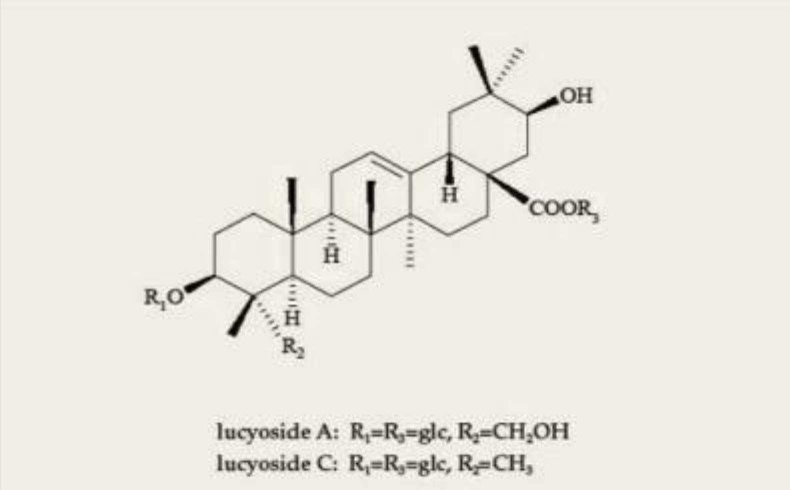

Figure 10 Triterpenoid saponins: lucyosides A and C

(93) **l** has carried out Hydrophilic antioxidant constituents in the fruits of the vegetable *Luffa cylindrica* (L.) which were separated by an antioxidant guided assay which was evaluated by radical scavenging effect on the DPPH radicals and concluded that the consumption of sponge gourds can supply some antioxidant constituents to human body (93).

