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Corrosion Inhibition of Low – Carbon Steel in aqueous Phosphoric acid Solution Using Poly acryl amide

A Thesis Submitted to the Council of the College of Science,
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by

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DEDICATION

*To the one whom God sent as a mercy to the worlds,
Muhammad (peace be upon him)*

*To the one who charted for me the path of my life and taught
me to follow in the footsteps of the beloved Prophet, peace and
blessings be upon him, my father, may God have mercy on him*

To the joy of home and the joy of the eye my dear mother

*To my beloved husband who was my support in the labor and
birth of this work*

*To everyone who gave me help and help from brothers and
friends*

I dedicate my humble effort .

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RASHA.....

ABSTRACT

The rates of the low carbon steel corrosion in (2M H₃PO₄) acid were studied at different temperatures of (300, 400, 500, and 600) °C with the presence and absence of the polymeric inhibitor (*Polyacrylamide*) as a corrosion inhibitor using the weight loss method and the electro polarization method. It was found that the corrosion inhibition efficiency increases with increasing the concentration of the inhibitor (PAM) and decreased with the temperature, where the maximum efficiency of the inhibitor efficiency was 91.02% at the concentration of (600 ppm) and the temperature 30°C. PAM adsorption was studied on the surface of the mild steel according to Langmuir adsorption isothermal. The value of the adsorption of free energy was negative and less than 20 kJ/mol, which indicates that the adsorption process was spontaneous and physical. The (equilibrium constant) k_{ads} values rise with increase of PAM concentration, indicating excellent surface adsorption. Negative(heat of adsorption) ΔG_{ads} values imply spontaneous adsorption of inhibitors onto metal surfaces. G_{ads} and ΔH_{ads} values were within the physical adsorption range. The activation energy was lower in the absence of the inhibitory material (PAM) and this increase in (ΔE_a) is attributed to the formation of a protective layer positive. The activation entropy, indicating that the system is becoming less organized.

The results of show that the values of resistance polarization (Rct) and solution polarization (Rs) are increased by adding 600 ppm of PAM. These results are attributed to the increase in the thickness of the protective layer on the steel surface.

In addition, the inhibition efficiency was measured by to the impedance measurements (% IE_{EIS}). By comparing the results with the result of the electrochemical polarization it was found that the adsorbed PAM particles remain stable on the surface of the mild steel even after five hours of immersion in the corrosive solution which shows the variation in the efficiency of PAM over time in the presence of a PAM inhibitor

The FTIR and UV-VIS tests revealed also the formation of a protective film on a metal surface.

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List of Abbreviation

Symbol	Definition
H₃PO₄	Phosphoric Acid
OES	Optical emission spectrometer
FTIR	Fourier transform infrared spectroscopy
EIS	Electrochemical impedance spectroscopy
PAM	Polyacrylamide
C ₆ H ₆	Benzene
C ₆ H ₆ O	Acetone
PDP	Potential dynamic polarization
OCP	Open circuit potentials

Icon List

units	Icon	full phrases
K	T	Absolute temperature
kJ/mol	E_a	Activation energy
$moleculumol^{-1}$	N	Avogadro number
-	R^2	Correlation coefficient
$g/m^2 d$	CR	Corrosion rate
$g/m^2 d$	CR_{inhib}	Corrosion rate of inhibited
$gm/m^2 d$	$CR_{uninhib}$	Corrosion rate of uninhibited acid
kJ/mol	ΔH^*	Enthalpy change
$kJ/mol.K$	ΔS^*	Entropy change
(L/ml)	K_{ads}	Equilibrium adsorption constant
$days$	t	Exposure time
-	A	Frequency factor
$J/mole.K$	R	Gas constant
ml/L	C_i	Inhibitor concentration
g/mol	MW	Molecular weight
$J.S$	h	Plank constant
-	n''	Slope
kJ/mol	ΔG°_{ads}	Standard adsorption free energy
V	E°	Standard electrode potential
m^2	A	Surface area
-	θ	Surface coverage
-	S_{θ}	Synergistic effect parameter
g	ΔW	Weight loss



Chapter One

Introduction & Literature Survey

1.1 Introduction

Corrosion is a chemical or electrochemical degradation of metals and alloys. It is a natural phenomenon that cannot be stopped, but can be prevented and avoided by adequate preventive techniques such as metallic coating, anodic and cathodic protections[1] . There are several factors that influence the rate and nature of corrosion reactions, including the material, the environment, and the temperature[2]. Corrosion is a major issue in the long-term longevity of metallic materials and buildings. Many attempts have been made to create a corrosion inhibition method that would extend the life of existing buildings and reduce corrosion damage [3]. Steel alloys are one of the most common materials used in industrial purposes. It can come into contact with a range of conditions, including acidic solutions during pickling and acid cleaning[4].

corrosion inhibitor is a chemical that, when added to a tiny amount of witer, effectively inhibits the corrosion of a metal exposed to that water[5]. The first stage in the adsorption of natural or chemical inhibitor molecules on a metal surface is usually the replacement of water molecules deposited there. [6]. Corrosion inhibitors may be chemical or natural substances. The inhibitors may be categorized based on inhibitory factors such as the chemical type of the inhibitors, their mechanism of action, and their mode of action.

Corrosion products, when in contact with rust (the first corrosion product), create an impenetrable layer on the metal surface.

[7]. Low-carbon steel is a metal with a broad variety of mechanical and industrial applications all over the globe. Due to its availability and mechanical properties, it is one of the most preferred construction materials for industries; however, it is highly susceptible to corrosion and poses a high economic burden to replace almost all of the environments in which metals are used serve as a potential hostile and aggressive medium to them. The effectiveness of their use is dependent on the protective mechanisms against corrosion. Corrosion occurs when defensive mechanisms are ignored or depleted, leaving the metal exposed to assault [8].

Economic, safety, and environmental harm are the three major reasons for the significance of corrosion prevention. It is the goal of corrosion engineers, together with the assistance of corrosion scientists, to reduce the amount of material lost as well as the resulting economic losses due to corrosion in various places including pipelines, storage tanks.

During the production and fabrication process, corrosion not only is the metal wasted, but so is the energy and human work that went into it.

For most of today's corrosion research, the economic element is a major driving force. Industry and government losses total hundreds of billions of dollars per year.

Corrosion, on the other hand, has a huge impact on the environment, since corrosion-related collapse of oil pipelines, gas pipelines, or oil tankers may have disastrous consequences for the environment in the form of water and air pollution.[9]

Limited researches are taking into consideration the using of polymeric corrosion inhibitors in acidic solutions, especially, H_3PO_4 .

1.2- literature survey

De Souza FS (2009) studied The inhibitory effect of naturally occurring biological molecule caffeic acid on mild steel corrosion in 0.1 M H_2SO_4 using weight loss, potentiodynamic polarization, electrochemical impedance, and Raman spectroscopy. Several techniques were used to verify the adsorption of caffeic acid onto the mild steel surface and the subsequent inhibition of the corrosion process. In order to reduce the available cathodic reaction area and change the activation energy of anodic reactions, caffeine acid acts [10].

Sherine et al. (2010) studied the weight loss technique to investigate the inhibition efficiency of inhibitor systems containing different combinations of hydroquinone and zinc ions (Zn^{+2}) in preventing corrosion of carbon steel submerged in well water. The inhibitory effectiveness of 100 ppm zinc was determined to be 50% (immersion time = 7 days). The IE rises to 97 percent when 150 ppm of hydroquinone is introduced. The nature of the protective layer formed on the metal surface has been studied using FTIR spectra, SEM, and AFM [11].

Deng et al. (2011) studied weight loss and potentiodynamic polarisation curves to investigate the inhibitory impact of sodium molybdate, (Na_2MoO_4) on aluminum corrosion in 1.0 M H_3PO_4 solution. The findings indicate that Na_2MoO_4 is a good inhibitor, with three techniques yielding inhibition efficiencies of more than 84 percent at 20 mM. At lower concentration (1–7 mM), the adsorption of Na_2MoO_4 follows the Freundlich isotherm, whereas at higher concentrations (7–20 mM), it follows the Langmuir isotherm. Na_2MoO_4 works as an anodic inhibitor, according to polarisation curves [12].

Chidiebere et al. (2014) studied electrochemical methods to evaluate the inhibitory characteristics of corrosion of Q235 mild steel in 0.5 M H_2SO_4 with phytic acid (PA). Researchers discovered that PA is both an anodic and cathodic inhibitor in 0.5 M H_2SO_4 . Synergy with KI had values higher than unity, which meant that suggesting that the two chemicals work together to enhance inhibitory performance. The Langmuir adsorption isotherm was followed by PA adsorption. On the basis of the trend of inhibitory efficiency with temperature and estimated activation energy values (E_a), a mechanism for chemical adsorption is suggested [13].

He X et al. (2014) studied The inhibitory behavior of imidazole (IM) and 2-phenyl-2-imidazoline (2-PI) for AA5052 was studied with weight loss, electrochemical measurements, contact angle measurements, and scanning electron microscopy. The findings indicate that both IM and 2-PI can prevent AA5052 corrosion, with 2-PI having a greater inhibition effectiveness. E_a and θ are used to calculate their adsorption characteristics. The findings show that the adsorption reactions are exothermic and follow the Langmuir adsorption isotherm, owing to use a monolayer chemisorption mechanism. On the AA5052 surface, inhibitory mechanisms are proposed to account for the differences in monolayer adsorption structure between IM and 2-PI [14].

khadom AA (2015) studied naphthylamine (NA), KI, and the synergism of KI and NA in inhibiting mild steel corrosion in HCl solution investigated at various temperatures using weight loss and electrochemical methods. The NA–KI mixture is a mixed-type inhibitor, according to electrochemical analysis. The effectiveness of inhibition improves as inhibitor concentration rises, but declines as temperature rises. An inhibitory mechanism that is both stable and spontaneous was found to follow the Freundlich isotherm with negative G_{0ads} values. When the concentration was high and the temperature was low, the blend efficiency was 95 percent [15].

Deepa et al.(2015) studied In the absence and presence of Zn^{+2} , the inhibitory efficiency (IE) of Trisodium Citrate (TSC) in reducing corrosion of carbon steel in RO water. It was assessed using a weight-loss technique. The IE of the formulation with 150 ppm TSC and 10 ppm Zn^{+2} is 72.5 percent. With the addition of Zn^{+2} ion, the inhibitory effectiveness of TSC is observed to improve. The carbon steel surface is protected by an inhibitive layer, according to polarization and characterisation surface analysis investigations. Based on the findings of the previous research, a plausible mechanism for corrosion inhibition has been suggested[16].

Kwolek et al. (2016) studied investigated the corrosion rates of aluminum in a mixture of phosphoric acid and sodium molybdate. The inhibitions were computed. Na_2MoO_4 offered the highest efficient corrosion inhibition for 0.5 M H_3PO_4 . Since insoluble corrosion products were found on the specimens, morphology of the specimens was examined using a scanning electron microscope[17].

Hassan KH et al. (2016) studied Citrus aurantium leaf extracts as a friendly inhibitor to study mild steel inhibition in 1 M H_2SO_4 . Weight loss methods were used to examine the effects of temperature, time, and inhibitor concentration . Citrus aurantium leaf extracts inhibited mild steel corrosion and reduced the corrosion rate in H_2SO_4 . The inhibition effectiveness rises with increasing . Temperature has no effect on inhibitor concentration. At 40 °C and 10 ml/l inhibitor concentration, the inhibition efficacy was 89% higher. Analyses of the adsorption of Citrus aurantium leaf extract using the Langmuir adsorption isotherm model. the adsorption free energy for charged molecules with a charged metal was around -20 kJ/mol We also utilized quantum chemical simulations to support the results of the research An electron microscope and Fourier transform infrared spectroscopy, respectively, were employed to examine the surface morphology and molecular structure of the inhibited molecules [18].

Hu K, Zhuang J et al.(2017) studied the to protect of X80 carbon steel from the green corrosive, DNA was utilized as a green corrosion inhibitor. The biological macromolecule DNA was then used to protect X80 carbon steel from the green corrosive. Compound DNA had a maximum inhibitory effectiveness of 91.9 percent. One-molecule-layer self-assembly on carbon steel resulted in chemisorption of DNA inhibitor. The theoretical simulation confirmed the connection between the DNA molecular structure and corrosion inhibition. Further, the DNA was shown to be a potential environment-friendly inhibitor, as the findings showed[19].

Abd El-Lateef HM et al.(2017) studied three new Schiff base compounds were produced and spectroscopically analyzed (FT-IR, ^1H NMR and ^{13}C NMR). They conductometrically and by surface tension measured the crucial micelle concentrations. Researchers looked at the ability to prevent corrosion on mild steel with different produced surfactants in H_2SO_4 . All surfactants prevent mild steel corrosion with an efficacy ranging from 99.21% at 1.0 mM. The investigated surfactants were mixed-type inhibitors. Substrate adsorption obeyed the Langmuir isotherm. the theoretical predictions support our experimental investigations[20].

Mu et al. (2017) studied Corrosion inhibitor for carbon steel in 8% amidosulfuric acid solution using 2-undecyl-N-carboxymethyl-Nhydroxyethyl imidazoline (UHCI) and potassium iodide (KI). The combination inhibitor significantly prevents the corrosion of 20 carbon. provide The mass to volume ratio is 9:1.s the optimum corrosion prevention for a total concentration of 0.4% steel in amidosulfuric acid solution. When a combination inhibitor is added to the blank solution, Carbon steel electrodes' impedance spectra change from having one time constant to having two. It is a chemisorption method that uses a combination based on the Langmuir adsorption isotherm[21].

Khadom AA et al. (2018) studied Mild steel corrosion resistance in 1 M HCl and the effects of potassium iodide were examined both in the absence of and presence of (KI). Temperature and inhibitor concentration were investigated using weight loss method. KI inhibited mild steel in HCl, decreasing the rate of corrosion. With a rise in both inhibitor concentration and ambient temperature, inhibition performance increased. 94% inhibition effectiveness was achieved at increased inhibitor dose and temperature. KI was shown to have preferentially co-adsorbed with Langmuir adsorption isotherm. Adsorption enthalpy is in the range of -20 to -40 kJ/mol, indicating mixed physical and chemical adsorption. Corrosion rate data was linked to other variables using different models[22].

