

# INHIBITION OF CORROSION ON MILD STEEL IN ACID MEDIUM USING INDOLE

*PROJECT REPORT*

*Submitted in partial fulfillment for the Award of the First Degree Programme in Chemistry under CBCSS  
to the University of Kerala*

**BY**



*Under the Supervision of*

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**MARCH 2015**

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## CERTIFICATE

*This is to certify that the thesis entitled “INHIBITION OF CORROSION ON MILD STEEL IN  
ACID MEDIUM USING INDOLE” submitted by  
, to the University of Kerala in partial fulfillment of  
the requirements for the First Degree Programme in Chemistry under  
CBCSS is an authentic record of work carried out under the supervision and guidance of Dr. Suja  
Mathai at the department of Chemistry, Mar Ivanios college, Thiruvananthapuram.*

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## CERTIFICATE

*This is to certify that the thesis entitled “INHIBITION OF CORROSION ON MILD STEEL IN ACID MEDIUM USING INDOLE” is an authentic record of the original work carried out by in the department of Chemistry, Mar Ivanios college, Thiruvananthapuram under my supervision and guidance in partial fulfillment of the requirements for the award of First Degree Programme in Chemistry under CBCSS to the University of Kerala.*

## DECLARATION

We hereby declare that the dissertation entitled “**INHIBITION OF CORROSION ON MILD STEEL IN ACID MEDIUM USING INDOLE**” is the report of original work carried out by us from January-March 2015 at Department of Chemistry, Mar Ivanios College (Autonomous), Thiruvananthapuram under the guidance of **Dr. Suja Mathai**, in partial fulfillment of the requirements for the Degree of Bachelor of Science in Chemistry from the University of Kerala and that no part of it has previously been presented for any other degree or diploma in any university.

Thiruvananthapuram  
March 2015

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## **ABSTRACT**

Corrosion inhibitions of mild steel in HCl medium have been studied using different electrochemical techniques. The corrosion of mild steel alloy in 1M HCl acid can be effectively inhibited by addition of Indole in different weight percentages. The results of Weight loss and Open circuit potential measurements shows that Indole in lower weight percentage is an effective corrosion inhibitor in the acid medium. The surface morphology is analyzed using Optical microscopy. From all these studies the optimum weight % of Indole shows better inhibition efficiency.

# **CHAPTER 1**

## **INTRODUCTION**

### **1.1 CORROSION**

The corrosion of steel is a fundamental academic and industrial concern that has received a considerable amount of attention [1]. Corrosion is the deterioration of metal by chemical attack or reaction with its environment. Metals generally tend to move to its original state by corrosion process.

Mild steel is an alloy form of iron and is extensively used fabrication material in sugar, petrochemical, food, paper and textile industries [2] which undergoes corrosion easily in acidic medium. Acid solutions are widely used in industry, chemical laboratories and in several industrial processes such as acid pickling, acid cleaning and oil wet cleaning etc [3]. In acid media, the use of hydrochloric acid in pickling of metals, acidization of oil wells and cleaning of scales is more economical, efficient and trouble free compared to other mineral acids [4]. Corrosion products are formed when a metal gives its electron to the oxidizing substances. Thus the use of corrosion inhibitors is one of the most practical methods for corrosion protection of steel especially in acid media [5-7]. Many of the well known inhibitors are organic compounds containing nitrogen, sulfur, and oxygen atoms [8-11]. It has been observed that many of the organic inhibitors act by adsorption on metal surface [12]. This phenomenon is influenced by the nature and surface charge of metal, type of aggression medium and chemical structure of inhibitors. The adsorption of corrosion inhibitors depends mainly on physico-chemical properties of molecules such as functional groups, steric factor, molecular size, molecular weight, molecular structure, aromaticity, electron density of the donor atom and  $\pi$ -orbital character of donating electron [13-17] and also the electronic structure of molecule [18, 19].

### **1.2 CONSEQUENCES OF CORROSION**

The consequences of corrosion are many and varied and the effects of these on the safe, reliable and efficient operation of equipment or structure are often more serious than the simple loss of a mass of metal. Failures of various kinds and the need for expensive replacements may occur even though the amount of metal destroyed is quite small. Some of the major harmful effects of corrosion can be summarized as follows.

1. Reduction of metal thickness leading to loss of mechanical strength and structural failures or breakdown.
2. Hazards or injuries to people arising from structural failure or breakdown (E.g.: bridges, cars, and aircraft).
3. Loss of time in availability of profile-making industrial equipment.
4. Reduce to value of goods due to deterioration of appearance.
5. Contamination of fluids in vessels and pipes goes cloudy when small quantities of heavy metals are released by corrosion.
6. Perforation of vessel and pipes allowing escape of their contents and possible harm to the surroundings.
7. Loss of technically important surface properties of a metallic component.
8. Mechanical damage to valves, pumps, etc. or blockage of pipes by solid corrosion products.
9. Added complexity and expense of equipment which needs to be designed to withstand a certain amount of corrosion, and to allow components to be conveniently replaced.



**Figure 1.1: Corrosion of Mild Steels in industries**

### 1.3 MECHANISM OF CORROSION

Corrosion of steel is an electrochemical reaction that requires the presence of water ( $\text{H}_2\text{O}$ ), oxygen ( $\text{O}_2$ ) and ions such as chloride ions ( $\text{Cl}^-$ ), all of which exist in the atmosphere. Atmospheric chloride ions are in greatest abundance anywhere near the coastline. This electrochemical reaction starts when atmospheric oxygen oxidizes iron in the presence of water.



**Figure 1.2: Schematically exhibits of corrosion process**

### 1.4 TYPES OF CORROSION

The different types of corrosion are:

### 1. **Uniform corrosion**

In this type, chemical or electrochemical reaction takes place uniformly on the metal surface. Uniform corrosion occurs when there are local anodic and cathodic sites on the surface of the metal. Due to polarization effect these location lift from time to time and a given area on a metal will act as both an anode and as a cathode over an extended period of time. The average effect of these shifting local action cells results in a rather uniform attack and general loss of material and roughing of the surface.