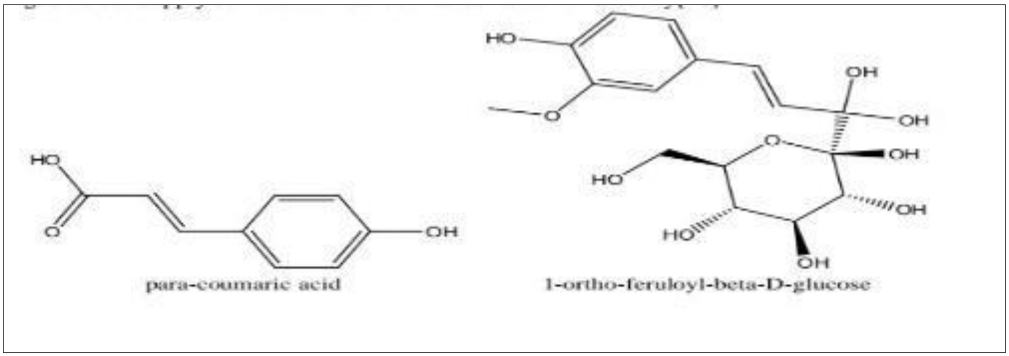

Figure 11 Para-coumaric and l-ortho-feruloyl-beta-D-glucose

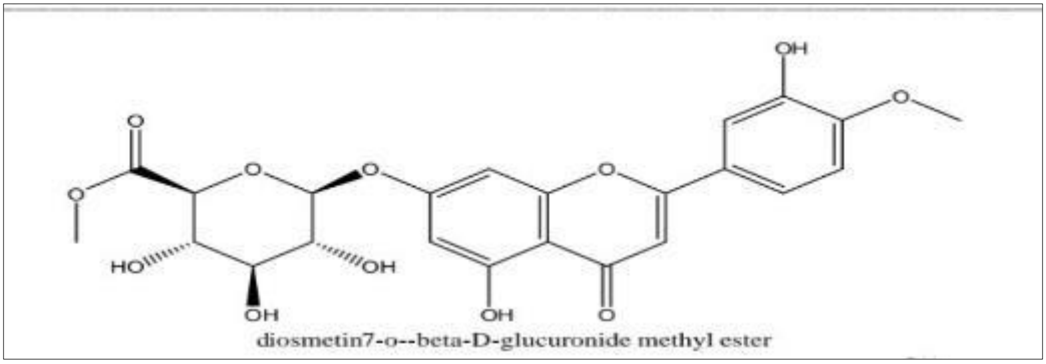

Figure 12 Diosmetin7-o- -beta-D-glucuronide methyl ester

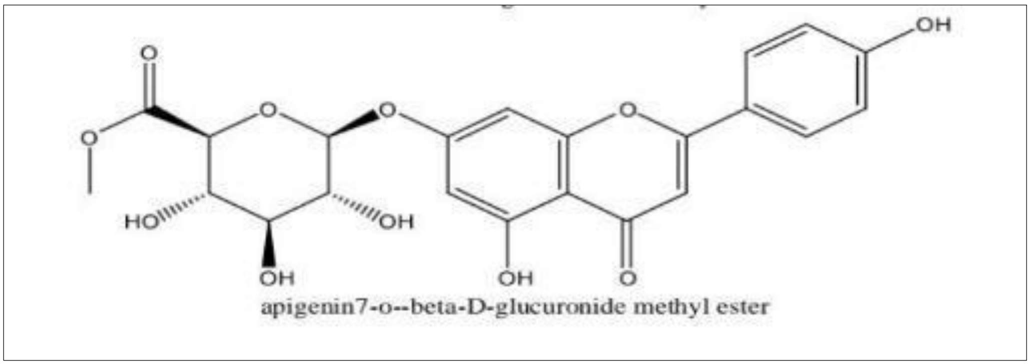

Figure 13 Apigenin7-o—beta-D-glucuronide methyl ester

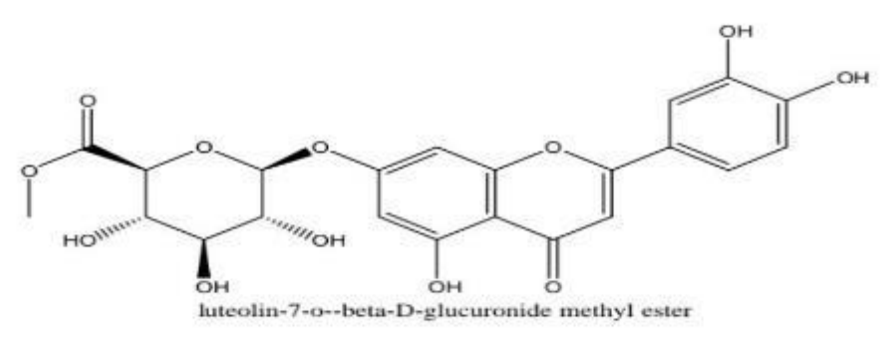

Figure 14 luteolin-7-o- -beta-D-glucuronide methyl ester

Anamika *et al.*, (2007) isolated Two triterpenoids sapogenins 1&2 from the ethanolic extract of the defatted powdered seed of L.cylindrica. Two triterpenoids sapogenins 1&2 were structure elucidation & characterized by ESIMS, 1DNMR and also 2NMR especially HSQC, HMBC and 1h-1H COSY techniques (94).

Okuyama *et al.,* (1991) two new fibrinolytic saponins, Lucyoside N and P, were isolated from the seeds of Luffa cylindrica Roem. (Cucurbitaceae) (95). On the basis of chemical and spectral evidence, Lucyoside N was characterized as 3-O-beta-D-galactopyranosyl- (87) beta-Dglucuronopyranosyl-28O-beta-D-xylopyranosyl- (96) -beta-D glucopyranosyl- -alphaL -rhamnopyranosyl- (82) alpha-arabinopyranosyl quillaic acid. Lucyoside P was characterized as a gypsogenin glycoside with the same sugar moiety as Lucyoside.

3. Materials and methods

3.1. Materials

The materials used includes Luffa *cylinderica* leaf extracts (LCLE), organic solvents (ethanol), separating bottle, Erlenmeyer flask, Buchner funnel, suction pump, filter papers, oven, reflux system, sample bottles, beakers, rotary evaporator, retort stand, glass funnel, distilled water, reagents,

3.2. Methods

3.2.1. Collection of Plant Extract

Fresh leaves of *Luffa cylinderica* were gathered in large quantity, from a nearby bush in Mission quarters Wukari, Taraba State. The leaves were selected from around same source, to ensure no contaminated leaves of different plant or species. The leaves were cleaned and dusted and finally washed with running tap water to remove impurities. The leaves were air-dried at room temperature, shredded into smaller pieces to reduce leaves surface area, thereby increasing drying. This is done until the leaves are no greener indicating they were dried, for about 3 weeks. In order to remove moisture content from the leaves and to aid pulverization, the dried leaves were oven-dried at 90 °C for about 2 hours until the sample were crispy. The crisp sample from the oven was immediately pulverized. It was then crushed (to reduce the size) and then pulverized using an industrial blender while still crispy. The powdered leaves (sample) were then stored in an air-tight glass bottled and covered (97).

Figure 15 Fresh leave of Luffa cylinderica dried leave of Luffa cylinderica pulverized Luffa cylinderica

3.2.2. Extraction Process

Using 70% ethanol for solvent extraction at room temperature, the pulverized sample of LCLE was soaked for 48 hours until a dark-green color was observed indicating that extraction has taken place. The darkish-green solution was filtered out which contains the plant extract while the residue is the chaff from pulverized leaves. The procedure was repeated on the chaff to ensure maximum extraction.

3.2.3. Purification and Concentration

Purification involved separation and refluxing for specific period of time. After separation of the filtrate using a separation funnel, the organic layer is the desired sample was then refluxed to ensure homogeneity of the sample. Here, bonds were broken and made within the molecules of the sample. The product of purification was then concentrated in other to be stored for further usage. The concentration of the extract of plants was carried out using a Rotary Evaporator which removes solvents from the extract thereby making it slurry.

3.2.4. Preparation of Metal Coupons

The specimen of mild steel was machined into rectangular coupons by lathe machine of dimension 20.21 mm by 14.63 mm as shown in Fig. 3 below. The edge of the coupons was polished with emery paper of 600 grades. The thickness was determined with Mitutoyo brand of analog micrometer screw gauge while the dimension by Mitutoyo digital venier gauge of 2 decimal places. The surface treatment of the coupons were without further polishing but grease on their surfaces were removed by immersion in absolute ethanol and then rinsed with double distilled water and acetone. They were then stored in moisture free desiccators to prevent rusting or contamination before use (98)

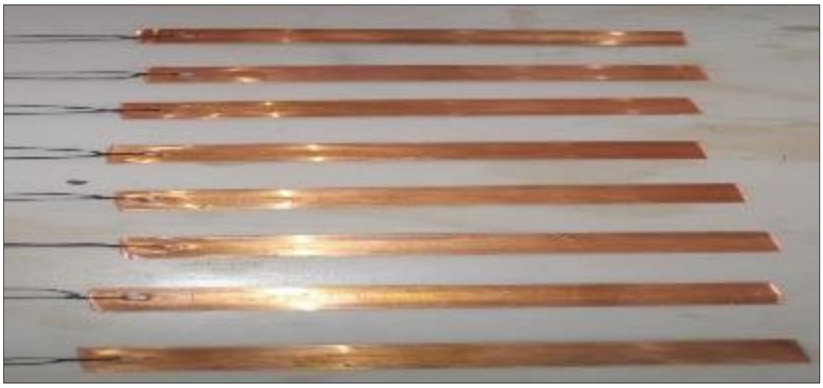

Figure 16 Metal coupons for corrosion test

3.2.5. Weight Loss Determination of Extract Inhibition

In the weight experience, five 250mL beakers (in two sets) were labeled A, B ,C ,D and E containing 0.01, 0.02, 0.03, 0.04 and 0.05 NaOH solution respectively. Another five sets of beakers containing 100, 300, 500, 700 and 1000 mg/dm3 concentration of the *Luffa cylindrical* leave extract respectively. One set of each was placed in a water bath maintained at 303K and 313K. The copper coupons were suspended in the beaker with the aid of glass hooks. The coupons were retrieved from their corrodent solution at 24hours interval for seven (7) days. Further measurement was carried (25). The weight loss was calculated using the formula:

$$W = (Wi - Wf) \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \; 3.1$$

Where W is the weight loss of coupons, Wi is the initial weight of copper coupon and Wf is the final weight of copper coupon after immersion. The inhibition efficiency of *Luffa cylindrica* leave extract acting as inhibitor was calculated using the formula:

$$I_E\% = \frac{W_0 W_1}{W_0} \times 100 \ldots\ldots\ldots..3.2$$

Where Wo and W1 are the weight losses (g) for copper in the presence and absence of additives respectively in NaOH solution at the same temperature (6)

The degree of surface coverage, Ө is given by the equation.

$$\theta = \frac{W_0 W_1}{W_0} \ldots\ldots\ldots..\mathrm{Eqn5}$$

The corrosion rate (CR) of copper in the different corrosion medium and *Luffa cylindrical* leave extract was determined for a 168 hours' immersion period from weight loss using the formula.

$$\mathrm{CR} = \frac{\Delta W}{DAT} \ldots\ldots\ldots\ldots..Eqn6$$

Where W = weight loss (mg), D = destiny of the specimen (g/cm^3), A = area of the specimen (cm^3) and T is the exposure time (99,100)

The activated energy values gotten in the study were computed from the transformed Arrhenius equations

$$E_a = \left[2.303\, R \,\frac{T_1 - T_2}{T_2 - T_1}\right] \left[\mathrm{Log}\, \frac{P_2}{P_1}\right] \ldots\ldots\ldots\ldots\ldots\ldots\mathrm{Eqn7}$$

Where R is the gas constant, P_1 and P_2 are corrosion rate at T_1 and T_2 respectively. The rate constants (K) were obtained from the linear plots of logarithm weight loss versus time, t (days) while the half-life, $t_{1/2}$, values were calculated using the following equation:

$t_{1/2} = 0.693/k$ ……………………………………………………………………………………………Eqn8

The value of heat of adsorption, $Q_{ads,}$ for the study were determined from the following

$$\text{Qads} = 19.147 \left[\text{Log} \frac{\Theta_2}{1-\Theta_2} - \, log \, \frac{\Theta_1}{\Theta_1} \right] \left[\frac{T_1 - T_2}{T_2 - T_1} \right] \text{…………… ………………Eqn9}$$

Where Θ_1 and Θ_2 are degree of surface coverage at 303K and 313K respectively (99,100)

Further analysis was carried out using SEM and XRD techniques to determine the surface morphology and crystal lattice structure of the samples respectively.

3.3. Scanning electron microscope (sem) analysis

Scanning electron microscopy model JSM-IT710HR provides morphology and structural image of nanomaterial. Surface analysis was done using SEM on copper metal, in the presence and absence of the inhibitor.

3.3.1. X-ray diffraction (xrd) determination

X-ray diffraction analysis of corrosion product was carried on electrochemically polarized copper surface using XRD machine, model TX55. An investigation was undertaken to examine the feasibility of X-ray diffractometry for the identification and characterization of corrosion products formed on copper surface in various media (in the present of the inhibitor and in NaOH solution).

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4. Results

4.1. Weight Loss Measurement

Table 4.1, 4.2 and 4.3 show calculated values for weight loss and corrosion rate at 303K and

313K Calculated values for Surface coverage and Inhibitor efficiencies at 303K and 313K and Kinetic data for copper belt in 0.01M NaOH Solution with inhibitor (*Luffa cylindrical*) at 303K and 313K. Figure 4.1 – 4.8. Show Graph of Weight loss against Inhibitor concentration at 303K and 313K, Plot of corrosion rate against Inhibitor concentration at 303K and 313K, Plot of corrosion rate against Inhibitor concentration at 303K and 313K, plot of Inhibition efficiency versus Inhibitor concentration at 303K and Plot of Inhibitor efficiency against Inhibitor concentration at 313K, Plot of inhibitor efficiency against inhibitor concentration at 323K, Plot of inhibitor efficiency against inhibitor concentration at 303K and 313K, Plot of log of Weight loss versus time, t (day) at 303K and313K, and the SEM analysis.

Table 2 Calculated values for Weight loss and Corrosion rate

Inhibitor conc. (mg/dm^3)	**Weight loss (Δw)**		**Corrosion rate (CR)**	
	303k	**313K**	**303k**	**313K**
100	0.5610	0.5900	2.6246×10^{-5}	2.7603×10^{-5}
300	0.4820	0.5201	2.2550×10^{-5}	2.4333×10^{-5}
500	0.3550	0.4750	1.6609×10^{-5}	2.2222×10^{-5}
700	0.3120	0.3810	1.4597×10^{-5}	1.7825×10^{-5}
1000	0.2710	0.3005	1.2679×10^{-5}	1.4059×10^{-5}

Table 3 Calculated values for Surface coverage and Inhibitor efficiencies at 303K and 313K

Inhibitor conc. (mg/dm3)	Inhibitor Efficiency (IE %)		Surface Coverage (Ɵ)	
	303k	**313K**	**303k**	**313K**
100	39.8700	32.1633	0.3987	0.3216
300	48.3712	381541	04837	0.3815
500	56.7149	51.1200	0.5671	0.5112
700	64.3532	56.2800	0.6435	0.5628
1000	72.1846	68.1242	0.7218	0.6812