Beniken M et al. (2018) studied the electrochemical impedance spectroscopy, the researchers used potentiodynamic polar and the effect of a polyacrylamide PA's adsorption and thermodynamic properties on corrosion prevention in a 1.0 M HCl solution was studied. Furthermore, temperature As well as immersion duration, researchers looked into the inhibitory efficacy at 3×10^{-6} mol L⁻¹ of PA. There were six linearized adsorption isotherms used to model the adsorption data, including Langmuir, Flory–Huggins, Temkin, and Freundlich. It was decided against using Langmuir's model since it had the greatest regression coefficient and slope. To put it another way, one PA molecule was used to replace three water molecules. As a third observation, we found that repulsive contacts existed in the adsorbed inhibitory layer, suggesting that the adsorbed inhibitory layer had lateral interactions. The charge transfer resistance and inhibitory efficiency were both substantially enhanced after six hours of use. thermodynamic (K_{ads} , ΔG_{ads}) and kinetic parameters (A , E_a) were factored in[23].

Ismael MH et al .(2019) studied shon in his research that the mild steel corrosion rate changes with temperature and inhibitor concentration in H_3PO_4 acid.As a corrosion inhibitor, potassium iodide was utilized. The corrosion rate data was evaluated using the weight loss method. The findings were interpreted using mathematical and statistical techniques. Descriptive statistical studies revealed that the data distribution deviates from normal distribution, indicating that the majority of corrosion rate data was distributed at greater inhibitor concentrations and lower temperatures, which is the preferred situation in the pickling process. Three mathematical models with strong correlation coefficients were proposed to describe the data[24].

Ahmed et al .(2019) studied weight loss measurements to investigate. six synthesized heterocyclic compounds' ability to prevent mild steel from corroding in 0.5 M hydrochloric acid The inhibition efficiency was more than 95%. The development of protective adsorption coatings on the steel surface was credited with the outstanding inhibitor effectiveness. Fourier transform infrared and nuclear magnetic resonance analyses were used to confirm the structures of the compounds. The Langmuir adsorption isotherm was followed by inhibitor adsorption on steel surfaces. To further understand the inhibitory mechanism, quantum chemical simulations were used[25]

Fadhil AA et al .(2020) studied the inhibitory effect of the *Portulaca grandiflora* leaf (PGL) extract on N80 carbon steel corrosion at different temperatures and inhibitor concentrations under 0.5 M HCl acidic conditions.. PGL's performance was evaluated via weight loss and electrochemical techniques such as polarization measurement, open circuit potential, and electrochemical impedance spectroscopy. Fourier transform infrared spectroscopy, ultraviolet spectra, and surface morphology results all confirm the corrosion rates. When tested in acidic circumstances at 60°C and inhibitor concentrations of 20 mL/L, the inhibitor effectively prevents carbon steel corrosion, with an inhibition efficacy of 95% at the highest

temperature and concentration. The similar pattern of PGL adsorption on the steel surface was seen using the Langmuir isotherm model. While adsorption on the metal surface is still in the negative energy range, an increase in the heat-of-adsorption value from negative to positive is indicative of an increase in heat-of-adsorption on the metal surface [26].

Rashid KH et al .(2020) studied polarization method to determine the corrosion inhibition effect of kiwi juice extract (KJE) in 2.5 M hydrochloric acid. At 45°C and 55 cm³/L concentration of inhibitor, the highest inhibition effectiveness was 87%, while the lowest was 81% at the same temperature and inhibitor concentration. The cathode is more polarized than the anode. Depending on the overvoltage data, the polarization curve curvature area near the possibility of corrosion may be studied. A mathematical model may be used to examine the effect of diffusion on the charge transfer process. The findings from electrochemical method and mathematical model are well-matched[27].

Kumar et al.(2017) studied the CA and its derivatives 6-OH-CA and 7-Me-CA were synthesized and examined for suppression of Low Alloy Steel corrosion in 1 M H₂SO₄ at various concentrations and temperatures utilizing Gravimetric, Polarization method, EIS, AFM, DFT and MD calculations. Inhibition efficiency improved with increasing CA compound concentration as measured by mass loss, PDP, and EIS. These chemicals are mixed type inhibitors, and the Langmuir adsorption isotherm fits well for adsorption. 7-Me-CA (88.00%) > CA (96.37%) > 6-OH-CA (96.77 percent). The hypothesized inhibitory mechanism is supported by all experimental and theoretical methodologies(28).

Chamovska et al. (2007) studied the Polyacrylamide (PAA) with number average molecular weight, Mn, between 15,000 - 1,350,000 g mol⁻¹ on mild steel and iron (99.99 percent Fe) in 3 M HCl at room temperature was tested by spectrophotometry (phenanthroline technique), weight loss method, and EIS (Electrochemical Impedance Spectroscopy). The corrosion protection efficacy of PAA-adsorbed

layers is substantially dependent on the molar concentration and molecular weight of PAA in the solution, with limiting values between 85 and 96%. It was also found that PAA had excellent adsorption capabilities on mild steel and iron in HCl acid, based on the high surface coverage achieved with low PAA concentrations (0.5 - 2 ppm). The data show a Langmuir adsorption isotherm. The adsorption coefficient B varied between 2107 and 4108 mol⁻¹ and was substantially dependent on the PAA molecular weight: $B = k n M$ (for $k = 0.67$ and 2.95104) or the polymer coil size. The EIS revealed that the adsorbed PAA layer was 1.1 nm thick (for $r = 15$) and exclusively consisted of polymer segments bonded to the metal surface. However, ellipsometry revealed that the adsorbed PAA molecules' limiting layer was voluminous and thick (100-200 nm), including entangled polymer coils (29).

Azzam et al. (2018) studied the Polyacrylamide (PACM) was synthesized utilizing the radical polymerization method with ammonium persulfate as initiator in nitrogen. The PACM produced had a viscosity average molecular weight of 177858. By oxidizing 2-methoxyaniline with APS in an acidic solution under nitrogen environment, the free amino groups of PACM were grafted. The grafting percentage is 96%. On the other hand, morphology of the obtained PACM and PACM-g-P2-MeOANI was investigated by SEM. Grafting improves the morphology of PACM and poly (2-methoxyaniline). The impact of the three polymers on mild steel corrosion in 1.0 M HCl was determined gravimetrically at 25, 40, and 55 °C and electrically at 25 °C. Temperature increased the inhibitory efficiency of all three polymers examined. At all temperatures studied, poly(2-methoxyaniline) is the best corrosion inhibitor. A combined chemical and physical adsorption of polymers on the steel surface was found to follow the Villamil isotherm. The tafel curves show that the studied polymers are anodic and cathodic. The double layer capacitance decreased as the inhibitor concentration increased(30).

Mishra et al. (2018) studied the An attempt is made to show the effect of electron withdrawing nitro and electron releasing methoxy substituents on the inhibition behavior of N-phenylbenzamide derivatives (BNAs) for mild steel acidic (1 M HCl) corrosion. The theoretical and experimental results indicated that methoxy (single bondOCH₃) increases inhibition efficiency whereas nitro (single bondNO₂) diminishes it. BNAs served as cathodic corrosion inhibitors in both EIS and polarization studies. Their greatest efficiencies were 89.56 percent for BNA-1, 93.91 percent for BNA-2, and 96.52 percent for BNA-3. Adsorption of the BNAs at metal/electrolyte interfaces was strongly (high K_{ads}) and spontaneously (negative G⁰), following the Langmuir adsorption isotherm. BNAs adsorb on mild steel surfaces and enhance the energy barrier for corrosive dissolution, as seen by greater values of E_a (activation energy). The experimental efficiency order is strongly supported by DFT studies using 3-21G, 6-31G, and 6-311G basis sets. All basis sets had DFT indices such EHOMO, ELUMO, electronegativity, softness, hardness, dipole moment, energy band gap, and proportion of electron transfer. MD simulations backed up the experimental and DFT studies(31).

studied Noor EA (2009) On the basis of hydrogen evolution (HE) and weight loss (WL) measurements, the corrosion and inhibitor adsorption processes in mild steel/1-methyl-4[4'(-X)-styryl]pyridinium iodides (X: single bondH, CH₃, and OCH₃)/hydrochloric acid systems were investigated. The investigated compounds showed excellent mild steel corrosion inhibition in 1.5 M HCl. The effectiveness of inhibition improves with lowering temperature and increasing inhibitor concentration. There was good agreement between hydrogen evolution and weight reduction measures. The adsorption of the investigated inhibitors on mild steel follows the Langmuir adsorption isotherm at all temperatures. We investigated and evaluated the kinetic and thermodynamic parameters for mild steel corrosion and inhibitor adsorption. We proposed physisorption and chemisorption for the examined inhibitors on mild steel surfaces using thermodynamic adsorption parameters. The Hammett connection

showed a high association between substituent type and inhibitor inhibition efficacy. The results reveal that increasing the substituent's donor property increases the inhibitor's efficiency: I-H II-CH₃ III-OCH₃(32)..

studied Kaddouri et al.(2013) The impact of inhibitor concentration and temperature on mild steel corrosion in molar hydrochloric acid has been studied using weight loss and electrochemical measurements (polarisation and impedance). The compounds significantly reduced the rate of corrosion, with one compound inhibiting corrosion by 98.2% at a concentration of 103 M. The influence of temperature on corrosion behaviour in the presence of two novel calixarenes was investigated between 45–75 °C. The chemicals' efficacy of inhibition increased with concentration and was independent of temperature. Inhibitors with mixed-type polarisation curves, calixarenes The Langmuir adsorption isotherm was followed by the carbon steel surface. We also calculated thermodynamic data for dissolution and adsorption (33)

studied Quraishi et al. (2010) The effects of *Murraya koenigii* leaf extract on mild steel corrosion in hydrochloric and sulphuric acid solutions have been examined utilizing weight loss, EIS, linear polarization, and potentiodynamic polarization. The concentration of the leaves extract increased inhibition. It was also investigated how temperature, immersion period, and acid concentration affect mild steel corrosion in 1 M HCl and 0.5 M H₂SO₄ with extract. Adsorption of inhibitor compounds on metal surfaces was considered to be the mechanism. The extract adsorbs to the mild steel surface according to the Langmuir isotherm. The activation energy and other thermodynamic parameters (Q, H*, and S*) were computed. These thermodynamic characteristics reveal substantial inhibitor-surface interaction. The findings suggest that *M. koenigii* leaf extract may effectively suppress mild steel corrosion in hydrochloric and sulphuric acid conditions (34)..

Mourya et al. (2015) studied The effect of quinolinium salts on mild steel (MS) corrosion in 0.5 M H₂SO₄ was studied by gravimetric and electrochemical methods. According to the polarization curves, these salts are combined inhibitors. A number of kinetic and thermodynamic parameters were determined and discussed. The Langmuir adsorption isotherm was discovered to govern salt adsorption on MS. SEM and AFM verified the presence of a protective coating on MS. A corrosion inhibition mechanism was established using XPS and quantum chemistry calculations (35).

1.3 Aim of study

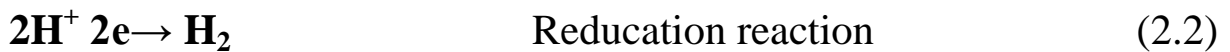
1. Corrosion control in industrial media induced by acid solutions
2. Low carbon steel corrosion rates in a solution of 2M phosphoric acid and at various temperatures.
3. Study of readily accessible and cheap inhibitors can also be conducted.
4. Infrared spectroscopy can be used to investigate inhibitor active groups.
5. Metal compounds may also be identified using a UV-VI spectrometer.
6. Study mechanism of adsorption of corrosion inhibitors on the surface of low carbon steel.



Chapter Two
(Theoretical part)

2.1. Mechanism of Corrosion

According to experimental evidence, the proposed appropriate method for steel corrosion includes an electrochemical mechanism arising from steel metal dissolving in acid. Equations (2.1) and (2.2) explain the anodic and cathodic processes, respectively .



In presence of oxygen the reduction reaction can be written as shown in equation (2.3)



Both the cathodic and anodic processes occur at the same time during electrochemical corrosion. As a result, the formation of a PAM monolayer on a steel surface inhibits the anodic, cathodic, or both regions. As a result, the corrosion process will be slowed. The findings of the experimental and theoretical techniques show that the PAM tested in this research inhibits steel corrosion efficiently [36].

2.2. Corrosion Types

Corrosion may be classified in a variety of ways, including

2.2.2.(Reaction of corrosion:)

2.2.2.1. Electrochemical corrosion

Corrosion may be broken down into two or more sub-reactions. These incomplete reactions are classified as either oxidation or reduction [37]. These two reactions are occurring at distinct locations on the metal surface. One of the processes (oxidation and reduction) is the anodic reaction, which is an oxidation reaction in which the metal undergoes a chemical change. Metal moves from a metallic to an ionic form, in which the valance of the metal is enhanced by releasing electrons.



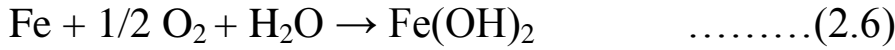
in various places than the anodic one. Cathodic reactions are possible. There are many options. Cathodic reactions are a kind of corrosion reaction that occurs often in metals. The most common are cathodic reactions.

There are a number of chemical reactions that explain many types of corrosion.

1. Metal-metal union with a non-metallic substance without water participating in this reaction.

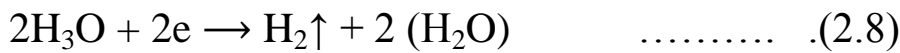


2- The union of metal with oxygen with the presence of water, such as iron rust.



3- Hydrogen evolution reaction

In an acidic medium, the cathodic reaction is:

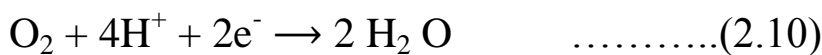


and in the alkaline medium the cathodic reaction is:



4- Oxygen reduction reaction

The cathodic reaction in the basic and neutral medium, where the oxygen is reduced according to the reactions below:



Or



2.2.2.2 Chemical corrosion

Direct chemical attack, also known as pure chemical corrosion, occurs when a bare metal surface is exposed to caustic liquid or gaseous chemicals. Unlike electrochemical attack, where anodic and cathodic changes may occur at a quantifiable distance apart, changes in direct chemical attack occur at the same time and at the same location. (1) spilled battery acid or fumes from batteries; (2) residual flux deposits arising from improperly cleaned, welded, brazed, or soldered joints; and (3) entrapped caustic cleaning solutions are the most frequent factors causing direct chemical assault on aircraft. and corrosion

Chemical (direct) also spreads in general in petroleum facilities due to the production of oil by the action of three gases

- 1- by oxygen gas O_2
- 2- by hydrogen sulfide gas **H₂S**
- 3- by carbon dioxide **CO₂**

2.2.3.. Medium of corrosion

2.2.3.1-Dry corrosion:

It happens when gases, such as Cl_2 , HF, fumes, and H_2SO_4 , act as corrosive agents in the absence of aqueous phases on the metal surface.

