

Investigation Of The Effect Of Green Leaf Inhibitor On Corrosion Rate Of Cold Work Mild Steel

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Abstract—Corrosion control of mild steel is technically and environmentally viable. One of the best approaches is the application of extracts of green leaf inhibitors which are environmentally biodegradable without any hazardous element or chemical compounds. Also, green leaves are cheap, renewable and readily available. A lot of organic and inorganic inhibitors employed in Nigerian industries as well as the world are hazardous and unfriendly to the environment. Inorganic inhibitors are hazardous to human health because of inhaling elements like phosphate, chromate, arsenic, nickel and carcinogenic substances. The objectives of this study were to investigate the microstructural conditionality of a specific organic inhibitor on corrosion rate of cold work mild steel in fresh water. The method of investigation employed involved immersion and gravimetric assessment of metal specimen, based on weight loss of coupons in different concentration of 0%, 5%, 10%, 15% and 20% of certified green leaves solution. The microstructures of inhibited and uninhibited coupons were examined under optical microscopic instrument. The results obtained indicate that the microstructure of heat-treated coupons had more pearlitic than the microstructure of the cold worked coupons and this phenomenon is due to less cementite on the structure. As a result of this structural re-orientation, the investigation reveal that the cold worked coupons corroded faster throughout the period of the experiment. The 15% inhibitor showed the best corrosion inhibition with the least corrosion rates for the entire experiment. The depletion of pearlite from the microstructure is as a result of corrosion effect. The study further indicates that in comparison to other green matters, the extract of Neem leaf has a high significant effect on the decrease of the corrosion of mild steel in fresh water as a result of the

formation of thin films on the substrate thereby displacing water molecules from the metal surfaces. The efficiency of the inhibitor increases with increase in concentration but decreases with increase in environmental factors such as wind and temperature.

Keywords—component; Corrosion inhibitor, Dogoyaro, Neem Leave, Mild steel, Microstructure, Pearlite, Ferrite.

I. INTRODUCTION

Mild steel is a well-known engineering structural material that has been used in chemical processing, petroleum production, refining, pipelines, mining, construction, etc., due to its exceptional mechanical properties and reduced cost, it is one of the most widely used steel in the industries. One of its limitations is that it undergoes corrosion in various operating environments in which it operates, such as saltwater, brackish water, air, acid, base and salt. Corrosion is an irrevocable process, and due to corrosion, there is restriction of progression in an industrial mild steel application. Examples of some of the applications of mild steel are bridges, ships, aircraft, food container, sheet metals used for domestic, automobile body components, building, and structural shapes and railways lines. Corrosion occurrence, control and prevention are the inevitable main scientific issues that must be looked into daily as far as there are increasing needs of metallic materials in all areas of technological development [1]. Corrosion is described as the gradual deterioration of materials due to its reaction with their environment. There are several consequences of corrosion, it varies, and their effects on the safe, reliable and efficient operation of equipment or structures are more severe than simple loss of a mass of a metal, in this regard, failure of various kinds and the need for expensive replacements may occur even though the amount of metal destroyed is quite small.

Corrosion which is a billion-dollar thief is regarded as great threat to the nation's economy. It was estimated that 10% of mild steel manufactured yearly are lost to corrosion [2]. Industrials machine parts, pipes line, bridges. Cars, aircraft, sheet metal etc are found to corrode rapidly thus constituting a health hazard to those who are directly engaged with the machines. However, Nigerian Oil and Gas Pipeline Standards, their investigation showed that 18% of pipeline failure is been generated by corrosion [3].

A lot of organic and inorganic inhibitors employ in Nigerian industry as well as the world are hazardous and unfriendly to the environment. Inorganic inhibitors are hazardous to human health because of inhaling elements like phosphate and chromate. As a result, there is need to search for environmentally friendly inhibitors which are free of health hazard. The aim and objectives of the study is to carry out an investigation of corrosion inhibition on mild steel by the use of Neem leave plant extract as an organic inhibitor in stagnated fresh water environment. The objectives are:

- To reduce the corrosion rate from the industries.
- Formulation and development of organic inhibitor from extract of Neem leaves.
- To investigate influence of organic inhibitor on micro-structure of cold worked mild steel.
- To conduct the micro- structural analysis of mild steel immersed in fresh water.
- To investigate the microstructure of mild steel when subjected to conditions of: (a) Cold worked and (b) Heat treated.
- To carry out corrosion test on mild steel using weight loss technique by varying concentration of inhibitors applied on mild steel.

II. LITERATURE REVIEW

A. Effects of Corrosion plate

Corrosion is deadly, dangerous, expensive and continuously causing rigorous damage to equipment, environment as well as mankind [4]. Corrosion is a big threat to the world because huge amount of money was wasted in maintenance, loses of life and properties during job execution. Most of the default could lead the way to structural failure, shut down of entire plant, caused defect to steel structure, machines failure, loss of production, injuries to the personnel in conjunction with the environment [5].

The Nigerian industries, which are generally dominated by marine operation suffered corrosion problem due to exposure of marine vessel to salty, brackish and fresh water environment. Corrosion is a natural existence which has different varieties of consequence on mankind. Community and individual surrounding. Corrosion failure occurs when an organic

inhibitor mechanism put in place to prevent or reduce it has been failed or unnoticed or neglected [6].

B. Causes of Corrosion

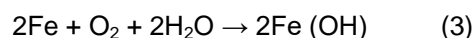
Corrosion is the reverting of unstable metal to stable form such as Iron (II) Oxide and Iron (III) Oxide which is known as hematite or iron ore. Corrosion is well-known as an indispensable method in which the breaking down of engineering component takes when the metal reacts with it environment. It is a natural process, irreversible process and it is not easy to eradicate entirely from the industry [7]. Often, metals get rust when combining with corrosive medium such as wettish or humid air, brackish water, fresh water salt water, bases and acids. Basically, corrosion is natural phenomenon which regress a refined metal to more chemically stable form, its oxide, hydroxide, sulphide. It is the gradual destruction of materials by chemical reaction with their environment [8]. Chemical reaction for rusting of metal (Fe) is iron plus water plus oxygen to form rust. For metal (Fe) to undergo corrosion at the surface of engineering materials, the following reactions occur [9].



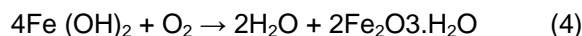
From equation (1), iron become positively charged ion after losing electrons. It is this factor that makes positively charged iron to undergo chemical reaction with other atoms of negatively charged ions [10]. Moist mild steel gets corrode to different types of oxides such as Iron (II) Oxide and Iron (III) Oxide so that redox half reaction of water (H₂O) and oxygen (O₂) must occur.



From the reaction (2), oxygen dissolved in water to form hydroxide ion. It is interesting to note that only negatively charged atoms that can chemically react with the iron and which are product of first chemical reaction [11].



Oxygen gradually dissolves in water and sometime in excess, the excess oxygen reacts with iron hydroxide to form hydrated iron (iii) oxide [12].



C. Factors that Influences Corrosion Rate

i. Nature of the Metal

Position in the electrochemical series: We have seen that different metal/metal ion combinations have different values of standard electrode potentials. Various elements can be arranged in order of increasing or decreasing values of their standard

reduction potentials. The extent of corrosion depends upon the position of the metal in the electrochemical series. Greater the oxidation potential, the greater is the rate of corrosion. When two metals are in electrical contact, the metal higher up in the electrochemical series becomes anodic and suffers corrosion. Furthermore, the rate and severity of corrosion depend upon the difference in their positions in the electrochemical series. The greater is the difference, the faster is the corrosion of anodic metal. The electrochemical series, also called activity series consisting of some electrodes along with their respective reduction reactions has been given in table 1 [13].

ii. Relative areas of the anode and cathode

The rate of corrosion is more when the area of the cathode is larger. When the cathodic area is larger, the demand for electrons will be more and this results in increased rate of dissolution of metals at anodic regions.