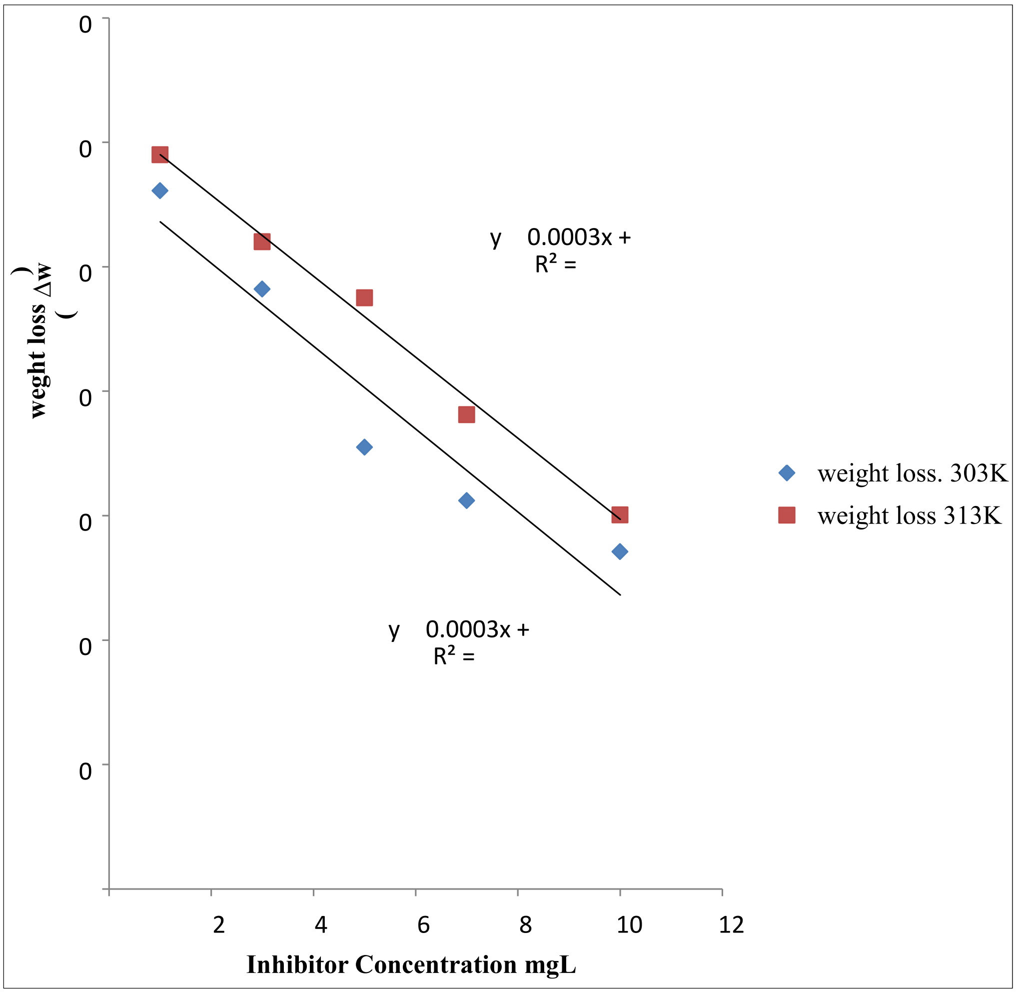

Figure 17 Graph of Weight Loss Against Inhibitor Concentration at 303K and 313K

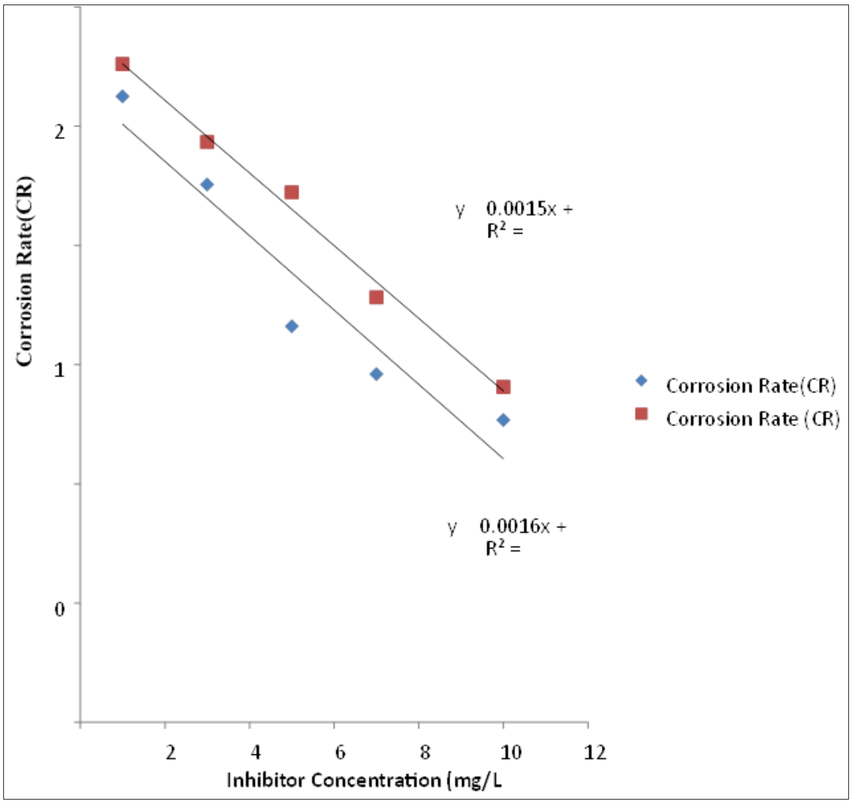

Figure 18 Plot of Corrosion Rate against Inhibitor Concentration at 303K and 313K

