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CORROSION AND CORROSION INHIBITION OF MILD STEEL

A GRADUATION RESEARCH PROJECT

SUBMITTED TO THE DEPARTMENT OF CHEMISTRY IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE COMPLETION OF THE DEGREE OF BACHELOR OF SCIENCE IN CHEMISTRY

BY

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UNDER SUPERVISION

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DEDICATION

I DEDICATE THIS WORK
TO MY FATHER , MOTHER , TO MY SON , MY WIFE
, TO MY FELLOW STUDENTS AND TO ALL
WHO CONTRIBUTED TO THE COMPLETION
I ASK GOD YOU AND ME TO RECONCILE
AND REPAYMENT

ACKNOWLEDGEMENT

I WOULD LIKE TO THANK GOD

THEN
I THANK THE PRESIDENT OF THE DEPARTMENT

OF CHEMISTRY, DR. SAMI HUSSEIN

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THANK DR. KAMAL K. K. HAMZA FOR HIS EFFORTS AND TIPS

THANK MY FAMILY

MY FATHER AND MY MOTHER THANK MY WIFE AND SON

THANK YOU, BROTHERS AND STUDENTS AND EVERYONE.

I OFFER MY RESEARCH.

ABSTRACT

Abstract:

In this research the corrosion of carbon or mild steel was studied in acidic and basic media as well as the sea water. Some corrosion inhibitors (acetamide, diaminobenzene and chalcone) were added to these corroding solutions. The amount of iron dissolved in pure solutions and solutions containing inhibitors was determined via spectrophotometric techniques. The corrosion rates of the steel sheets were calculated using the appropriate equations. The corrosion inhibition of the inhibitors used were also calculated.

مستخلص البحث :

في هذا البحث تم دراسة تآكل الحديد في وسط حمضي و قاعدي و ماء البحر . تمت إضافة مثبطات للتآكل في هذه الأوساط والمحاليل وتم تحديد كمية الحديد المتآكل في كلا من المحاليل النقية والمحاليل المحتوية على مثبطات التآكل . وحسبت كل معاملات التآكل للحديد وتم حساب درجة كفاءة تثبيط التآكل لكل من المركبات .

1.0 Introduction

Most of metals have natural tendency to corrode to their oxides and hydroxides as a result of environmental factors such as oxygen, humidity and acidic gases. The following electrochemical equation explains this phenomenon.

Metal + Environment → Soluble product (oxides & hydroxides) + Energy

Taking mild steel as an example,

$$\text{Fe}^{+2} + 2e \rightarrow \text{Fe (s)} \quad E° = -0.44 \text{ V}$$

This reaction is nonspontaneous in this direction as it has negative potential ($\Delta G° > 0$). This indicates that iron is not stable as metal [Fe (s)] but as an oxidized form. On the other hand noble metals such as gold is stable as metal as can be seen from its reduction potential;
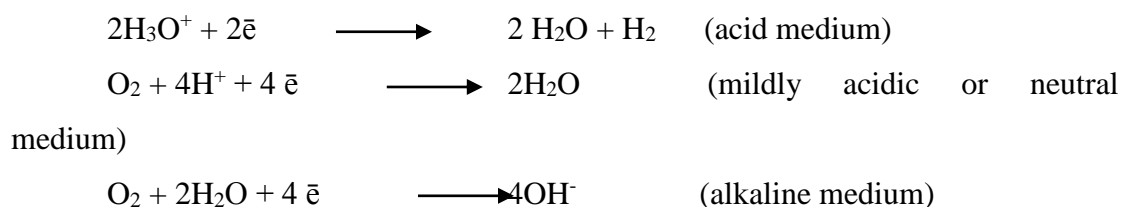
$$\text{Au}^{+3} + 3e \rightarrow \text{Au (s)} \quad E° = +1.34 \text{ V}$$

where the positive value of E° indicates a negative value free energy. This is why iron needs protection against corrosion.