Table 1: Standard Electrode Potentials

Cathode (Reduction) Half – Reaction	Standard Potential E^0 (volts)
$\text{Li}^+ + e^- \rightleftharpoons \text{Li}$	-3.05
$\text{K}^+ + e^- \rightleftharpoons \text{K}$	-2.93
$\text{Ca}^{2+} + 2e^- \rightleftharpoons \text{Ca}$	-2.87
$\text{Na}^+ + e^- \rightleftharpoons \text{Na}$	-2.71
$\text{Mg}^{2+} + 2e^- \rightleftharpoons \text{Mg}$	-2.37
$\text{Al}^{3+} + 3e^- \rightleftharpoons \text{Al}$	-1.66
$\text{Ti}^{2+} + 2e^- \rightleftharpoons \text{Ti}$	-1.63
$\text{Zn}^{2+} + 2e^- \rightleftharpoons \text{Zn}$	-0.76
$\text{Cr}^{3+} + 3e^- \rightleftharpoons \text{Cr}$	-0.74
$\text{Fe}^{2+} + 2e^- \rightleftharpoons \text{Fe}$	-0.44
$\text{Cd}^{2+} + 2e^- \rightleftharpoons \text{Cd}$	-0.40
$\text{Ni}^{2+} + 2e^- \rightleftharpoons \text{Ni}$	-0.25
$\text{Sn}^{2+} + 2e^- \rightleftharpoons \text{Sn}$	-0.14
$\text{Pb}^{2+} + 2e^- \rightleftharpoons \text{Pb}$	-0.13
$2\text{H}^+ + 2e^- \rightleftharpoons \text{H}_2$	-0.00
$\text{Cu}^{2+} + 2e^- \rightleftharpoons \text{Cu}$	+0.34
$\text{Fe}^{3+} + e^- \rightleftharpoons \text{Fe}^{2+}$	+0.77
$\text{Ag}^+ + e^- \rightleftharpoons \text{Ag}$	+0.80
$\text{Hg}_2^{2+} + 2e^- \rightleftharpoons 2\text{Hg}$	+0.85
$\text{F}_2 + 2e^- \rightleftharpoons 2\text{F}^-$	+2.87

iii. Purity of the metal

The impurities present in a metal create heterogeneity and thus galvanic cells are set up with distinct anodic and cathodic areas in the metal. The higher the percentage of impurity present in a metal, the faster is the rate of corrosion of the anodic metal. For instance, impurities such as Pb and Fe in zinc lead to till formation of tiny electrochemical cells at the exposed part of the impurity and the corrosion of zinc around the impurity takes place due to local action. It is

evident that the corrosion resistance of a metal may be improved by increasing impurity.

iv. Physical state of the metal

Metal components subjected to unevenly distributed stresses are easily corroded. Even in a pure metal, the areas under stress tend to be anodic and suffer corrosion. Caustic embrittlement takes place in stressed parts such as bends, joints and rivets in boilers [14].

v. Nature of the Oxide film

Metals such as Mg, Ca and Ba form oxides whose volume is less than the volume of the metal. Hence, the oxide film formed will be porous, through which oxygen can diffuse and bring about further corrosion. On the other hand, metals like Al, Cr and Ni form oxides whose volume is greater than that of the metal and the nonporous oxide film so formed will protect the metal from further corrosion.

vi. Solubility of the products of corrosion

If the corrosion product is soluble in the corroding medium, the corrosion of the metal will proceed faster. On the other hand, if the corrosion product is insoluble, then the protective film formed tends to suppress corrosion [15].

D. Types of Corrosion

The classifications of corrosion are

i. Dry corrosion

This type of corrosion takes place in the needfulness of a liquid phase or above the dew point of the environment. Vapours and gases are usually the corrodents. Dry corrosion is often associated with high temperature; a good example is the attack on steel by furnace.

ii. Wet Corrosion

Wet corrosion takes place when a water or liquid is present. It usually involves an aqueous solution or electrolyte. This is the most common type of corrosion. A good example is corrosion of turbine blade by water [16].

E. Forms of Corrosion

Corrosion occurs in difference forms. Its classification is usually based on one or all of the three factors

- Nature of the corrodents: Corrosion can be classified as wet or dry. Liquid or moisture is usually required for wet corrosion and dry corrosion involves reaction with high temperature gases
- Mechanism of corrosion: This is usually involving electrochemically or direct chemical reactions
- Appearance of the corroded metal: Corrosion may be uniform and the metal corrodes at the

same rate over the entire surface, or it may be localized, in which case only small

The forms are as follows

i. Uniform Corrosion

Uniform Corrosion is also known as general corrosion. General corrosion is the result of chemical or electrochemical reactions which proceed over the entire exposed surface at about the same rate. General corrosion results in the metal becoming thinner and usually alters the appearance of the surface. General corrosion could result in failure through lowering the mechanical strength of components or by reducing wall thickness until leaking results. It can be prevented or reduced by coating, cathode protection or use of inhibitors [17]. This form of corrosion, however, is not of too great concern from the technical standpoint because the life of equipment can be accurately estimated on the basis of comparatively simple tests or, experience [18]. Most of the other forms of corrosion are insidious in nature and they are considerably more difficult to predict. They are the forms that cause unexpected or early failures and consequent problem or headaches to the management [19]. Figure 2 shows an example of uniform corrosion.



Fig. 2. Uniform Corrosion.

ii. Galvanic or Bimetallic Corrosion

Galvanic corrosion is common form of corrosion in marine environments. It happens when two (or more) dissimilar metals are brought into electrical contact under water [20]. This method of corrosion occurs with an electrolyte like seawater. Metals that have positive and negative charges of ions potentially become electrically connected and put in an electrolyte, the more functional metal which has a high negative potential becomes the anode.

Due to its high negative potential, it corrodes fast. But the less active metal becomes the cathode. The flow of electric current continues till the potentials are equal between both electrodes. So at the joint where the two non-similar metals meet, the galvanic corrosion appears. Hence galvanic corrosion can be break by choosing the two metals which are close in electrochemical series. As platinum is the least active,

it is also less active for corrosion [20]. Figure three shows an instance of galvanic corrosion.

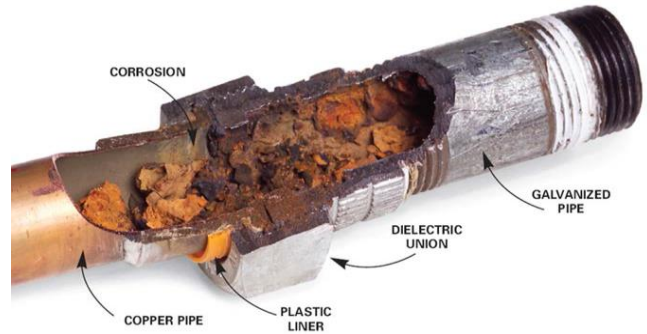


Fig. 3. Galvanic Corrosion

iii. Pitting Corrosion

Pitting is perhaps the most destructive and insidious form of corrosion, containers fail because of perforation, yet only a little amount of metal is lost in the entire structure [21]. In addition, pitting is difficult to evaluate on an experimental basis, and the service life of equipment is correspondingly uncertain. Pitting is probably responsible for more unexpected plant equipment failures than any one of the other forms of corrosion. This happen because of random attacks on particular parts of the metal's surface. This makes holes which are large in depth. These holes are called "pits". The pit acts as the anode while the undamaged part of the metal is the cathode. It begins with a chemical breakdown in the form of a scratch or spot. The pitting method makes the metal thinner and increases fatigue. For example, it can be very harmful in gas lines [19]. Figure four shows an example of this kind.