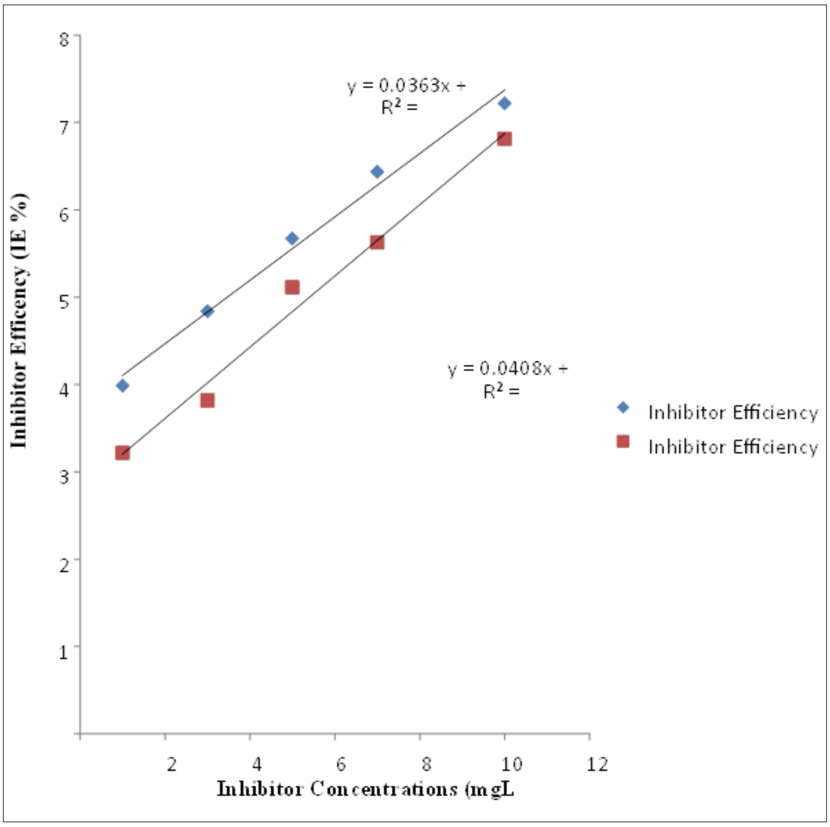

Figure 19 Plot of Inhibitor Efficiency against Inhibitor Concentration at 303K and 313K

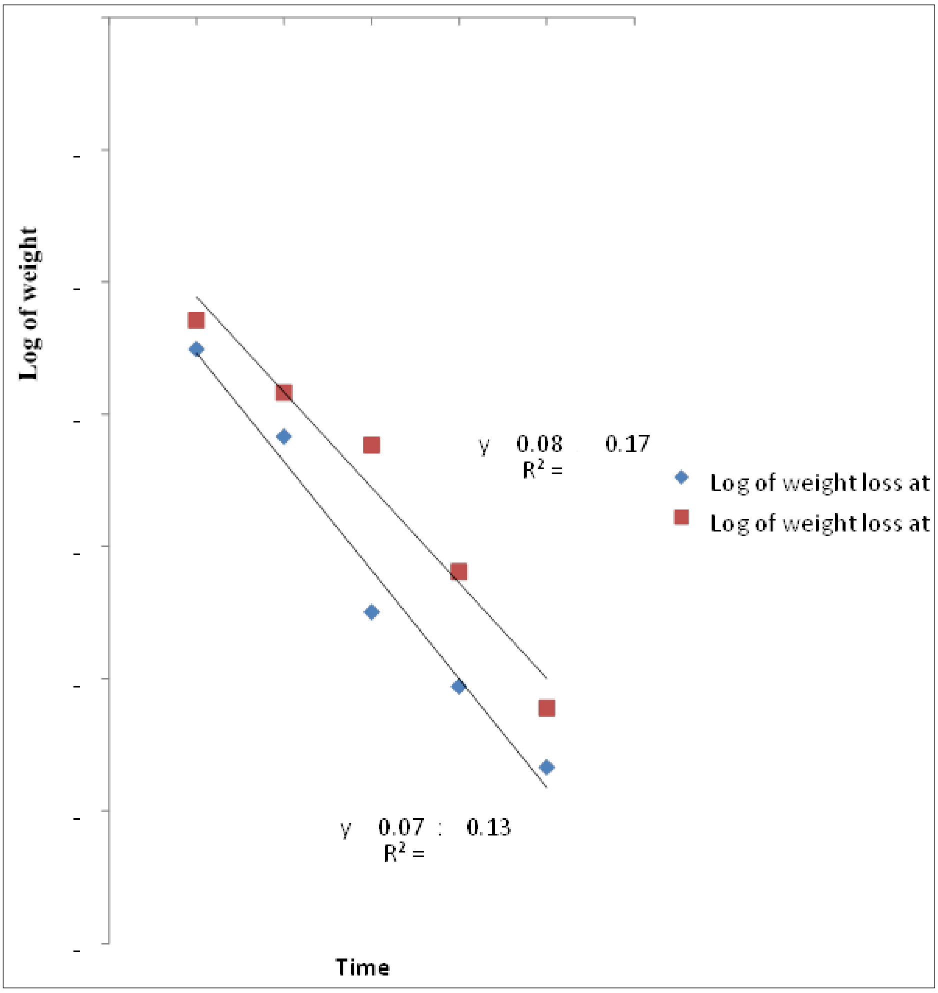

Figure 20 Plot of Log of Weight Loss Versus Time, t (days) at 303k and 313K

Table 4 Kinetic Parameter for Copper Corrosion in 0.01M NaOH Solution with Inhibitor from Weight Loss Measurements

Inhibitor Conc (Mg/dm^3)	**Rate constant K (min^{-1})**		**Half-life $t_{1/2}$ (min)**		**Activated energy ($kJmol^{-1}$)**	**Heat of absorption Qads ($kJmol^{-1}$)**
	303k	313K	303k	313K		
100	0.0790	0.0730	8,7722	9.4932	3.8933	-26.4619
300	0.0659	0.0549	10.5159	12.6229	5.8772	-32.9617
500	0.0442	0.0131	15.6787	15.6787	22.4851	-17.7496
700	0.0281	0.0258	24.6619	26.8604	15.4298	-26.6599
1000	0.0200	0.0344	34.6500	20.1453	7.9793	-15.3085

