

**CORROSION BEHAVIOUR OF LOW CARBON STEEL USED IN  
OIL AND GAS ABOVEGROUND STORAGE TANKS**

**A**

**THESIS**

**SUBMITTED TO THE GRADUATE FACULTY IN PARTIAL FULFILLMENT  
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**BY**

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## **ABSTRACT**

The cost and losses associated with corrosion annually is approximately \$ 276 billion or 3.1% of the GDP in the USA, 3 - 4% of GNP in Australia, Great Britain, Japan and other countries. The associated cost and losses include aboveground storage tanks for petroleum products. For example, Liberia Petroleum Refining Company (L.P.R.C.) is the national refining company in Liberia. It imports all petroleum products such as lightweight cooking gas, kerosene, diesel, petrol, jet fuel, gasoline, etc. Above-ground storage tanks are used to store these petroleum products. The storage tanks corrode, which leads to wall thinning and possible fuel leakage/losses. This research focuses on the corrosion behaviour of low carbon steel used in oil and gas above-ground storage tanks that may lead to leakages, failure and possible contaminations. The proposed work includes experiments on coated and uncoated carbon steel used in oil and gas storage tanks for storing petroleum products in understanding corrosion rate and how the low carbon steel corrodes when exposed to such environments. The petroleum products used as corrosive media are diesel and gasoline. The experiments will include corrosion weight loss measurements, pH measurement, surface analysis, etc.

## **ACKNOWLEDGEMENT**

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## **DEDICATION**

I am grateful to God for bringing me thus far in my academic sojourn. To HIM belongs all the glory and honor. I dedicate this thesis work to the earthly guardians HE gave me, Mr. and Mrs. Joseph S. D. Suah Sr. and my amazing older siblings who are always there with their support. To Stelvin, Kiki, and my wonderful nieces and nephew, the best future and legacy I want you all to have that's why I strive to achieve excellence in my works so that one day you all can follow as I light the way.

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## **CHAPTER ONE: BACKGROUND AND INTRODUCTION**

### **1.0 BACKGROUND AND INTRODUCTION**

#### **1.1 Background**

When a metal reacts with its environment such that it deteriorates or its properties, it is termed as corrosion. [21] Corrosion is a natural phenomenon for metal because the processes that are involved reverts the metal to its natural state of low energy that was changed when it was mined and processed. It is the reverse of extractive metallurgy. There are several forms which corrosion may take. Some of which are: Stress Corrosion Cracking, Galvanic Corrosion, Pitting Corrosion, Filliform corrosion, Sulfur induced corrosion, fatigue corrosion, etc. The corrosive environment may be acidic, basic or neutral in some given circumstances. Examples are gases - hydrogen sulfide, fuel gases, chlorine, ammonia; air; acids – nitric acid; organic acids – acetic, naphthenic; several kinds of food products; alkalies; solvents; soils; and petroleum oils. [1] However, in the petroleum industry corrosion is caused mainly by sulfur, sulfuric acids, hydrochloric acids, sodium chloride and water than gasoline, oil, or naphtha. [21]

Corrosion affects safety, finances, and preservation of metal. A research published in NACE International Corrosion Journal estimates that annually the United States of America spends \$276 billion which is equivalent 3.1% of their Gross Domestic Product (GDP) directly on corrosion. [21] The failure of engineering structures has resulted into loss of lives and the structure, which can be unrepairable sometimes; and effect on the environment. Examples of such situations are failure with catastrophic consequences of sulfur storage tanks, boilers, bridges, oil rigs, automotive steering mechanisms, pipelines, metallic containers for toxic

chemicals, etc. Due to corrosion, the statue of Liberty in the USA had to be repaired because corrosion had affected the structure. [22]

The effect of corrosion cuts across all industries. In the oil and gas industry, the cost attached to corrosion is about \$1.4 billion in oil and gas exploration and production. [21] In addition it was given that in the USA annually for direct cost of corrosion in the storage of hazardous materials (HAZMAT) which are chemicals, petrochemicals such as gasoline, diesel, etc., is \$7 billion. This value includes \$4.5 billion for aboveground storage tanks and \$2.5 billion for underground storage tanks. The study determined that the total annual direct cost of corrosion for HAZMAT storage is \$7 billion—\$4.5 billion for ASTs and \$2.5 billion for USTs [21].

Aboveground storage tanks are used to store petroleum products. They may store fuels that are used in stationary engines, vehicles or serve as storage for used oil [23]. They store up to 500,000 gallons of petroleum products and more. Their design, construction, and maintenance can be done accordance with ASTM, API and local regulatory bodies. A typical aboveground storage tank basically has a bottom, shell, and roof [24]. The central portion also known as the bottom plate and a sketch plate also called annular plate are two separate sections the bottom has. [24] The height of the tank shell is divided into high courses or rings based on desired construction of 2.5 m (8 ft.) thereabout. The roof may be one of the three major kinds- internal or external floating roof and cone roof. Most of the failure in these tanks is caused by corrosion [24].

However, there are several ways in which these structures can be protected. The most common way is by applying coatings that are corrosion resistant on the surface of the metal to protect the metal beneath the coatings from corroding.

## **1.2 Problem Statement**

Corrosion of oil storage tanks has been identified as a major problem that the oil and gas industry is challenged with. [25] Emissions from these failed tanks pose great threat to the environment. There are also indirect and direct losses associated with the failure of these tanks. The corrosion mechanisms occurring internally and externally vary with environments as well.

In Africa, we have emerging oil and gas industries. For countries that are neither refining nor exploring oil nor gas, they import refined petroleum products and store them in above ground storage tanks and later distribute to other entities. One of such countries is Liberia, found in the tropical region of West Africa.

The Liberia Petroleum Refining Company (L.P.R.C) is an entity that store, supply and distribute petroleum products amongst others. [26] They store petroleum products such as Gasoline (PMS); Fuel (AGO); Kerosene, Jet fuel A-1. [26] There are above ground storage tanks that have been in service for about ten years. Controlling and preventing corrosion in these tanks are done in accordance to international standards such as ASTM approved testing methods with local regulations.

However, corrosion is still a profound issue and challenges in repairing and protecting these tanks from corrosion still exist. To avoid losses, failure, etc., of aboveground storage tanks, there is a need to investigate the corrosion behavior of low carbon steel used in oil and gas above ground storage tanks.

## **1.3 Study Objectives**

The objectives of this study are; (1) To investigate the corrosion behavior (corrosion rate) of low carbon steel used in oil and gas ASTs in diesel and gasoline corrosive environments, and (2) To

study the internal corrosion mechanisms associated with corrosion of low carbon steel used in oil and gas ASTs and it affects the petroleum products

#### **1.4 Scope of the Study**

The works involved in this study are:

- I. Exposure of 456 hours of low carbon steel to experimental corrosive media of gasoline and diesel in order to simulate internal environment of the above ground storage tanks.
- II. Weight loss measurement of corroded samples
- III. Analysis involving corrosion rate of low carbon steel.
- IV. Implications of analyses and microscopic study of samples after 456 hours of exposure.