### 2. **Galvanic corrosion**

Galvanic corrosion is a type of electrochemical corrosion in which two different types of metals in contact are jointly exposed to corrosive atmosphere. Here the metals with more negative electrode potential become anode and goes into solution or corrode.

### 3. **Pitting corrosion**

Pitting corrosion is a localized attack resulting formation of holes in the metal otherwise relatively unattached surface. A pit may be considered as a self formed crevice [20].

### 4. **Fretting corrosion**

It is a wear phenomenon involves wear of a metal or alloy when it is in contact with another solid material in dry or humid air.

### 5. **Underground and soil corrosion**

The various factors that responsible for soil corrosion are acidity of soil, moisture contents, electrolyte degree of aeration and physical properties of soil. Source having low receptivity has high corrosivity.

### 6. **Erosion corrosion**

When the movement of a corrodant over a metal surface increases the rate of surface increases the rate of attack due to both mechanical wear and corrosion, the type of attack so

produced is known as erosion corrosion. It is evidenced by the appearance of groves and trough on the metal surface.

#### 7. **Waterline corrosion**

This type of corrosion results from differential aeration leading to the formation of oxygen concentration cells. When water is stagnant in a steel tank for a long period of time, the corrosion takes place just below the water level.

#### 8. **Crevice corrosion**

If a crevice between different metallic objects like bolts, nuts and rivets is in contact with liquids, the crevice becomes an anodic region and suffers corrosion. This type of corrosion is known as crevice corrosion.

#### 9. **Exfoliation and selective leaching**

Exfoliation is a sub surface corrosion which starts on a clean surface and spreads below it. Due to this the whole layers of material are corroded. Selective leaching is the removal of elements of an alloy. This type of corrosion is highly undesirable as it yields a porous metal with poor mechanical properties.

#### 10. **Microbiological corrosion**

Some type of bacteria, exert some influence on the corrosion process. Such type of corrosion is known as microbiological corrosion. Oxygen consuming bacteria present in water or soil decreases the concentration of oxygen in the medium. If the metal is totally exposed to this medium, it may reduce corrosion. But if the structure is only partially exposed, then the bacterial action may intensify the corrosion.

#### 11. **Stress corrosion cracking**

Metal components are subjected to unevenly distributed stresses during their manufacturing process. The electrode potential thus varies from one point to another area under great stress and acts as an anode and undergoes corrosion.

## **1.5 METHODS OF CORROSION PREVENTION**

These are several ways of preventing corrosion and the rate at which it can propagate with a view of improving the lifetime of metallic and alloy materials.

### **1. Cathodic protection**

The principle involved in cathodic protection is to force the metal (iron) behave like a cathode since there will not be any anodic area on the metal, corrosion does not occur. Impressed current cathodic protection and sacrificial protections are of two types. In impressed current cathodic protection an impressed current is applied in an opposite direction to nullify the corrosion and converting the corroding metal from anode to cathode [21].

In sacrificial anodic protection a more active metal is connected to the metal to be protected. The more active metal loses electrons in preference to other and converts itself into ionic state. With the passage of time, the more active metal gets consumed but so long as it is present there, it will protect the iron (metal) from rusting and does not allow even the nearly exposed surface of iron to react. The metal which is most often used for covering iron with more active metal is zinc and the process is called “galvanization”. The layer of zinc on the surface of iron, when comes in contact with moisture, oxygen and carbon dioxide in air, a protective invisible thin layer of basic zinc carbonate  $\text{ZnCO}_3 \cdot \text{Zn(OH)}_2$  is formed due to which the galvanized iron sheets lose their luster and also tends to protect it from further corrosion.

### **2. Anodic protection**

This is another important form of corrosion control in which metal is passivated by applying current in a direction that renders it more anodic. The technique is only applicable to such metals and alloys which exhibit active-passive behavior. Like cathodic protection, this is also applicable when the corrodant is an electrolyte. The important parameters in anodic protection are,



- Potential range over which the metal is passive should be wider
- The current density needed to start the protection should be wider

The lower the passive current needed to maintain protection the lesser will be the operating costs.

### 3. **Protective coating**

Another important method for protecting a metal from corrosion is to apply a protective coating, i.e., for separating the surfaces that are susceptible to corrosion from factors in the environment which cause corrosion to occur. The protective coating may be metallic, nonmetallic, inorganic or organic substances. The metallic coating often used are Zn, Sn, Ni, Cu, Cr, An, Pb. Metallic coatings are usually imparted by hot dipping, electroplating, metal spraying, metal cladding and cementation. The inorganic nonmetallic protective coating includes surface conversion or chemical dip coating, anodized oxide coatings and vitreous enamel and lacquers. The protective coating can never provide 100% protection of 100% of the surface. If localized corrosion at coating defect is likely to cause rapid catastrophic failure, additional corrosion control measures must be taken.

### 4. **Addition of inhibitors**

A corrosion inhibitor is a chemical additive, which when added to a corrosive aqueous environment reduces the rate of metal wastage. Common inhibitor species used include alkyl quaternary ammonium salts and imidazoline derivatives that are strongly surface active and contain a cationic hydrophilic head group coupled to one or more alkyl chains that form the hydrophobic tail group.

Inhibitor performance is strongly dependent on the composition of the aqueous solution in contact with steel surface for a variety of factors. Inhibitor adsorption is different for different surfaces. Inhibitor adsorption to the relevant surface is also strongly dependent on factors such as electrolyte concentration and temperature. Inhibiting agents are effective only

if their solubility in the corrosive environment is in the right range. Very low solubility of inhibitor leads to lack of active agent at the metal interface and consequently to weak the inhibition. If the solubility is too high the substrate will be protected, but for only a relatively short time since the inhibitor will be rapidly leached from the coating.

## **1.6 INHIBITORS**

Corrosion potential of each metal solution system depends on the exchange current densities of anodic and cathodic reactions. The optimal inhibition by inhibitors at the point of zero charge and the inhibition are best if the corrosion potential coincides with this. A good inhibitor should have one of the following characteristics.