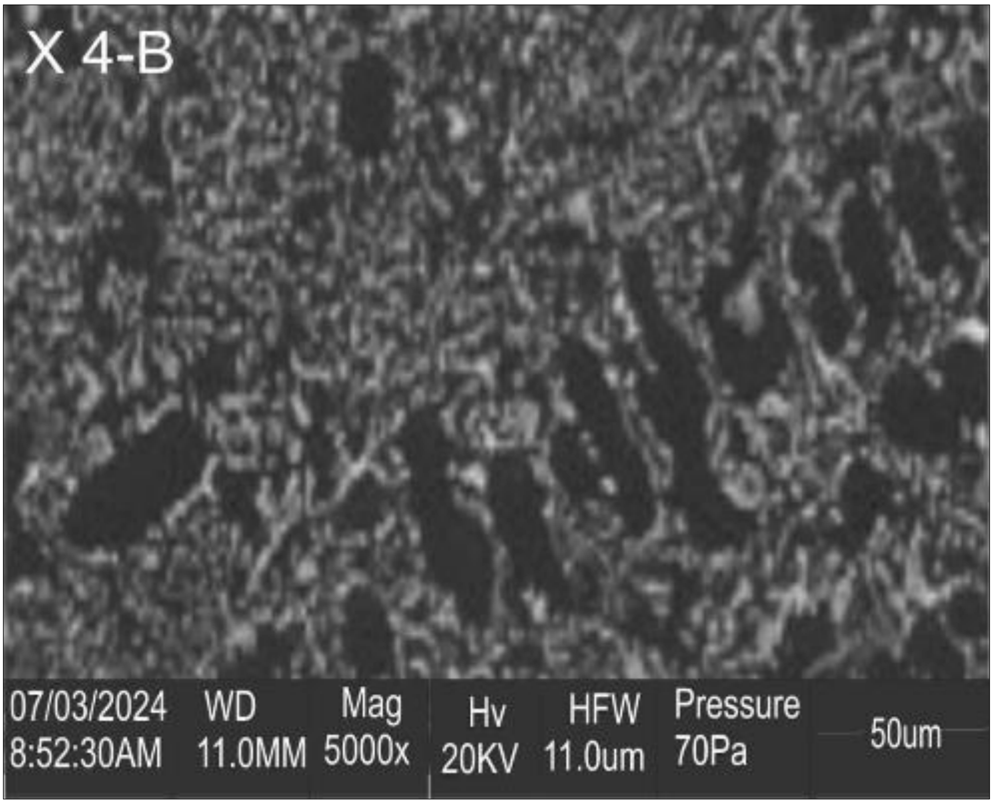

Figure 20 Pictorial View of Copper Metal from SEM Analysis in the Presence of Inhibitor

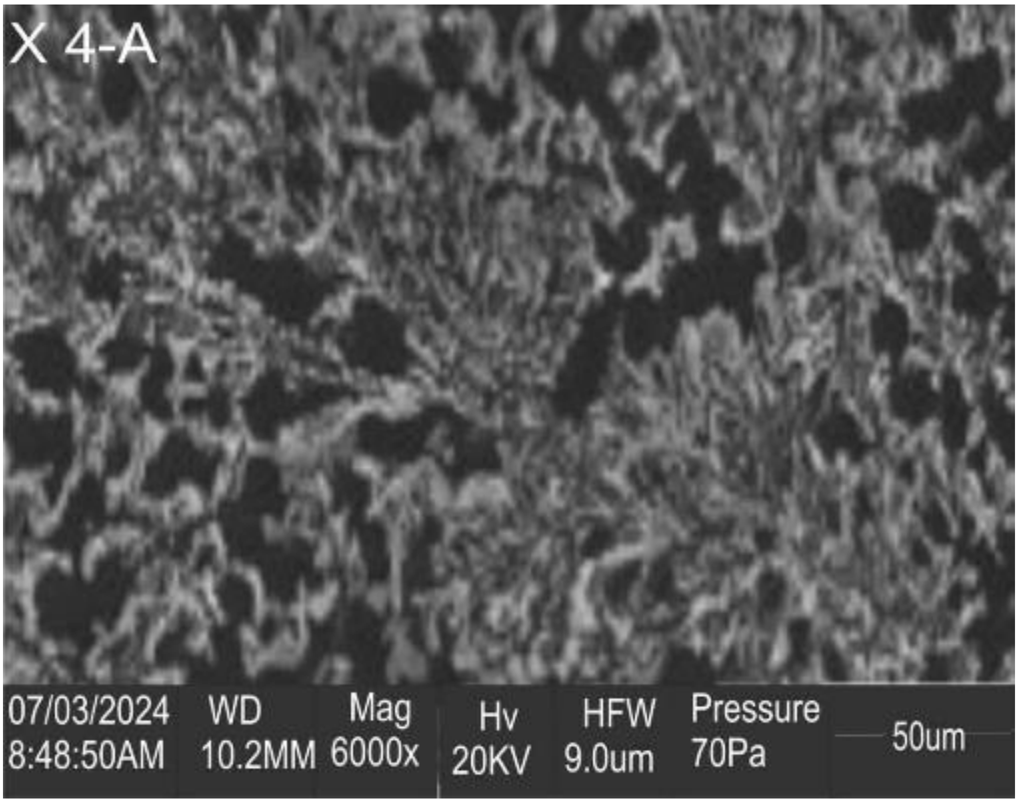

Figure 21 Pictorial View of Copper Metal from SEM Analysis in the Absence of Inhibitor