2.2.3.2-Wet corrosion:

It happens when aqueous phases are present on the metal's surface [37]. Wet corrosion is more rapid than dry corrosion because the dipolar water molecule stabilizes the free metal ions in solution, and the metallic structure and the water in contact with it both transmit electric current. As a result, several types of wet corrosion, such as uniform, pitting, crevice, erosion, intergranular, selective leaching, and stress corrosion cracking, have been discovered and categorized [38]: as shown below

a.. General (uniform) corrosion It's a uniform rate of metal loss over the exposed surface, characterized by corrosive attack that spreads evenly across the entire surface area, or a significant portion of it.

Uniform corrosion is the most basic type of corrosion. It is also one of the easiest to measure and predict, making catastrophic failures uncommon. As a result, it is not always the most important in terms of cost or safety. If surface corrosion is allowed to continue, the surface can become rough, and it can lead to more serious types of corrosion [38, 39].

b. Pitting corrosion

Pitting corrosion is characterized by metal loss that is concentrated in a small area. On the metal, it appears as a deep, tiny hole. The width of the pit may widen over time, but not to the same extent as the depth. Most of the time, the corrosion product covers the pit opening, making it difficult to detect during inspection [38, 39].

Pitting can happen to most metals that have a protective film on them. As a result, the breakdown of the protective film on the metal surface is linked to the formation of a pit. Many metals and alloys, including carbon steels, stainless steels, titanium, nickel, copper, and aluminum, are susceptible to pitting in various environments [38].

c. Crevice corrosion (CC)

This is a type of localized corrosion that occurs in crevices with a gap large enough for liquid to pass through but small enough to keep the liquid in the crevice stagnant.

Crevice corrosion can affect a variety of metals, including Al, Fe, Cr, and Ni. CC is influenced by a variety of variables, including metallurgical, ambient, electrochemical, surface physical, and geometrical considerations. In heat exchangers, for example, CC occurs under flange gaskets, nail and screw heads, and paint coating edges in overlap joints between tubes and tube plates. Underneath deposits of, the same kind of corrosion occurs.

Corrosion products, dirt, sand, leaves, and sea creatures, for example, are all examples of deposit corrosion. Mechanisms, modeling, test techniques and findings, practical experience, protective measures, and monitoring are all part of the package. A recent review of the mechanisms of CC was published [38]

d. Intergranular corrosion

Intergranular corrosion is a kind of corrosion that occurs only on or near grain boundaries, with little corrosion elsewhere on the surface. The assaults spread throughout the substance. This is a hazardous kind of corrosion because the cohesive forces between the grains may be insufficient to resist tensile stresses; the material's toughness is significantly decreased at an early stage, and fracture may occur without warning [39].

e. Selective corrosion (Selective leaching)

In alloys when one element is obviously less noble than the other, this kind of corrosion occurs (s). The corrosion process indicates that the material's less noble element is eliminated. Dezincification of brass is the most frequent example of selective corrosion, which is the phrase used to describe the leaching of zinc from brass [39].

f. Erosion corrosion

The word "erosion" refers to the degradation of materials caused by mechanical forces. A gaseous or corrosive liquid flowing across the metal typically causes erosion corrosion. Velocity, turbulence, impingement, the presence of suspended particles, and temperature all have an impact.

Because turbulence may dissolve protective coatings and produce very high corrosion rates in materials, any equipment that is exposed to a moving fluid is susceptible to erosion corrosion [39].

g. Cavitation corrosion

This corrosion type is similar to erosion corrosion, however the assault looks different from the erosion corrosion attacks discussed in the previous section. Cavitation attacks, on the other hand, are deep holes that develop perpendicular to the surface and have a pattern that reflects the flow direction. The resistance to cavitation assaults is improved by both strong corrosion resistance and high hardness. References [38] detail the relative resistance of different steels, stainless steels, irons, copper alloys, and nickel alloys.

h. Stress corrosion cracking

Due to the combined action of tensile mechanical tension and a corrosive media, stress corrosion cracking (SCC) is a process involving the start of cracks and their propagation, potentially up to total failure of a component. This stress may be imposed (external load) or residual stress in the metal (for example, owing to the manufacturing process or heat treatment) [38].

2.3. Parameters affect the wet corrosion

2.3.1. Effect of temperature

Temperature increases the rate of almost all chemical reactions, which leads to increase in corrosion rate. Like most chemical reactions, the rate of corrosion of iron and steel in aqueous acid solutions increases

with increasing temperature [40]. This effect can be expressed by Arrhenius equation in which the rate of corrosion change with temperature

$$C_R = A \exp\left(\frac{-E_a}{RT}\right) \dots\dots\dots(2.13)$$

Where A represents the frequency factor, E_a represents the activation energy (kJ/mole), R represents the gas constant (8.314 J/mole.K), and T represents the absolute temperature[41]. The activation energy and frequency factor may be determined using the Arrhenius equation by using the linear log form of the equation: -

$$\ln C_R = \ln A - \frac{E_a}{R} \cdot \frac{1}{T} \dots\dots\dots(2.12)$$

So, given a slope of $(-E_a / R)$ and an intercept of $\ln A$, if $\ln (C_R)$ is plotted versus $(1/T)$. When the activation process is the rate determining phase, temperature variations have the largest impact. It's not unexpected, therefore, that at high coverages, the activation energy of inhibited reactions [42] may be greater or smaller than that of uncontrolled reactions. In the presence of inhibitor, the relationship $[\ln (C_R) = f (1/ T)]$ is very often, but not always, linear, according to the literature. while may be used to determine the enthalpy and entropy of activation while the equation of transition state[42] .

$$C_R = \frac{RT}{Nh} \exp\left(\frac{\Delta S^*}{RT}\right) \exp\left(\frac{-\Delta H^*}{R}\right) \dots\dots(2.14)$$

$$\frac{C_R}{T} = \frac{R}{Nh} \exp\left(\frac{\Delta S^*}{R}\right) \exp\left(\frac{-\Delta H^*}{RT}\right) \dots\dots(2.15)$$

$$\ln\left[\frac{C_R}{T}\right] = \ln\left(\frac{R}{Nh}\right) \exp\left(\frac{\Delta S^*}{R}\right) \exp\left(\frac{-\Delta H^*}{RT}\right) \dots(2.16)$$

$$\ln\left[\frac{C_R}{T}\right] = \ln\left(\frac{R}{Nh}\right) + \ln \exp\left(\frac{\Delta S^*}{R}\right) + \ln \exp\left(\frac{-\Delta H^*}{RT}\right) \dots(2.17)$$

$$\ln\left[\frac{C_R}{T}\right] = \ln\left(\frac{R}{Nh}\right) + \left(\frac{\Delta S^*}{R}\right) - \left(\frac{\Delta H^*}{R}\right) \cdot \frac{1}{T} \dots\dots\dots(2.18)$$

Where; h is the Planck's constant, (J.s) N is the Avogadro's number

(molecule mol⁻¹) ΔH* is the enthalpy of activation (kJmol⁻¹) ΔS* is the apparent entropy of activation (kJmol⁻¹K⁻¹). we can plot ln(CR/T) against (1/T) and the slope of the straight line show a value (-ΔH*/R) and intercept show a value of (ΔS*/R + lnR/Nh) from which (ΔH*) and (ΔS*) is possible to compute.

2.3.2. Effect of inhibitor concentration

Investigators of acid corrosion quickly discover that there is a link between the two. The concentration of inhibitors and the weight loss of the metal specimen with a distinct relationship. The weight loss

reduces as the inhibitor concentration rises, tending to approach a low constant value that relies on the characteristics of the individual inhibitor [40]. The inhibitor concentration may be linked to the amount of inhibitors adsorbed on the metal surface

using an adsorption isotherm. Langmuir, Freundlich, and Kinetic-thermodynamic models are the most often employed isotherms [43]. To get the adsorption isotherm, the degree of surface coverage (θ) for different inhibitor concentrations must be determined, and the various models must be evaluated using the equations below to demonstrate that the model is compatible with the data.

$$\theta = \frac{IE}{100} \dots\dots(2.19)$$

Where θ is the surface coverage and IE is the inhibitor efficiency.

a . Langmuir Adsorption Isotherm

Many instances of strong adsorption do not match the Langmuir isotherm model, which is a collection of uniform adsorption sites. This isotherm is expressed mathematically as:

$$\theta = \frac{K_L C_i}{1+K_L C_i} \dots\dots\dots(2.20)$$

Where K_L is the Langmuir adsorption isotherm's equilibrium constant (L/mL) indicating the degree of adsorption

(i.e., the higher the value of K_L , the more firmly the inhibitor is adsorbed on the metal surface), C_i is the inhibitor concentration

(mL/L), and is the surface coverage [40]. If you rearrange equation (2.20), you'll get:

$$\frac{C_i}{\theta} = \frac{1}{K_L} + C_i \quad \dots\dots\dots(2.21)$$

$$K_L = \frac{1}{55.5} \exp \left(\frac{-\Delta G_{ads}^0}{RT} \right) \quad \dots\dots\dots(2.22)$$

$$\Delta G_{ads}^0 = - RT \ln 55.5 K_L \quad \dots\dots\dots(2.23)$$

Where (55.5) is the concentration of water in the solution in mole/L (M). The intercept values of K_L may be displayed as C_i / θ versus C_i in equation (2.20). The behavior of the equilibrium constant of adsorption (K_L) was computed and shown to increase with increasing temperature [44].

2.4. Thermodynamics of corrosion

Thermodynamics can tell you which reactions are possible and if they are likely to happen. This aids in the comprehension of corrosion phenomena and is necessary for the investigation of corrosion cells. Chemical thermodynamics researches the role of entropy in chemical processes, defining equilibrium as a function of the elements and compounds present, as well as ambient factors like pressure, temperature, and chemical composition.

It's used to see whether corrosion is possible and to forecast which stable corrosion products will develop. The most stable state for a collection of reactants is the one with the lowest free energy, according to a natural rule. As a result, metal surfaces in contact with a solution tend to be in the lowest possible free-energy state.

There is no further change after the system reaches this point. The equilibrium state is ultimately this final, unique, lowest-energy state. When a system is stable, there are no driving factors that may cause it to change [44]

2.5. Methods for corrosion prevention

2.5.1. Corrosion Control

Corrosion control is primarily concerned with (i) materials and (ii) surroundings. The goal of corrosion management is to improve the performance of technical systems by focusing on "people." As a result, successful corrosion control requires a comprehensive management approach that includes both people and equipment. Its goal is to bring the corrosion rate down to an acceptable level (or predictable limits).

Sheir et al. [45] proposed a five-category framework plan for 'Methods of Corrosion Prevention.' Design, material selection, coating, environmental modification (adding inhibitors), electrochemical techniques, and cathodic protection, Anodic protection.

2.5.2 Inhibitors of Corrosion

An inhibitor is a substance that slows or stops a chemical process from happening. As a result, a corrosion inhibitor is a chemical that, when introduced to an environment, reduces the rate of environmental assault on a metal. To avoid severe corrosion, corrosion inhibitors are frequently supplied in tiny quantities to acids, cooling fluids, steam, and other conditions, either constantly or occasionally [46].

Inhibitors reduce or inhibit the metal's interaction with the environment.

media. They lower the rate of corrosion by [47] :

- (i) Ion/molecule adsorption on metal surfaces,
- (ii) modifying the anodic and/or cathodic reactions,
- (iii) lowering the rate at which reactants diffuse to the metal's surface,
- (iv) lowering the electrical resistance of the metal surface
- (v) Using inhibitors that are typically simple to use and may be applied in situ.

2.5.3 Corrosion inhibitor types

The mechanism of inhibition is used to classify inhibitors.

2.5.3.1- Anodic Inhibitors

Which are soluble salts above the surface of the positive electrode that slow down or cancel anode reactions (positive electrode reactions) on a metal surface and are usually coupled with corrosion products and block interactions. To promote the anodic reactions of various chemicals, the added value must be higher than the stated critical value and not less [48].

2.5.3.2 - Cathodic Inhibitors

They are chemicals that are provided with positive ions that interact with the cathodic base to produce insoluble compounds clustering on the cathode and neutralize or eliminate cathodic reactions [48]. Because there is no critical concentration for cathodic inhibitors, they are safer to employ than anodic inhibitors. Even low doses provide a modest level of protection. Cathodic corrosion inhibitors include, for example (sulfates, zinc, magnesium and nickel).

2.5.3.3 - Inorganic Inhibitors

Inorganic oxidizing inhibitors and inorganic sweeping inhibitors operate on the surface of the metal that serves as a positive pole, primarily silicates, phosphates, chromates, borates, ions and antimony arsenic. Cathodic interaction inhibitors that are inorganic [48].

2.5.3.4 - Organic Inhibitors

Because of their use in preventing corrosion in a variety of hostile settings, organic compounds are widely used to inhibit corrosion. These compounds have proven to be very efficient in preventing aqueous corrosion caused by the development of a film on the metal surface. Organic chemicals' inhibitory effect is due to adsorption interactions with metallic surfaces.

Organic inhibitors remove water molecules from the metal surface, then interact with anodic and/or cathodic corrosion processes to prevent water and corrosive-active species from reaching the surface, thus inhibiting cathodic and anodic reactions, or both at the same time. The effect is achieved by either creating a diffusion barrier or inhibiting reaction sites. By displacing surface water molecules, most organic inhibitors are adsorbed on the metal, creating a tight barrier [49]

2.5.3.5 - Mixed Inhibitors

They are inhibitors that induce corrosion on the feet by slowing both anodic and cathodic processes. Polyphosphate and silicate are examples of inhibitors that fit within this category [50].

2.5.4 - Mechanism of Inhibitor

Corrosion inhibitors adsorb on the metal surface and alter the structure of the electrical double layer, influencing the kinetics of the electrochemical processes that make up the corrosion process. The majority of effective inhibitors employed in industry are organic compounds containing oxygen, sulfur, and nitrogen atoms, which cause the compounds to adsorb on the metal surface [51]. Corrosion inhibitors work by creating different kinds of coatings to prevent corrosion. Adsorption, the development of bulky precipitates, and/or the creation of a passive layer on the metal surface are all ways inhibitors create films. The majority of organic inhibitors work by adsorbing into a thin, invisible film that is just a few molecules thick [52]. Most organic inhibitors are adsorbed on the metal surface by displacing water molecules on the surface and creating a compact protective barrier, according to the available data. Inhibitor molecules with no bonded (lone pair) and π -electrons promote electron transfer from the inhibitor to the metal. It is possible to form a coordinate covalent bond via electron transfer from the inhibitor to the metal surface.