- Aromatic and the presence of  $\pi$  bonds
- High electric polarity of substituent groups in the organics.
- Availability of lone pairs in the substituent groups
- Long side chains up to about 11 carbon atoms.
- Energy of HOMO and it's compatibility with the energy of LUMO in the metal [22].

Inhibitors reduce corrosion by either acting as a barrier by forming an adsorbed layer or retarding cathodic or anodic process [23].

### **1.6.1 Classification**

Inhibitors are divided into two broad categories.

- Inhibitors which enhance the formation of a protective oxide film through an oxidizing effect.
- Inhibitors that are selectively absorbing on the metal surface and creating a barrier that prevents access of corrosive agents to the metal surface.

The inhibitors which retard cathodic process are called Cathodic inhibitors. They function by reducing the available area for the cathodic reaction. Those which retard anodic process are called Anodic inhibitors. Those which influence both processes are called mixed inhibitors.

These observations are generally referred to as “synergism” and demonstrate the synergistic action which exist between zinc and chromate ions.

Depending on the mechanism and mode of protection, they are classified as,

***Chemical passiveters:*** These inhibitors have a sufficiently high equilibrium potential and sufficiently low over potential are called chemical passiveters or oxidizing inhibitors.

E.g.: Nitrites, chromates, silicates, ferric cyanide etc..

***Adsorption inhibitors:*** They get adsorbed on the metal surface and provide a blanketing effect.

E.g.: 0.1% hexamethylenetetramine in 10% HCl at a maximum temperature of 70°C

***Film forming inhibitors:*** They form a blocking of barrier films of a material other than actual inhibiting species.

For E.g.:  $\text{Zn}^{2+}$  from cathodic inhibitors reacts with cathodic alkali  $\text{OH}^-$  to form  $\text{Zn}(\text{OH})_2$  films.

***Vapour phase inhibitors:*** These are substances of low but significant vapor pressure, the vapor of which has a corrosion inhibiting property.

E.g.: diphenyl and cyclic amines.

### **1.6.2 Mechanism of inhibition**

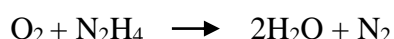
A common mechanism for inhibiting corrosion involves formation of a coating, often a passivation layer, which prevents access of the corrosive substance to the metal.

The nature of the corrosive agent depends on:

- The material being protected, which are most commonly metal objects
- On the corrosive agent to be neutralized

The corrosive agents are generally oxygen, hydrogen sulphide and carbon dioxide.

Oxygen is generally removed by reductive inhibitors such as amines and hydrazine.



In this example, hydrazine converts oxygen, common corrosive agent to water, which is generally benign. Related inhibitors of oxygen corrosion examine phenylenediamine, and dimethyl ethanolamine and their derivatives.

Anti oxidants such as sulfites and ascorbic acid are Benzotriazole is one such species used to protect copper. For lubrication, zinc dithiophosphates are common, they deposit sulfide on surface. The suitability of any given chemical for the task in hand depends on many factors, including their operating temperature.

### **1.7 Review of Literature**

The ancient Greek Historian Herodotus (Fifth century BC) and the ancient Roman naturalist, Piny the Elder (First century BC) mentioned the adsorption of tin for the protection of iron from corrosion. Alchemists through centuries made fertile attempts to transform base metals in to noble ones. Early attempts to mitigate corrosion of metals were empirical and centered largely on the use of organic and metallic coatings. Inhibitors for acid corrosion of metals were known from middle ages. These were obvious measures to protect metallic structures constructed by early artisans, often at the expense of much time and very hard labor.

**Lomonosov (1743 – 1750)** was the first to make broad systematic experiments on the study of the action of acids on metals.

**Faraday (1820 – 1882)**, established a very important relationship between chemical action and the generation of electric current.

**Davy (1826)** proposed an electrochemical method using sacrificial anode for the protection of copper sheathed ocean going ships.

**De La Rive (1830)** attributed the pronounced corrosion of impure zinc metal to the operation of short-circuited microcells on the metal surface.

**Marangoni and Stephanelli (1872)** used extracts of glue and gelatin and bran to inhibit the corrosion of iron in acids. This and subsequent discoveries of effective corrosion inhibitors were to large extent: the result of empirical studies.

**Wagner (1938)** proposed a mixed potential theory. The theory proclaimed that (i) any electrochemical reaction comprised of two or more partial oxidation and reduction reactions, (ii) there can be no net accumulation of electrical charges during an electrochemical reaction and the potential at the entire surface of an isolated electrode should be the same.

The first patent in corrosion inhibition was awarded to **Baldwin (1960)** British patent 2327 which involved the use of molasses and vegetable oils for pickling steel sheet in acid. Later, increased research activities in corrosion inhibition studies were started.

Recently, plant extracts have become important source of corrosion inhibitors because of its environmental friendliness and availability [24]. Several scientists have used natural products as corrosion inhibitors for various metals and alloys in aggressive media [25-26].

## CHAPTER 2

### AIM AND SCOPE OF PRESENT WORK

#### 2.1 BACKGROUND OF THE PRESENT PROBLEM

Mild steel is extensively used fabrication material in sugar, petrochemical, food, paper and textile industries. Mild steel corrodes easily because all common structural metals form surface oxide films when exposed to pure air but the oxide formed on mild steel readily broken down, and in the presence of moisture, it is not repaired. Therefore a reaction between steel (Fe), moisture (H<sub>2</sub>O) and Oxygen (O<sub>2</sub>), takes place to form rust. This reaction is complex but it can be represented as chemical equation of the following type:



2Fe<sub>2</sub>O<sub>3</sub>·H<sub>2</sub>O is the rust, and is not usually protective; therefore, the corrosion process is not impeded [27]. The corrosion of mild steel and other metals in many industries, construction, installations, and civil services such as electricity, water, sewage supplies is a serious problem.

