Chapter 1

CHAPTER-1

INTRODUCTION

1.1 CORROSION

Corrosion is the destructive attack of a material by reaction with its environment. Corrosion is a universal phenomenon and many times in spite of all precautions it has led to the failure of machines and structures. Corrosion is a surface-active phenomenon and a mere knowledge of the bulk composition is insufficient to understand the mechanism of corrosion of alloys.

1.1.1 Need for corrosion studies

Corrosion becomes a serious problem in our day today life and we are in need to pay more attention to corrosion due to¹.

- The economic importance in reducing the material loss
- The need to conserve metal resources
- Increasing use of metals due to rapid industrialization
- Pollution of air and water resulting in a corrosive environment
- Requirement of safety measures from fire hazards, explosion or release of toxic products.

1.1.2 Factors influencing corrosion

The nature of metals and environment determines the rate and extent of corrosion².

Nature of the metals

- Position of the metal in emf series
- Hydrogen over voltage
- Relative areas of anode and cathode
- Physical state of the metal
- Oxidation potential and purity of the metal
- Nature of the oxide film
- Solubility and volatility of the corrosion products

Nature of the environment

- Humidity and temperature
 - Effect of pH
- Nature of the cations and anions present
- Concentration and conductivity of the medium
- Formation of oxygen in concentration cells
- Polarization of electrodes

1.1.3 Principles of corrosion

It includes metallurgical, electrochemical and thermodynamic principles. Out of these electrochemical and thermodynamic principles are more important.

Electrochemical principles³

The electrochemical process of corrosion takes place by anodic and cathodic reactions of the reducible species present in the environment. This can be illustrated by the attack of iron in hydrochloric acid. When the iron is dipped in acid, a vigorous reaction occurs resulting in the evolution of hydrogen gas, where iron dissolves in acid to give Fe^{2+} ion in solution.

Fe
$$\longrightarrow$$
 Fe²⁺ + 2e⁻ (at the anode)
2H⁺ + 2e- \longrightarrow H₂ (at the cathode)

Thermodynamic principle

The spontaneity of a chemical reaction can be studied by thermodynamic principles. The tendency of the metal to corrode during corrosion depends upon the energy associated with the given chemical reaction which determines the feasibility of the corrosion reaction.

1.1.4 Theories of corrosion

Homogeneous theory

A metal subjected to corrosion can be regarded as a single electrode on which reactions take place, irrespective of the presence or absence of any micro heterogeneity. Charge transfer reaction takes place at the interface of the metal. As a result, the metal becomes unstable. It is necessary that the potential difference across the interface must be more negative than the equilibrium potential for the metal dissolution reaction or it must be more positive than the equilibrium potential for the electronation reaction to occur.

Heterogeneous theory

According to this theory, corrosion occurs when the metal is exposed to moist atmosphere. Corrosion is caused by local galvanic elements arising on the surface of the metal, as a result of the chemical structure heterogeneity.

Mixed potential theory

According to this theory, any electrochemical reaction can be divided into two or more partial oxidation and reduction reactions⁴. The metal immersed in an electrolyte cannot spontaneously accumulate electrical charge. The total rate of oxidation equals the total reduction for an electrically isolated metal sample during the process of corrosion.

The three important parts of a corroding metal are

- An electron source area where electronation reaction occur
- An electron sink area where de-electronation reaction occur
- An ionic conductor to keep the ion current flowing

1.1.5 Types of Corrosion⁵

(i) Galvanic corrosion

Galvanic corrosion occurs when metals having different electrode potentials are coupled in presence of an electrolyte come into contact in an electrolyte. The potential difference between the dissimilar metals is the driving force for the accelerated attack on the anode member of the galvanic couple. The electrolyte provides a means for ion migration where by the metallic ions can move from anode to cathode. This leads to the anodic metal corroding more quickly than the other. The corrosion of cathodic metal is retarded. The galvanic corrosion is maximum at the junction of the two metals. The presence of an electrolyte and an electronic conducting path between the metals is essential for galvanic corrosion to occur.