Fig. 4. Pitting Corrosion

iv. Erosion Corrosion

Erosion corrosion often causes unexpected and rapid deterioration of plant process equipment. Erosion corrosion is the acceleration or increase in rate of deterioration or attack on two metals because of relative movement between a

corrosive fluid and the metal surface [22]. The rate of corrosion can be highly accelerated when mechanical or abrasive conditions are around, such as liquids moving at substantial speed, presence of solids in suspension (that is, slurries), marked turbulence, and impingement of fluid streams. Examples of equipment that may be subject to erosion-corrosion are pumps, valves, centrifugals, measuring devices such as an orifice, agitators, pipe lines, particularly at elbows and tees, and heat exchanger tubes. Erosion corrosion can be controlled by selection of materials design consideration which are resistance to erosion corrosion [23]. An instance of this kind of corrosion is shown in figure 5.



Fig. 5. Erosion Corrosion (Jappes et al, 2005)

v. Filliform Corrosion

Filliform corrosion is also known as "under film". It is majorly caused by contaminants that were left on the surface or trapped between two surfaces that come in contact with each other before the primers and paint were applied. Once trapped by the paint, the corrosion develops and has the look of a spidery growth or a lake bed pattern under painted surface [17]. Filliform corrosion is shown in figure 6.

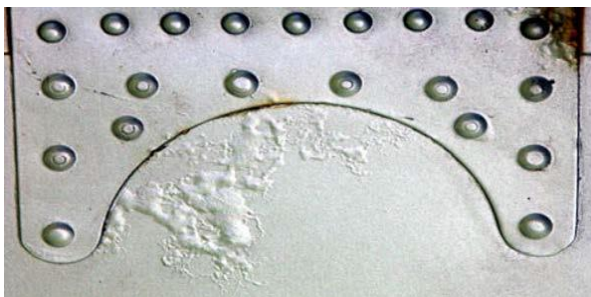


Fig. 6. Filliform Corrosion

vi. Graphitic Corrosion

Graphitic corrosion is the corrosion of gray cast iron in which the metallic iron component is transformed into

corrosion products which reinforces the residual graphite [24]. Figure 7 shows this type of corrosion.



Fig. 7. Graphitic Corrosion

F. Adsorption of Green Leaf Inhibitors

Adsorption of organic inhibitor unto the mild steel surface thereby forming thin-film on the surface of adsorbent by preventing or reducing oxygen and water from reacting with mild steel is a major footstep in organic inhibition. Absorption of organic inhibitor occurs as a result of electrostatic interaction between the positively charged nitrogen atom and the negatively charged mild steel surface through Weak Van der Waals forces. The polar organic ions first developed a physical absorption surface film which may in turn developed chemical adsorption to form a donor type reaction [25].

Inhibitors are inorganic or organic substances which react with corroding surfaces or the environment, to give the surface a level of protection. This may be by forming a film thereon or forming an inert compound. Corrosion inhibitors are chemical compounds which, when added in small concentration stop or slow down corrosion of metals, alloy and they have a continuing and major role to play in the cost-effective control of corrosion [26].

G. Types of Adsorption

Adsorption is the adhesion of molecules, atoms, ions from liquid or dissolved solid to a surface of mild steel. This process creates a film of the adsorbate, the adsorbed organic inhibitor molecules accumulate on the surface of the adsorbent thereby reducing the rate of corrosion by preventing the oxygen from reacting with metal [27]. The adsorbed substance is known as adsorbate while the adsorbing mild steel surface is called adsorbent. The chemical reaction formed between the adsorbate and adsorbent, if it is chemically combined together, the reaction will be non-reversible but if it is the reaction is physical, it is reversible. The type of adsorptions are:

i. Physical Adsorption

Physical adsorption occurs, if the forces of attraction existing between adsorbate and adsorbent are Vander Waals forces, the adsorption is called physical adsorption. It is also known as Vander Waals adsorption. In physical adsorption the force of attraction between the adsorbate and adsorbent is very weak, therefore this type of adsorption can be

easily reversed by heating or by decreasing the pressure [26].

ii. Chemical Adsorption

Chemical adsorption occurs, if the force of attraction existing between adsorbate and adsorbent are almost same strength as chemical bonds, the adsorption is called chemical adsorption. It is also called langmuir adsorption. In chemisorption the force of attraction is very strong and therefore adsorption cannot be easily reversed [26].

H. Corrosion Inhibitors

Inhibitors generally have heteroatoms, Oxygen, Nitrogen, and Sulphur are found to have higher basicity and electron density and hence act as corrosion inhibitor. O, N, and S are the active centres for the process of adsorption on the metal surface [28]. The inhibition efficiency should follow the sequence Oxygen < Nitrogen < Sulphur < Phosphorous [29].

Several factors are considered during selecting of an organic inhibitor which includes cost and simplicity in processing it, easy availability and most importantly safety to environment in which it operates and its species need to be considered when choosing an inhibitor. Basically, there are two types of corrosion inhibitors; these are organic and inorganic corrosion inhibitors. But at the moment, an intensive research is been undertaken by researchers in the area of plants being used as a corrosion inhibitor due to their non-hazardous and non-toxic to mankind and the environment. Researchers are trying to classified corrosion inhibitor substances gotten from plant extracts (leaves, fruits, seed, bark) as green corrosion inhibitors [29].

i. Inorganic Corrosion Inhibitors

Inorganic substances such as phosphates, chromates, dichromate's, silicates, borates, tungstates, molybdates, and arsenates have been found effective as inhibitors of metal corrosion. Pyrrole and derivatives are believed to exhibit good protection against corrosion in acidic media [30]. These inhibitors have also found useful application in the formulation of primers and anticorrosive coatings, but a major disadvantage is that their toxicity and as such their uses has come under severe criticism because of their threat to the environment [29].

ii. Organic Corrosion Inhibitors

Organic substances containing polar functions with oxygen, nitrogen and sulphur atoms in a adjoin system have been described as good inhibiting properties [29]. The use of organic compounds containing oxygen, sulphur, and especially nitrogen in their molecular structure to reduce corrosion attack on

mild steel has been studied in some detail. The existing data show that most organic inhibitors adsorbed onto the metal surface by displacing water molecules and forming a film on the adsorbent. Availability of non-bonded (lone pair) and p-electrons in inhibitor molecules facilitate electron transfer from the inhibitor to the adsorbent [31]. A coordinate covalent bond involving transfer of electrons from inhibitor to the metal surface was formed. The strength of the chemisorption bond depends upon the electron density on the donor atom of the functional group and also the Polaris ability of the group. However, most of the organic and inorganic inhibitors are hazardous and carcinogenic, as a result, there is need to develop cheap, non-toxic and ecofriendly which are the focus of the use of natural products [29].

iii. Green Corrosion Inhibitors

Plant extracts are viewed as an incredibly rich source of naturally synthesized chemical compounds that can be extracted by simple procedures with low cost and are biodegradable in nature. The use of these natural products such as extracted compounds from leaves or seeds as corrosion inhibitors have been widely reported by several authors [32]. Environmental concerns worldwide are increasing and are likely to influence the choice of corrosion inhibitors in the future. Some chemicals are excellent inhibitors, but are quite toxic and readily absorbed through the skin. The known hazardous effects of most synthetic organic inhibitors and restrictive environmental regulations have now made researchers to focus on the need to develop cheap, non-toxic and environmentally friendly natural products such as green corrosion inhibitor which non-hazardous and toxic to the environment.