2.0 Literature review

2.1 Corrosion – Significance and Importance

With the exception of noble metals, all other metals used in industries are unstable to varying degrees in the natural environmental conditions due to corrosion. Metallic structures in practice are generally exposed to corrosive environments. The loss due to corrosion is a serious engineering problem and the national economies have suffered great losses due to corrosion. Protection of metals and alloys from corrosion is therefore a subject of great technological and industrial importance and an enormous amount of research effort has been directed towards the study of the process of corrosion and the stability of the materials in different environments.

In practice the cathodic reactions that occur in aqueous solutions are reduction of either H_3O^+ or dissolved oxygen.

$$2H_3O^+ + 2\bar{e} \longrightarrow 2 H_2O + H_2 \quad \text{(acid medium)}$$

$$O_2 + 4H^+ + 4 \bar{e} \longrightarrow 2H_2O \quad \text{(mildly acidic or neutral medium)}$$

$$O_2 + 2H_2O + 4 \bar{e} \longrightarrow 4OH^- \quad \text{(alkaline medium)}$$

2.2 Thermodynamics of Corrosion

As a general rule metallic corrosion in aqueous medium is the resultant of two partial processes namely metal dissolution and hydrogen evolution i.e.,

$$M + nH^+ = M^{n+} + n / 2 H_2 \qquad (1.1)$$

According to thermodynamics, for the above process to be spontaneous

$$\Delta G = -nF\Delta E \qquad (1.2)$$

2.3 Kinetics of Corrosion

In corrosion process the rate is expressed in terms of current.

2.4 Types of corrosion

The main types of corrosion are: uniform corrosion, galvanic corrosion, pitting corrosion, parting corrosion, stress corrosion cracking, erosion corrosion·

2.5   Factors affecting corrosion

There are many mutually interdependent factors relating to both the metal and the medium which affect the corrosion process. However, for the sake of convenience they may be discussed under primary factors (relating to metal) and secondary factors (relating to the medium).

2.5.1   Primary Factors

a) Nature of the Metal

The tendency of a metal to corrode in an aqueous solution is related to its characteristic electrode potential as listed out in the "Electrochemical series". According to this series metals with more positive potentials are relatively stable and those with more negative potentials are unstable.

b)   Surface State of the Metal

A highly polished and smooth surface resists corrosion while a rough surface containing various types of imperfections such as cleavage steps, dislocations, point defects etc. is liable to severe attack.

c) Protective films

Certain metals like Al have a tendency to form protective films on the surface which act as physical barriers between the metal and the medium thereby curtailing corrosion.

2.5.2   Secondary Factors

a) pH

For metals like Zinc, Iron, Magnesium etc., hydrogen evolution is the thermodynamically favoured cathodic reaction. Corrosion of such metals in acidic medium is therefore highly pH dependent. A decrease in pH facilitates the rate of hydrogen evolution and hence increases the corrosion rate of metals.

b) Dissolved oxygen

Dissolved oxygen in the medium plays an important role when corrosion involves its reduction as the cathodic reaction

c) Effect of other ions present in solution

The presence of other ions usually Fe^{+3}, NO^-_3, Cl^-, SO_4^{-2} etc, in certain concentration ranges affect the rate of corrosion. For instance Cl^- ions interfere with

the formation of protective films on metals such as iron, aluminium and copper and cause extensive damage because of the specific activity of Cl^-. Similar effects with less severity are observed in the presence of SO_4^{-2}

e) Temperature of the medium

An increase in the temperature of the medium usually enhances the rate of corrosion process. This is especially significant in media in which corrosion is accompanied by hydrogen evolution.

2.6 Corrosion control

Corrosion control is achieved by several methods depending upon the type of corrosion and environmental conditions. Some methods commonly used to control corrosion are:

  i.   Cathodic protection
  ii. Anodic protection (Passivation)
  iii. Coatings (metallic, Organic, Inroganic, …etc)
  iv. Chemical control of the environment using inhibitors.

Of these the last method has been extensively investigated.