(ii) Pitting corrosion

It is a form of extremely localized corrosion that leads to the creation of some small holes in the metal. The driving force for pitting corrosion is the depassivation of a small area, which becomes anodic while the other area becomes cathodic leading to galvanic corrosion. It is one of the most destructive and insidious forms of corrosion. The corrosion penetrates in to the metal with limited diffusion of ions. The acidity created inside the pit is maintained by the spatial separation of the cathodic and anodic half reactions which creates a potential gradient and electro migration of aggressive ions in to the pits. The formation of pits, damages the structure of the metal. This type of pitting corrosion is initiated by small surface defect or damage to protective coating.

(iii) Crevice corrosion

Intense localized corrosion occurs normally within crevices and other shielded areas on the metal surface when exposed to corrosive environment. This type of attack is usually associated with small volumes of stagnant solution caused by holes, gasket surface, tap joints, surface deposits and crevices under bolt and river heads.

(iv) Microbial corrosion

It is otherwise called as bacterial corrosion, biocorrosion or microbial induced corrosion (MIC). MIC is caused by microorganisms, usually chemoautotrophs. It can apply to both metals and non-metallic materials. Microorganisms associated with corrosion are of two types, aerobic and anaerobic. Bacterial corrosion appears in the form of pitting corrosion, for example in pipelines of the oil and gas industry. This type of corrosion also occurs in plastics, concrete and many other materials.

(v) Stress corrosion cracking

Stress corrosion cracking is a type of growth in cracks in a corrosive environment. This type of corrosion leads to unexpected sudden failure of normally ductile metals subjected to a tensile stress, especially at elevated temperature. It is highly specific that certain alloys are likely to undergo this type of corrosion only when exposed to some chemical environments.

(vi) Intergranular corrosion

This type of corrosion occurs in certain alloys when exposed to a particular temperature. It is characterized by a localized attack at the boundaries with relatively little corrosion at the inner core of the metal. As a result of this corrosion the alloy disintegrates and loses its strength. It is caused by the impurities at the grain boundaries, enrichment of one of the alloying element or depletion of one of these elements in the grain boundary areas.

(vii) Uniform attack

Uniform corrosion is the most common form of corrosion. It is usually characterized by chemical or electrochemical reaction that proceeds over the entire exposed surface or over a large area. Due to this electrochemical reaction, the metal becomes thinner resulting in a failure. It can be prevented by use of inhibitor, coatings and cathodic protection.

(viii) Stray current corrosion

Stray current corrosion is caused by direct current. Great damage may thus be caused by direct current from welding generators used in electric welding.

(ix) Corrosion fatigue

This type of corrosion is caused by the combined action of corrosion and cyclic stress on the metal. This is a special form of stress corrosion leading to a drastic reduction in the life of the metal when placed in a corrosive environment. The corrosion fatigue mechanism involves the formation of extrusions and intrusions at the surface thereby destroying the protective surface on the metal.

(x) Erosion corrosion

Erosion corrosion or impingement corrosion is associated with increasing corrosion rate caused by the relative motion between the metallic surface and the corrosive environment. It is caused by the breakdown of a protective film at the spot of impingement and its subsequent inability to repair itself under existing abrading conditions. This type of corrosion normally occurs in systems where high velocities of the fluid are encountered.

(xi) Cavitation corrosion

Combined action of high liquid flow rate and corrosion results in cavitation corrosion. The word cavitation denotes the occurrence of a gas or vapor bubbles in a liquid. Two types of cavitation are distinguished as gas cavitation and vapor cavitation. Example: Hydraulic turbulence, ship propellers

(xii) Filiform corrosion

It is a special case of crevice corrosion which results in irregularly developed hair fine lines of filaments of corrosion products below coatings of paint, rubber, lacquer, and enamel etc., Filiform corrosion tends to follow grinding and polishing marks and is independent of metallurgical factors.