## **CHAPTER TWO: LITERATURE REVIEW**

### **2.1 Corrosion Mechanism**

Corrosion can be defined as a reaction between a material and its environment that leads to its deterioration or destruction [1]. Corrosion progresses with time and may not be easily detected.

In some cases, it leads to thinning of the material, weight loss. Whereas, in some cases, there may not be noticeable weight loss and structures may fail catastrophically due to corrosion. [2, 2]

The processes begin when a material is attacked by a corrosive medium and this happens on the material's surface. Two kinds of corrosion reactions that are of importance for this research are: chemical reactions of corrosion and electrochemical reactions of corrosion. [3, 3]

### **2.2 Corrosion Reactions**

Corrosion occurs by chemical or electrochemical reactions between the material and its environment. [4] Corrosion reactions are used to describe the reactions and/or processes that take place as the material corrodes.

#### **2.2.1 Chemical reaction of corrosion**

This chemical reaction of corrosion simply excludes electrochemical reactions of corrosion. [3]

Reduction-oxidation (redox) reactions are one of the reactions that can be used to describe the chemical reactions that can be used to describe a metal as it undergoes corrosion. Since the reactions are redox reactions, the metal is the oxidized species while the oxidizing agent, which is reduced, may be any other material or solution (or corrosive environment).[3] Hence, from basic chemistry, two half reactions can be deduced and they are oxidation reaction where the metal loses electrons and reduction - gaining of electrons. [5] At the anode, oxidation occurs and reduction takes place at the cathode.

Similar to redox reactions are acid-base reactions which are like match pair occurring together or not without the other. [6]

### **2.2.2 Electrochemical Corrosion Reactions**

We consider the following conditions that must be met before electrochemical corrosion occur.

There must be the anode which is the metal that corrodes; the cathode; a constant path that conducts, electrolyte; and a medium that carries the flow of current from the anode to cathode.

[7]

In corrosion we have three (3) important types of cells that participate in the process. They are dissimilar electrode cells, which refers to for example, the surface of a metal that has electrically conducting impurities and another example is, two single crystals that are in contact with each other having different orientations; concentration cells which are two identical electrodes but are in contact with solutions that are not the same and there are two kinds, salt concentration cell – having to do with reactions that takes place in order to bring the solutions to the same concentration, and differential aeration cell – crevice corrosion is a typical example of this being that the oxygen difference yields a potential difference that causes flow of current; and differential temperature cells which less is known about the theory of its occurrence but they are identical metals with different temperature and are put into electrolyte having electrolyte composition initially the same. In real life practice these three combined may be the cells causing corrosion. [4]

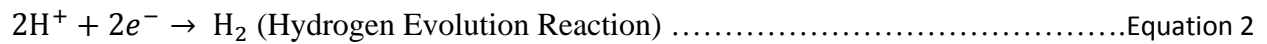
A corrosion system can be regarded as a short-circuited electrochemical cell. [8] The two principal reactions of electrochemical corrosion reactions are anodic and cathodic reactions. The

two reactions may not occur at the same site but they are simultaneous. The anodic reaction is in this general form:



M indicates metal as it dissociates and give out a free electron, thus, oxidized.

Several cathodic reactions are given as:



These reactions occur on the surface of the metal and may penetrate beneath the surface of the material. [11]

### 2.3 Corrosion Mechanisms of Steel

Steels are used for most engineering structures nowadays. The corrosion process is natural for metals because they are in a state where they seek to revert to their natural state which is more stable. [3]

Basically, steels are iron-carbon alloys [12]. The oxidation states of iron (Fe) are:

Fe (II)..... previously known as ferrous iron and is one of the most common oxidation state.

Fe (III)..... previously known as ferric iron and is one of the most common oxidation state.

Fe (VI) is not of high importance in this study. It is only stable in alkaline media that are highly oxidizing and it is the highest state in the ferrate ion,  $\text{FeO}_4^{2-}$  [14].

The two most common states of oxidation result in the forming of oxides, hydrous oxide phases, soluble cations, corresponding series of salts, and in consequent reactions may lead to change in pH of a solution or corrosive media they are immersed in. [14]

Of more importance is the oxidation of steels. Carbon, manganese, silicon, and due to deoxidation, excess aluminum are present in plain carbon steels; while higher concentration of these elements in addition to alloying elements like chromium, vanadium, nickel and molybdenum are present in alloy steel. [14] The roles of these alloying elements help also us to understand how steels corrode in certain environments. Alloying elements and their functions are briefly described as follows:

- Carbon: High carbon contents in steels at temperatures higher than 700 degree Celsius is likely to have negative effects on the oxide because of the oxidation carbon to carbon monoxide. Although this negative effect is not so at lower temperatures.
- Silicon: High concentrations of Silicon, about 3% or 5%, promote internal oxidation to some extent in electrical appliances. It stifles oxidation, provides to some extent resistance to gases that are sulfur-bearing which stimulate oxidation and this is done by promoting the formation of glassy silicates in oxide layers.
- Manganese: Its oxidation behavior is similar to iron. Based on its concentration, which may be moderate, is likely to not have large effect on the rate of oxidation.

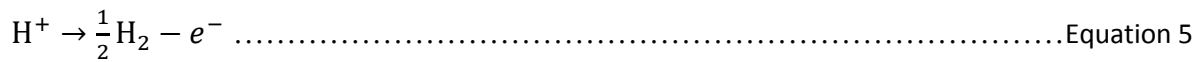


- Nickel: Nickel's oxidation behavior is quite similar to iron's. It also has not much effect on oxidation rate when its concentration is moderate but, it has influence on scale structure formation when in air. FeO and NiO form in initial oxidation stages as they both have related oxygen potentials even less than air. The process continues as scales become thicker and the amount of oxygen reduces but FeO becomes responsible for the oxygen potentials. NiO goes back to being in the formed scales in the form of metallic nickel.
- Aluminum: Doesn't have much concentration effect. By forming under the main scale a protective layer of  $\text{FeAl}_2\text{O}_4$ , the oxidation resistance can be improved with addition of Aluminum in steel.
- Molybdenum: The explanations of Molybdenum effects in flushing away scales and not being protective are quite confusing since it is known to have benefits but its benefits really can't be explained scientifically or adequately understood.
- Vanadium: Along with Molybdenum, vanadium and molybdenum give rise to disastrous oxidation. They produce oxides that with other scale components form low melting-point eutectics, thereby, fluxing away the scale formed.
- Sulfur: Sulfur has small effect and they are present in small residual concentrations.
- Phosphorous: Also in small concentration, its effect is little.
- Chromium: It is known for being corrosion resistance. [14]

Some of these constituents are there to stabilize the phases of some of the elements that are present in steels.