4.2. Quantitative Analysis Report

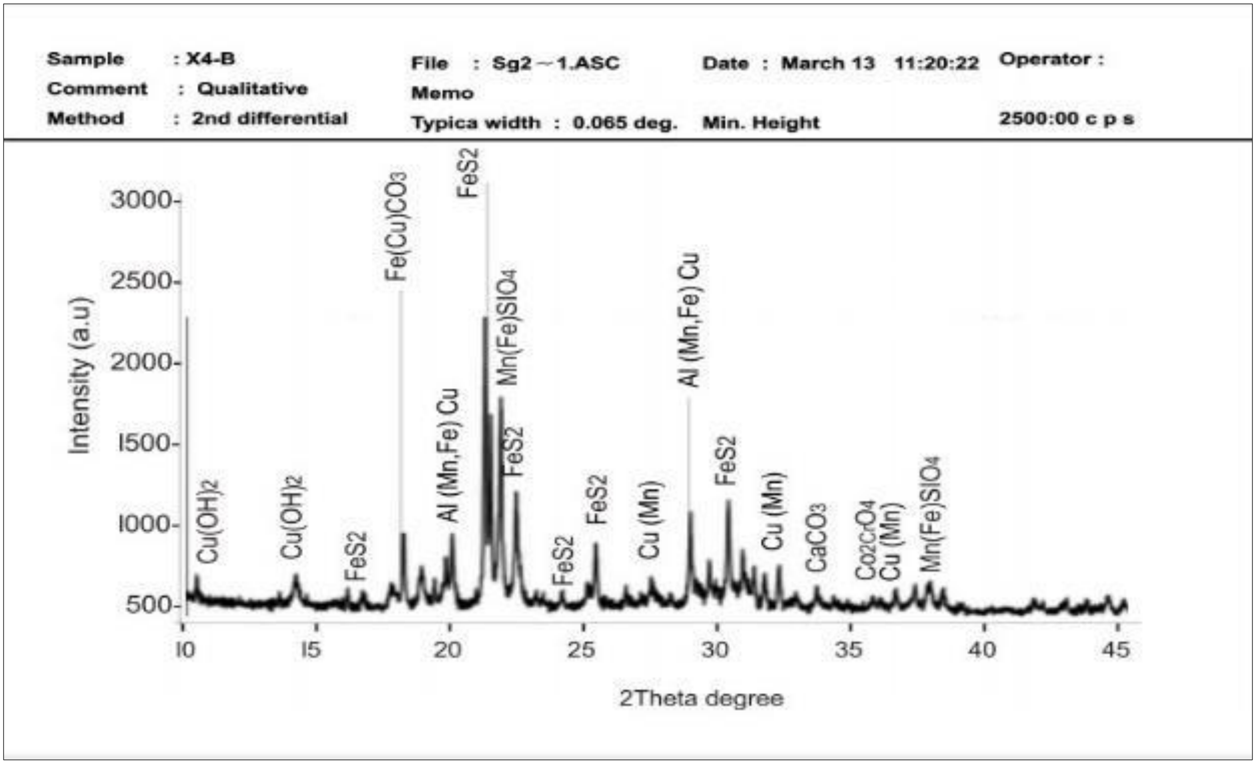

Figure 22 Phase Data View of Copper Metal from XRD Analysis in the Absence of the Inhibitor

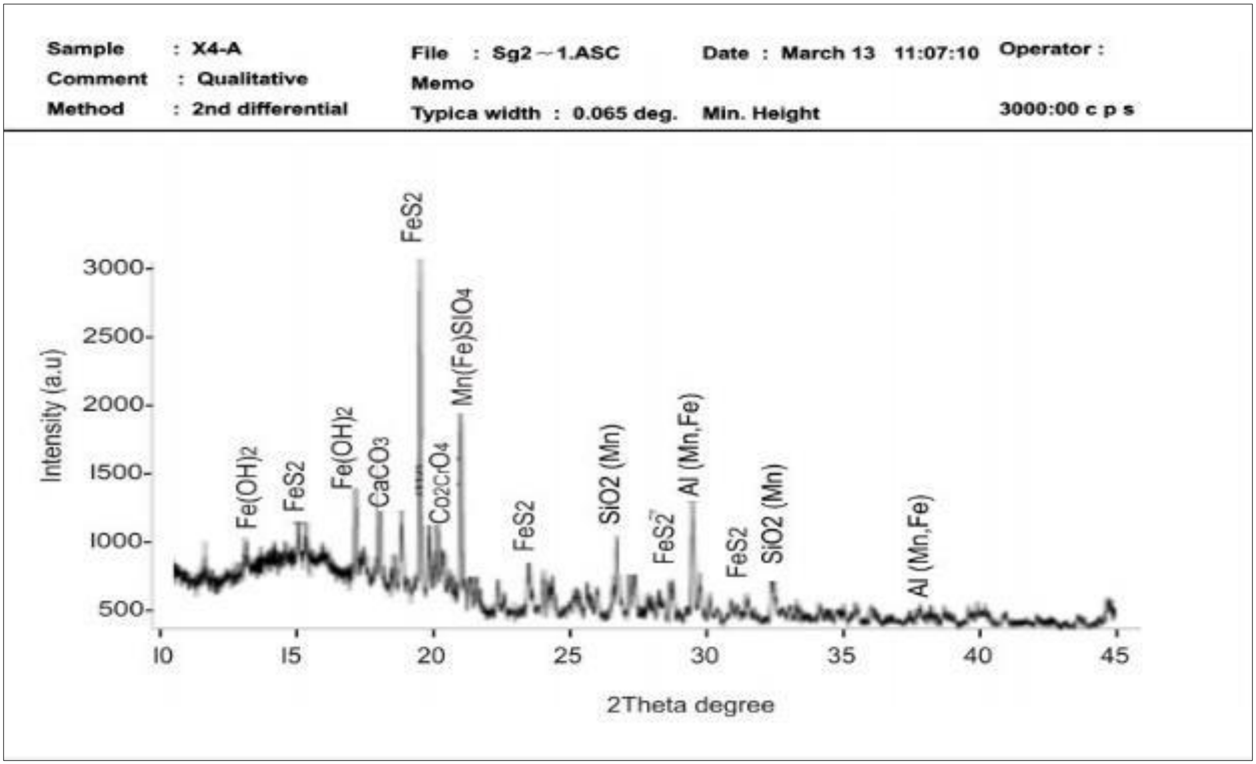

Figure 23 Phase Data View of Copper Metal from XRD Analysis in the Presence of the Inhibitor