1.1.6 Methods to control corrosion

The rate of corrosion can be reduced by either retarding anodic or cathodic reactions which can be achieved in several ways.

(a) Conditioning the metal

(i) Coating the metal

Corrosion resistant coating can be applied on the metal which may consists of another metal Eg: Zn or tin coating on steel, a protective coating derived from the metal itself, organic coating such as resin, plastics, paints, enamels etc.

(ii) Alloying the metal

To produce more corrosion resistant alloy, the metal can be alloyed with another suitable metal. Eg: Stainless steel in which ordinary steel is alloyed with Cr and Ni.

(b) Conditioning the corrosive environment

(i) Removal of Oxygen

By the removal of oxygen from water systems in the pH range 6.5-8.5 one of the components required for corrosion would be absent. The removal of oxygen could be achieved by the use of strong reducing agents. Eg: Sulphite.

(ii) Using corrosion inhibitors

A corrosion inhibitor is a chemical additive, which when added to corrosive aqueous environment, reduces the rate of metal wastage.

(c) Electrochemical control

Corrosion is an electrochemical reaction whose progress may be studied by measuring the changes which occur in the metal potential with time or applied electric current. The rate of the corrosion reaction may be controlled by passing anodic or cathodic current to the metal.

(i) Anodic protection

This method of protection is based on the phenomenon of passivity in which the metal to be protected is given a fixed potential so as to produce a passive film on it thereby preventing from corrosive environment. This method is applicable to metals with requirements such as,

- The metal or alloy should have active passive transition.
- The metal must require a small current to maintain passivity.
- It must have a wide passive range.
- The aggressive medium must have sufficient electrical conductivity to which the metal is exposed.

(ii) Cathodic protection

Cathodic protection of a metal is done by making the entire metallic surface to function as cathode without the existence of any anodic site in the electrolytic cell so that the metal does not undergo corrosion. The two methods of applying cathodic protection are galvanic and impressed current method. A direct current supply is available in both the methods.

1.2 CORROSION INHIBITORS

1.2.1 Definition

Corrosion inhibitors are chemical substances or mixtures which when added in low concentration to an aggressive environment minimizes or prevents corrosion⁶. They minimize the corrosion rate by either acting as a barrier by forming an adsorbed layer or retarding the cathodic and anodic process⁷.

1.2.2 Functions of inhibitors

The significant functions of an inhibitor are

- It forms a thin film by adsorption on the surface of a corroding metal.
- It induces the formation of thick corrosion product.
- It leads to the formation of protective precipitate on the metallic surface or removes the aggressive constituents thereby preventing the metal from corrosion.

1.2.3 Characteristic features of a good inhibitor

- It should be free from toxicity and pollution problems
- It should be effective over a long period of time
- It should be economically and technically competitive with other inhibitors
- It must have high magnitude of suppressing the corrosion rate.

1.2.4 Classification of corrosion inhibitors

Based on electrode process, inhibitors are classified as follows

(a) Anodic inhibitors

Anodic inhibitors are also called as passivating inhibitors. They facilitate the formation of protective oxide film on the metallic surface and inhibit the anodic metal dissolution reaction⁸. The concentration of the inhibitor plays an important role in the anodic inhibition. The effect of these anodic inhibitors depends on the nature and concentration of aggressive ions.

(b) Cathodic inhibitors

Cathodic inhibitors shift the corrosion potential in the negative direction. Here migration of cations towards the cathodic surface takes place. The inhibitor then precipitates merely blocking the metallic surface. Cathodic inhibitors are considered as safe because they do not cause localized corrosion.

The three main inhibiting mechanisms of cathodic inhibitors are,

(i) Cathodic poisons

Cathodic poisons such as arsenic and antimony are used to suppress the rate of cathodic reactions. They increase the susceptibility of the metal to hydrogen induced cracking.

(ii) Cathodic precipitators

Cathodic precipitators such as calcium and magnesium will precipitate insoluble compounds on the metallic surface by increasing the alkalinity at the cathodic sites.