2.5.5 - Effective inhibitor properties.

Metallic inhibitors of the materials in issue must be able to comply with the following procedures to prevent or reduce corrosion [52].

- 1- It must work at a very high degree of corrosion resistance. Concentrations of the inhibitor are low.
- 2- It is necessary to safeguard devices that are susceptible to corrosion.
- 3- In severe working circumstances, it must maintain its performance, high velocity, and temperature.
- 4- During the submission process. Under the inhibitor dose, the incidence of corrosion must not substantially rise.
- 5- No deposit on the inhibitor or inhibitor ingredients is possible. Specifically, surface metal in heat transfer areas.
- 6- It must minimize both localized and uniform rusting. The efficiency range must be extensive.
- 7- It must not pose a health risk or emit any pollutants [52].

2.6 - Mechanism of Adsorption

The charge and nature of the metal surface, the electronic characteristics of the metal surface on adsorption of solvent and other ionic species, the temperature of the corrosion reaction, and the electrochemical potential at the metal solution interface all influence the adsorption of organic compounds. There are two kinds of potential interactions between the inhibitor and the metal surface during adsorption.

a- The first is a weak undirected contact between inhibitory organic ions or dipoles and the metal's electrically charged surface owing to electrostatic attraction. Physical adsorption, often known as physisorption, is the name given to this interaction.

b- The second kind of interaction happens when the adsorbate and adsorbent come into contact. Chemical adsorption or chemisorption is a kind of contact that includes charge sharing or charge transfer from the adsorbate to the atoms of the metal surface in order to create a coordinate type bond.

Organic inhibitors' adsorptive activity is described by this term. The type and charge of the metal surface, solvent and other ionic species adsorption, electrical properties of the metal surface, and the temperature of the corrosion reaction at the solution interface are the key factors [53,54].

2.7- Properties of an efficient inhibitor (inhibitor properties).

Metallic inhibitors of the materials in issue must be able to comply with the following procedures to prevent or reduce corrosion [52].

1- It must work at a very high degree of corrosion resistance. Concentrations of the inhibitor are modest.

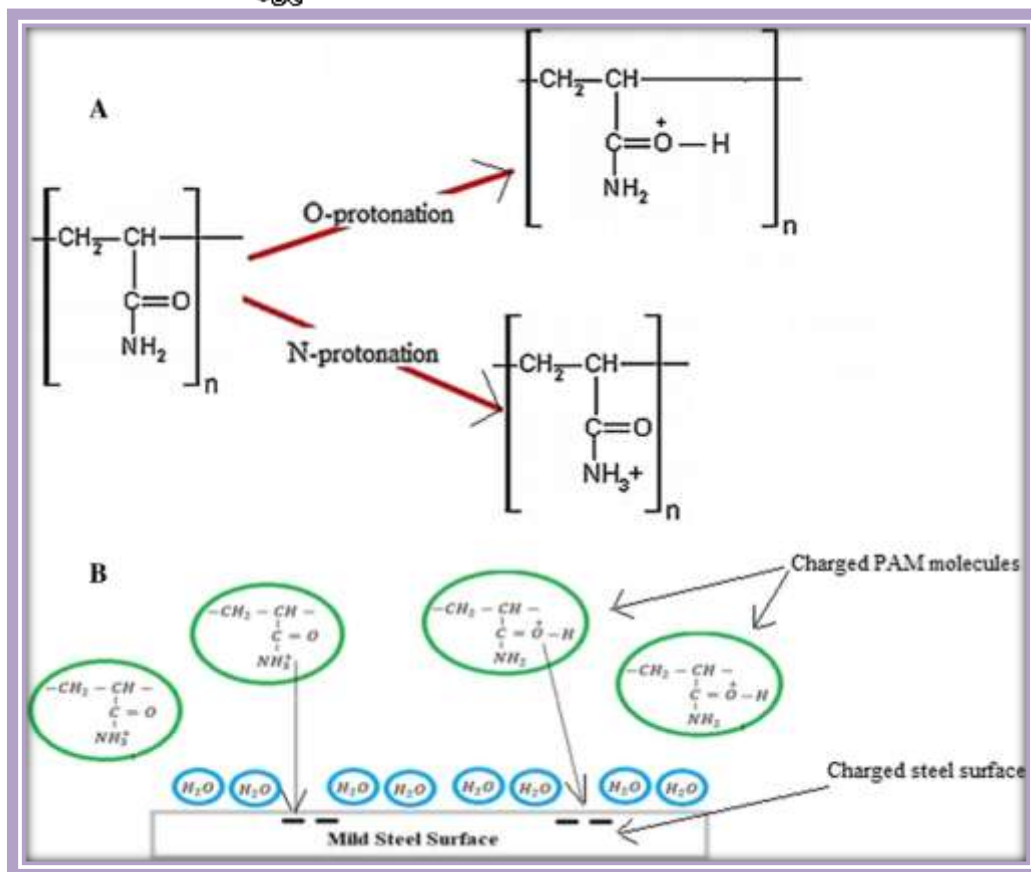
2- It is necessary to safeguard devices that are susceptible to corrosion.

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- 4- During the submission process. Under the inhibitor dose, the incidence of corrosion must not substantially rise.
- 5- No deposit on the inhibitor or inhibitor ingredients is possible. Specifically, surface metal in heat transfer areas.
- 6- It must minimize both localized and uniform rusting. The efficiency range must be extensive.
- 7- It must not pose a health risk or emit any pollutants [52].

2.8- Polyacrylamide (PAM)

It is a white, odorless, solid crystal that dissolves in water, methanol, ether and chloroform. It is usually non-ionic, but due to the hydrolysis of some amide groups it can be converted into carboxylic groups, giving PAM some weak ionic properties. Industrially prepared by hydrolysis of Acrylonitrile



The application or use of polymers as corrosion inhibitors has attracted several researchers. Polymeric compounds are important as corrosion inhibitors in acidification valves. Whereas, the use of polymers and organic materials containing heterogeneous atoms of (S, O, N) is one of the most efficient inhibitors for carbon steel.



Chapter Three
Experimental part

Corrosion rates of carbon steel in phosphoric acid in absence and presence of PAM were studied at different temperatures and inhibitor concentrations is standard. Weight loss and electrochemical techniques were used to evaluate the corrosion parameters.

3.1 Utilized Materials

3.1.1 Phosphoric Acid

Phosphoric acid was used in all experiments as a corrosive solution after dilution to 2M.

It is a transparent colorless liquid supplied by [Scharlab] company and it was made in Spain. Its chemical formula is (H_3PO_4) and it is a, odorless, and non-volatile. In addition of having adequate degree of acidity to cause corrosion.

The phosphoric acid's purity is 99.9%, its molecular weight is 98.00 g/mol, and its density is 1.7 g/ml.

3.1.2- Acetone

Its chemical formula is ($\text{C}_3\text{H}_6\text{O}$), its purity is 99.9, and its molecular weight is (58.0) g/mol. Acetone is supplied by (ROMIL) company and it was used to clean the sample before and after each test.

3.1.3 Benzene

Its purity was 99.7% and it was used to clean the sample and is supplied by [HMEDIA] in Europe.

3.1.4 Distilled Water

It was used to clean the samples and it was obtained from the laboratories of the Chemistry Department, College of Science, University of Diyala.

3.1.5 - The Carbon steel

The sample used is the low carbon steel, which contains the following elemental composition ,as given in Table (3.1).

Table (3.1) Carbon steel compositions .

Element	Wt %
Fe	(99.5)
Mn	(0.201)
C	(0.0762)
Cu	(0.0343)
Cr	(0.005)
Ni	(0.0198)
Si	(0.0306)
mo	(0.0040)
Al	(0.0312)
Co	(0.0020)
Nb	(0.0030)
Ti	(0.0010)
V	(0.0020)
W	(0.0448)
Zr	(0.0030)
Pb	(0.004)

3.2 -The Utilized Equipment and Tools

The utilized equipment and tools are listed in Table (3.2) with their origin and specifications.

Tabale(3.2) Instruments utilized in this research.

No.	Instrument	Model	Origin
1	Electronic Balance	KERN & Shone GmbH, Type ACS 120-4, NO. WB12AE0308,CAPACITY 120g, READABILITY 0.1mg.	Germany
2	Thermostatic Water Bath	THERMOSTAT Water Bath HH-2	China
3	Hot Plate Magnetic Stirrer	(AREC	Germany
4	Laboratory Thermal Oven	BINDER, Hotline International	Germany
5	Hot Plate Magnetic Stirrer	AREC	AREC
6	(Corrosion Cell)	Homemade	Baghdad workshop,Iraq
7	Optical Emission spectrometer(OES)	PMI MASTER Pro2, In Put: 24VDC ,Power:500vA	Oxford-UK
8	Spectrophotometer (UV-1800, UV-Vis)		Shimadzu, Japan
9	Fourier Transform Infrared Spectroscopy(FTIR)	Perkin Elmer Spectrum 65,type (400-4000cm ⁻¹), Germany	Chemistry Department, of College Science, University of Diyala,Iraq

3.3 - Preparation of the Corrosion Solution

The corrosion solution was prepared from phosphoric acid (H_3PO_4) with a molarity of (2M) by the dilution method, as shown in Figure (3.1).

3.4 - Preparation of the PAM Solution

The polyacrylamide, which is supplied by BAD Chemical Ltd. – United Kingdom, was used as a corrosion inhibitor. The PAM was added with different concentrations of (300, 400, 500, and 600ppm) to the phosphoric acid solution (H_3PO_4) of concentration of (2M).



Figure (3.1) Corrosion diluted phosphoric acid solution

3.5 Preparation of the Corrosion Samples

Samples with dimensions ($3 \times 1 \times 0.1$ cm) were cut from a large slice to have a rectangular shape with the surface area exposed to the corrosion solution.

The samples are first heated in an electric oven at $600\text{ }^{\circ}\text{C}$ for one hour (1h) and then they are cooled to room temperature, as shown in Figure (3.2).



Figure(3.2) The preparation steps of the low carbon steel samples

Then, the samples were smoothed using the standard (emery paper) gradually with (60, 80, 100, 230, and 400),mesh.

After that, the samples were washed using tap water, followed by distilled water, and then, they were dried with clean tissues that were immersed in acetone and benzene. The samples are then dried for another period and kept in the desiccator, which contains (silica gel), until use.

Subsequently, the samples are weighed using a four-digit electronic balance, the dimensions are measured by the electronic ruler, and then the samples are suspended and completely immersed in the corrosion solution and transferred to the water bath, as illustrated in figure (3.3).



Figure(3.3) preparation of the Corrosion low carbon steel

3.6 . Weight Loss Measurement

To measure weight loss, the metal samples were completely immersed in 250 ml of the corrosion solution of (2M H₃PO₄) in a conical flask and then they are transfer to a water bath, as depicted in (3.4). The samples are exposed to the corrosion solution for three hours (3h) at the desired temperature. The metal samples were then cleaned by washing them with tap water, followed by distilled water. After that, they were dried with clean tissues, immersed in acetone and benzene, and dried again. Weights were determined in the presence and absence of the inhibitor at (30, 40, 50, and 60 °C) and concentrations of (300, 400, 500, and 600) ppm of (PAM) inhibitor. Figure (3.4) shows the steps of the experimental work.

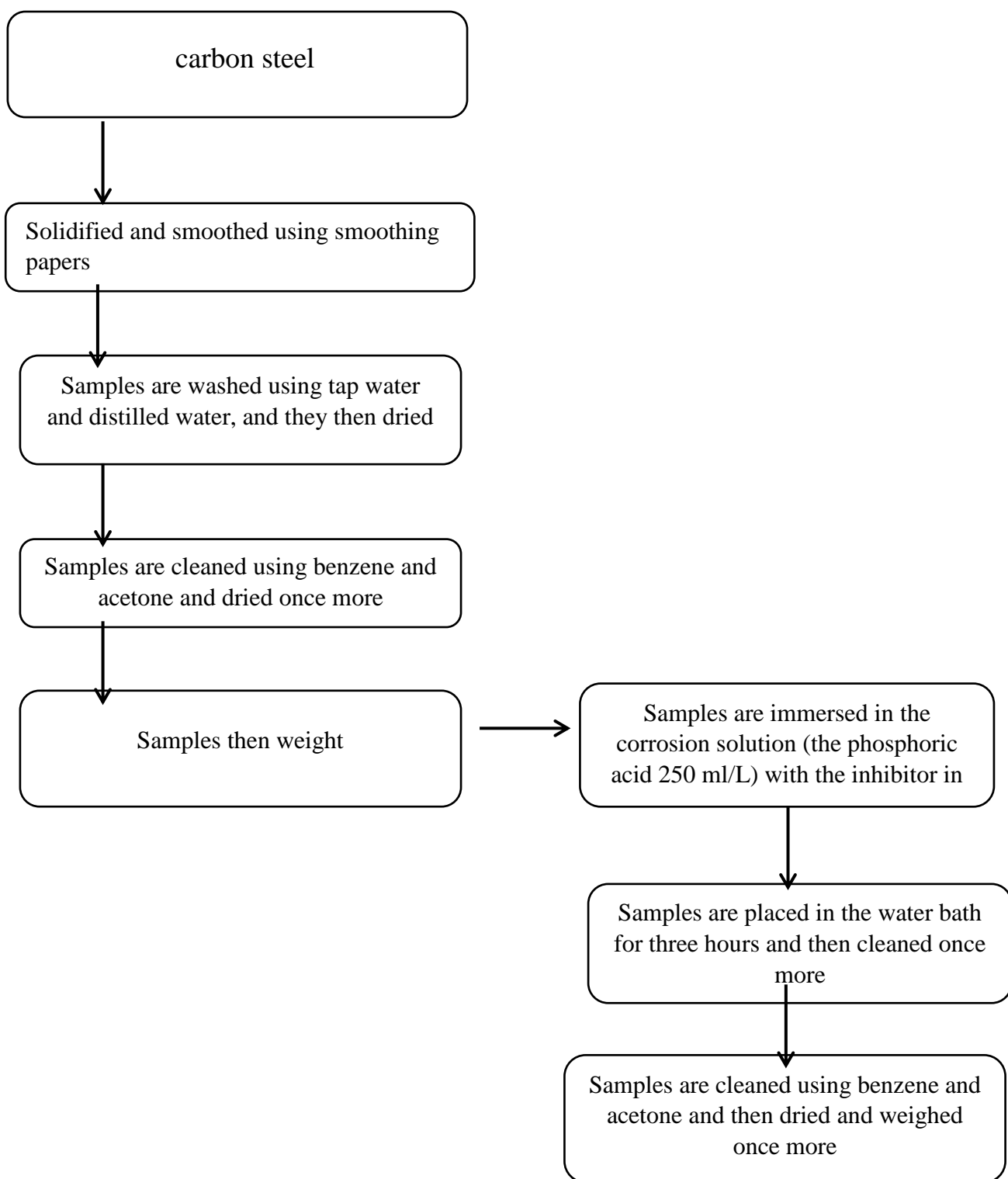


Figure (3.4) Steps of the experimental work.