5. Discussion

5.1. Effect of Inhibitor (*Luffa cylindrica*) Concentration on Copper Corrosion

Figure 4.1 show variation of weight loss with inhibitors concentration for copper in 0.01NaOH with various concentration of *Luffa cylindrica* leaf extract (LCLE) at 303K and 313K respectively. The graph reveals that weight loss was lowest at 1000mg/dm^3, this indicates that (LCLE) inhibits corrosion of copper to some extent, with inhibition increase with increasing inhibitor at lower temperature. With the addition of inhibitor (LCLE) the inhibition efficiency (%I $_E$) and surface coverage were found to increase while corrosion rates decrease with increasing concentration in the order: 100mg/dm^3 1000mg/dm^3 at 303K (72.1846 % efficiency), and at 313K (68.1242% efficiency).

5.2. Effect of Inhibition Efficiency (%I$_E$) on Inhibitor Concentration

Table 4.2 shows the evaluation of inhibition efficiency of (LCLE) against inhibitor concentration of copper corrosion in 0.01M NaOH solution after 24 hours immersion respectively for different temperatures. The increase in the inhibition efficiency with concentration may be due to adsorption of (LCLE) onto the copper surface through the non-bonding electron pairs of N and O atoms as well as π-electron of the aromatic ring. This is mainly because of the phytochemical detected.

5.3. Effect of Corrodent Concentration and Temperature on Copper Corrosion

Corrosion rate was observed to be slightly higher at 313K and 303K. A plot of log of weight loss of copper specimen against time (days) in 0.01M NaoH solution for 168 hours as shown in Figure 4.4 indicates that the weight loss decreases as the number of days increase and increase as the temperature increases.

Table 4.1 and 4.2 shows variation of weight loss and corrosion rate with days for copper in 0.01M at 303K and 313K with (LCLE). It was observed that weight loss and corrosion rate of copper increase with concentration of NaOH solution. This could be because the rate of chemical reaction increases with increase in concentration of the alkaline and the temperature.

5.4. SEM Analysis

Surface analysis was carried out using SEM (model JSM-IT710HR) on copper metal, in the presence and absence of inhibitor. A significant surface damage was observed on copper metal which was immersed in 0.01 NaOH Figure4.6 indicating that the surface was highly corroded.

The surface of the corroded area was protected by the addition of the inhibitor as evident from Figure 4.5. the surface morphology and the presence of the inhibitor at the optimum concentration exhibited a smooth surface indicating restriction to corrosion unlike Figure 4.6, the SEM image clearly indicates that the metal surface was protected from corrosion in the presence of plant extract which is acting as an inhibitor. The result is a good agreement with the weight loss measurement

5.5. XRD Analysis

Structural analysis was carried out on copper using XRD (model TX55). It was observed that the copper metal was crystalline in nature with elemental composition of $Fe(OH)_2$, FeS_2, $CacO_3$, CO_2CrO_4, Mn(Fe) SiO_4, SiO_2(Mn), Al(Mn, Fe) at different intensities (500, 1000, 1500, 2000, 2500, 3000 a.u).

5.6. Inhibition Mechanism

Calculated values of activation energy, (E_a) were obtained. The average value of the activation energy, E_a for inhibitor is 11.1329kJmol^{-1} is lower than 40.0kJmol^{-1} as shown inTable 4.2 indicating that the compound is physically adsorbed on the copper.

Physical adsorption requires that the average Ea be ≤ 40.0kJmol^{-1} and present inhibition efficiency increases at lower temperatures. This agrees with the earlier report by (25).

5.7. Thermodynamic Consideration

Adsorption is the process of attracting molecules of adsorbate to the adsorbent's surface. As a result, energy is released, and the heat of adsorption is negative, implying that adsorption is always exothermic. The values of adsorption are negative for copper corrosion as shown in Table 4.3. Indicating that the process is spontaneous, which is a characteristic of strong interaction of the inhibitor with metal surface

6. Conclusion

The efficiency of *Luffa cylindrica* leaf extract (LCLE) as a green corrosion inhibitor has been assessed in this research by using the weight loss, depth of attack and surface analyses. The efficiency (% I_E) of inhibitor increases with increase in the inhibitor concentration and decreases with increase in temperature. This implies that the inhibition rate increases with an increase in inhibitor concentration and the corrosion rate increases with temperature. The values of Q_{ads} obtained at 303K and 313K are negative, indicating that: the reaction is exothermic; the inhibitor is strongly adsorbed on copper surface and the adsorption process is spontaneous. The XRD spectra revealed the presence of functional groups containing hetero atoms. The SEM analysis showed that the inhibitor adsorption forms a protective film on the copper surface. The adsorption of extract film was found to follow Langmuir isotherm and pseudo-second-order kinetics. The values of the activation energy, enthalpy, Gibbs free energy, heat of adsorption and parameters estimated from this research suggested that the adsorption mechanism of LCLE on the copper is physisorption and exothermic.

Recommendations

In view of the results obtained from this research, I therefore recommend the use of *Luffa cylindrica* leaf extract as a corrosion inhibitor of other metals and alloys in various environments. *Luffa cylindrica* leaf extract should be used in industries as replacement for toxic chemicals, hence the extract should be used as alternative for highly expensive and nonenvironmentally friendly corrosion inhibitor of copper. Furthermore, more of the eco-friendly plants should be planted, preserved and worked on to know their inhibitor efficiency.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest to be disclosed.

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