(iii) Oxygen scavengers

They reduce the corrosion by forming a product by reaction with oxygen present in the system.

(c) Mixed inhibitors

Mixed inhibitors work by retarding both the anodic and cathodic reactions. They protect the metallic surface by acting through physical adsorption, chemisorption and film formation. Adsorption plays important role in deciding the effectiveness of these inhibitors and the adsorption depends on the structure of the inhibitor, type of the electrolyte and on the surface charge of the metal.

Classification of inhibitors based on the environment

Acid inhibitors

Acid inhibitors may be further classified into (i) Inorganic inhibitors and (ii) Organic inhibitors

Inorganic inhibitors

The action of these inhibitors have been attributed to the reduction of electropositive ions and deposition on the surface of the metal by lowering the over voltage of the cathodic depolarization reaction⁹. Inhibitors such as bromide and iodide have been found to be effective in strong acid solution. The use of heavy metal ions such as Pb^{2+} , Ti^{2+} , Mn^{2+} and Cd^{2+} as inhibitors have been recently studied in acid medium.

Organic inhibitors

A wide range of organic substances have been studied as corrosion inhibitors in acid solution¹⁰⁻¹¹. Organic compounds containing oxygen, nitrogen, sulphur and multiple bonds have been reported as good corrosion inhibitors¹²⁻¹⁴. These inhibitors act as anodic, cathodic or mixed type depending on the molecular size, carbon chain length, aromaticity, conjugation and nature of bonding atoms.

Neutral inhibitors

These inhibitors are highly effective in acid solution but failed to function in neutral solutions. This is because in neutral solutions these inhibitors interact with oxide film coated on the metallic surface and prevent the oxygen reduction at the cathodic sites.

Alkaline inhibitors

The metals which form amphoteric oxides are susceptible to corrosion in alkaline solution. Naturally occurring organic compounds can form metal complexes in basic solution and can be used as inhibitors. Example: Thiourea, Naphthol.

Vapour phase inhibitors

Vapour phase inhibitors are also called as volatile corrosion inhibitors. These inhibitors inhibit corrosion by volatilization from the source temporarily. Volatile solids such as cyclohexylamine, hexamethylene amine are reported as effective inhibitors¹⁵. The vapours of these inhibitors condense and get hydrolyzed by moisture and liberate protective ions with good inhibiting property. The effectiveness of these inhibitors depends on the inhibition efficiency and pressure of the vapour.

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Classification of inhibitors based on the mode of protection

(a) Chemical passivators (b) Adsorption inhibitors (c) Film forming inhibitors

Chemical passivators

Chemical passivators are substances with higher equilibrium potential and lower over potential and they decrease the corrosion rate on attaining passitivity. Examples: Nitrites and chromates

Adsorption inhibitors

Compounds containing lone pair of electrons are used as adsorption inhibitors. These inhibitors act by adsorption on the metal surface and form a blanket over the surface. Generally these inhibitors affect both cathodic and anodic reactions.

Film forming inhibitors

These inhibitors tend to form a barrier film of materials other than the actual inhibitory species itself to prevent corrosion. Such inhibitors affect either cathodic or anodic reactions. Zn and Ca salts are most commonly used cathodic film forming inhibitors.

1.2.5 Mechanism of corrosion inhibition

The principle mechanism of corrosion inhibition reported by several researchers in the field of corrosion is based on the concept of adsorption. This concept has been successful in the corrosion of metals in acid medium. These inhibitors get adsorbed on the surface of the corroding metal forming a protective layer thereby preventing it from corrosion. The basic processes involved in corrosion are

- Oxidation process
- Reduction process
- Electronic charge transfer process
- Ionic charge transfer process

Corrosion inhibition takes place by interfering with any one of the above process and brings down the rate of the reaction or may bring about a limitation in rate of one or other of the remaining three processes¹⁶.

1.2.6 Theories of inhibition

Corrosion inhibitive action of the inhibitor has been explained by adsorption theory or film formation theory ¹⁷.