Most engineering metals have very thin oxide films, 3 to 10nm thick that protect them at ordinary temperatures. [9]

As the electrochemical theory of corrosion was briefly discussed, corrosion can be explained in terms of short – circuited galvanic cells on a metal’s surface. This means ions from steel go into solution at areas that are anodic which is chemically equivalent in amount to what is going on at cathodic sites. The rate at which steel corrodes is determined by the cathodic reactions and generally this is slow.



Cathodic reaction in deaerated solutions and it occurs faster in acids due to the low pH than in alkaline or neutral aqueous solutions. At a specific pH, hydrogen evolution rate depends on the presence of hydrogen over potential impurities in the steel. [4]

### **2.4 Forms of Corrosion**

Corrosion of metal may physically take on many forms as it reacts with the environment. The forms are distinct and can be described and classified in many ways. For this research purpose, they were divided into two; general or uniform corrosion and localized corrosion. Few forms of corrosion are discussed below.

In some forms of corrosion, there is almost no visible weight change or degradation, yet properties change and the material may fail unexpectedly because of certain changes within the material [2].

### **2.4.1 General or Uniform Corrosion**

General or uniform corrosion as the name implies occurs uniformly on the surface of the metal without any localization. It results in thinning or reduces the material and by visual inspection it can be detected. [10]

### **2.4.2 Localized Corrosion**

Localized corrosion is whereby there's an intense dissolution of metal at a particular site on the surface of the material but on the rest of the metal surface the corrosion is occurring at a lower rate. The reasons are: variation along the surface in corrosive environment, variation in alloy composition/microstructure and properties of the oxide film, mechanical factors such as stress or oxide film within the metal, and the metal geometry. [10]

#### **2.4.2.1 Stress Corrosion Cracking**

For stress corrosion cracking (S.C.C.) to occur it must fulfill these three requirements that there must be the presence of a tensile stress and this could be residual, locked in or applied stress; a material that is susceptible having a favorable microstructure; and an enabling environment such as a favorable temperature, pH, dissolved oxygen, chloride ion, carbon dioxide, hydrogen sulfide, etc. SCC may also initiate from pits and it occurs in different media (e.g. alkaline SCC also known as Caustic cracking). SCC could also be aggravated in the presence of chloride concentration and some other species. [10] This form of corrosion is common in boilers, tanks, etc.

#### **2.4.2.2 Pitting Corrosion**

Pitting corrosion, leads to the development of small holes in the metal. It has similar mechanism to crevice corrosion (driven by local chemistry of electrolyte), but occurs on a free surface. The alloy has to have a passive external surface for pitting to occur and be able supply a high

potential to cause current to flow into the pit. This driving force is not available for surfaces that are active. [10]

#### **2.4.2.3 Galvanic Corrosion**

Galvanic corrosion occurs when two dissimilar metals are in contact with each other. This will lead to one corroding preferentially to the other. The different electrochemical potentials can help in determining how these metals will corrode. The galvanic series and electromotive force series (EMF) are used. EMF is series having standard well-arranged electrochemical potential of metals while galvanic series can be determined by experiment and it deals with metals and alloys relative activity in certain environments. [10]

#### **2.4.2.4 Crevice Corrosion**

Crevice corrosion is usually associated with a stagnant solution in a narrow gap or crevice which is formed under gaskets, washers, fastener heads, surface deposits and coatings. Mechanism is similar to differential aeration (driven by changes in local chemistry of electrolyte), except that metal is initially passive. [10]

### **2.5 Causes of Corrosion?**

Even though corrosion has been said of a natural phenomenon, there are some factors that account for occurrence and/or progression of corrosion of metals when they interact with different environment.

#### **2.5.1 Thermodynamics of Corrosion**

Corrosion thermodynamics is very important as it tells us if the reaction of a metal and its environment is possible. Change in Gibbs free energy provides insight into this mechanism. It gives the spontaneity criterion for the occurrence of corrosion. This criterion is given as

$$\Delta G = -RT \ln K_{eq} \quad [13]$$

Where  $\Delta G$  is change in Gibbs free energy,  $R$  is a constant and  $K_{eq}$  equilibrium constant. [4]

Relationship between  $\Delta G$  in joules and emf in volts,  $E \quad \Delta G = -nF\Delta E \quad \dots\dots\dots$ Equation 5

$\Delta G$  = Gibbs free energy change

$n$  is the number of electrons (or chemical equivalents) taking part in the reaction

$F$  is the Faraday (96,500 C/eq)

The Nernst's Equation expresses emf exactly in terms of a cell's activities of products and reactants. [4]

$$E = E_o - RT/nF (\ln K_{eq}) \quad [13] \quad \dots\dots\dots \text{Equation 6}$$

Reactions are more likely to occur when the value of  $\Delta G$  is more negative. [4] But, it doesn't imply that the corrosion rate will be high as the value of  $\Delta G$  becomes more negative, other factors are to be taken into consideration for how fast or slow the corrosion rate is. The tendency for corrosion to occur in environments where  $\Delta G$  is positive is unlikely. [4]

Once more, the tendency for a material to corrode is independent of reaction rate and kinetics. [4]

### 2.5.1.1 Effect of pH

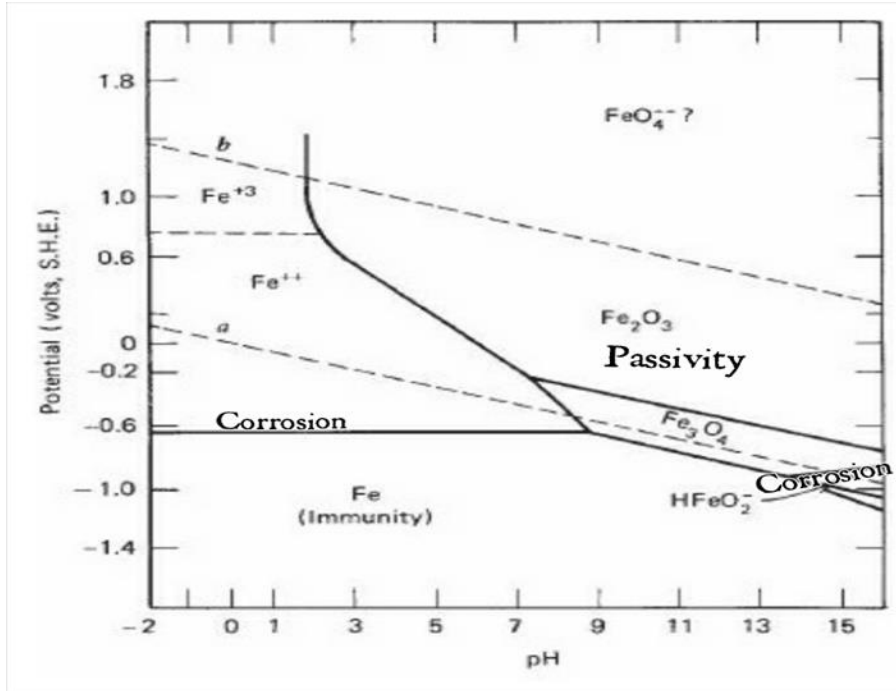
pH of a chemical measures between 0 and 14 which indicates its neutrality, acidity or basicity.[16] 7 is neutral, below 7 is acidic, and above 7 is basic. When metals are placed in any of these environments it will corrode, depending on the metals composition and other factors.