2.7 Types of Inhibitors

The use of inhibitors for corrosion prevention is a standard practice. An inhibitor is a chemical substance, organic or inorganic in nature added in traces to the corrosive medium in order to retard the corrosion rate. Corrosion inhibition by inhibitors can thus be defined as the treatment of a metal surface by the addition of low percentages of chemicals to the environment to control the corrosion process. Based on the mode of action, corrosion inhibitors are classified as

  (i)   Anodic inhibitors
  (ii)   Cathodic inhibitors and
 (iii) Mixed (adsorption) inhibitors.

Iron displaces hydrogen :

from hydrochloric acid to form pale green iron(II) chloride:

$$Fe(s) + 2\ HCl(aq)\ \ FeCl_2(aq) + H_2(g)$$
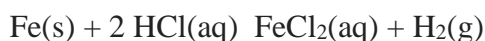
The chloride crystallizes as $FeCl_2 \cdot 4\ H_2O$. Exposure to air gradually oxidizes the iron(II) to $FeCl_3$ and Fe2O3. Oxidizing $FeCl_2$ with Cl_2 produces orange-yellow $FeCl_3$, which has metal-nonmetal bonds with covalent character.

- Iron corrosion reactions in NaOH :

Rust is oxidation, the addition of oxygen to a molecule.
$4Fe + 3O_2\ \ ---> 2Fe_2O_3$
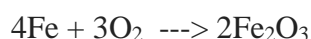
Sodium hydroxide is a base. It does not cause an oxidizing reaction.

$Fe + 2NaOH ---> 2Na + Fe(OH)_2$
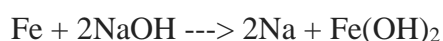
Iron corrosion reactions in seawater:

Corrosion of iron, and in general of metal in seawater is a function of the amount of oxygen: more oxygen more corrosion. This is due to the fact that in a corrosion process in water or seawater there are two basic reactions:

iron that corrodes, oxidation reactions:

$$2Fe\ \ ----> 2\ Fe^{2+} + 4e\text{-}$$
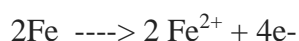
and the reduction of Oxygen

$$O_2 + 2H_2O + 4e\ \ \text{-} ------> 4\ OH\text{-}$$

the overall reaction is

$$2Fe + O_2 + 2H_2O ---> Fe(OH)_2 \text{ (rust)}$$

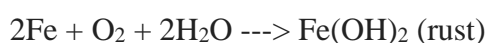as you can see the "rate" of the corrosion reaction is a function of the concentration of O2 in the seawater. Seawater is more corrosive than fresh water 'cause due to the presence of high quantity of NaCl has a greater electrical conductibility.

2.8 Corrosion rate measurement:

There are two main methods for the measurement of corrosion rate.

1. Weight loss methods:

Here the corrosion rate is measured by the amount of metal (mg) dissolved in a specific period of time. It can also be calculated as mm/year:

$$mm/year = \frac{87.6xW}{dxAxt} \qquad (2.1)$$

where:

W = weight loss (mg).

d = metal density (g.cm^{-3}).

A = metal sample surface area (cm^2).

t = immersion time (h).

2. Electrochemical methods:

In these methods the rate is expressed as corrosion current (i_{corr}). The i_{corr} can be obtained from the weight loss by the formula:

$$i_{corr} = \frac{nxFxW}{mXt} \qquad (2.2)$$

where:

n = number of electrons.

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F = Faraday's constant.

W = weight loss (g).

m = atomic mass of the metal.

t = time of immersion (s).

The addition of inhibitors reduces the rate of corrosion and consequently the inhibition efficiency is calculated as:

$$Inhibition\% = \frac{W_0 - W}{W_0} \qquad (2.3)$$

where:

W_0 = corrosion rate (mg/h) **without** inhibitor.

W = corrosion rate (mg/h) **with** inhibitor.

Also it can be calculated from the corrosion current values:

$$Inhibition\% = \frac{i_0 - i}{i_0} \qquad (2.4)$$

where:

i_0 = corrosion rate (mg/h) **without** inhibitor.

i = corrosion rate (mg/h) **with** inhibitor.