(i) Adsorption theory

According to the adsorption theory put forth by Machu and co-workers¹⁸ corrosion inhibition is due to the formation of a layer of inhibitive substances with high electrical resistivity on the surface of the metal. It is further classified as physical or chemical adsorption based on the mode of adsorption.

(a) Physical adsorption

Physical adsorption is due to the electrostatic attraction existing between the ions of the inhibiting species and electrically charged surface of the metal. The surface charge of the metal depends on free corrosion potential and potential with respect to zero charge.

(b) Chemisorption

Chemisorption is the result of interaction between the metal surface and an inhibitor molecule. Chemisorptions favor the formation of a coordinate bond by the charge transfer from the inhibitor molecule to the metal surface¹⁹. Temperature plays an important role in chemisorption. During chemisorption electron transfer from inhibitor to the metal is facilitated by the presence of unshared lone pair of electrons, Π-electrons due to multiple bonds and aromaticity.

(ii) Film theory

According to this theory formation of slightly soluble or insoluble corrosion products on the metal surface provides effective protection. Earlier research reveals the formation of thin protecting film of slightly soluble or insoluble complex compounds of iron sulfide with thioethers and thioalcohols ²⁰.

(iii) Hydrogen over voltage theory

According to this theory the inhibitor gets adsorbed on the metal surface retarding either the anodic or cathodic or both the reactions. This causes anodic or cathodic polarization of the reaction sites there by reducing the overall corrosion rate.

(iv)Quantum chemical approach

According to Quantum chemical approach corrosion inhibition is due to the chemisorption favored by the direct interaction of outer most electrons of the inhibitor molecule with the surface of the metal. The chemical adsorption of the inhibitor can be considered as linear combination of the wave function of the surface metal atoms.

The binding energy can thus be attributed to the energy difference between lowest unoccupied molecular orbital (LUMO) and highest occupied molecular orbital (HOMO)

1.2.7 Factors affecting the applicability of inhibitors

- Nature of the metal surface
- Nature of the environment
- Temperature and pH of the system
- Concentration of the inhibitor
- Scale formation
- Disposal of toxic substances and effluent problems

1.3 CORROSION MONITORING TECHNIQUES

Corrosion monitoring is important through which systematic measurement of corrosion rate of the materials has been done with the objective of diagnosis and controlling corrosion. Corrosion measurement is the quantitative method which allows us to estimate the corrosiveness of the environment as well as the weight loss experienced by the material.

1.3.1 Non-Electrochemical methods

Weight loss measurement

This is the most reliable method. In this method, the weight loss of a metal due to corrosion is measured by exposing the metal specimen of known area to the environment for a known period of time. The difference in weight before and after the exposure is then calculated.

The corrosion rate is expressed in mils per year (mpy)

 $Corroion rate = \frac{534 \text{ X weight loss in milligrams}}{\text{Density X AreaX Time}}$

Gasometric method

Gasometric technique is based on the principle that a corrosion reaction in aqueous media is characterized by the evolution of gas resulting from the cathodic reaction of the corrosion process which is proportional to the rate of $corrosion^{21}$.

The rate of evolution of the gas (RVH) is determined from the slope of the graph plotted between volume of gas evolved (v) and time (t). The main drawback of this method is it is time consuming and cannot be applied to strong medium.

Hydrogen permeation method

In acidic solutions the cathodic reaction on the metal surface involves hydrogen evolution. The discharged hydrogen atoms on the surface of the metal penetrate in to the metal. When this system is connected to an external circuit a current flow can be recorded. This is known as permeation current which is a measure of corrosion rate.