### 2.5.1.2 Pourbaix Diagram

The pourbaix diagram for iron/steel summarizes their corrosion thermodynamic behavior in different pH regions. This is done by mapping out the state of corrosion, passivity, and nobility, of iron using its thermodynamic data relative to its potential and the pH. The diagram is typically plotted on Cartesian coordinates having E on y axis and pH on the x axis for different equilibria. Pourbaix diagrams contains no information about the kinetics of corrosion, however, the thermodynamic boundaries for corrosion species and reactions that are important are defined by it. It defines corrosion behavior thermodynamic boundaries.

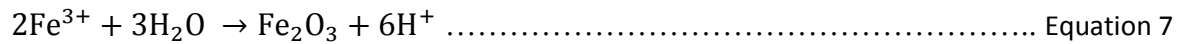
Corrosion potential and pH are important variables when it comes to wet corrosion; corrosion in aqueous media. [15] A metal's voltage when immersed in a solution is its potential. This is usually measured against a reference electrode. [11] When changes in other variables such as concentration of oxygen, changes are seen in the corrosion potential. [15]

Shown below in figure 2.1, the pourbaix diagram for iron/steel has 4 regions and they are regions of passivity, two areas of corrosion and immunity. The regions labeled corrosion indicate the occurrence of corrosion is favorable in these regions. Passivity implies that protective oxide or layer will form rather than corrosion occurring on the metal's surface. Immunity indicates corrosion will not form and this is use in formulating cathodic protections for steels. [16]

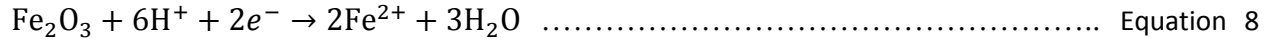


**Figure 1 E-pH diagram, pourbaix diagram or Predominance diagram at 25 degree Celsius 18 degrees Fahrenheit. Adapted from Reverie Corrosion handbook with modification.**

Equilibrium reaction on the vertical line at pH 1.76 is



pH>1.76 which on the right of the vertical line Fe<sub>2</sub>O<sub>3</sub> phase is stable and there's a possibility to form an oxide film that will protect the surface. pH<1.76 on the left shows stability of ferric ions. Therefore, with the absence of formation of oxide film to protect the surface, corrosion will occur. The sloping line between Fe<sup>++</sup> and Fe<sub>2</sub>O<sub>3</sub> has equilibrium reaction



[4]

**2.5.1.3 Pilling–Bedworth Ratio**

Pilling- Bedworth ratio is used to measure the occurrence of oxidation. This tells us about the corrosion product volume as compare to the metal on which it was formed. It however has some challenges. [4, 4]

The ratio is given as  $Md/nmD$ ..... **Equation 9**

M = molecular weight of corrosion product scale formed during oxidation

d = density of the metal

n = number of metal atoms in a molecular formula of scale

m = atomic weight of the metal

D = density of corrosion product scale formed during oxidation on the metal

$Md/nmD < 1$  Indicates corrosion product formed is less than the metal it was formed on. In such case, the scale formed is not protective. [4]

$Md/nmD > 1$  The scale of the corrosion product is greater than the metal it formed on. When such condition exist, the scale is protects the metal because it is in compression. However, this is not adequate for predicting corrosion resistance. [4]

$Md/nmD \gg 1$  Because of high stresses that may have developed, the scale(s) formed on the surface of the metal may buckle and detach. [4]



## **2.6 Corrosion Protection and Monitoring**

In order to properly control corrosion there must be a system where the structures are monitored and they are well protected. The cost of protecting and monitoring metallic structures in industries, specifically, oil and gas industries, cost several billion dollars. These two are important for safety, and unforeseen additional lost in finances that failure, fracture or spill, corrosion may cause. There are two ways in which protection is done; active corrosion protection and passive corrosion protection. Active corrosion protection is done to limit or rid corrosion off of a material using materials selection, project engineering, design, manufacturing and manipulation of the corrosion process. [3] Passive corrosion protection limits or prevents corrosion by applying protective layers to isolate the metal from corrosive environment. [3] How the protection and monitoring processes work are subsequently discussed.

Before applying corrosion protection layers, you must ensure that the protective layer is pore - free; adheres to the base metal properly; is resistant to mechanical stress; possesses ductility to some extent; and above all, has good corrosion resistance characteristics. [3] Most importantly, before applying protection layers there are certain requirements and they are; preparation of the surface the coating is to be applied on and quality when it comes to corrosion protection in design. [3]

### **2.6.1 Coatings**

Coatings are materials that act as barrier between the metal and its environment so that it is protected against corrosion. There are several types and these types are classified as organic, inorganic and metallic.

### **2.6.1.1 Organic Coatings**

Organic coatings serve as a barrier which is relatively thin, between a substrate material and its environment. [1] It is estimated in the United States of America that 2 billion dollars per year are used on organic coatings. [1]

There are different organic coatings used in different industries to combat corrosion. Some include: lacquers, epoxy, polyamide, paints and varnishers.

#### **2.6.1.1.1 Epoxy Coatings**

Epoxy coatings are versatile organic coatings that can be used anywhere for any application but may fade in sunlight. [1]

### **2.6.1.2 Inorganic and metallic coatings**

Inasmuch as they provide good barrier between the metal and its environment, inorganic coatings are brittle and metallic coatings are quite formidable. [1] When break in in these coatings occur, localized corrosion is accelerated since the metals are in contact. [1]

Application of metallic coatings is done by cladding, hot dipping, electrodeposition, vapor deposition, and flame spraying. Example of coated metallic material is tin can. 'Glassed' steel tanks are inorganic ceramic coatings. Inorganic coatings can be applied by diffusion, chemical conversion, or spraying. [1]

However, when the coatings are chosen based on how corrosive the environment is, the period in which the coatings serve as protection is known to be the expected service life until the first repairs. [3]

### **2.6.2 Cathodic and Anodic Protection**

Cathodic protection is one of the best ways to protect structures from corroding. The protection is done by supplying electrons to metallic structures that need to be protected. [1] Two ways by which this is done are by; carrying on proper galvanic coupling; or, supplying power externally. [1] Cathodic protection makes use of the immune region of iron on the pourbaix diagram to design cathodic protections for iron.