#### 2.2 AIM AND SCOPE OF THE PRESENT WORK

Mild steel is an alloy form of iron and is extensively used fabrication material in sugar, petrochemical, food, paper and textile industries which undergoes corrosion easily in acidic medium. Acid solutions are widely used in industry, chemical laboratories and in several industrial processes such as acid pickling, acid cleaning and oil wet cleaning etc. Some of the important fields of application being acid pickling of iron and steel, chemical cleaning and processing, ore production and oil well acidification. The present work aims to explore and evaluate efficient inhibitor for effective protection of mild steel in acid media. In order to prevent or minimise corrosion, inhibitors are especially used especially in flow cooling system [28].

### **2.3 SELECTION OF INHIBITORS**

The use of inhibitors for the control of corrosion for metals and alloys which are in contact with aggressive environment is an accepted practice. Large numbers of organic compounds were studied to investigate their corrosion inhibition potential. All these studies reveal that most of the excellent acid inhibitors for corrosion of steel in acidic medium are organic compound containing nitrogen, oxygen and/or sulphur atoms [29-41]. But, unfortunately most of these compounds are not only expensive but also toxic to living beings. The selection of inhibitor is controlled by its economic availability, its efficiency to inhibit the substrate material and its environmental side effects. Here the inhibitor used is a non-nitrogenous polymer named Indole. Indole is taken in different volume % in a solution of 1M HCl.

### **2.4 PLAN OF THE WORK**

The inhibition characteristics of different weight % of Indole will be explored and its concentration will be optimized. If there is any critical concentration involved during the course inhibition, then the role of the preferred inhibitor will be optimized based on the mechanism of action. It is also decided to evaluate the variation in the extent of corrosion attack as the result of depletion of the inhibitor and by means of in-situ addition of excess inhibitors. The optimized combinations are planned to be evaluated under simulated condition also.

## CHAPTER 3

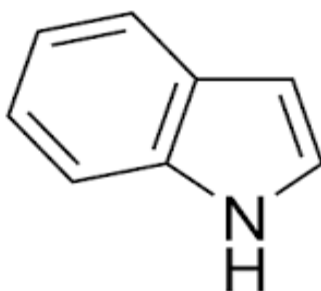
### EXPERIMENTAL METHODS

#### 3.1 MATERIALS

##### 3.1.1 Inhibitor structure of compound

The composition of the inhibitor used is as shown below:

| NAME OF INHIBITOR | FORMULA   | COMPANY  |
|-------------------|-----------|--|
| Indole            | $C_8H_7N$ | LOBACHEMIE, Laboratory reagents and fine chemicals, Mumbai |



**Indole**

##### 3.1.2 Solutions

An aggressive solution where made of AR grade HCl. 1 M concentration of acid was prepared with double distilled water. In this a known concentration of inhibitors were employed.

#### 3.2 ELECTRODES AND SPECIMENS

The working electrode was mild steel strip of dimension  $3 \times 2 \times 1 \text{ cm}^3$ . The composition of mild steel used for the study is shown in Table3.1.



**Table 3.1: Elemental Composition of Mild steel in weight (%)**

| <b>C</b> | <b>Mn</b> | <b>Si</b> | <b>P</b> | <b>S</b> | <b>Cr</b> | <b>Mo</b> | <b>Ni</b> |
|----------|-----------|-----------|----------|----------|-----------|-----------|-----------|
| 0.128    | 0.942     | 0.127     | 0.020    | 0.017    | 0.013     | 0.021     | 0.011     |

Prior to all measurements the electrodes were polished with different grades emery papers (grades 220, 400, 500 and 600), degreased with acetone and rinsed with distilled water.

### **1. Reference electrodes**

A saturated calomel electrode was used as reference electrode.

### **2. Counter electrode**

Platinum grid of large surface area (2X2 cm<sup>2</sup>) is used as the counter electrode.

### **3. Solutions used**

The solutions used were 1M HCl (blank) and different weight % of Indole in 1M HCl,

## **3.3 EVALUATION METHODS**

### **3.3.1 Physical methods**

#### **1. Visual tests**

The passive film formed on the mild steel surface was examined carefully. The film formation indicates the ability of Indole for corrosion inhibition in high concentration in HCl solution.

#### **2. Weight loss method**

The experiment is carried out under room temperature, which consists of five 250ml beakers, which separately contained 150 ml of 1M HCl (blank) and Indole in different weight % in 1M HCl. Previously weighed mild steel coupons were each suspended in each beaker with the help of glass hooks. The mild steel coupons were retrieved from these solutions at 120 hours interval, and weighed, progressively for 600 hours (25 days).

The percentage inhibition efficiency was calculated by the equation,

$$IE (\%) = (W1 - W2) \times 100 / W1$$

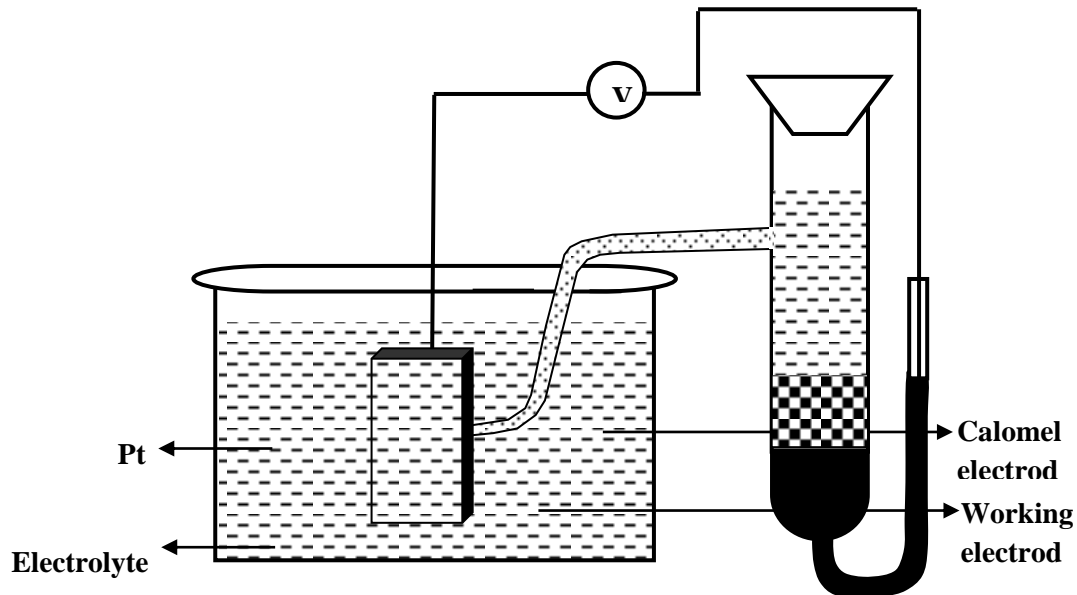
Where, W1- weight loss of mild steel in uninhibited solution

W2- weight loss of mild steel in inhibited solution.