Mild steel is an important industrial metal used in various applications. Hydrochloric acid solutions are widely used for the pickling, cleaning, descaling and etching of mild steel [2].Various methods are followed to stop metals corrosion but the use of inhibitors is the most practical methods for preventing their corrosion, especially in acidic media [3]. Most of the well known acid inhibitors are organic compounds containing nitrogen, sulfur and oxygen atoms [4] Compounds containing heteoatoms like sulfur and nitrogen are considered to be very effective corrosion inhibitors [5].

The structure of two of the metal used are shown in Figs. (2.1, 2.2).
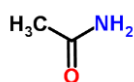
Fig. (2.1): Acetamide
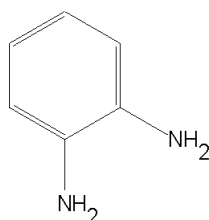
Fig. (2.2): Diamnobenzene

The inhibitive reactivity of an inhibitor is fundamentally affected by the molecular structure of the inhibiting molecules. Most prominent corrosion inhibitors are organic compounds containing nitrogen, sulphur, oxygen, and phosphorus in their functional groups [6]. The mechanism of these compounds has been proposed to be the adsorption, by means of lone pairs of electron, of the organic functional groups on the metal surfaces [7].

The research objective:

1. To study the corrosion of mild steel in acid, base and sea water.

2. To investigate the effect of inhibitors on the corrosion of mild steel in the different media.

3. To calculate the corrosion parameters from the experimental data.

4. To calculate the corrosion rate and inhibition efficiency of these inhibitors.

3.0 Experimental:

3.1 Chemical:

The chemicals HCl, NaOH, acetamide, diaminobenzene were from Loba India. Distilled water was used to make all solutions except sea water.

3.2 Corrosion studies:

To study the corrosion and corrosion inhibition, about 4.0 cm2 sheet of mild steel was cut. The sheet was immersed in 25.0 ml solution of pure HCl, NaOH and sea water without and with different concentration of inhibitor for specific time interval. Then the solution was kept in a plastic container for analysis.

3.3 Iron determination: [8]

Iron (II) reacts with 1,10-phenanthroline to give an orange-red complex that absorbs at 515 nm. The complex is stable at pH 2-9 for long time. Due to the tendency of Fe^{+2} to get spontaneously oxidized to Fe^{+3} as the electrochemical reaction has positive value:
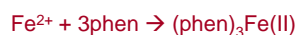
$$Fe^{+2} \rightarrow Fe^{+3} + e \qquad\qquad E° = 0.771 \text{ V.}$$

To avoid this reaction Fe^{+3} is reduced using hydroxylamine hydrochloride.

Tris(1,10-phenantholine)iron(II)

1,10-Phenanthroline

Fe^{2+} + 3phen → (phen)$_3$Fe(II)
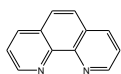
orange-red complex
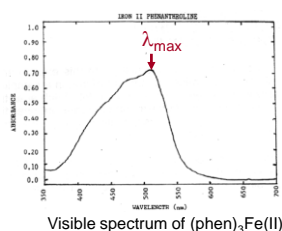
Visible spectrum of (phen)$_3$Fe(II)

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$$n\,Fe^{2+} \; + \; m\,phen = Fe_n(phen)_m^{2+} \; (orange\text{-}red)$$
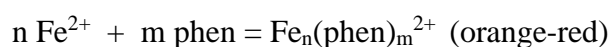
Apparatus

1. A spectrophotometer or a photometer.

2. Sample cells.

3. pH Meter.

Chemicals and Reagents

1. 0.1 M Acetate buffer, pH 4.0.

2. 0.5 M Hydroxylamine hydrochloride.

3. Fe(NH$_4$)$_2$ (SO$_4$)$_2$ 6H$_2$O standard solution (100 ppm).

4. 5x10^{-4} M 1,10- phenanthroline solution.