I. Structural Mechanism of Organic Inhibitors

Although, Researchers has not clearly defined the structural mechanism behind organic inhibitor on the surface of metallic plate. But some Researchers has confirmed that Imidazole, Acetamide, I-Ppenyl-4-methyl Imidazole Schiff bases which has O, N.P. and S in their molecular structure is good corrosion inhibitor. Inhibitor structural mechanism formed by weak Vander Waal forces by forming a film on the adsorbent surface, film adheres to the surface creating a barrier between the adsorbent and the corrosive medium or in blocking the metallic surface by the activities of inhibitor molecules through adsorption. In general, the phenomenon of absorption is influence by the nature and surface charge of the metal, the type of aggressive electrolyte and by the chemical structure of the inhibitors [33]. The absorption of inhibitor onto the surface of mild steel is as a result of formation of links between the vacant d-orbital's of mild steel atoms and lonely sp² pairs of electrons present on Oxygen (O) and Nitrogen (N) atoms found in organic compound or heteroatom's involving the displacement of water molecules from the mild steel surface. Figure 8 shows the mechanism of structural Inhibitor

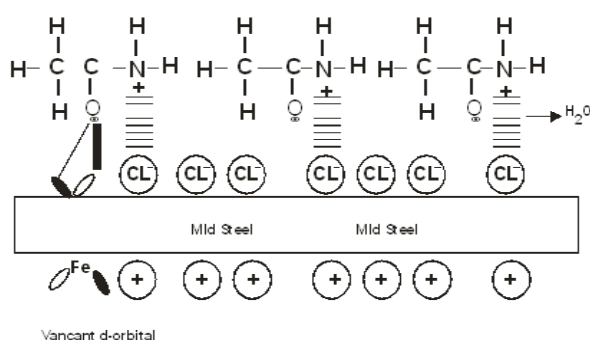


Fig. 8. Structural Inhibitor (Duncan, 1996)

Adsorption of organic inhibitor occurs as a result of electrostatic interaction between the positively charged nitrogen atom and the negatively charged mild steel surface [34].

Deposition of chlorine ions on the surface of adsorbent causes small amount of hydration and due to this its first get adsorbed on the adsorbent surface creating negative charge on the solution side of the adsorbent and thereby favors the absorption of inhibitor onto the surface of mild steel [35].

J. Overview of Interrelated Previous Studies

Scientist research work on the use of green leaves as corrosion inhibitors is on the development as year's passes by. Because most of the green plants are ecologically friendly and environmentally-safe [36].

Green plans are birthplace of naturally developed organic compounds, a few with complicated atomic structures and having distinctive biological and synthetic properties. The naturally rich organic compounds are often used because they are ecologically acceptable, non-expensive, non-hazardous and environmentally friendly as well as abundantly available on earth. Based on these facts, the use of green plant extracts and their production as organic corrosion inhibitor for mild steel and alloys in divergent environment [37].

In some cases, the effectiveness of some of the green plants extracts has been accredited to the presence of tannin in their chemical elements [38]. Divergent green plants extract can also be used as green corrosion inhibitors. Most of the researchers have previously investigated green leaves as organic inhibitor. Below are some of the following.

Anyankuo [39] researched on the effectiveness of green plant extract of *Cassia Auriculata* on corrosion inhibition of mild steel in 1M HCl by gravimetric method and electrochemical investigation. It was concluded that the extract of *Cassia Auriculata* can reduced rate of corrosion of mild steel in HCl media with efficacy up to 74. It was also found that increase in concentration of inhibitor decreases the rate of corrosion of mild steel in 1M HCL.

Malik [17] investigated the inhibition efficiency of *Zenthoxylumalum* plant extract on corrosion rate of mild steel in 5% and 15% aqueous hydrochloric acid solution by gravimetric method and electrochemical impedance spectroscopy (EIS). Their finding showed that the effectiveness of inhibition increases with increase in *Zenthoxylumalum* plant extract concentration [40].

Marcelo [41] investigated inhibition efficiency of *Nypa fruiticans wurmb* leaves extract on corrosion rate of mild steel in HCl. The same investigation was carried out using *Nypa fruiticans wurmb* fruits and leaves extracts by Orubite and Hamilton (2004). The result shows that the fruits extract inhibited corrosion rate of mild steel a little more excellent than the leaves of the same plant [42].

Lukovits [43] Investigated inhibition on the effectiveness of aqueous extracts of spent coffee on the corrosion of carbon steel in a 1M HCl. Two means of extraction were investigated: infusion and decoction. The finding showed that the effectiveness inhibition of C-steel in 1M HCl increased as the extract concentration and temperature increased [44]. After carefully examination of overview of interrelated studies. It was observed that green leaves are effective inhibitor of mild steel in most of aggressive environment. It was also observed that increase in temperature affect the inhibitive properties of green leave [45].

III. MATERIALS AND METHODOLOGY

A. Materials and Method

The preparation of the coupons involves mechanically cutting of plate of mild steel sheet with dimensions of 45mm x 35mm x 1mm by to obtain ten pieces from the plate and the specimens were descaled by surface cleaning using wire brushing and No 600grit abrasive paper for smoother surface. The coupons were examined carefully to check for rough edges, which could influence the rate of corrosion and micro structure monitoring process, these rough edges were filed out using a rotary filing machine (Liu et al., 2009). The mild steel utilized in this investigation is Grades C- 1026 (0.31), TSL- (0.69) which is used in forging of vehicle, hydraulic shafts and pump shaft is the chemical composition of mild steel are shown in table 2

Table 2. Composition of Mild Steel

Element	Weight (%)
Fe	96.14
Au	1.38
Pd	1.33
Ti	0.52
Mn	0.39
Nb	0.15
Cr	0.08

B. Positive Material Identification (X- Met 7000) Equipment Report

The sample was cleaned to remove dirt, rust or adhering grease. An XRF spectrometer works on the principle that if a sample is illuminated by an intense x-ray beam, which is known as the incident beam, a few of the energy is scattered, but some are also absorbed within the sample in a method that is dependent on its chemistry. The incident x-ray beam is typically produced from a RH target. When this primary x-ray beam illuminates the sample, it is said to be excited. The excited sample in turn emits x – rays along a spectrum of wavelengths characteristic of the types of atoms present in the sample. This sample was further tested for its chemical composition with the Oxford instrument x – met 7000 series.

C. Heat Treatment Process

Heat Treatment: Heating and cooling process is to maneuver the materials properties or structural changes of the mild steel. The plastic deformation of metal above the recrystallization temperature is called Annealing. Annealing is heating a metal at an exact temperature and allowing it to cool naturally or mechanically. The strip of mild metal was heated at a temperature of 800°C and holding it for 60 minutes in blast furnace. The main purpose of annealing is to relieve the stress impacted by cold work on the mild steel materials, refine its grains and improve the ductility. Figure 9 shows the effect of heat.

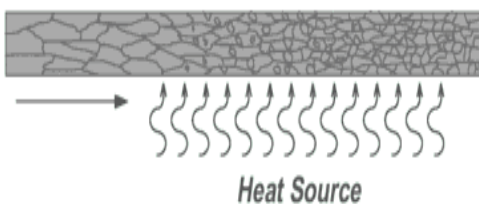


Fig. 9. Growth of new grains within old grains during annealing

D. Cold Working

Cold working is the process whereby metal is plastically deformed below its recrystallization temperature. The strip of metal was passed through in between two rollers which impact pressure on the mild steel. The metal strip becomes much longer, thinner and stronger. The grains in the metals become also elongated. The metal is also more brittle and more liable to fracture as the elongation grows up. Figure 10 shows a cold working process.

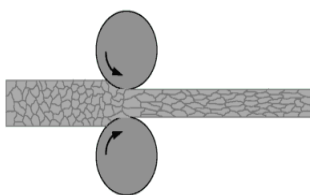


Fig. 10. Formation of elongated grains during cold working of mild steel

E. Water Analysis

Water analysis was carried out to ascertain the compositions of fresh water in Amassoma, Bayelsa State and Distilled water as shown in table 3 and 4.