Using electrode kinetics principles, anodic protection was devised. [1] This method reduces the rate of corrosion meaningfully. [1] Potentiostat – an electronic device that with respect to a reference electrode maintains a metal at constant potential- is required for anodic protection. [1]

Anodic protection can be applied on active-passive metals only, whereas, cathodic protection can be applied on all metals. [1] It has high installation cost but very low operating cost while Cathodic protection has low installation cost but operating cost is between mediums to high.[1]

### **2.6.3 Corrosion monitoring and measurement**

Corrosion monitoring gives thorough information about corrosion of all structures in plants, facilities, assets, industrial objects, etc. [16] it identifies components that may not be working properly and helps in estimating or providing alternative life extension solutions for components that are failing based on information gathered. [16] The use of "probes" which may be electrical, electrochemical or mechanical devices provides such data. [17] There are many techniques being used to monitor corrosion, some of which are: Corrosion Coupons (weight loss measurements), Electrical Resistance (ER), Linear Polarization Resistance (LPR), Galvanic (ZRA) / Potential etc.

Corrosion measurement involves techniques that are used to determine the corrosivity of an environment and the loss metal. [17] This process quantitatively evaluates the effectiveness of the monitoring and protection processes as it measures corrosion through methods such as:

Non-Destructive Testing (NDT): Ultrasonic Testing

Radiography

Eddy Current

Analytical chemistry: pH measurement

Dissolved gas ( $O_2$ ,  $CO_2$ ,  $H_2S$ )

Metal ion count ( $Fe^{2+}$ ,  $Fe^{3+}$ )

Microbiological analysis

Fluid Electrochemistry: Potential measurement

AC impedance

Operational Data: pH

Flow rate (velocity)

Temperature

Pressure

## **2.7 Corrosion Rate Determination**

Expressions for good corrosion rate should take into account:

Penetration

Units that are familiar

Calculations that can be done easily but has small amount of error

The use of whole numbers to avoid problems with decimals [1]

Determining the rate at which corrosion is occurring can be done several ways. One way is to calculate using the formula

$$\text{cpy} = \frac{87.6W}{DAT} \dots\dots\dots\text{Equation 10}$$

Where cpy corrosion penetration per year in millimeter per year, W is weight loss in mg, D is density of specimen in g/cm<sup>3</sup>; A is the area of the specimen in sq.in. And T is exposure time, hr. Data are taken from the weight loss corrosion experiment. In the United States, mpy is commonly used because corrosion rate is expressed with small integer values of penetration. [1]

Similar expressions can be given as

$$\frac{\mu\text{m}}{\text{yr}} = 87,600 \frac{W}{DAT} \dots\dots\dots\text{Equation 11}$$

Where W represents the weight loss in milligrams, D density in g/cm<sup>3</sup>, an area in cm<sup>2</sup>, and T time of exposure in hours.

Electrochemical methods can be used to determine corrosion rate also. Electrochemical methods make use of current density. The conversion into penetration rates derived from Faraday's law

$$\text{Corrosion penetration rate} = K \frac{ai}{nD} \dots\dots\dots \text{Equation 12}$$

$a$  = the atomic weight of the metal

$i$  Is the current density in  $\mu\text{a}/\text{cm}^2$

$n$  Is the number of electrons lost (valence change)

$D$  Is the density in  $\text{g}/\text{cm}^3$

$K$  is a constant depending on the penetration rate desired

$K$  values are 0.129, 3.27, and 0.00327, for  $\text{mpy}$ ,  $\frac{\mu\text{m}}{\text{yr}}$  and  $\frac{\text{mm}}{\text{yr}}$ , respectively. [1]

### 2.7.1 Weight Loss Measurement

Weight Loss measurement is the simplest, most common and cheapest way of monitoring and measuring corrosion. The process by which this is done is by exposing the coupon (sample of metal to be experimented on) to an environment that may be corrosive and then remove the coupon for analysis, usually weight loss.[16] The range of application for this method is very wide because it is can be used for all environments; provides medium for analyzing deposits of corrosion on the material; easily calculate and analyze corrosion rate; provides better visual; performances of inhibitors are examined; measures and identifies localized corrosion; etc. [16]One of the disadvantages is that it may not give significant corrosion rate given the duration

of the experiment which may not experience weight loss that is significant. [16] Hence, the correlation between some other methods like ER and LPR becomes helpful. [16]

### **2.7.2 Linear Polarization Resistance (LPR) Technique**

This technique is based on electrochemical theory that is complex but can be broken to fundamental concepts for use in industries [16].

The way it works is, electrode in a solution has voltage applied to it and current needed to maintain a specific voltage shift relates directly to corrosion occurring on the surface of the electrode. Corrosion rate is calculated from measuring the current. The advantage of this technique as compare to others is that it is instantaneous but it doesn't work well in gases or oily environments, only in clean aqueous electrolytic environments [16].

## **2.8 Petroleum Products**

Petroleum products are made from crude oil that consists of a mixture of many different hydrocarbon compounds. Their composition varies and considers the location it was produced. The different products that come out of a refinery are defined by their boiling temperatures during distillation in order of increasing boiling point. [17]

### **2.8.1 Gasoline**

A refined product of crude oil, gasoline consists of mixture of hydrocarbons, blending agents and additives. The composition of gasoline varies on where it was produced and the processes involved in its production. Typically, gasoline has 4-8% alkanes; 2-5% alkenes; 25-40% isoalkanes; 3-7% cycloalkanes; 1-4% cycloalkanes; 20-50% total aromatics (0.5-2.5% benzene) according to the international agency for research on cancer in IARC summary & Evaluation, Volume 45, 1989. Additives and blending agents such as anti-knock agents, anti-oxidants, metal

deactivator, detergents, and dyes are included in gasoline to enhance its performance and maintain its stability. [19, 19]

### **2.8.2 Diesel**

Diesel fuel like other fuels has the following typical composition of complex mixtures: aliphatic alkanes (paraffins) and cycloalkanes (naphthenes) having 80-90% of the fuel oils, 10 – 20% and 1% of aromatics and olefins. [18]

## **2.9 Prior Work on Corrosion in Above-ground Storage Tanks**

Recently, Hobbs et al. in 2013 studied in their research *Catastrophic failure of a carbon steel storage tank due to internal corrosion* the failure of an aboveground storage tank in 2009 that contained about 350m<sup>3</sup> waste organic solvents such as heptane, methyl acetate, ethyl acetate, acetone, etc. The tank was high 10 m with a diameter of 8 m. They took samples from the fractured tank, investigated the environment and the storage tank materials. The thickness and hardness were measured, metallographic analyses were done on the fractured steel and the chemical analysis was taken. They also modeled the surface the storage tank. Corrosion in the tank was observed. Pitting corrosion and the content of the tank may have attributed to residual stresses in the tank, and also the design and inspections were faulty.

The intriguing aspect of this research is that pH was incorporated into single well tracer test (SWTT). Wellington et al. in 1994 studied *Redesigned Ester Single-Well Tracer Test That Incorporates pH-Driven Hydrolysis Rate Changes*. Even though their work didn't include storage tanks, the idea of using pH is of relevance to this research. They observed ester



hydrolysis reactions were sensitive to pH change and with that they could monitor reactions, their variations at different periods.