### 3.3.2 Electro chemical methods

#### 1. Open circuit potential measurements

The potential of the working electrode was measured with respect to saturated calomel electrode (SCE) in 1M HCl from the time of immersion in different % of inhibitor at room temperature. The variation of OCP was then followed as a function of time until a steady value is attained using a multi meter. The setup for OCP measurement is shown in Figure 3.1.



**Figure 3.1: Experimental set up for OCP measurement**

#### 3.3.4 Surface analysis

The specimens dipped in different inhibitor solutions in OCP analysis were dried without disturbing and examined for their structural and topographical features using OPTICAL MICROGRAPH METHOD.

## CHAPTER 4

### RESULTS AND DISCUSSION

#### 4.1 WEIGHT LOSS MEASUREMENTS

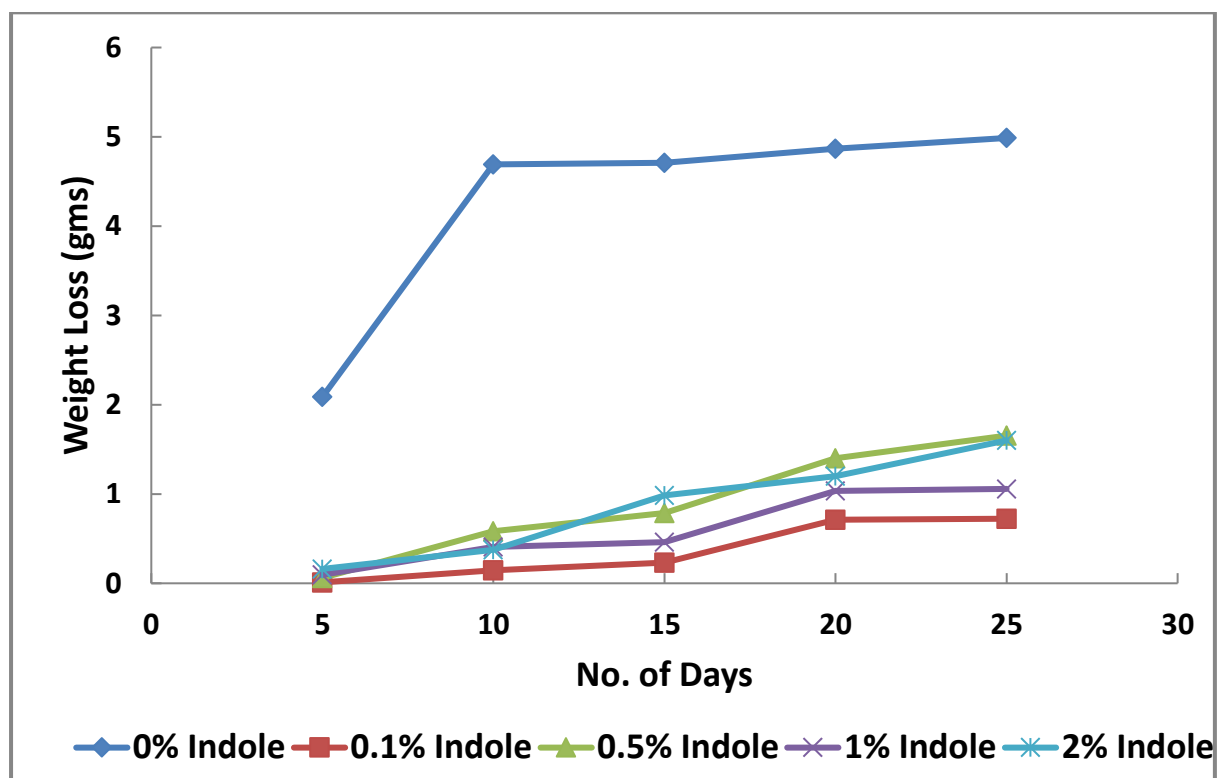
The corrosion rate and inhibition efficiency of mild steel exposed to blank, and in different wt. % of Indole is shown in Table 4.1.

**Table 4.1: Weight loss of MS, with and without different inhibitor solutions**

| No : of Days | Weight loss(g) of Mild Steel samples in HCl medium |             |             |           |           |
|--------------|--|-------------|-------------|-----------|-----------|
|              | HCl  | 0.1% Indole | 0.5% Indole | 1% Indole | 2% Indole |
| 5            | 2.087  | 0.011       | 0.061       | 0.095     | 0.161     |
| 10           | 4.693  | 0.144       | 0.408       | 0.582     | 0.374     |
| 15           | 4.71   | 0.231       | 0.461       | 0.787     | 0.986     |
| 20           | 4.867  | 0.713       | 1.034       | 1.199     | 1.401     |
| 25           | 4.987  | 0.723       | 1.057       | 1.6       | 1.654     |

It is observed that the rate of corrosion of mild steel vary with the concentration of Indole. This behavior is due to the increase in adsorption of Indole at the metal solution interface. The weight loss is found more when the metal sample is kept in blank (HCl) solution and is least in the case, when the solution contains Indole. It is found that as the concentration of Indole in the acid solution is at minimum %, the loss of weight due to corrosion is found to be decreased considerably. At the end of the day 25, the weight loss of steel sample in blank solution is about 4.987 gms, whereas in the case of 0.1% Indole, which is proved as a better inhibitor it is found to be 0.723 gms, which shows the effective

inhibition efficiency of Indole. The variation of weight loss of Mild steel is shown in Figure 4.1.