Table 3. Composition of fresh water in Amassoma, Bayelsa State

Sample Code	Fresh water (wt%)
P ^H	6.4
SAL	0.03
COND	102.5
TURB	31.95
TDS	51.25
TSS	3.71
NO ₃	0.122
CL	13.5
SO ₄	0.63
HCO ₃	0.65
TA	7.5
TH	20.5
Ca	7.44
Mg	1.92
Na	3.97
K	1.33
Fe	0.14
Mn	0.021

Table 4. Composition of Distilled water

Sample Code	Eva water (wt%)
P ^H	7.2
SAL	0.16
COND	205
TURB	0.02
TDS	1.03
TSS	0.01
NO ₃	0.031
CL	27.5
SO ₄	0.05
HCO ₃	0.50
TA	12.5
TH	28.5
Ca	14.5
Mg	3.59
Na	7.23
K	1.94
Fe	0.011
Mn	0.00

F. Preparation of Inhibitor

Healthy, disease free, mature leaves of Dongoyaro leaf was used for this experiment. The raw material utilized in this experiment was Dongoyaro leaf, which was purchased from Opolo market in Bayelsa State. The sample was identified to be a neem plant (*Azadirachta indica*) and subsequently weighed and found to be (1230g) grounded into powder form locally, using mortar and pestle. The grounded leaves were mixed with 3000ml of distilled water so as to enable the extraction of the green content from the

grounded leaves. The leaves were filtered using chemical laboratory filtering apron so as to have fine extracts without particles. The resultant solution was stored and kept for 24 hours, plant extracts (PE) test solutions were prepared at concentrations of

$$\frac{1230g}{3000mol} = 0.41g/mol$$

G. Test Solutions Preparation

The fresh water which was collected from Amassoma River and prepared solution was gradually poured into 500ml beaker and subsequently transferred to different plastic bowl respectively. Each of the weighed mild steel was suspended with the aid of a thread, sticks and completely immersed in the entire plastic bowl containing 0%, 5%, 10%, 15% and 20% of the solution. After seven days interval (168hours). The immersed specimens, as shown in figures 10 and 11, were retrieved and washed with warm water, degreased in acetone, air dry and reweighed to notice the rate of corrosion for each week after the



immersion of specimen.

Fig. 10. Coupons in Corrosive Media (Fresh water)

The process was continued for a period of eight. After the eight weeks, the coupons were subjected to micrographic structure test experimentation to determine the structural changes during the immersion test.



Fig. 11. Coupons in Corrosive Media (Distilled Water)

IV. RESULTS AND DISCUSSION

A. Results

Table 5. Cold Worked Mild Steel

Code	Time (hr)	Initial Weight W1(g)	Final Weight W1(g)	Change In Weight W(g)	Corrosion Rate (mm/yr)
0% C1	168	8.1473	8.0956	0.0517	0.1044
0% C2	336	8.1473	8.0463	0.1010	0.1019
0% C3	504	8.1473	8.0005	0.1468	0.0988
0% C4	672	8.1473	7.9542	0.1931	0.0974
0% C5	840	8.1473	7.9064	0.2409	0.0972
0% C6	1008	8.1473	7.8461	0.3012	0.1013
0% C7	1176	8.1473	7.7850	0.3623	0.1045
0% C8	1344	8.1473	7.7294	0.4179	0.1054

Table 6. Heat Treated Mild Steel

Code	Time (hr)	Initial Weight W1(g)	Final Weight W1(g)	Change In Weight W(g)	Corrosion Rate (mm/yr)
0% H1	168	8.6431	8.5991	0.0440	0.0888
0% H2	336	8.6431	8.5525	0.0906	0.0914
0% H3	504	8.6431	8.5092	0.1339	0.0900
0% H4	672	8.6431	8.4777	0.1654	0.0834
0% H5	840	8.6431	8.4240	0.2191	0.0885
0% H6	1008	8.6431	8.3743	0.2688	0.0904
0% H7	1176	8.6431	8.3337	0.3094	0.0892
0% H8	1344	8.6431	8.2972	0.3459	0.0872

Table 7. Cold worked Coupon (5%)

Code	Time (hr)	Initial Weight W1(g)	Final Weight W1(g)	Change In Weight W(g)	Corrosion Rate (mm/yr)
5% C1	168	8.6643	8.6435	0.0208	0.0419
5% C2	336	8.6643	8.6236	0.0407	0.0410
5% C3	504	8.6643	8.6073	0.0570	0.0383
5% C4	672	8.6643	8.6041	0.0602	0.0304
5% C5	840	8.6642	8.6002	0.0641	0.0258
5% C6	1008	8.6642	8.5952	0.0691	0.0232
5% C7	1176	8.6642	8.5893	0.0750	0.0216
5% C8	1344	8.6642	8.5828	0.0815	0.0205

Table 8. Heat Treated Coupon (5%)

Code	Time (hr)	Initial Weight W1(g)	Final Weight W1(g)	Change In Weight W(g)	Corrosion Rate (mm/yr)
5% H1	168	8.3342	8.3182	0.0180	0.0363
5% H2	336	8.3342	8.3037	0.0305	0.0308
5% H3	504	8.3342	8.2925	0.0417	0.0281
5% H4	672	8.3342	8.2842	0.0500	0.0253
5% H5	840	8.3342	8.2773	0.0569	0.0229
5% H6	1008	8.3342	8.2725	0.0617	0.0208
5% H7	1176	8.3342	8.2688	0.0654	0.0188
5% H8	1344	8.3342	8.2621	0.0721	0.0182

Table 9. Cold worked Coupon (10%)

Code	Time (hr)	Initial Weight W1(g)	Final Weight W1(g)	Change In Weight W(g)	Corrosion Rate (mm/yr)
10% C1	168	8.7682	8.7492	0.0190	0.0384
10% C2	336	8.7682	8.7292	0.0390	0.0396
10% C3	504	8.7682	8.7202	0.0480	0.0322
10% C4	672	8.7682	8.7172	0.0510	0.0257
10% C5	840	8.7682	8.7092	0.0590	0.0238
10% C6	1008	8.7682	8.7072	0.0610	0.0205
10% C7	1176	8.7682	8.6992	0.0690	0.0198
10% C8	1344	8.7682	8.6937	0.0745	0.0187

Table 10. Heat Treated Coupon (10%)

Code	Time (hr)	Initial Weight W1(g)	Final Weight W1(g)	Change In Weight W(g)	Corrosion Rate (mm/yr)
10% H1	168	7.8562	7.8407	0.0155	0.0313
10% H2	336	7.8562	7.8273	0.0289	0.0292
10% H3	504	7.8562	7.8171	0.0391	0.0263
10% H4	672	7.8562	7.8142	0.0420	0.0212
10% H5	840	7.8562	7.8070	0.0492	0.0199
10% H6	1008	7.8562	7.8041	0.0521	0.0175
10% H7	1176	7.8562	7.7961	0.0601	0.0173
10% H8	1344	7.8562	7.7854	0.0708	0.0178

Table 11. Cold worked Coupon (15%)

Code	Time (hr)	Initial Weight W1(g)	Final Weight W1(g)	Change In Weight W(g)	Corrosion Rate (mm/yr)
15% C1	168	8.4552	8.4430	0.0122	0.0246
15% C2	336	8.4552	8.4327	0.0225	0.0227
15% C3	504	8.4552	8.4248	0.0304	0.0205
15% C4	672	8.4552	8.4172	0.0380	0.0192
15% C5	840	8.4552	8.4102	0.0450	0.0182
15% C6	1008	8.4552	8.4032	0.0520	0.0175
15% C7	1176	8.4552	8.3942	0.0610	0.0176
15% C8	1344	8.4552	8.3852	0.0700	0.0177

Table 12. Heat Treated Coupon (15%).