3.7 The Techniques Used in the Analysis

The study of effects of acids on of the surface the low carbon steel and the composition of the used inhibitor was carried out using the following techniques:

3.7.1 Infrared Spectroscopy

The FTIR nique is used to identify the effective groups before and after the immersion of carbon steel in the corrosion solution. The FTIR device used is made in Germany (PerkinElmer Spectrum, Model 65)and Figure (3.6) illustrates it.



Figure (3.5) FTIR Infrared spectrophotometer used.

3.7.2 Optical Emission Spectrometer

The Optical Emission Spectrometer (OES) technology is used to study the elements composition of the metal sample. The device model is (Foundry Master X Pert-Oxford Instruments). Figure (3.6) demonstrates the Optical Emission Spectrometer (OES) being used.



Figure (3.6) Optical Emission Spectrometer(OES) used.

3.7.3 UV-VIS Spectrometer

The UV-VIS technology is used to identify the effective groups before and after the immersion of sample in the corrosion solution. The of the UV-VIS device are (shimadzu, UV-VIS, 1800, Japan).

3.7.4 The Corrosion Cell

The electric cell was used to study the steel corrosion and its inhibition. This cell is made of pyrex glass (homemade), as shown in Figure (3.7).



Figure (3.7) The Corrosion Cell used.



Chapter Four
(Results and Discussion)

4.1 Indroduction

The corrosion rate of low-carbon steel in 2M H₃PO₄ in absence and presence of different concentration of PAM .weight loss, polarization, and electrochemical impedance microscopy are used to evalut the corrosion parameters.

4.2 Weight Loss Measurement

4.2.1 Corrosion Rate under Different Conditions

The weight loss measurement was used to calculate the corrosion rates at different temperatures after three hours of the immersion time. It was found that the corrosion rate of the low carbon steel in (2M) (H₃PO₄) increases sharply with the increase in temperature and the rate of dissolution of mild steel decreases with the increase in the concentration of PAM. Table(4.1) shows the corrosion rate as afuntion of temperature and inhibiter concentration.

The results obtained show that the addition of inhibitors limits the dissolution of the low carbon steel by blocking the corrosion sites and thus reducing corrosion .It is also5 clear that the corrosion rate decreased with increasing the concentration of the inhibitor at all used temperatures of (30, 40, 50, and 60)°C as a result of the successful surface coverage increase due to the reaction of PAM with the electrically charged metal surface.

4.2.2- Effect of inhibitor concentration and adsorption isotherm

It was found that increasing the concentration of the PAM inhibitor from 300 to 600 PPM reduces the corrosion rate to very low values as Table (4-1) show this that

Table(4.1) Corrosion rate of mild steel in 2M H₃PO₄ as function of temperature and polyacrylamide inhibitor concentration

run	C (ppm)	T (°C)	Corrosion rate (gmd)	IE%
1	0	30	37.31	0
2	0	40	63.26	0
3	0	50	123.33	0
4	0	60	223.16	0
5	300	30	8.53	77.14
6	300	40	33.27	47.41
7	300	50	85.47	30.69
8	300	60	181.11	18.84
9	400	30	6.33	83.03
10	400	40	28.32	55.23
11	400	50	74.11	39.91
12	400	60	162.2	27.32
13	500	30	5.22	86.01
14	500	40	20.53	67.55
15	500	50	67.39	45.36
16	500	60	156.8	29.74
17	600	30	3.35	91.02
18	600	40	15.11	76.11
19	600	50	54.32	55.96
20	600	60	149.2	33.14

the concentration of the inhibitor is sufficient to cover the metal surface in the temperature range of 30-60°C..

Figure (4-1) demonstrates the increase in the inhibition efficiency with increasing the concentration of the inhibitor and decreasing the temperature, where the maximum inhibition efficiency is at a temperature of (30 °C) and inhibitor concentration of (600ppm).

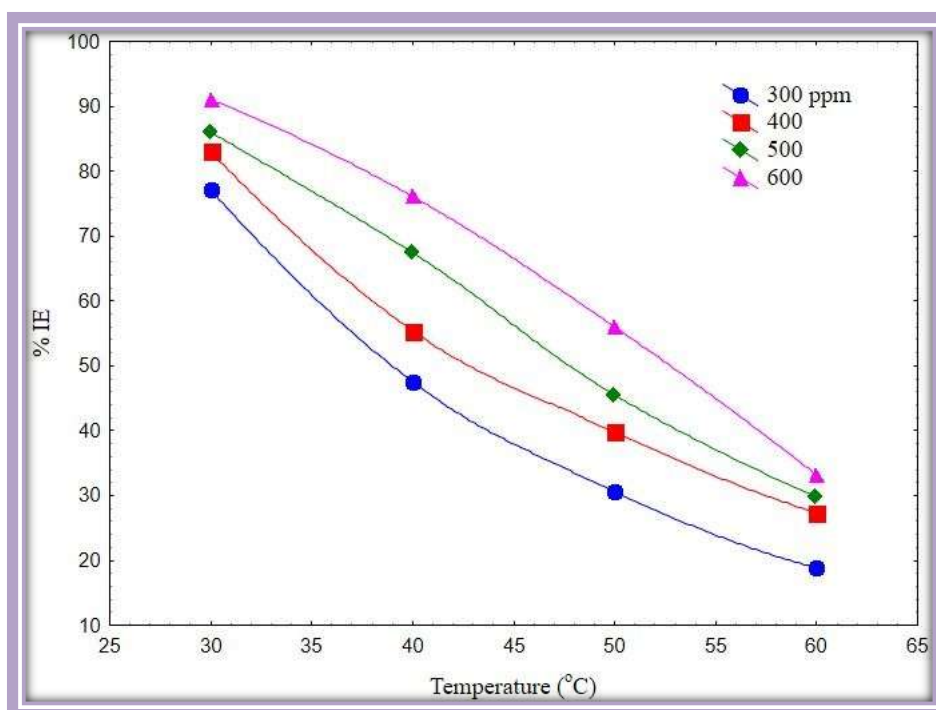


Figure (4.1) Effect of (PAM) concentration on the corrosion inhibition efficiency of low carbon steel

4.2.3- Effect of temperature and activation parameters

Figure (4-2) illustrates an increase in corrosion rate with the increase in temperatures in the absence of the inhibitor, then this increase begins to decrease by increasing the concentration of the inhibitor gradually from (300) to (600) ppm.

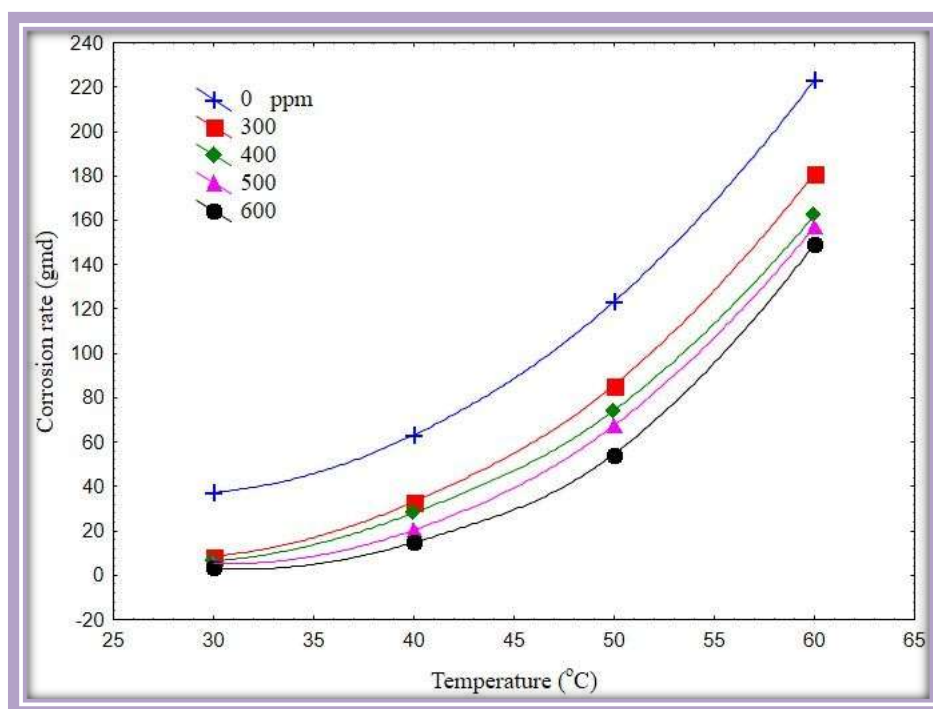


Figure (4.2) Effect of temperature on the corrosion rate of immersed low carbon steel in $H_3PO_4(2M)$.

Surface coverage data (θ) plays an important role in evaluating the properties of inhibitors and is useful while discussing adsorption properties. In particular, when the inhibitor molecules are added, they adsorb on the surface of the metal and the interaction between them can be described by the adsorption isotherm. The surface coverage of inhibitors at a certain concentration is calculated using Equation (2.19). The corrosion rate data can be used to study the Langmuir adsorption isotherm.

The Langmuir isotherm is plotted from Equation (2.20) and according to it the data give straight lines indicating that the inhibitor adsorption follow Langmuir isotherm modelw3.

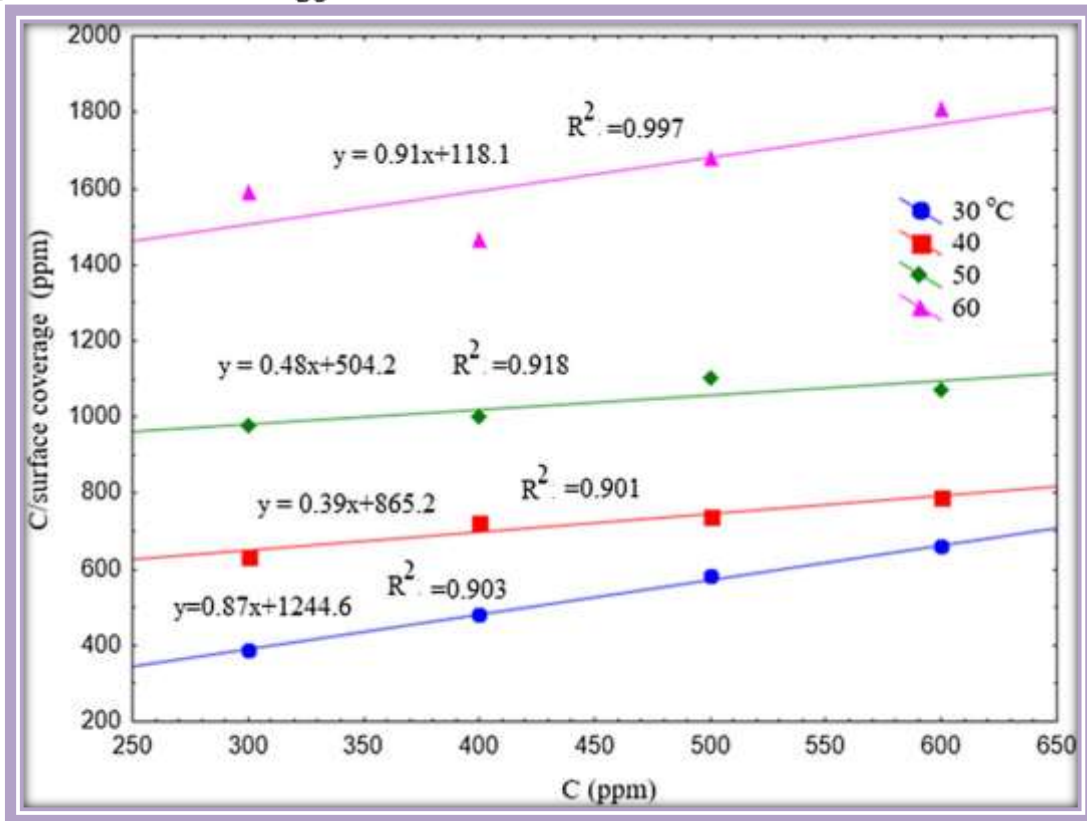


Figure (4.3) Langmuir adsorption isotherms of PAM on the mild steel surface in 2 M H_3PO_4 solution at different temperatures.

These obtained straight lines reveal that the main process of inhibition is adsorption and can also explain the increase in the efficiency of the inhibition with the increase in the concentration of the inhibitor, which indicates that the inhibitor molecules adsorb on the surface of the low carbon steel and block the active sites from direct acid attacks and protect the metal from corrosion [55].

Plotting $\frac{c_i}{\theta}$ versus c_i of Equation (2.21) shows the concentration of PAM and the high structure of the lines means that each active site on the metal surface occupies one inhibitor molecule. Since the

adsorption process was according to the Langmuir isotherm, the k_{ads} values are listed in Table (4.2).

Table(4.2) Adsorption parameters of polyacrylamide on mild steel surface in 2M H₃PO₄ at different temperatures.

T(oC)	K_{ads} (ppm ⁻¹)	$\Delta G_{o_{ads}}$ (kJ/mol)	$\Delta H_{o_{ads}}$ (kJ/mol)	ΔS_{ads} (kJ/mol K)
30	8.5×10^3	- 22.78	- 64.36	- 0.137
40	1.9×10^3	- 19.75		0.143
50	1.1×10^3	- 18.94		- 0.141
60	0.8×10^3	- 18.52		- 0.138

The k_{ads} values increase with the concentration increase of PAM, which indicates that the absorption is good at the surface of the metal. Negative values of ΔG_{ads} indicate that the inhibitors are spontaneously adsorbed onto the metal surface. $\Delta G_{o_{ads}}$ values of about 20 -kJ/mol⁻¹ or less indicate the electrostatic interaction between the charged metal surface and the organic is molecules physical adsorption one (ption physisor). Standard free energy values of adsorption of about 40 - kJ/mol or more indicate that the sharing or transfer of charge from the inhibitor molecules to the metal surface to form a kind of coordination bond (chemical adsorption). In the present work and as shown in Table (2), the ΔG_{ads} values were within the physical adsorption. ΔG_{ads} values are found according to Equation (2.22). The inhibition enthalpy

(ΔH^*) and the inhibition entropy (ΔS^*) are important thermodynamic parameters which are calculated from Equation (2.18). ΔS^* was negative, which indicates rate of corrosion (56) and as in Figure (4.3) and Table (4.2), the ΔH_{ads} was within the physical adsorption range.