**Figure 4.1: Variation in weight loss of MS, with and without inhibitor**

The inhibition efficiency of different combinations is shown in the Table 4.2.

**Table 4.2: Kinetic parameters for mild steel in absence and presence of different combination inhibitions from weight loss measurements**

| Solutions          | Weight loss at the 25 <sup>th</sup> day<br>(grams) | Inhibition efficiency (%) at<br>the 25 <sup>th</sup> day |
|--------------------|--|--|
| BLANK(HCl)         | 5.194  | -  |
| 0.1% Indole in HCl | 4.198  | 85.50  |
| 0.5% Indole in HCl | 4.1  | 66.83  |
| 1% Indole in HCl   | 2.715  | 78.93  |
| 2% Indole in HCl   | 1.996  | 67.92  |

The inhibition efficiency of Indole is already proved and 0.1% Indole shows an efficiency of 85.50%. But the inhibitor solution containing 2% Indole in HCl shows an inhibition efficiency of only 67.92%. This indicates that, there is an optimum concentration for Indole to show efficiency to a greater extend.

#### **4.2 OPEN CIRCUIT POTENTIAL MEASUREMENTS**

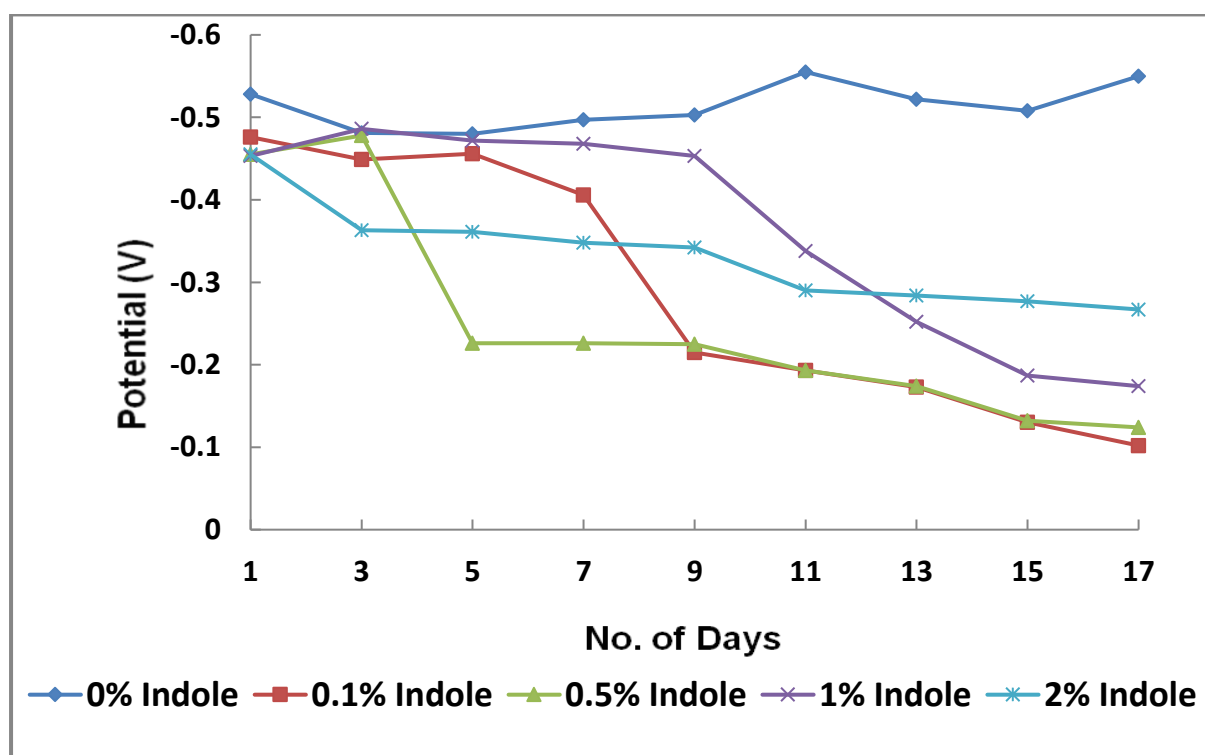
All the metal samples were prepared and inserted in appropriate inhibitor solutions. These plates are working electrodes and were immersed in different corrosive media for two week. Saturated calomel electrode is used as the reference electrode. The loss of water due to evaporation was compensated by addition of de ionized water twice daily. The solution was kept in stagnant condition. Table 4.3 shows the trend of OCP with different % of Indole.

**Table 4.3: Variation of OCP for mild steel immersed in blank and different concentration of inhibitor solution**

| Days | Open circuit potential of |             |             |           |           |
|------|---------------------------|-------------|-------------|-----------|-----------|
|      | HCl                       | 0.1% Indole | 0.5% Indole | 1% Indole | 2% Indole |
| 1    | -0.528                    | -0.476      | -0.455      | -0.453    | -0.455    |
| 3    | -0.481                    | -0.449      | -0.478      | -0.486    | -0.363    |
| 5    | -0.48                     | -0.456      | -0.226      | -0.472    | -0.361    |
| 7    | -0.497                    | -0.406      | -0.226      | -0.468    | -0.348    |
| 9    | -0.503                    | -0.215      | -0.225      | -0.453    | -0.342    |
| 11   | -0.555                    | -0.193      | -0.193      | -0.338    | -0.29     |
| 13   | -0.522                    | -0.173      | -0.174      | -0.252    | -0.284    |
| 15   | -0.508                    | -0.13       | -0.132      | -0.187    | -0.277    |
| 17   | -0.55                     | -0.102      | -0.124      | -0.174    | -0.267    |

The table contains the OCP data per two days. From the beginning, the value of blank solution is almost a constant but it changed very fast to more anodic values, after 7 days,

which means that, the strip dipped in blank solution is undergoing severe corrosion attack and the OCP value is -0.696. The metal strip dipped in Indole, which is already proved as a better inhibitor have an OCP value of -0.227V. The less the anodic values, more will be the efficiency of the inhibitor. So from the values it is proved that the concentrations having ascorbic acid in a considerable amount have greater inhibition efficiency. Figure 4.2 shows the potential–time curves of mild steel in 1.0 M HCl without and with various concentrations of inhibitors at room temperature.