Code	Time (hr)	Initial Weight W1(g)	Final Weight W1(g)	Change In Weight W(g)	Corrosion Rate (mm/yr)
15% H1	168	8.6554	8.6446	0.0108	0.0218
15% H2	336	8.6554	8.6363	0.0191	0.0193
15% H3	504	8.6554	8.6266	0.0288	0.0194
15% H4	672	8.6554	8.6230	0.0324	0.0163
15% H5	840	8.6554	8.6170	0.0384	0.0155
15% H6	1008	8.6554	8.6064	0.0490	0.0165
15% H7	1176	8.6554	8.6028	0.0526	0.0152
15% H8	1344	8.6554	8.5901	0.0653	0.0165

Table 13. Cold Worked Coupon (20%)

Code	Time (hr)	Initial Weight W1(g)	Final Weight W1(g)	Change In Weight W(g)	Corrosion Rate (mm/yr)
20% C1	168	8.7854	8.7672	0.0182	0.0367
20% C2	336	8.7854	8.7520	0.0334	0.0337
20% C3	504	8.7854	8.7438	0.0416	0.0280
20% C4	672	8.7854	8.7374	0.0480	0.0242
20% C5	840	8.7854	8.7342	0.0512	0.0207
20% C6	1008	8.7854	8.7263	0.0591	0.0199
20% C7	1176	8.7854	8.7241	0.0613	0.0176
20% C8	1344	8.7854	8.7196	0.0658	0.0166

Table 14. Heat Treated Coupon (20%)

Code	Time (hr)	Initial Weight W1(g)	Final Weight W1(g)	Change In Weight W(g)	Corrosion Rate (mm/yr)
20% H1	168	8.5492	8.5352	0.0140	0.0283
20% H2	336	8.5492	8.5227	0.0265	0.0267
20% H3	504	8.5492	8.5114	0.0378	0.0254
20% H4	672	8.5492	8.5115	0.0377	0.0190
20% H5	840	8.5492	8.5077	0.0415	0.0168
20% H6	1008	8.5492	8.5012	0.0480	0.0161
20% H7	1176	8.5492	8.5003	0.0489	0.0141
20% H8	1344	8.5492	8.4942	0.0550	0.0139

B. Graphical Representations from Corrosion Test

To aid easy comparison of the weight loss and corrosion rate of mild steel samples immersed in fresh water and with different percentages of the inhibitor used, a graph involving weight loss vs. time and corrosion rate vs. time were plotted for all the cases.

Figure 4.1 shows the variation of the weight loss of mild steel in the absence of inhibitor according to the time of immersion. It is seen that the two coupons show linear curves and experienced higher weight loss from the first week to eight weeks of immersion. This implies that the absence of inhibitor in the corrosive media. There was a closed weight loss from the first (1st) week to the third (3rd) weeks of immersion. But after the third (3rd) week, the heat-treated coupon gradually separated itself from the cold work by losing less weight. From figure 13, it was observed that all two coupons had a higher corrosion rate because there was no inhibitor in the control sample to decrease the rate of corrosion.

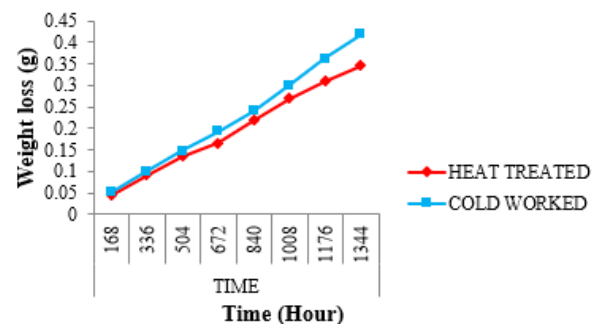


Fig. 12. Weight loss with respect to time.

It was also identified that there was a minor decrease in weight loss from second weeks to six weeks of immersion of cold work and an increase in weight loss at seventh (7th) weeks and eight (8th) weeks of immersion.

From figure 13, it was observed that Cold worked coupon experienced higher weight loss at third (3rd) week and eight (8th) week of immersion and decrease in weight loss from fourth (4th) week to seventh (7th) week of immersion.

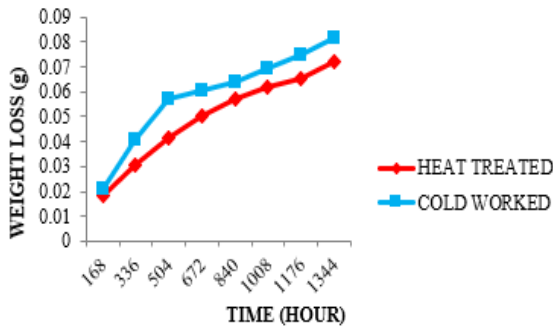


Fig. 13. Weight loss with time for (5%) Inhibitor.

While Heat treated coupon had a linear weight loss from the first week to the seventh week of immersion.

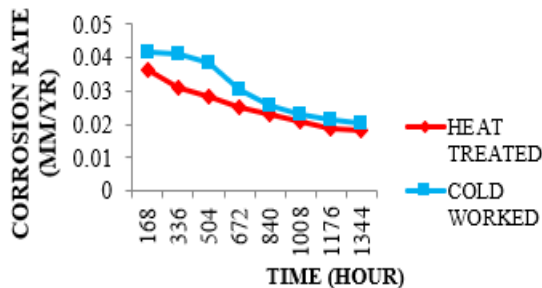


Fig. 14. Corrosion rate vs time for (5%) Inhibitor.

From fig 14, it was observed that Cold worked coupon had a higher corrosion rate from first (1st) week to fourth (4th) week of immersion and experienced close weight with Heat treated from fifth (5th) week to eight (8th) week.

The deduction from figure 15, revealed that both Heat Treated coupon and Cold worked coupon had close weight loss during first (1st) week and eight (8th) week of immersion. Also, there is a minor decrease in weight loss compared to 5% inhibitor.

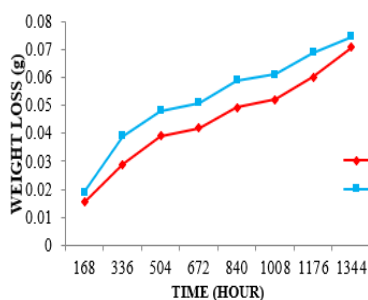


Fig.15. Corrosion rate with time 10% inhibitor

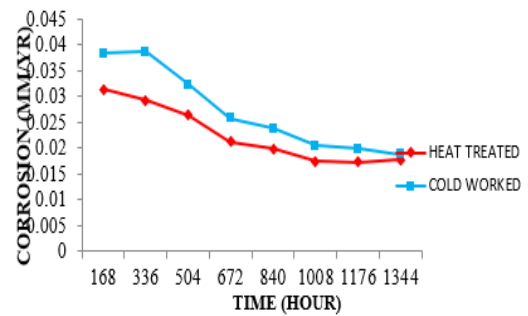


Fig. 16. Corrosion rate with time 10% inhibitor.

From figure 4.6, it reveals that all two coupons experience higher corrosion rate during first (1st) and third (3rd) week of carrying out the immersion test. After the third (3rd) week of immersion, all the two mild steels (coupons).

Heat Treated and Cold worked gradually decreases in rate of corrosion during the fourth (4th) and eight (8th) week of immersion test compared to the corrosion rate of 5% inhibitor. It can also be deduced that the organic inhibitor Neem leave extract (Dongoyaro leave) which has been used as a corrosion control reduces the rate of corrosion uniformly from fourth (4th) week to eight (8th) week of immersion test. By the end of the eight (8th) week of immersion all coupons experienced close rate of corrosion. From figure 17, it can be observed that all coupons had close weight loss during first (1st) and third (3rd) week of immersion.

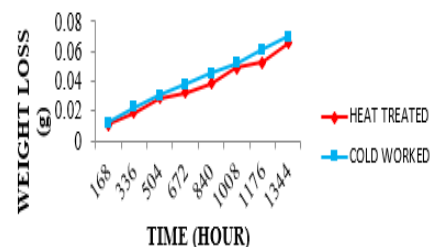


Fig. 17. Weight loss with time for 15% inhibitor.

It can also be deduced that Cold worked mild steel (coupon) had straight line graph which shows that the rate of corrosion is proportional to the time. After third (3rd) week of immersion, Heat treated coupon experienced a higher weight loss from fourth (4th), fifth (5th) and seventh (7th) week of immersion.

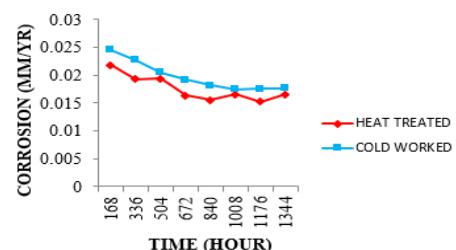


Fig. 18. Corrosion rate with time 15% inhibitor.