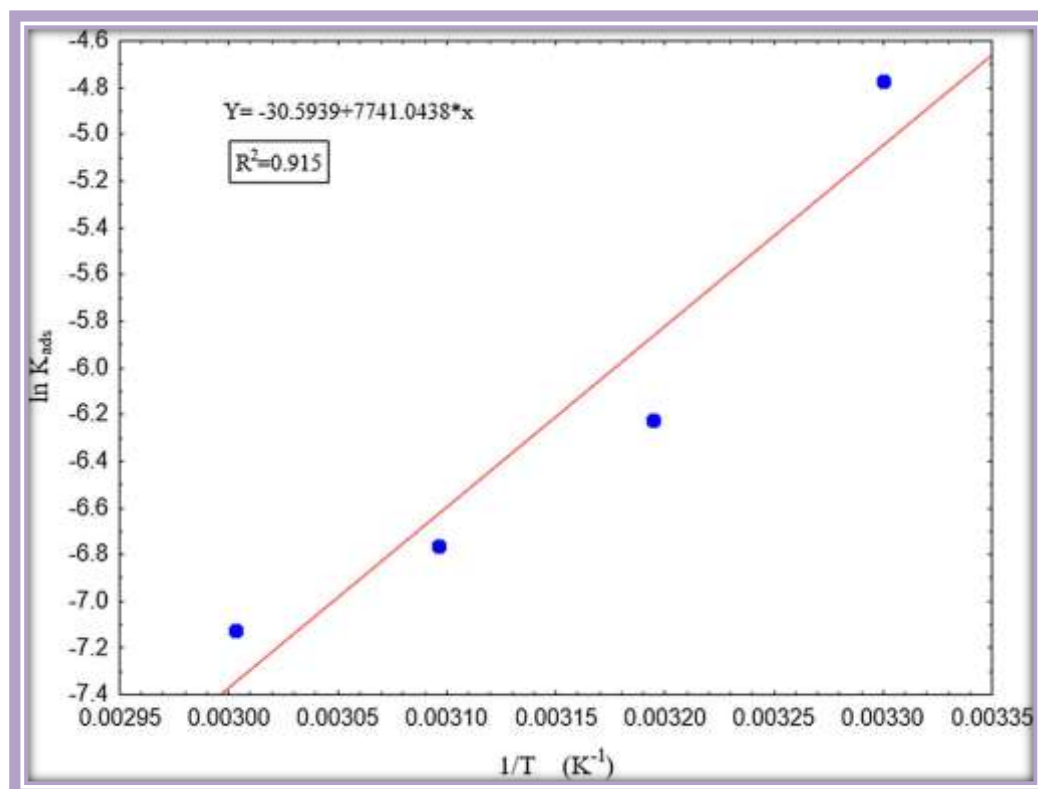


Figure (4.4) Van't Hoff equation

Experimental corrosion rates from the low carbon steel corrosion weight loss measurements in (2M) (H_3PO_4) in the absence and presence of the inhibitor were used to find the inhibition enthalpy ($H\Delta^*$) and the inhibition entropy (ΔS^*) to form the activation complex from the transition state equation, where the effect of the inhibitor and the motion can be studied from Arrhenius equation (Equation (2.12)) and the transition state equation (Equation (2.18)). In Figure (4.5) (Arrhenius plots) and Figure (4.6) (transition state),

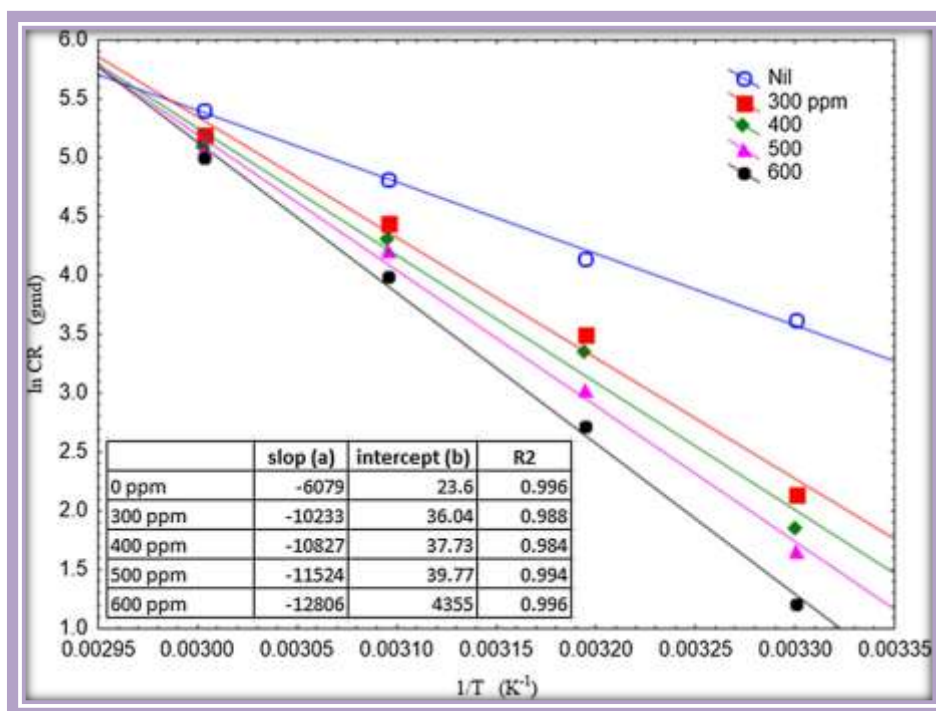


Figure (4.5) Arrhenius plots of steel in inhibited and uninhibited 2 M H_3PO_4

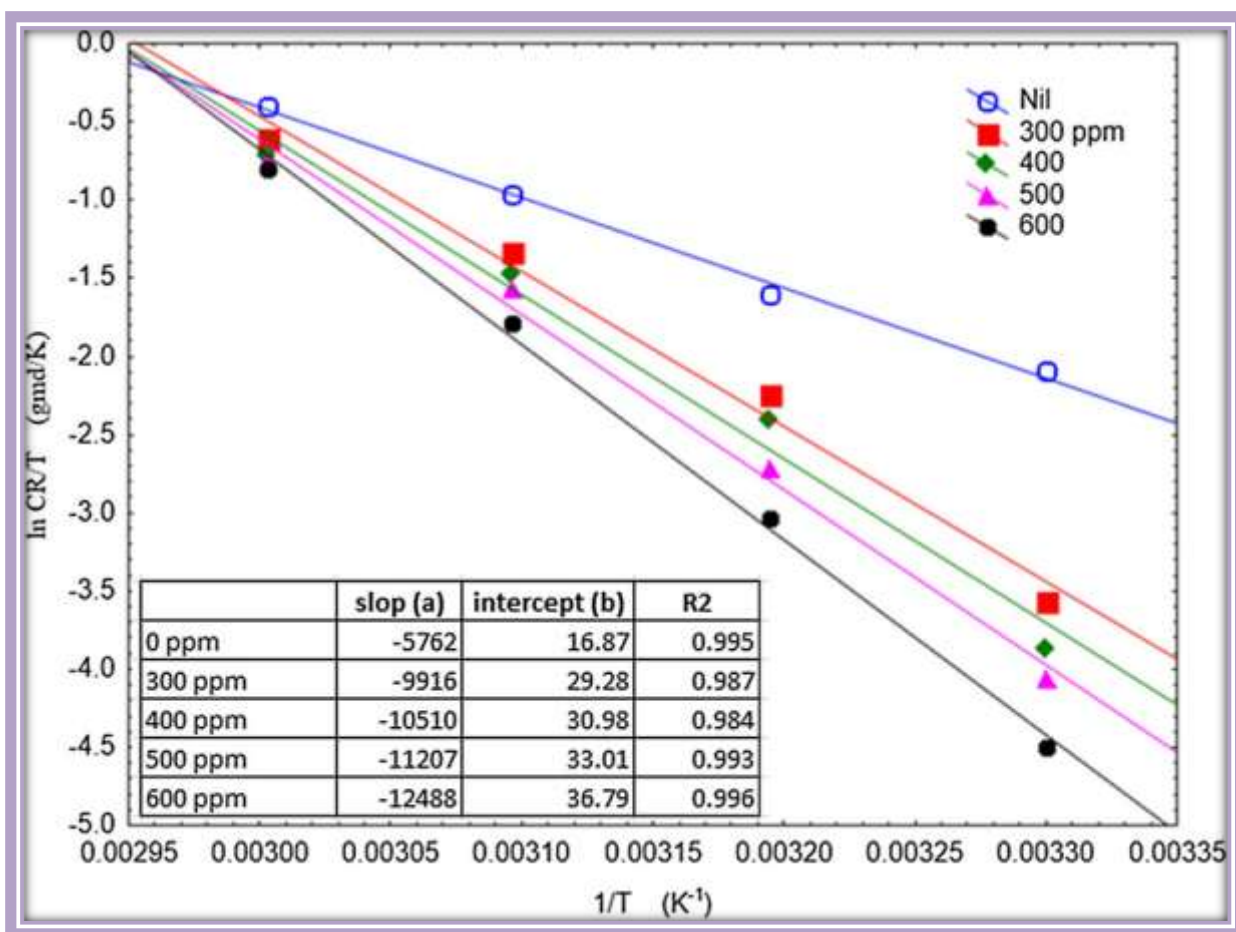


Figure (4.6) Transition-state plots of steel in inhibited and uninhibited 2 M H₃PO₄.

The equations were plotted as (C_R) in and (C_R/T) in versus estimating the values of activation energy [E_a], the enthalpy of inhibition (ΔH), and the activation entropy [ΔS], respectively. Table (3) shows these values.

Table (4.3) Kinetics parameters for corrosion of mild steel in absence and presence of polyacrylamide in 2M H₃PO₄

C (ppm)	$\exp^{(-Ea/RT)}$	Ea (kJ/mol)	ΔH_a (kJ/mol)	ΔS_a (J/mol \square K)
0	0.151	50.54	47.91	-64.52
300	0.041	85.08	82.44	43.57
400	0.33	90.02	87.38	59.36
500	0.026	95.81	93.17	76.91
600	0.017	106.47	103.82	108.41

We can notice that the activation energy was lower in the absence of the inhibitory material (PAM) and this increase in (Ea) is attributed to the formation of a protective layer across the surface of the metal which needs more energy for corrosion to occur [57].

In the Arrhenius equation (($\exp (Ea/RT)$) is the fraction of collisions that have enough energy to overcome the motion barrier at temperature T. From Table (3), $\exp (Ea/RT)$ was reduced with the (PAM) concentration due to the restriction of collisions having energy or equal to Ea. The enthalpy or adsorption enthalpy was also positive in the absence of PAM, indicating that the decomposition of steel is

difficult, especially in the presence of the inhibitor and the slow decomposition of mild steel [58].

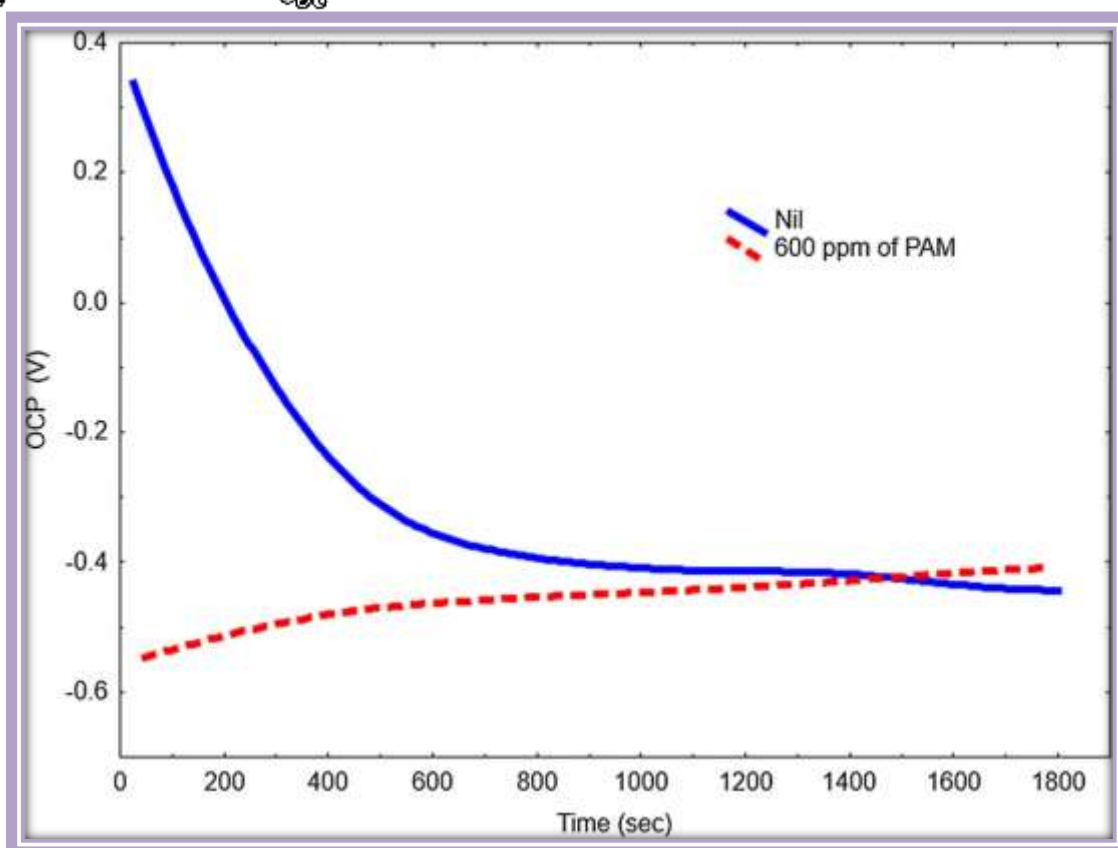
In general, the value of the activation entropy was positive, which means that the system moves from a less ordered arrangement to a more random arrangement [59].

4.3- Electrochemical Measurements

Electrochemical reactions occur on the surface of the metal (anodic and cathodic reactions) and they are important in providing information about the porosity of the surface layer and the corrosion behavior of steel .