**Figure 4.2: The variation of OCP for mild steel immersed in distilled water with different concentrations inhibitor solution**

The corrosion potential of steel electrode in 1M hydrochloric acid solution for the blank sample (blank curve) tends towards more negative value, giving rise to short steep. This behaviour corrosive behaviour of the MS in acid solution. Addition of the inhibitor molecules produce a positive shift in the potential but the shift towards anodic value is found to be more for lower % i.e., for 0.1% and 0.5% of the inhibitor. In fact, it is important to

mention that the addition of inhibitor at any % to the corrosive medium produces a positive shift in the open circuit potential due to the retardation of the corrosive nature.

### **4.3 VISUAL OBSERVATIONS**

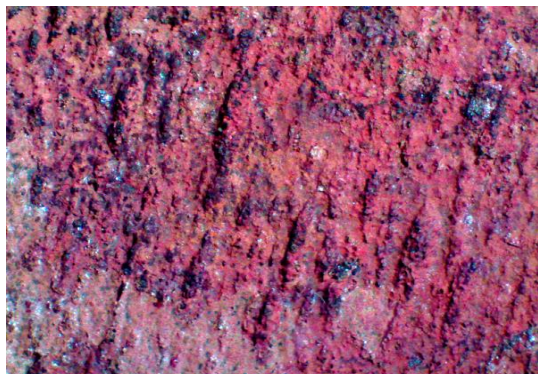
All these experiments were supported by the visual evaluation of electrodes as well as electrolyte. Visual examination shows that in the blank solution the systems show much corrosion. The electrolyte turns to yellowish brown color. The solution containing 0.1% Indole in HCl seems to be colorless but as the end of second week it seems to be little faded and a visible corrosion is appeared. Due to the action of Indole, an effective passive film formed on the surface of mild steel and prevents from corrosion. As a result steels with immersed in the solution containing 0.1% Indole, there is no visible corrosion or appearance of rust. The surface of the specimen which is not in contact with the inhibitor solution is found to be rusted. But the portion in contact with the inhibitor solution seems to be perfectly clean.

### **4.5 SURFACE ANALYSIS**

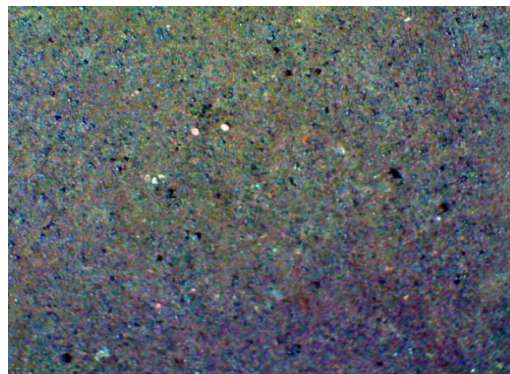
#### **4.5.1 Optical micrographic method**

The optical micrographs of the surface of polished mild steel (a), mild steel immersed in blank (b) and inhibitor solutions of varying concentrations(c) for the period of 25 days are represented in Figure 4.4. The surface of the polished mild steel surface is very clear and is free of any irregularities. In case of the mild steel after immersion in the HCl solution, the surface obtained is rough and consist of cracks as well as delaminated portions. This can be attributed to the occurrence of corrosion process in the blank solution. At the same time, the surface of the mild steel after immersion in the each concentration of inhibitor solutions, there is some surface changes occurs depending on the concentration of inhibitor. In the case of optimum inhibitor solution i.e., 0.1% Indole, the surface of the mild steel is almost in the same appearance as that observed in case of the polished mild steel surface. This is an

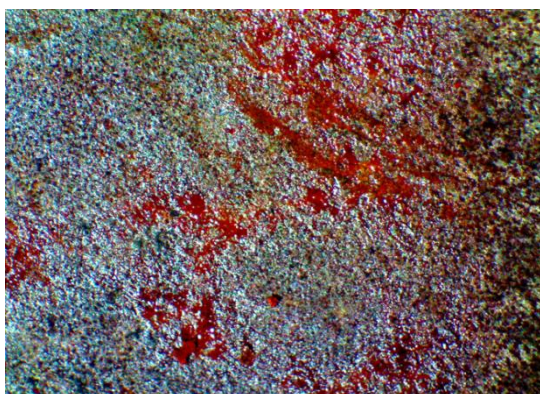
indication of the better inhibiting action of the inhibitor. That is the inhibitor leads to the formation of a passive film on the mild steel surface and it will protect the mild steel surface from corrosion.



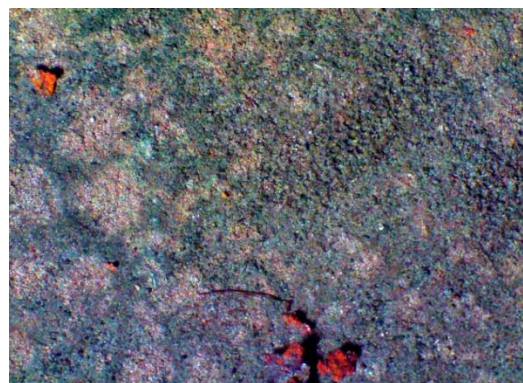
(a)



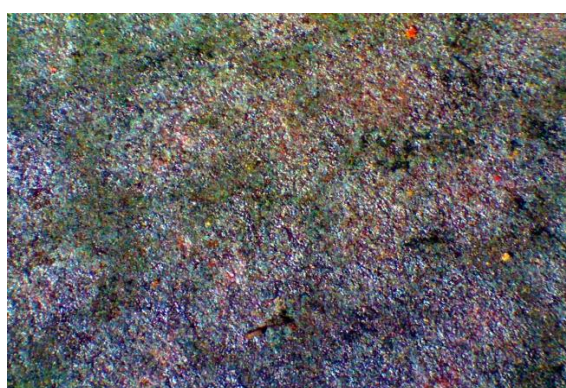
(b)