## CHAPTER THREE: MATERIALS AND METHODS

### 3.1 Materials

For the experiment in this study, carbon steel was the steel selected. The steel was obtained from the Liberia Petroleum Refining Company (L.P.R.C). It was used for repair of the storage tanks. The steel was then sectioned at S.C.C., Abuja, Nigeria with a dimension of 1 cm x 1 cm x 0.6 cm. These cut low carbon steels served as samples/coupons in the experiment. The basis for these selections is for simulation of the actual interaction between the steel, coatings and petroleum products.

#### 3.1.1 Carbon Steel

Steels are alloys made with iron-carbon that may have other alloying elements present but with appreciable concentrations relative to iron and carbon. <sup>[12]</sup> They are categorized under ferrous alloys which fall under metal alloys. The types of carbon steel are based on the composition of carbon present in the alloy even though Iron is the major constituent. Below 0.25 wt. % C we have low carbon steel, between 0.25 wt. % C and 0.60 wt. % C is medium carbon steel, and between 0.60 and 1.4 % C is high carbon steel. [12, 12]

##### 3.1.1.1 Chemical Analysis

C%	Si%	Mn %	P%	S%	Cr%	Mo%	Ni%	Cu%	Fe%
0.184	0.088	0.37 8	0.01 8	<0.001	0.007	<0.002	<0.002	<0.001	99.291

**Table 1 Chemical Analysis of low carbon steel**

### 3.1.2 Carbon Steel Preparation

The samples were grinded in the multi-functional lab at AUST Abuja, Nigeria. The coupons in total were 8. Their containers were labeled with the sample's number, dimensions of the sample, mass, density, surface area and medium. Air was not allowed in these containers.

#### 3.1.2.1 Grinding

Grinding machine was used with 60 and 120 grits abrasive papers to grind the rough surface of the samples. Water was added as the grinding took place to avoid high temperature as the metals and grinding papers come in contact so as to maintain the sample microstructure and rid of small particles contamination due to the grinding. The uncoated samples were grinded with 60 and 120 grits paper to remove corrosion from the surface.

### 3.1.3 Corrosive Media

#### 3.1.3.1 Diesel

Diesel fuel served as one of the solutions in which the coupons were placed in. Measured in eight (4) containers, four (4) uncoated samples were placed in them. All of them were under the same

conditions. Different containers were selected to avoid interruptions or contaminations from tests as the experiment proceeded especially air exposure.

Chemical Composition of Diesel from literature is:

Hydrocarbon type	Diesel	Volume %	
		Fuel oil no. 1	Fuel oil no. 2
Paraffins ( <i>n</i> - and iso-)		52.4	41.3
Monocycloparaffins		21.3	22.1
Bicycloparaffins		5.1	9.6
Tricycloparaffins		0.8	2.3
Total saturated hydrocarbons		79.7	75.3
Olefins		No data	No data
Alkylbenzenes		13.5	5.9
Indans/tetralins		3.3	4.1
Dinaphthenobenzenes/indenes		0.9	1.8
Naphthalenes		2.8	8.2
Biphenyls/acenaphthenes		0.4	2.6
Fluorenes/acenaphthylenes		No data	1.4
Phenanthrenes		No data	0.7
Total aromatic hydrocarbons		23.6	24.7

\*Derived from IARC 1989; provided by the American Petroleum Institute

**Figure 2** Obtained from <http://www.atsdr.cdc.gov/toxprofiles/index.asp>

### 3.1.3.2 Gasoline

Gasoline was made as a corrosive media where coupons were placed. The coupons placed were four (4) uncoated coupons. Literature gives the following composition of Gasoline:

Component	Percentage Composition <sup>a</sup>
<i>n</i> -alkanes	3.0
C <sub>10</sub> -C <sub>11</sub>	11.6
C <sub>12</sub> -C <sub>13</sub>	1.2
C <sub>14</sub> -C <sub>15</sub>	0.7
C <sub>16</sub> -C <sub>17</sub>	0.8
total of <i>n</i> -alkanes	17.3
branched alkanes	2.2
C <sub>10</sub> -C <sub>11</sub>	15.1
C <sub>12</sub> -C <sub>13</sub>	8.0
C <sub>14</sub> -C <sub>15</sub>	1.9
C <sub>16</sub> -C <sub>17</sub>	1.8
C <sub>18</sub> -C <sub>19</sub>	2.1
total of branched	32.0
cycloalkanes	3.0
C <sub>6</sub>	1.4
C <sub>7</sub>	0.6
total of cycloalkanes	5.0
olefins	1.8
C <sub>9</sub>	1.8
total of olefins	1.8
aromatics	
benzene	3.2
toluene	4.8
xylenes	6.6
ethylbenzene	1.4
C <sub>7</sub> -benzenes	4.2
C <sub>8</sub> -benzenes	7.6
others	2.7
total aromatics	30.5
carboxylic acids	

<sup>a</sup>Adapted from Air Force 1989

Figure 3 Adapted from <http://www.atsdr.cdc.gov/toxprofiles/index.asp> and modified.

### 3.2 Samples Preparation for Experiment

After sectioning with electronic cutting machine at S.C.C. Nigeria Limited in Abuja, Nigeria, the samples were grinded with 60 and 120 grits size abrasive paper, 60 for coated and 60 and 120 for uncoated samples respectively. They were dried and weighed using weigh balance in AUST biology lab. After weighing and measurement, they were rinsed in acetone for degreasing and cleaning of their surfaces from any contamination. [1] Petri dishes and glass bottles were gathered.

45 milliLiter (mL) of each; Diesel and Gasoline were measured using erlenmeyer flasks, funnel, beakers and graduated cylinders. They were first poured into 100ml beakers with a funnel, then,

with funnel 45ml of each solution was poured into graduated cylinders and poured into designated and marked petri dishes or glass bottles. With gloved hands handling a tong, the samples were placed in specific petri dish or glass bottle, lid closed using crucible in the space of maximum 5 minutes to avoid air contamination. This was repeated for every sample. The containers were left in the multifunctional lab at AUST under normal room temperature.

### 3.3 Method

#### 3.3.1 Weight Loss Measurement

Weight loss measurement is the simplest way of measuring corrosion and is to a significant extent accurate because corrosion may result in appreciable loss of metal. This method involves measuring the weight of corroded sample relative to its weight before exposure.

The samples after the first 24 hours and from henceforth 72 hours exposure time over the period of 456 hours of corrosive environment exposure were removed using tong. Chemical cleaning procedure for removal of corrosion products was achieved by cleaning samples with acetone using cotton. After cleaning, the samples were dried and weighed on Mettler model AE 100 model weighing balance. These were performed at AUST labs.