4.3.1 Open circuit potentials

through open circuit potentials (E_{cop}) [60]. experiments in this work, in to Figure (4.7), show that the presence of (PAM) transformed E_{cop} to the positive direction, that is, the values of E_{cop} increased and turned to the positive direction with an approximate value of 450 ± 30 millivolts (mv), which indicates that the corrosion inhibitor is of a mixed type.



Figure(4.7) OCP–time curves for mild steel corrosion in 2 M H_3PO_4 solution with and without 600 ppm of PAM at 30 °C

4.3.2- Polarization measurements

Figure (4.8) shows the dynamic potential curves of the mild steel in 2M phosphoric acid in the absence and presence of 600 ppm of PAM at (30) °C, where PAM significantly reduces the current corrosion densities.

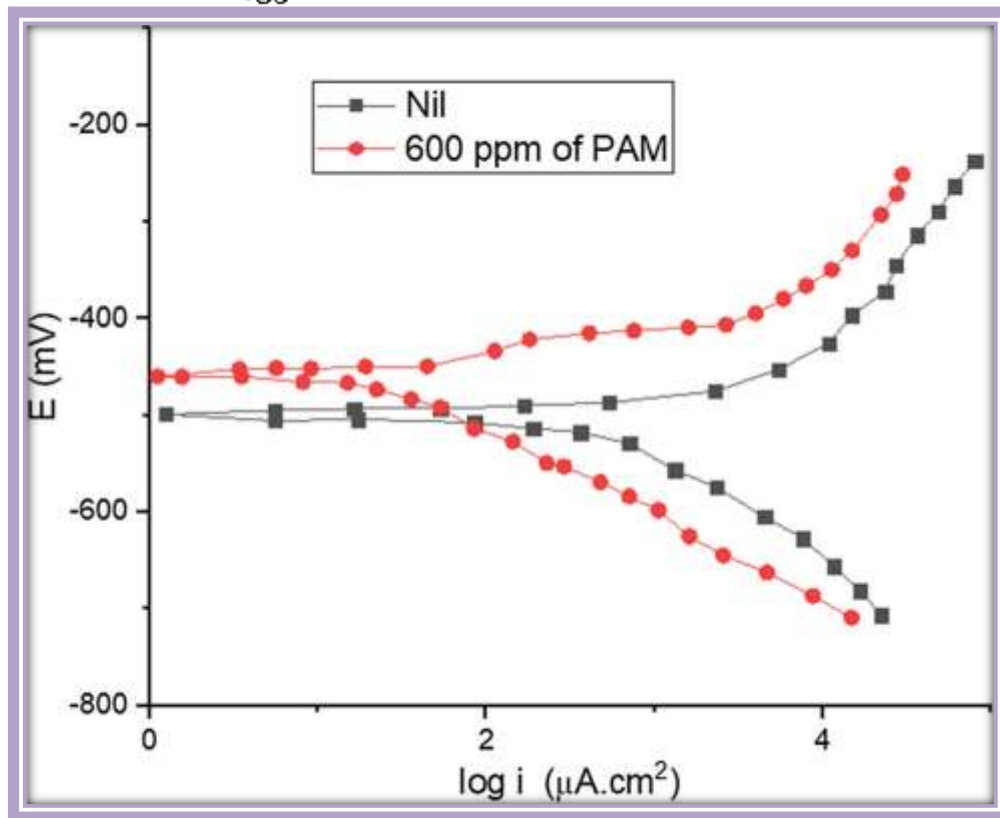


Figure (4.8) Polarization curves for mild steel in 2 M H_3PO_4 solution with and without 600 ppm of PAM at 30 °C

The (E_{corr}), corrosion density (i_{corr}) and the anodic and cathodic Tafel curves (b_a and b_c) were found and they are listed in Table (4,a,b).

(Table 4.4a) PDP (Potential dynamic polarization)

C (ppm)	i_{corr} (mA cm ⁻²)	E _{corr} (mV)	bc (mV/dec)	%IEPDP
Nil	2.97	-490	-120	-
600	0.356	-440	-118	88

(Table 4.4b) EIS results (Electrochemical impedance spectroscopy)

	R _s (ohm□ (cm ²))	R _{ct} (ohm (cm ²))	C _{dl} (μF (cm ⁻²))	%IEEIS
Nil	0.042	0.91	34.36	-
600	0.187	4.68	7.681	82

The inhibition efficiency was calculated from the polarization measurements (%IEPDP) using the following equation:

$$\%E = \frac{i^{\circ}_{cor} - i_{cor}}{i^{\circ}_{cor}} \times 100$$

where, i_{cor} and i°_{cor} are the corrosion current density with and without the inhibitor (PAM).

Here, the shift in the value of the corrosion current (E_{corr}) at the anodic and cathodic sites (a mixed type inhibitor). We also note that the products of anodic and cathodic polarization shift towards the low current in the presence of PAM, which indicates the mixed nature of the inhibitors.

4.3.3- electrical impedance

The electrical impedance EIS of the mild steel in (2M H₃PO₄) was conducted in the absence and presence of 600 ppm of PAM at 30°C.

Figure (4.9) illustrates the impedance diagram, which displays a semi-circle with a high frequency. The radius of the circle is increased by adding 600 ppm of PAM, which indicates the large corrosion inhibitory effect of PAM.

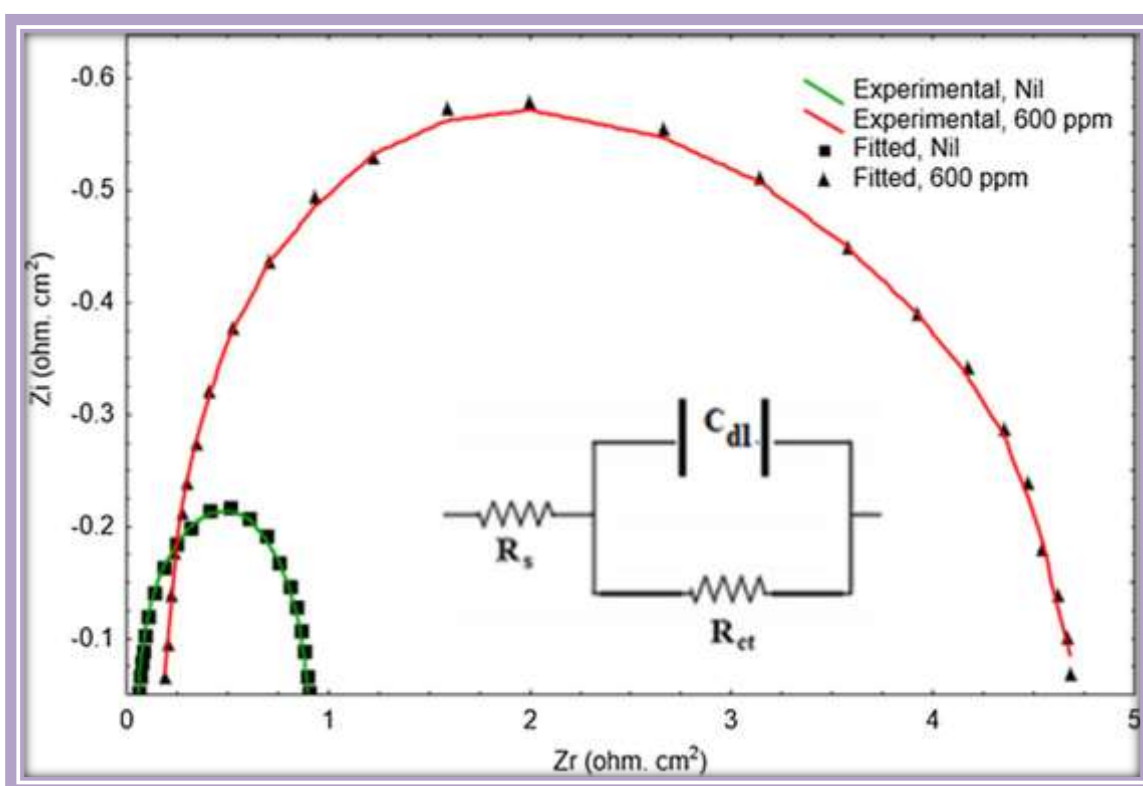


Figure (4.9) Nyquist plots for mild steel in 2 M H₃PO₄ solution with and without 600 ppm of PAM at 30 °C

The results in Table (4) show that the values of R_{ct} and R_s are increased by adding 600 ppm of PAM. These results are attributed to the increase in the thickness of the protective nature on the steel surface due to the gradual replacement of water molecules by the adsorption of PAM molecules on the surface of mild steel [61].

In addition, the inhibition efficiency was measured according to the impedance measurements (IE_{eis}), according to the following equation:

$$IE_{Eis} \% = \frac{R_{ct} - R^0_{ct}}{R_{ct}} \times 100$$

By comparing the results with the results of the electrochemical polarization .

4.4- FTIR measurements

The PAM powder and the protective layer formed on the metal surface were analyzed by FTIR spectra, because the main step in the corrosion inhibition is the adsorption of inhibitors on the metal surface to protect it. Subsequently, an FTIR analysis is performed to get some clarification on the possible interactions between the adsorbed inhibition and the surface of the low carbon steel in the acidic environment of 2M H_3PO_4 with 250 M/L of PAM.

The degree of activation depends on the molecular structure of the inhibitors, where PAM is tested to assess the main functional groups.

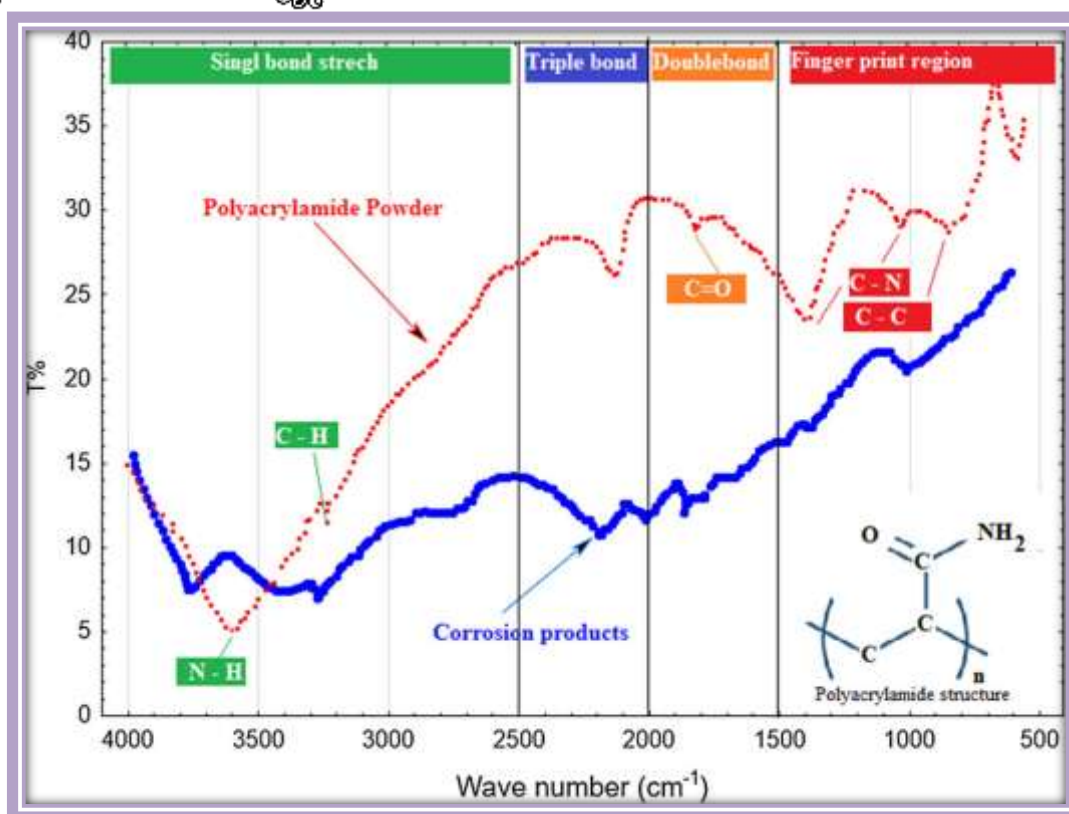


Figure (4.10) FTIR spectrum of PAM powder and protective

The FTIR spectrum of the pure PAM material, is depicted in Figure(4.10)

film (scratched corrosion products) from mild steel surface after 3 h immersion in 2 M H_3PO_4 with 600 ppm of PAM at 30 °C

(4.10) showed the first absorption peaks at 3442 cm^{-1} , which indicates the vibrations of the N-H bond stretching. The peak at 1687.83 cm^{-1} refers to the vibrations of the stretching of the bond C=O and the peak at absorption 1021 cm^{-1} indicates the stretching of the C-N bond. While the FTIR spectrum of the protective layer formed on the metal surface was shifted after immersion in the solution containing 600 ppm of PAM and 250 ml/L of (2M H_3PO_4) acid. Particularly, the

transition was made to a lower wave number, as shown in Figure (4.10), where the frequency of stretching the N-H band was shifted from 3655.18cm^{-1} to 3431.43cm^{-1} .

Moreover, the stretch of the bond C=O was shifted from frequency of 1687.83 to 1640 cm, where these observations show that PAM was absorbed on the metal surface [62].

4.5- UV-VIS measurements

The UV-VIS measurement also provides a technique for the studying of metals and compounds [63], The UV-visible absorption spectra of (H_3PO_4 2m- 600ppm. PAM) solution were obtained before and after three hours of immersion of the steel sample. Figure (4.11) shows both cases.