Electrochemical methods

Electrochemical methods are widely used in inhibitor testing. These methods require short duration which is a definite advantage and gives more accurate results. The most important methods are,

(i) Polarization method

In this method corrosion rate is determined by measuring the electric current flow between the test and auxillary electrode while polarizing the test electrode with respect to reference electrode. In order to measure and control the potential and current flow a three electrode system- the specimen or working electrode, an auxillary electrode and a reference electrode is used. A standard polarization test cell recommended by ASTM G5 is used. For a small change in potential (+10 mV), the relation between current flow and the potential is linear and follows the Stern-Geary equation which is used to determine the corrosion current.

$$I_{corr} = \frac{b_{a^*}b_c}{2.303(b_a + b_c)} \cdot \frac{\Delta I}{\Delta E}$$

Where, b_a and b_c are the anodic and cathodic Tafel constants respectively, ΔE and ΔI are the changes in potential and current respectively and I_{corr} is the corrosion current.

The measurement of open circuit potential (OCP) is used for monitoring corrosion. The main advantage of the polarization method is that the corrosion reaction can be monitored continuously. This is useful in determining the effect of process changes with time on the corrosion rate.

AC impedance method ²²

AC impedance technique has been used for the measurement of the corrosion rate, due to the main advantage that the double layer capacitance (C_{dl}) and charge transfer resistance R_t can be determined. The charge transfer resistance, R_t is a measure of the corrosion rate. In this method an alternating voltage of 10-20 mV is applied to the cell and the resulting current and phase angle is measured for various frequencies.

The cell impedance is resolved in to two parts

Real part $Z'=|Z|\cos\Phi$ Imaginary part $Z''=|Z|\sin\Phi$

The charge transfer resistant R_t is related to I corr as

$$R_t = \frac{b_a * b_c}{2.303 (b_a + b_c) * I_{corr}}$$

The impedance Z is the total resistance to the alternating current and it is given by

$$Z=Z'-Z''$$

Where Z' =

$$R_{s} + \frac{R_{t}}{1 + \omega^{2} C^{2}_{dl} R^{2}_{t}}$$
$$Z'' = \frac{\omega C_{dl} R_{t}^{2}}{1 + \omega^{2} C^{2}_{dl} R^{2}_{t}}$$

The plot of Z' against Z" for various frequencies results in a semicircle which cuts the real axis at higher and lower frequencies. At the higher frequency end the intercept corresponds to R_s and at lower frequency end the intercept corresponds to $R_s + R_t$.

The double layer capacitance is obtained from the frequency at which Z'' is maximum.

$$\omega(\mathbf{Z}''\max) = \frac{1}{C_{dl}*R_t}$$

Advantages

- 1. It is a more valuable technique which involves the measurement of both capacitance and charge transfer resistance
- 2. It provides mechanistic information as it is applicable to low conductivity system also.
- 3. This method employs amplitude in the range of 5-10 mV peak to peak. Hence polarization is minimum and reduces the errors caused by the measurement.
- 4. Even small changes in the specimen can alter the impedance plot as this method is very sensitive.

Faradaic rectification method ²³⁻²⁵

In this method corrosion rate of the metal is measured from the rectified current and potential when a voltage of frequency ω is applied to the electrode. The open circuit potential (OCP) in this method is steady. By adjusting the back off voltage the current passing through the cell is reduced to zero. The potential now corresponds to the corrosion potential (E_{corr}). An alternating voltage in the range 30-40 mV is applied and the direction of the current and magnitude are recorded. From the recorded values, the corrosion rate (I_{corr}) can be calculated. The important characteristic feature of this method is that single AC frequencies are measured.

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Objectives of the Present Study

OBJECTIVES OF THE PRESENT STUDY

The objectives of present work is to

- Synthesize differently substituted benzofused seven membered heterocyclic compounds such as benzodiazepines, benzothiazepines and benzoxazepines.
- Evaluate the corrosion inhibition performance for mild steel in 1M H₂SO₄ under varied conditions.
- Compare the corrosion inhibition efficiency of some selected benzodiazepine for mild steel, copper and aluminium.
- > Analyze the effect of surfactants on corrosion inhibition efficiency.
- Carry out quantum chemical calculation with the title compounds in order to correlate the molecular parameters with corrosion inhibition efficiency.