#### 3.3.2 Corrosion Rate Determination

The overall corrosive environment exposure time for this experiment was 540 hrs. Corrosion rate can be determined many ways involving faraday's equation, polarization, weight loss measurement etc. Corrosion rate calculation from mass loss formula is given below <sup>[12]</sup>:

$$\text{Corrosion penetration rate} = \frac{(K \times W)}{(A \times T \times D)} \dots\dots\dots \text{Equation 13}$$

Where

$K$  = a constant which is 87.6

$T$  = time of exposure in hours to the nearest 0.01

$A$  = area in  $\text{cm}^2$

$W$  = mass loss in g, to nearest 1 mg (corrected for any loss during cleaning) and

$D$  = density in  $\text{g/cm}^3$

### **3.3.3 pH Measurement**

The pH measurement was gathered for understanding of corrosion and its rate. pH data was measured against time for each corrosive medium and sample type. Solutions' pHs were measured at Nigeria Turkish Nile University, Abuja, Nigeria. The pH of Gasoline and diesel were measured before and after corrosive exposure.

#### **3.1 Surface Morphology**

##### **3.1.1 Optical Microscope**

Optical microscope was used to obtain micrographs of the microstructure and the surface.

##### **3.1.2 Scanning Electron Microscopy**

Scanning electron microscope was used in viewing in depth surface of the low carbon steel.

## CHAPTER FOUR: RESULTS AND DISCUSSION

### 4.1 Weight Loss Measurement and Corrosion Rate

The weight loss of coupons was measured after exposure. The data earned are tabulated below.

Sample No.	Corrosive Medium	Weight loss (mg)									Corrosion Penetration Rate Millimeter per year (mm/y)
		24 hrs.	72 hrs.	120 hrs.	168 hrs.	216 hrs.	312 hrs.	360 hrs.	408 hrs.	456 hrs.	
1	Diesel	0.6	3	4	6	2					0.3250
8	Diesel						0.325	0.322	0.326	0.328	0.0160
2	Gasoline	0	0	5	6	4					0.3125
7	Gasoline						1	2	1	2	0.0740

**Table 2 Weight loss measurement for the coupons with their respective media**

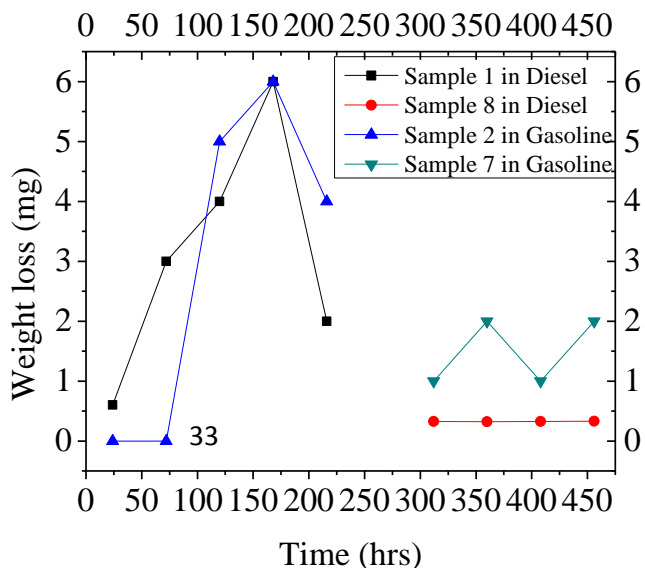
After 24 hours of immersion of low carbon steel in gasoline and diesel, samples 1 and 7 were weighed using a weighing balance. It showed that after 24 hours for both diesel and gasoline



there wasn't any significant corrosion occurring. After 48 hours, the weight of the sample immersed in diesel was reduced, corrosion had started occurring. It took another 48 hours for the low carbon steel in the gasoline medium for corrosion to start occurring and when it did, the weight loss was greater than the weight loss of sample 1 in diesel by a difference of one at the same time at 120 hours. At 168 hours both samples had the same weight loss, 6 mg. There was a drop in weight loss of both samples 1 and 2 at 216 hours. The weight loss decreased by a difference of 4 and 2 mg, for samples 1 and 2 in diesel and gasoline, respectively. It was observed even though corrosion initiated late in sample 2 in gasoline, at the end of 216 hours, the weight loss was a bit higher than sample 1 in diesel. However, the average weight loss for samples 1 and 2 when rounded up to whole numbers was the same 3 mg.

The same pattern of weight loss that is the increase in weight loss followed by decrease in weight loss was observed for samples 8 in diesel and 7 in gasoline. The only difference was the weight loss, even though it fluctuated between and 1 and 2 mg, sample 7 in gasoline had more weight loss than sample 8 in diesel.

The losses in weight of samples in gasoline was more but slower on the overall. Nevertheless, the weight loss of the low carbon steel in gasoline and diesel exhibited an interesting similarity in their mechanisms of corrosion.



#### Figure 4 Plot of weight loss vs time using OriginPro.

The increase and decrease in weight loss can be attributed to several reactions that may have taken place but the formation of passive film by the steel to protect itself under the conditions it was exposed to is an alternative explanation. As such, when there was a break in the passive films, the metal corroded and vice versa.

The average corrosion rate for each sample with their time of exposure was calculated. It showed that even though there was corrosion occurring, it was well below 0.50 mm/yr. [12]

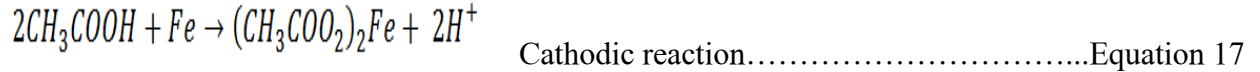
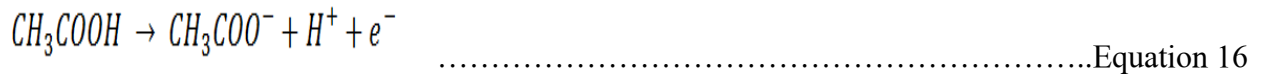
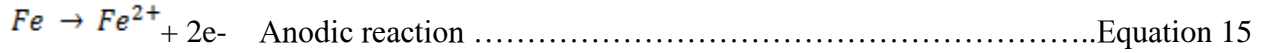
#### 4.2 pH measurement

The following table shows the readings of pH taken after 456 hours of exposure when the samples were immersed. After the removal of the samples, the pH was taken after 24 hours (470 hours).