Procedure

1. About 5 mL of Fe^{2+} standard solution was added to 5 mL of the acetate solution into a 25 mL volumetric flask.

2. Exactly 2 mL of hydroxylamine hydrochloride solution was added followed by a 5 mL portion of the standard 1,10-phenanthrolyine solution.

3. The volume was adjusted to the mark using distilled water and allow to stand for 10 min.

4. The blank was placed in one of the absorption cells provided and adjust the absorbance reading to zero at 515 nm.

5. The previous steps were followed to determine the

7. The results were plotted on an x-y graph with absorbance on the y axis and wavelength on the x axis and draw the absorption spectrum.

The concentration of Fe+2 in the samples was calculated using Beer's law:

$$A = \varepsilon bC$$

where:

A = absorbance.

ε = absorbtivity coefficient or molar absorption coefficient $(M^{-1}.cm^{-1})$.

b = path length or cell length (cm).

C = analyte concentration (M).

4.0 Results and Discussion:

The standard calibration curve of the iron absorption is shown in figure (1).
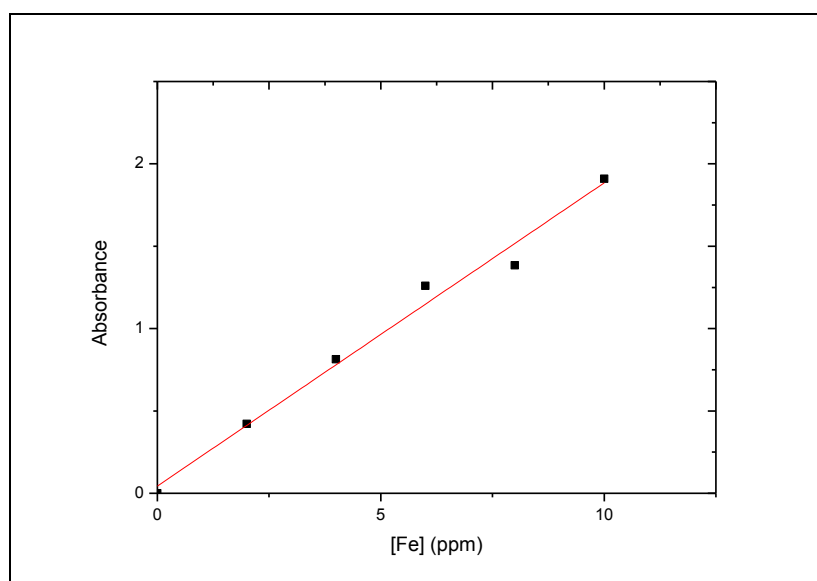

Fig. (3.1): Standard calibration curve for iron absorption

The curve was used to obtain the concentration of iron in the samples. The amount of iron dissolved was calculated using the formula:

$$\text{Amount of Fe (mg)} = C \times V \times D_f$$

where:

c = concentration $(mg.L^{-1})$

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V = volume of sample (L)

D_f = dilution factor

The amount of iron corroding in pure 2.0 M HCl, 2.0 M NaOH and sea water without and with inhibitors is shown on table (1). The amount of iron dissolved was found to decrease with the addition of inhibitors compared with pure HCl. The amount of iron dissolved at different concentrations of acetamide was found to increase as the concentration was decreased. Similarly the corrosion inhibition efficiency reduce with the acetamide solution dilution. The inhibition efficiency of iron in chalcone solutions was high even at lower concentration. This indicates that the chalcone is an excellent inhibitor. The diaminobenzene also shows less weight loss and high percentage inhibition, indicating that its ability to protect steel in acid medium is high.