From figure 18, it can be clearly seen that Heat treated mild steel (coupon) behaves like sinusoidal wave curve. It is almost repetitive of smooth oscillation. The wave form in which the amplitude is always proportional to the sine of its displacement angle at every point in time. Also, Cold worked coupon experienced a higher weight loss throughout the immersion test. From figure 19, it can be observed that Cold worked coupon experienced a close weight loss during the first (1st) and third (3rd) week of immersion.

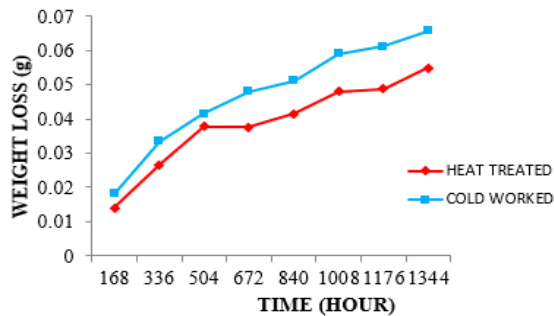


Fig. 19. Weight loss with time for 20% inhibitor.

Heat Treated mild steel (coupon) gradually loses lesser weight from the fourth (4th) week to eight (8th) week of immersion test. From figure 20, it can be deduced that Cold worked coupon had higher corrosion rate at first week of immersion and close corrosion rate at third week.

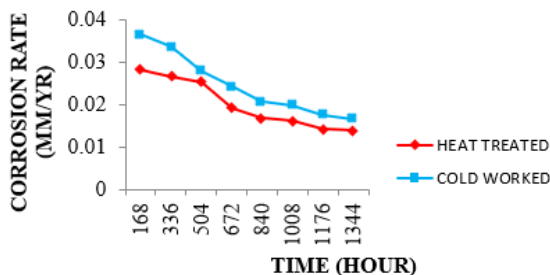


Fig. 20. Corrosion rate with time for 20%

After the third week both had a sharp decrease in corrosion rate from fourth week to eight weeks of immersion. Figure 21. shows that the coupon without inhibitor had a higher weight loose than 5%, 10%, 15% and 20% inhibitor. Also, 15% Inhibitor experienced lesser weight loose throughout the experiment.

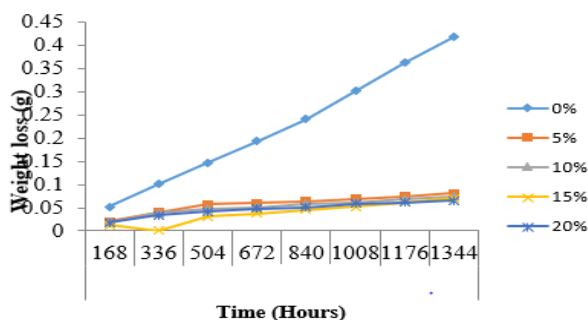


Fig. 21. Weight losses with time for all cold worked coupon.

From figure 4.22, it be seen that 15% inhibitor also shows lesser corrosion rate compared to other percentage. It can also be deduced that 0% inhibitor had the highest corrosion rate.

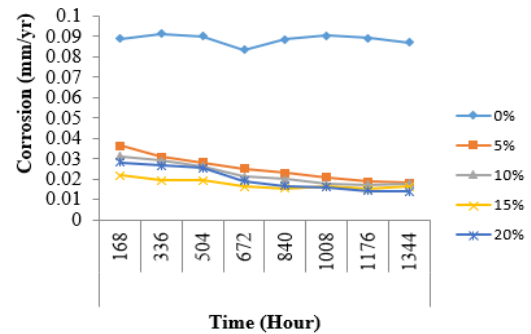


Fig. 22. Corrosion rate with time for all heat-treated coupon

C. Microstructural Evaluation

The micro structure of heat-treated mild steel (coupon) shown in figure 23 has two major constituent, which are Ferrite and Pearlite. The micrographs of each samples appear below at a magnification of X 400

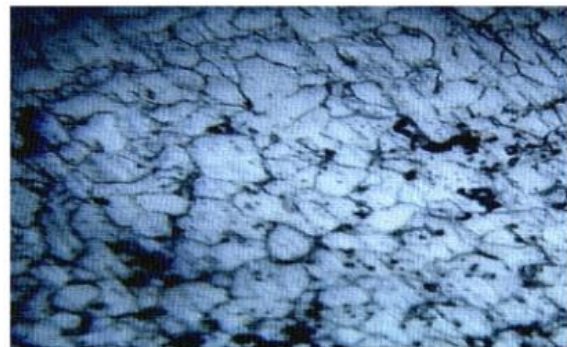


Fig. 23. Micrograph of heat-treated coupon

The white coloured patches of the microstructure are the ferrite while the black coloured patches are the pearlite. It was observed that the pearlite seems to cover much more area than the ferrite. It can also be noticed that the grains and grains boundary can be clearly seen in the micrograph. In plate 24, ferrite and pearlite are elongated due to cold rolling of mild steel coupon. It can also be deduced that ferrite cover much more area than pearlite.

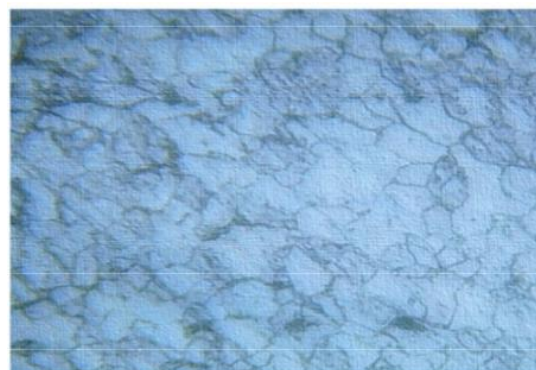


Fig. 24. Micrograph of cold worked coupon.

Ferrite is known to be soft because it has 0.008% of carbon and the rest is Iron while pearlite is basically a microstructure of 88%alpha ferrite and 12%cementite. Cementite is comparatively harder phase which contains 6.67% of carbon. With much pearlite on the microstructure, the metal is hard and strong but not particularly tough.

In figure 25, it can be observed that the grains boundary cannot be clearly seen as a result of corrosion. It can also be observed that there is a reduction of grain sizes due to corrosion effect on the microstructure of the coupon

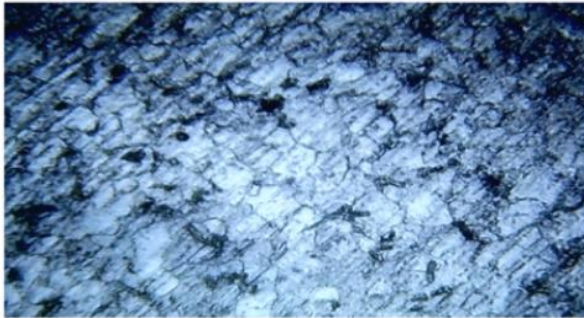


Fig. 25. Micrograph of 0% inhibitor (without inhibitor) for heat treated coupon

Figure 26 shows micrograph with much ferrite than pearlite. It can be noticed that there is a major reduction of grains sizes compared to the zero percent of heat-treated coupon.

The gradual disappearance of grain boundary and reduction of sizes as a result of corrosion effect on the microstructure of mild steel. Decreasing grain size are prone to corrosion.

From the micrograph shown in figure 27, it can be observed that the grain boundary can be clearly seen. It is also observed that the volume of pearlite is much less than the ferrite. There is a reduction of grains sizes due to the corrosion.