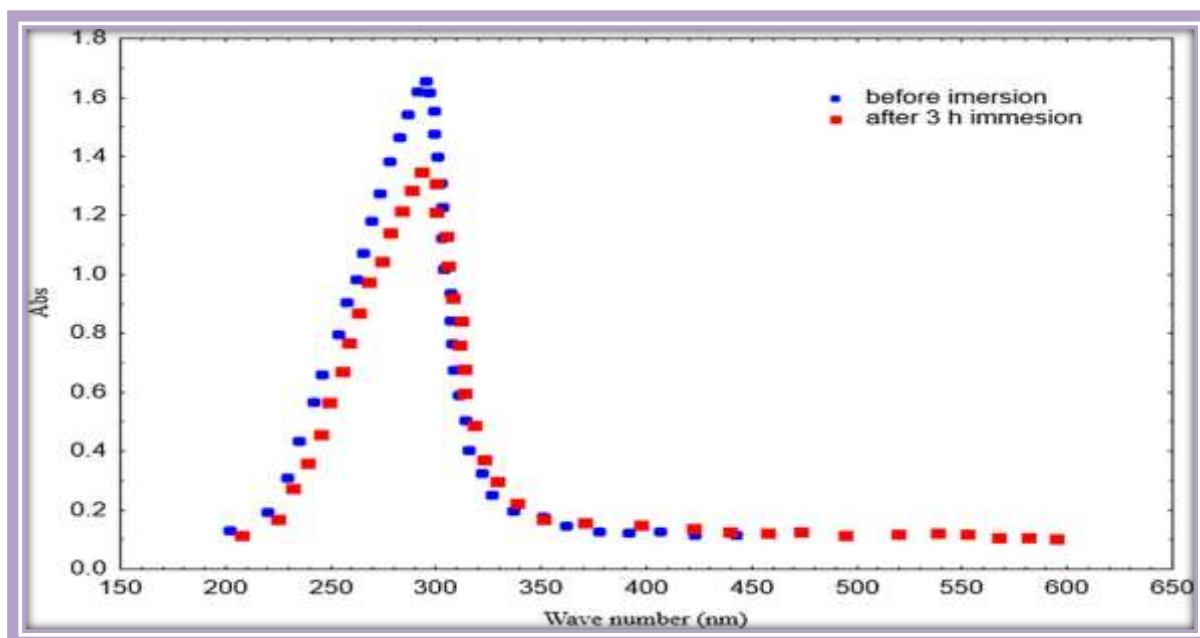


Figure (4.11) UV–Vis spectrum of 600 ppm PAM—2 M

H₃PO₄ before and after 3 h immersion at 30 °C

where the absorption spectrum of PAM before immersion of the metal showed one band in the ultraviolet region, but after three hours of immersion, the change in the absorption mode and shift to high wave length region indicates a complex formation between PAM and the steel surface, which indicates the possibility of forming a layer over the iron surface[63].

4.6- A Study of the Mechanism of Inhibition and Stability

An increase in inhibition efficiency was observed with increasing the PAM concentration. The reason for this is due to the increase in the surface of the mild steel covered with the adsorbent. Furthermore, it was found that the adsorbed PAM particles remain stable on the surface of the mild steel even after five hours of immersion in the corrosive solution. This can be illustrated in Figure (4.12), which shows the variation in the efficiency of PAM over time in the presence of a PAM inhibitor with a concentration of 600 ppm in H_3PO_4 acid with a concentration of 2M and at a temperature of 30 °C.

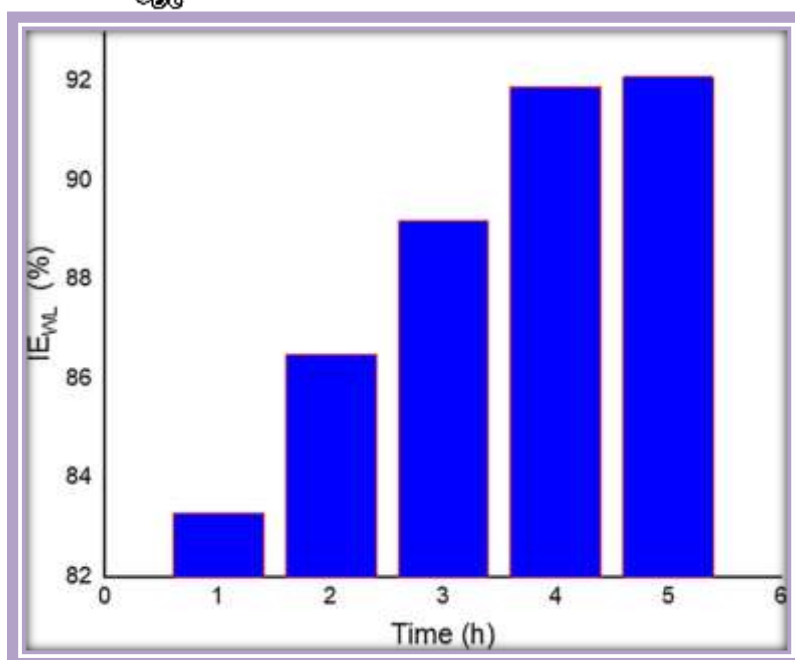


Figure (4.12) Variation of inhibitor efficiency with time in presence of 600 ppm PAM in 2 M H₃PO₄ at 30 °C

It is expected that the cause is due to the PAM molecules, which contain heterogeneous atoms of oxygen and nitrogen, where the transfer of an electron to the surface of the mild steel, and thus a greater adsorption capacity was obtained in acidic solutions. PAM can exist in a protonated state and two possibilities for proton states can be proposed, as shown in Figures (13A) and (13,B) [21].

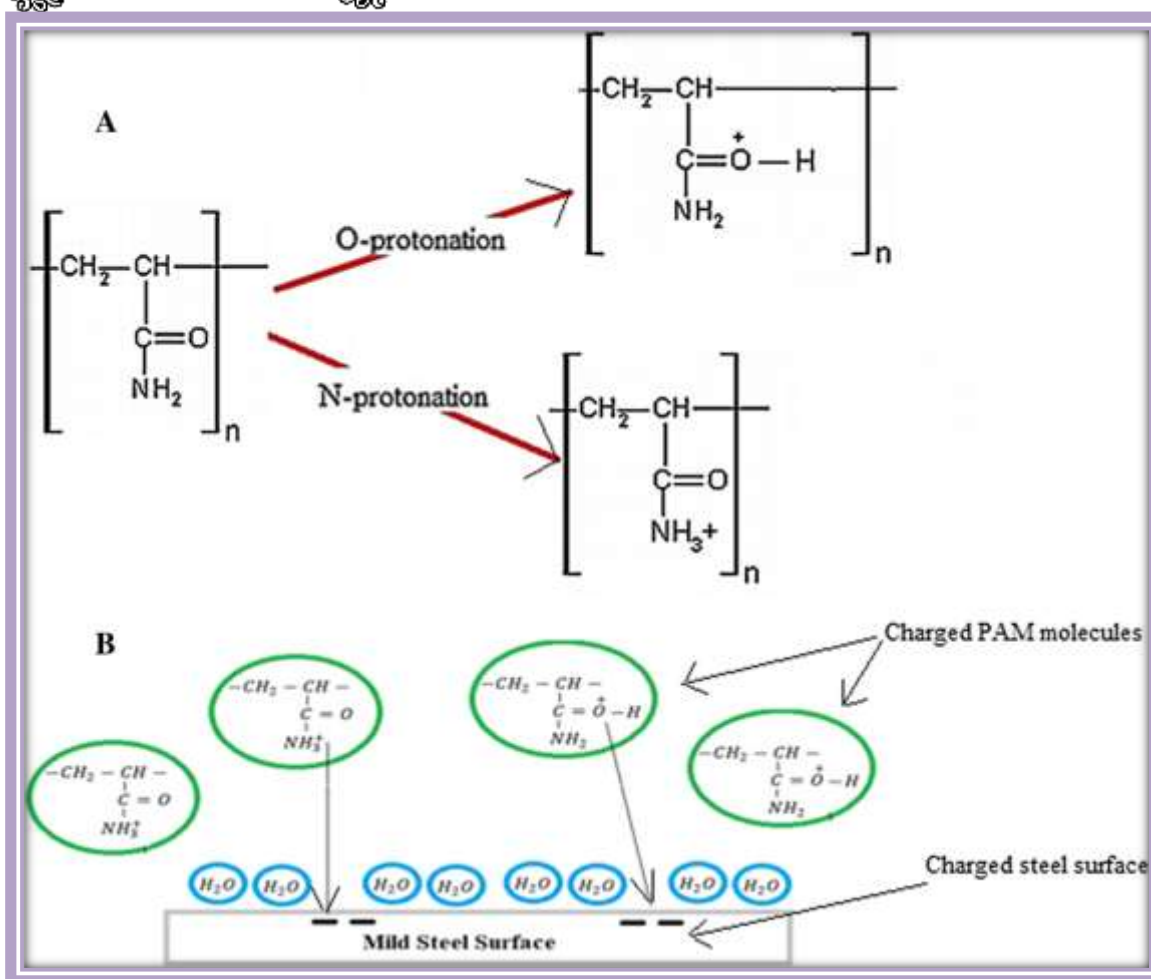


Figure (4.13.A.B)Suggested protonated states of PAM. B. A. Suggested adsorption mode of PAM

The adsorption of PAM on a mild steel surface was also facilitated due to the presence of positively charged protonated types in the acids. The corrosion of steel occurred in the anodic (oxidation) equation (2.1) and in the cathodic (reduction), equation (2.2) or equation (2.3) during the electrochemical reaction.

Both the cathodic and anodic reactions proceed at the same rate, so the formation of a PAM film on the surface of the steel will inhibit the anode and cathode regions, thus reducing the corrosion

The results obtained from experimental and analytical methods indicate that the tested PAM in this study effectively inhibits the corrosion of steel.

Corrosion is a complex phenomenon in general and the phenomenon of preventing corrosion cannot be classified as a physical or chemical phenomenon only.

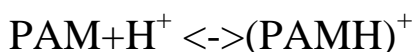
The thermodynamic factors for adsorption obtained from this study revealed that PAM is adsorbed on the surface of the steel.

The PAM reaction is activated by the presence of oxygen and nitrogen atoms, which leads to a higher inhibition efficiency. It suggests physical and chemical adsorption as mechanisms to bind the inhibitor molecules to the metal surface.

In general, physical adsorption requires the presence of the quality charged in the bulk of the solution and the electrically charged metal surface, while the chemical adsorption process involves the transfer of charges from the inhibitor molecules to the metal surface.

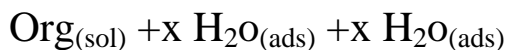
It has been assumed that the PAM adsorption mechanism can be explained by one or more of the following explanations:

1/ In acidic solutions, PAM is expected to be in equilibrium with the corresponding neutral form according to the equation (64)



The protonated forms of PAM are adsorbed onto the negatively charged metal surface by chromatography.

2/ For organic corrosion inhibitor, in general the adsorption on a mild steel surface may be attributed to the alternative adsorption between water molecules adsorbed on the metal surface $H_2O_{(ads)}$ and the organic molecules in the aqueous solution ($org_{(sol)}$) [12].



where X represents the number of water molecules replaced by one inhibitor molecule. Because of the electrostatic attraction, the protonated PAM particles were actually adsorbed on the mild steel according to the mechanism (1) and as shown in Figure (12B).

4.7- Conclusions

Referring to the results obtained through the study, the following can be concluded:

1. Polyacrylamide acts as an excellent a mild carbon steel corrosion inhibitor in a (2M H₃PO₄) solution
2. Inhibition efficiency increases with the PAM concentration and decreases with temperature approaching the maximum value of 91.02% at 30°C and 600 ppm
3. According to the Langmuir adsorption isotherm, PAM was adsorbed to the steel surface.
4. According to the standard free energy negative values (ΔG_{ads}) , the adsorption process was spontaneous and physical.
5. Analyses using UV and FITR revealed that a protective layer had formed on the metal's surface.
6. Study proved PAM was an electron donor for the surface of steel.

4.8 - Recommendations

- 1/ Finding new, high-efficiency, renewable and cheap inhibiting materials with minimal effort.
- 2/ Repetition of similar work for other metals such as copper and aluminum.
- 3 / Studying the efficiency of inhibitors in alkaline and saline media.
- 4/ Study the efficiency of inhibitors under the influence of other temperatures.
- 5/ Using organic or inorganic inhibitors other than PAM.



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الخلاصة

تمت دراسة معدلات تآكل الفولاذ منخفض الكربون في حامض ($2M H_3PO_4$) عند درجات حرارة مختلفة (300 ، 400 ، 500 ، 600) درجة مئوية مع وجود المثبط البوليمري (PAM) وغيابه كمثبط للتآكل باستخدام طريقة فقدان الوزن وطريقة الاستقطاب الكهربائي. وجد أن كفاءة تثبيط التآكل تزداد مع زيادة تركيز المثبط (PAM) وتتنخفض مع درجة الحرارة ، حيث بلغت أقصى كفاءة للمثبط 91.02% عند تركيز (600 جزء في المليون) ودرجة حرارة 30 درجة مئوية. تم الحصول على امتزاز PAM على سطح الفولاذ الطري وفقاً لامتناس Langmuir متساوي الحرارة. كانت قيمة امتناس الطاقة الحرة سالبة وأقل من 20 كيلو جول / مول ، مما يدل على أن عملية الامتزاز كانت تلقائية وفيزيائية. ترتفع قيم k_{ads} (ثابت التوازن) مع تركيز PAM ، مما يشير إلى امتزاز السطح.القيمة السالبة لحرارة الامتزاز تشير إلى الامتزاز التلقائي للمثبطات على الأسطح المعدنية. كانت قيم ΔG_{ads} ضمن نطاق الامتناس الفيزيائي. كانت طاقة التنشيط أقل في غياب المادة المثبطة (PAM) وهذه الزيادة في (ΔE_a) هي يُعزى إلى تكوين الطبقة الواقية. كانت إنتروبيا التنشيط إيجابية ، مما يشير إلى أن النظام أصبح أقل تنظيماً.

أظهرت النتائج أن قيم استقطاب المقاومة (R_{ct}) واستقطاب المحلول (R_s) تزداد بإضافة 600 جزء في المليون من PAM. وتعزى هذه النتائج إلى زيادة سمك الطبقة الواقية على سطح الفولاذ.

الخلاصة

بالإضافة إلى ذلك ، تم قياس كفاءة التثبيط وفقاً لقياسات المعاوقة (% IEEIS). بمقارنة النتائج
بنتيجة الاستقطاب الكهروكيميائي وجد أن جزيئات PAM الممتزة تظل مستقرة على سطح الفولاذ
الطري حتى بعد خمس ساعات من الغمر في محلول التآكل مما يدل على التباين في كفاءة
PAM بمرور الوقت في وجود مثبت PAM بينت اختبارات FTIR و UV-VIS عن تكوين طبقة
واقية على سطح معدني.



وزارة التعليم العالي والبحث العلمي
جامعة ديالى
كلية العلوم
قسم الكيمياء

تشبيط تآكل الفولاذ منخفض الكربون في المحلول المائي لحامض الفوسفوريك
باستخدام بولي اكريل امايد

رسالة مقدمة الى

مجلس كلية العلوم – جامعة ديالى

وهي جزء من متطلبات نيل درجة الماجستير في علوم الكيمياء

من قبل

رشا جمال جداع حبيب

بكالوريوس. في علوم الكيمياء ٢٠٠٨

كلية العلوم- جامعة ديالى

باشرف

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