Table (1): Weight loss and percentage corrosion inhibition of iron in the different solutions

Solution	Fe (mg\h)	% Inhibition
HCl pure	53.0	
1 M AcM	3.14	94.07
0.75 M AcM	5.02	90.53
0.50 M AcM	10.26	80.64
0.25 M AcM	12.56	76.30
0.59 M Chal	0.094	99.82
0.25 M Chal	0.094	99.82
0.125 M Chal	0.120	99.77
0.25 M Di-Ph	0.612	98.84
0.01 M Di-Ph	0.361	99.31
NaOH pure	70.16	
1M AcM	3.56	94.92
0.75 M AcM	4.39	93.74
0.50 M AcM	4.81	93.14
0.25 M AcM	23.30	66.79
0.25 M Di-Ph	0.19	99.72
0.05 M Di-Ph	0.04	99.94
Sea Water	5.55	
1 M AcM	1.085	84.47
0.50 M AcM	4.871	11.34
0.25 M AcM	7.204	-129.8

The table also shows the weight loss and the percentage inhibition of the compounds under study in 2.0 M NaOH. The amount of iron dissolved was found to decrease with the addition of inhibitors compared with pure NaOH. The amount of iron dissolved at different concentrations of acetamide was found to increase as the concentration was decreased. Similarly the corrosion inhibition efficiency reduce with the acetamide solution dilution. The diaminobenzene also shows less weight loss and high percentage inhibition, indicating that its ability to protect steel in basic medium is high.

In sea water only acetamide was used as inhibitor. The corrosion inhibition was less when the acetamide concentration was high. At lower acetamide concentration the weight loss increased and there was no inhibition but the iron corroded more.

The corrosion rate in mm/year is tabulated in table (2). The corrosion rate was found to decrease when inhibitors were added. The corrosion rate was lower at high acetamide concentration and increase when the concentration is less. Similar results are observed for the other inhibitors.

Table (2): Corrosion rate in (mm/year) for iron in different solutions

Solution	Fe (ppm) mg\h	Corrosion Rate (mm\Year)
HCl pure	53.0	73.93
1 M AcM	3.14	4.38
0.75 M AcM	5.02	7.01
0.50 M AcM	10.26	14.31
0.59 M Chal	0.094	0.13
0.25 M Chal	0.094	0.13
0.125 M Chal	0.120	0.16
0.25 M Di-Ph	0.612	0.85
0.01 M Di-Ph	0.361	0.50
0.25 M AcM	12.56	17.52
NaOH pure	70.16	48.93
1M AcM	3.56	2.48
0.75 M AcM	4.39	3.06
0.50 M AcM	4.81	3.35
0.25 M AcM	23.3	16.25
0.25 M Di-Ph	0.19	0.26
0.05 M Di-Ph	0.04	0.05
Sea Water	5.55	38.7
1 M AcM	1.085	7.56
0.50 M AcM	4.871	48.26
0.25 M AcM	7.204	112.46

In table (2), the corrosion current, corrosion current density and percentage inhibition of iron corrosion in pure corroding solutions and solutions containing inhibitors are shown. The values were obtained using equations2.2, 2.3 and 2.4. Corrosion current results from the process of corrosion and therefore increase with the extent of corrosion. From the table we can see that the corrosion current and current density are highest when iron is immersed in pure HCL, NaOH or sea water. This is an indication of the high corrosiveness of these solutions. The addition of inhibitors had greatly reduced the corrosion current and current density. This is an indication of the inhibition action of these compounds. The percentage inhibition calculated using the corrosion current values were almost equal to those obtained from the weight loss measurements. The corrosion current and current density at 0.25 M acetamide in sea

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water is higher than that of pure sea water. This is an indication of the corrosion increase at such lower concentration.

Table (3): Corrosion current and current density for iron in different solutions

Solution	I_corr (A)	% Inhibition	j(A\cm²)
HCl pure	0.0507		0.0063
1 M AcM	0.0030	94.07	0.0003
0.75 M AcM	0.0048	90.53	0.0006
0.50 M AcM	0.0098	80.64	0.0012
0.25 M AcM	0.0120	76.30	0.0015
0.59 M Chal	0.00004	99.82	0.000005
0.25 M Chal	0.00004	99.82	0.000005
0.125 M Chal	0.00006	99.77	0.000008
0.25 M Di-Ph	0.00033	98.84	0.000041
0.01 M Di-Ph	0.00019	99.31	0.000023
NaOH pure	0.0671		0.0083
1M AcM	0.0034	94.92	0.0004
0.75 M AcM	0.0042	93.74	0.0005
0.50 M AcM	0.0046	93.14	0.0005
0.25 M AcM	0.0223	66.79	0.0027
0.25 M Di-Ph		99.72	
0.05 M Di-Ph		99.94	
Sea Water	0.053		0.0060
1 M AcM	0.0103	84.47	0.0012
0.50 M AcM	0.047	11.34	0.0082
0.25 M AcM	0.069	-129.8	0.0192