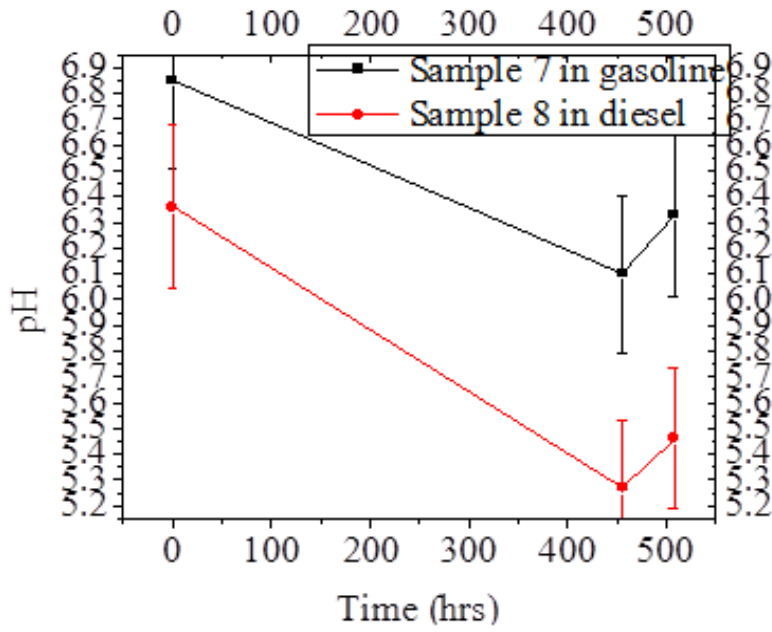
Sample No. and Medium	pH after Exposure		pH before Exposure
	456 hrs.	508 Hrs.	
7 (Gasoline)	6.10	6.33	6.85
8 (Diesel)	5.27	5.46	6.36

**Table 3 pH of solutions containing samples 7 in gasoline and eight in diesel.**

The results show that as the low carbon steel corroded, the solutions became acidic. From literature, it was gathered that one possible reaction for this is as follows



Carboxylic acid, acetic acid had the tendency to react with the metal. The corrosion product formed was iron acetate. We have the evolution of hydrogen ion which increased the concentration of hydrogen ion thereby giving a reduction in pH after 456 hours. When the steel was removed and exposed to air, oxygen acted as base and reacted with the solutions in the form of neutralization reaction forming water and the pH shifted up wards again. See plot and below



**Figure 5** pH versus time for samples 7 and 8.



**Figure 6** Image shows formation of water

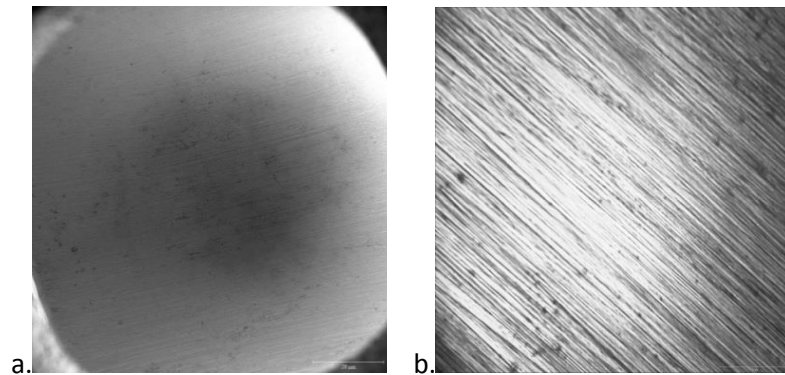
#### 4.3 Surface Morphology

##### 4.3.1 Microstructure of low carbon steel before immersion

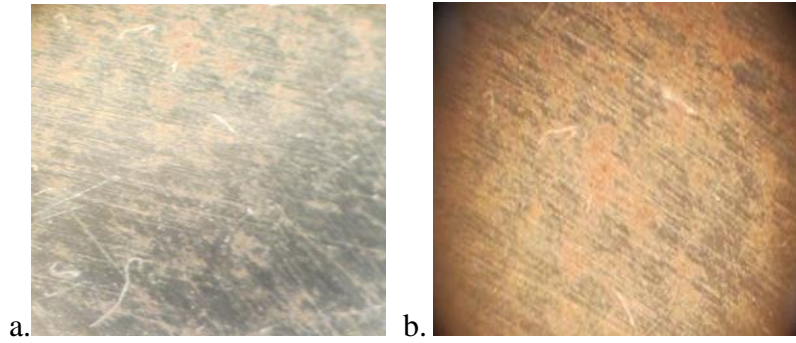


**Figure 7 Microstructure of low carbon steel before exposure. 3x400**

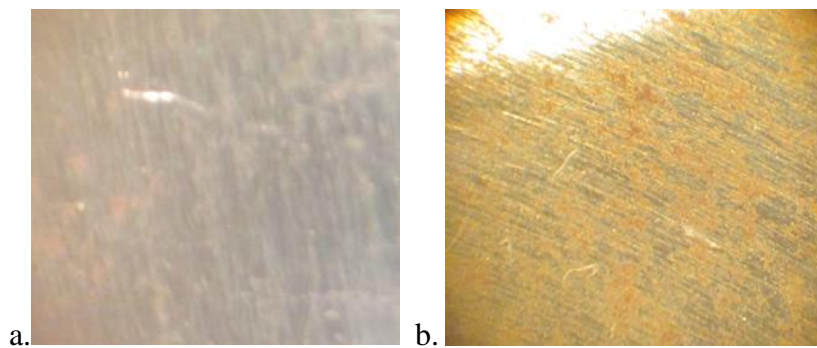
This low carbon steel consists of ferrite and pearlite phases, with the pearlite phase dominating. The microstructure provides insight on how the low carbon steel will behave exposed to our corrosive environments.



**Figure 8 a. Sample 7 and b. Sample 8 before immersion into gasoline and diesel respectively**



**Figure 9: Sample 1 after 264 hrs. of immersion in diesel b. Sample 8 after 456 hours of exposure in diesel. Optical Microscope x4**



**Figure 10 a. surface of Sample 2 after 264 hrs. of exposure in gasoline medium and b. surface of sample 7 after 456 hrs. of exposure to gasoline medium using optical microscope x4**

It can be seen from the micrographs that the corrosion product on the surface of the metal is not uniform. And this can be related to the inhomogeneity on the surface of the metal as seen from the microstructure. The sites where the corrosion products were first formed were the sites having ferritic composition since they are more susceptible to corrosion than the pearlite with alternating layers of ferrite and cementite.

## **CHAPTER FIVE: CONCLUSION AND RECOMMENDATIONS**

### **5.1 Conclusion**

To conclude, the low carbon steel corroded at a very low and acceptable rate in diesel and gasoline due to the noncorrosive nature of the organic hydrocarbons in diesel and gasoline. The behavior of the steel based on its microstructure experienced corrosion first at selective regions which were at areas having the ferrite phase due to its susceptibility to corrosion. A form of reaction between carboxylic acid in the diesel and gasoline with the metal contributed to the corrosion of the low carbon steel. When the metals corroded, they experienced weight loss. The dissolution of metallic ions contributed to the corrosion reactions, thus, reducing the pH of the two by evolution of hydrogen ions. After the samples were removed, the pH started to increase due to the introduction of oxygen and neutralization reaction occurring forming water. From these results a simple and cheap method of inspection for corrosion can be designed.

### **5.2 Recommendations**

I recommend that this experiment be carried out for hours more 456 hours and temperature included as a variable to examine the effect of temperature. Additional analysis should be done, such as analysis of the surface using EDS. In addition to pH, diesel and gasoline composition should be analyzed as the experiment goes on. The pilling Bedworth ratio can also be used in addition to corrosion rate to tell us more about the ratio between the scales formed and the corroded metal for further knowledge on the scales formed.

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