Fig. 26. Micrograph of 0% inhibitor for cold worked coupon

It can also be noticed that there is an elongation of grains due to cold working of the coupon

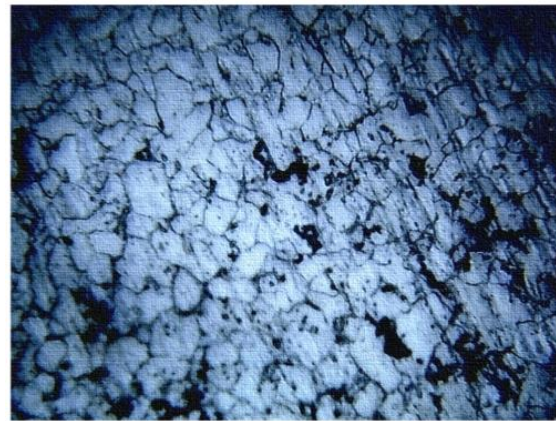


Fig. 27. Micrograph of 5% inhibitor for heated coupon

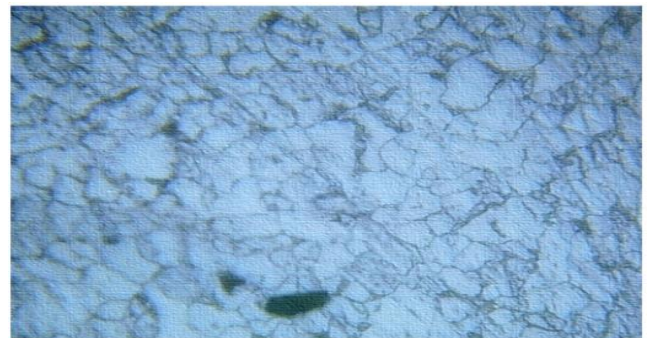


Fig. 28. Micrograph of 5% inhibitor for cold work.

The micrograph shown in figure 29 shows less volume of pearlite than the ferrite.

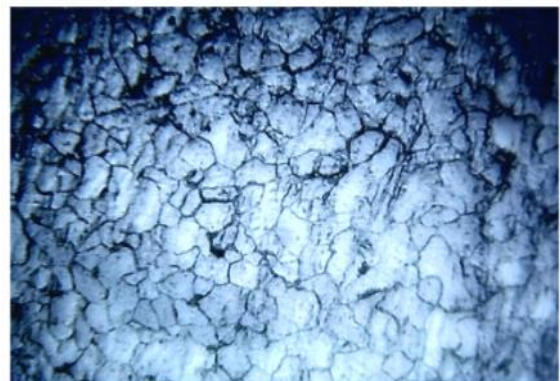


Fig. 29. Micrograph of 10% inhibitor for heat treated coupon.

The depletion of pearlite structure patches and slightly reduction of gains is as a result of corrosion. It can also be noticed that in 10% inhibitor, the grains boundaries can be clearly seen in the presence of inhibitor. The micrograph shown in figure 30 displays higher concentration of ferrite structure as well as reduction of grains sizes.

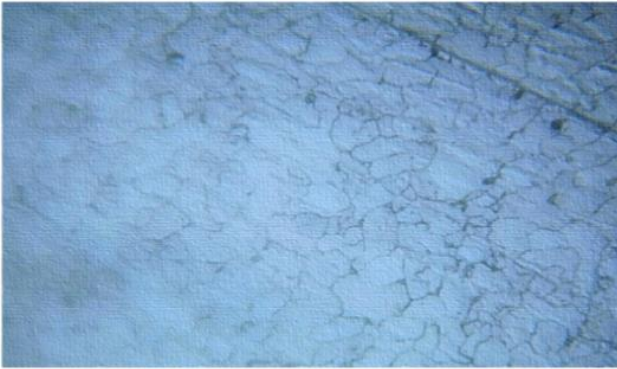


Fig. 30. Micrograph of 10% inhibitor for cold worked coupon.

It can also be observed that there is no clear definite grain boundary due to the corrosion of the microstructure.

From the micrograph shown in figure 31, the microstructure of 15% inhibitor of coupon at a magnification of 400. The microstructure shows a fine distribution of pearlite and ferrite while the grains and grain boundary can be clearly seen as result of the inhibitor present

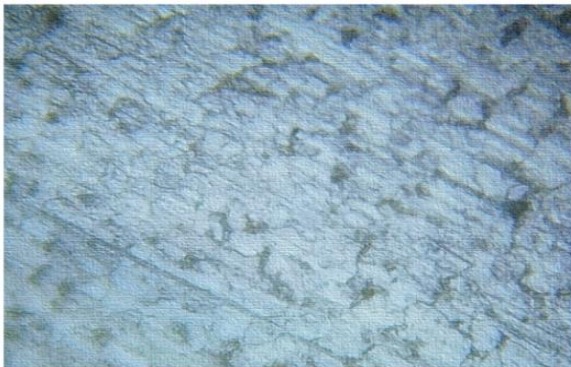


Fig. 31. Micrograph of 15% inhibitor for heat treated coupon

It can also be deduced that there is a slightly reduction of grains sizes due to the corrosion. It also observed that 15% inhibitor showed the best corrosion inhibition with the least corrosion rates for all the heat-treated coupon.

From the microstructure shown in figure 32, the microscopic view of the micrograph shows less pearlite than the ferrite. It can also be observed that there is a uniform distribution of ferrite and grains boundary as a result of 15% of inhibitor. The differences between pearlite/ferrite ratios is as a result of corrosion of the microstructure. It can also be deduced that there is a reduction of grains sizes resulting from corrosion. It also observed that 15% inhibitor showed the best corrosion inhibition with the least corrosion rates for the entire experiment.

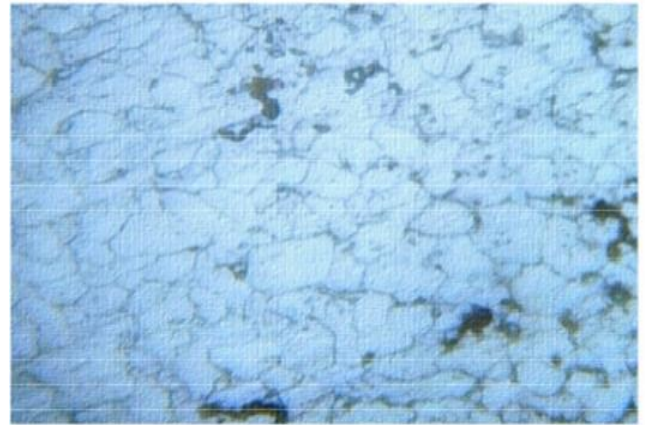


Fig. 32. Micrograph of 15% inhibitor for cold worked coupon.

Figure 33 shows that there is much depletion of pearlite region and a high concentration of ferrite region due to corrosion. Also, there is a slight reduction of grains sizes. It can also be observed that grain boundary can be seen in the microstructure.



Fig. 33. Micrograph of 20% inhibitor for heat treated coupon.

From the micrograph figure 34, there is a significant reduction of grains sizes compared to the microstructure of 15% percent inhibitor. It can be deduced that 20% microstructure corrode more as the inhibitor is gradually losing it strength against the microstructure. Gradually depletion of grain boundary is as a result of corrosion



Fig. 34. Micrograph of 20% inhibitor for cold worked coupon.

V. CONCLUSION AND RECOMMENDATIONS

A. Conclusion

This research work reveals that mild steel is the major metal used in oil and gas industries. From the results of the microstructure analyses carried out in this study, the following conclusion was made:

The Heat-Treated microstructure (coupon) had more pearlite than Cold worked coupon due to less Cementite on the structure, as a result, Cold worked coupon corrodes more throughout the investigation. The 15% inhibitor showed the best corrosion inhibition with the least corrosion rates for the entire experiment. The depletion of pearlite from the microstructure is as a result of corrosion effect. The extract of Dongoyaro leave has a high significant effect on the decrease of the corrosion of mild steel in fresh water as a result of the formation of thin films on the substrate thereby displacing water molecules from the metal surface. The efficiency of the inhibitor increases with increased in concentration but decreases with increase in environmental factors such as wind and temperature. Mild steel to be used in fresh water environment should be coated to achieve a useful service life and with minimum maintenance.

B. Recommendations

In light of the experimentations carried out and the results obtained, it is recommendable that

- I. Mediums such as crude oil and distillate fuels should be used to characterize the inhibition propensity of dogonyaro (Neem) leaves.
- II. Researchers should study the organic compounds that reduce corrosion in Dongoyaro leave so as to improve on the use of dogonyaro as corrosion inhibition.

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