From the data obtained for the corrosion of iron using the acetamide, diaminobenzene and chalcone we can see that they reduce the rate of iron corrosion in 2.0 M HCl and 2.0 M NaOH. This may be explained by the adsorption of these compound on the surface of iron. The lone pair of electron in the structure of these compounds can may them attracted to the metal surface by electrostatic forces [3, 6]. Therefore these compounds can make protective films that isolates the metal surface from the corrosion medium.

The less inhibition efficiency shown by solutions of acetamide in sea water may be due to the presence of aggressive ions such as chlorides (Cl⁻) and sulfates (SO_4^{-2}) that can eliminate such protective films formed. This has been reported in literature [2].

5.0 Conclusions:

1. The corrosion of iron (mild steel) in 2.0 M HCl, 2.0 M NaOH and sea water was investigated.

2. The concentration of iron was determined using spectrophotometric methods employing 1,10 phenanthroline as complexing agent with Fe^{+2} ions.

3. The compounds acetamide, diamnobenzene and chalcone were added to inhibit the corrosion process.

4. The weight loss, corrosion rate, inhibition efficiency, corrosion current and corrosion density were obtained.

5. The results showed that the compounds used could reduce the corrosion of iron in the different solution.

6. The inhibition was attributed to the presence of lone pairs of electrons in the structures of the compounds used. These lone pairs make the compounds adsorb on the metal surface forming protective films.

7. The corrosion inhibition of acetamide for iron in seawater was less compared to other solutions. The presence of aggressive ions like chloride and sulfate may have damaged protective films formed on iron surface.

6.0 Recommendations:

1. Study the corrosion of other metals in these solutions.

2. Use more techniques such as electrochemical   polarization. Also the use of imaging techniques to study   the structures of metals surfaces.

7.0 Acknowledgement:

8.0 References:

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	سيـــــرة ذاتيـــــة

بيانات شخصية

الاســـــم : عبدالكريم خليف العنزي
العنـــــوان : الرياض ١ حي الندوة
رقم الجوال : ٠٥٩٩٩٨٠٨٣٩
تاريخ الميلاد : ١٤١٥\٧\١٤
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تقـدير التخــرج : جيد

اللغات والمهارات

- **الحاسب الالي-:**
  التعـامل مع برامـج الحـاسب الألى (Word - Excel - Power point)

- **اللغات-:**
  اللغة العربية ـــ إجادة تامة (قراءة + كتابة + تحدث).
  اللغة الانجليزية ـــ إجادة متوسطة (قراءة + كتابة + تحدث).

الخبرات العلمية

- شهادة حاسب آلي ثلاث شهور
- خبرة ٧ شهور في مجال خزانات المياة
- خبرة سنة و شهرين في مصنع للبلوك والخرسانة
- خبرة الى الان ٦ شهور في شركة ادوية واجهزة ومستلزمات طبية

الخبرات المهنية

الوظيفة الحالية :
موظف اداري وميداني في الشركة المتحدة الاولى للخدمات الطبية المحدودة
وظيفة سابقة (١)
مشرف في شركة امال للبلوك والخرسانة من ١٥ \ ٤ \ ١٣٣٤ الى ١ \ ٨ \ ١٤٣٦
وظيفة سابقة (٢)
مندوب في شركة المهيدب لخزانات المياة من ٩ \ ٧ \ ١٤٣٣ الى ١ \ ٢ \ ١٤٣٤