(c)



(d)



(e)

**Figure 4.4: The optical micrograph of mild steel dipped in different composition of inhibitor solution (a): Blank (HCl), (b): 0.1% Indole in HCl, (c): 0.5% PEG in HCl (d): 1% Indole in HCl (e): 2% Indole in HCl**

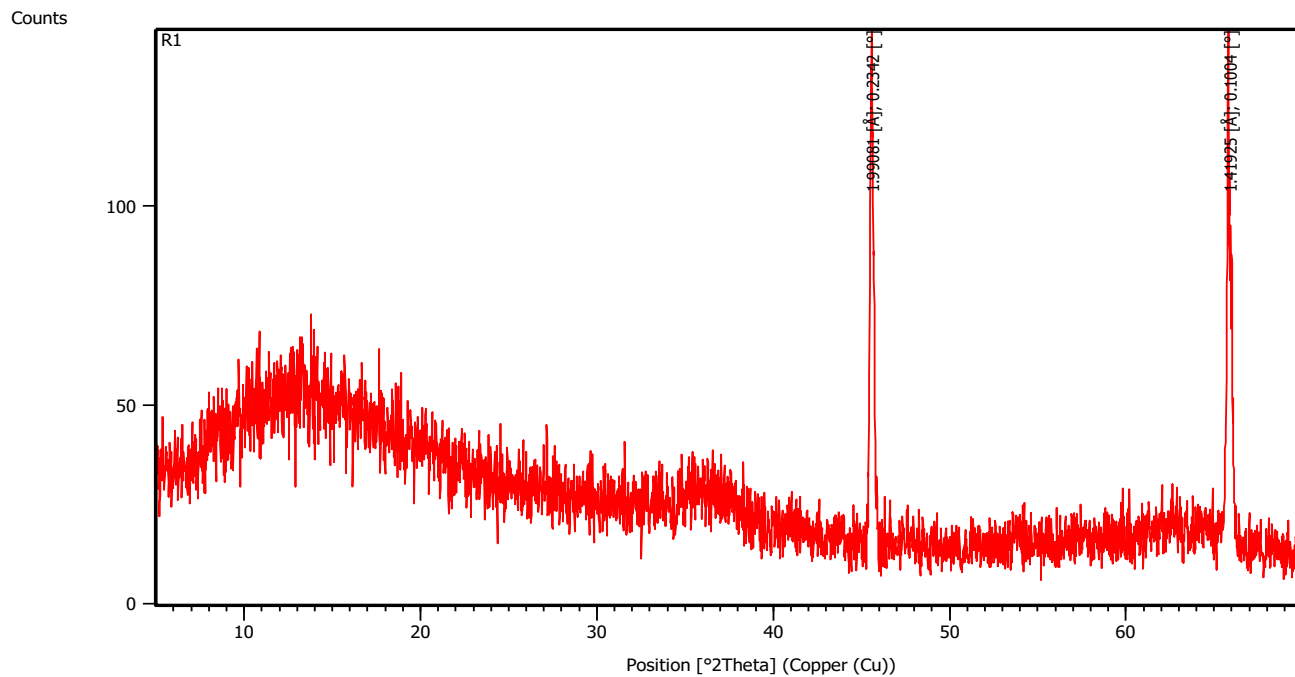


#### 4.5.2 XRD analysis

The XRD values of mild steel pattern immersed in optimum concentration is represented in Table 4.4.

**Table 4.5: Peak List from XRS analysis**

| Pos. [°2Th.] | Height [cts] | FWHM Left [°2Th.] | d-spacing [Å] | Rel. Int. [%] |
|--------------|--------------|-------------------|---------------|---------------|
| 45.5667      | 123.96       | 0.2342            | 1.99081       | 100.00        |
| 65.8036      | 121.61       | 0.1004            | 1.41925       | 98.11         |



**Figure 4.5: The XRD analysis of mild steel in optimum concentration**

The highest peak from Figure 4.5 is defined as the 100% peak and the intensity of all other peaks are measured as a percentage of the 100% peak. The intensity of the X-rays is measured on the y- axis, and increasing in values of 2Theta is shown on the x-axis. The X-rays used are of the Copper K-Alpha1 [Å], wavelength  $1.54060 \times 10^{-10}$  m, the scan is taken between 2Theta of  $10^0$  and 2Theta of  $70^0$  at a scan rate of  $1.2^0/\text{min}$ .

## **CHAPTER 5**

### **SUMMARY AND CONCLUSIONS**

#### **5.1 SUMMARY**

Mild steel is an alloy form of iron and is extensively used fabrication material in sugar, petrochemical, food, paper and textile industries which undergoes corrosion easily in acidic medium. The metals against corrosion are thus important. Modification of the environment of the metal will reduce corrosion. Use of corrosion inhibitors is a technique that is normally used for such systems. Many organic and inorganic inhibitors are used, which have high or low inhibition efficiencies. Some are toxic and are not environmental friendly. So a need for a better inhibitor became much essential, for protecting metals from corrosion. So in the present work Indole is used as inhibitor in acid medium. It was decided to evaluate the performance of the different % of Indole in acid medium. All the results have been discussed in Chapter 4. The inhibition efficiency of the inhibitor was evaluated by weight loss method, open circuit potential measurements and surface analysis.

#### **5.2 CONCLUSIONS**

Indole in optimum % i.e., 0.1% is a very effective corrosion inhibitor of mild steel in acidic medium. The inhibition efficiency do not increases with the increasing the % of inhibitor.

#### **5.3 SCOPE FOR FURTHER WORK**

The present work may be modified by using other low cost inhibitors like Ce (IV). Other organic co-inhibitors with ascorbic acid, may also improve its inhibition efficiency. The present inhibitor can be evaluated for other metals like aluminium, zinc, copper etc. and also in other media like coal water slurries etc. Multi component systems may also be studied based on this combination for other metals and in other environments. SEM studies should be carried out.

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