

FULL PAPER

Synthesis of new corrosion inhibitors with high efficiency in aqueous and oil phase for low carbon steel for missan oil field equipment

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The Iraqi oil field environment is filled with metal pipes and other components that are often exposed to chemicals that can cause corrosion, especially when the metal and chemicals are in a solution such as downhole fluids. Inhibitors are among the most important industrial additives that are commonly used in Iraqi petroleum industry to decrease the corrosion that cause damage to pipelines, water and waste water system. This fact evoked the urgent need to prepare corrosion inhibitor materials; one of the most important properties of these compounds is to act as surfactants working to remove or reduce the surface tension between the two immiscible phases with each other and make it easier to mix. Three types of corrosion inhibitors were prepared by using fatty acids and converted them into the fatty alkyl halides in presence of hydrochloric acid. Finally, the products reacted with various mole ratios of tertiary amine in various mole ratios, and then they were identified by classical methods melting point measurement, solubility test and [FTIR, ¹H-NMR and ¹³C-NMR]. Morphology of the carbon steel was examined using scanning electron microscopy (SEM) in presence and absence of inhibitors. In the last step, the inhibitors of low carbon steel in acidic medium were assessed as a case study by using potentiostat device and weight loss to calculate the corrosion rate and inhibition efficiency. The result was as follows: P1 was 81.30%, P2 was 81.79% and P3 was 97.56%. In addition, inhibitors prepared in this study were evaluated in organic phase (Bazrcan iraqi oil fields) by weight loss method using different concentrations of corrosion inhibitors (50,75,100 ppm) and the inhibition efficiency as follows:

P1: inhibition efficiency (91.8%) at a concentration of inhibitor (100 ppm);

P2: inhibition efficiency (83.8%) at a concentration of inhibitor (100 ppm); and

P3: inhibition efficiency (81.2%) at a concentration of inhibitor (100 ppm).

KEYWORDS

Corrosion inhibitor; corrosion; quaternary ammonium salt.

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Introduction

The use of inhibitors is one of the most practical methods for protecting materials against corrosion, especially in acidic media.

They have been widely studied in many industries to reduce the corrosion rate of metal materials in contact with aggressive medium[1]. In general, inhibitors which reduce corrosion of metallic material can be

divided into three kinds: (i) inorganic, (ii) organic, and (iii) mixed material inhibitors.

The most well-known acid inhibitors are organic compounds containing nitrogen, sulfur, and oxygen atoms. The efficiency of these molecules mainly depends on their abilities to be adsorbed on the metal surface with the polar groups acting as the reactive centers[2]. It would be inept to include mechanisms of inhibition in the definition of a corrosion inhibitor because inhibition is accomplished by one or more of several mechanisms. Some inhibitors retard corrosion by adsorption to form an invisible thin film only a few molecules thick, others forms visible bulky precipitates which coat the metal and protect it from attack. Another common mechanism consists of causing the metal to corrode in such a way that that a combination of adsorption and corrosion product forms a passive layer. Hydrocarbon production is often associated with corrosive species such as acid gases (H_2S , CO_2), the extraction and the existence of other factors regarding the nature of oil extracted and the component of metals in addition to the alloys used in petroleum industries (equipment, tools, devices and pipelines) [3]. The first condition for any corrosion process to develop is the presence of water, and corrosion in production well is usually driven by the presence of corrosive elements containing the produced fluids when considering down hole tubing corrosion of producing well, transportation of crude or oil

products and other industrial form of corrosion. One of the most characteristic features specific to the Iraqi crude oil is that the produced fluid has high levels of water cut. Also, the Iraqi oil field environment includes a huge number of metal pipes and the components that are often exposed to different conditions (like chemicals) that can cause corrosion, especially when the metal and chemicals are in a solution such as down hole fluids [4]. This research adopted the activity and effectiveness of the chelating compounds like, EDTA and modified its chemical structure to obtain a new combination as Gemini surfactants [5], where the term "Gemini" was coined by Menger and Littan in 1991 [6]. Gemini, dimeric or twin surfactants contain identical polar head groups and identical hydrophobic tails with easier methods to synthesize compared with their unsymmetrical counterparts. Gemini surfactant is a new generation of surfactant developed in recent years, constituted by two hydrophilic groups and two hydrophobic groups in the molecule, and separated by a rigid or flexible spacer[5], forming micelles with lower critical micelle concentration (CMC) [one to two orders of magnitude lower than for the corresponding conventional surfactants], and about 3 orders of magnitude more efficient at reducing surface tension, better solubilization and greater efficiency in lower the surface tension of water [7].

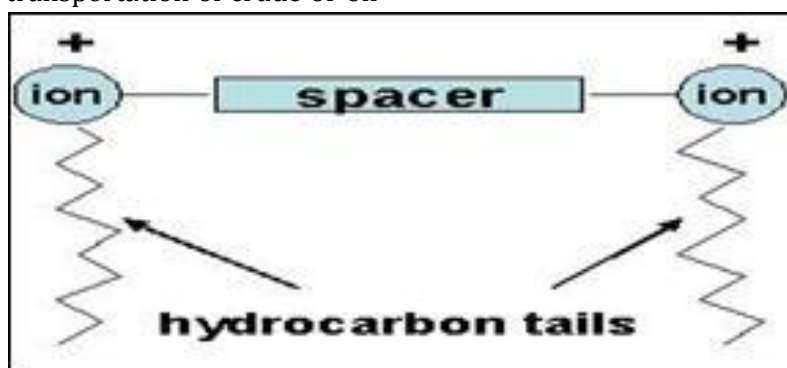


FIGURE 1 The Gemini structure

Many Gemini surfactants have been synthesized with their unusual physicochemical properties, including their high surface (or interfacial) activity, unusual changes of viscosity, unusual micelle structure, aberrant aggregation behavior, better foaming properties and the ability to make organic compounds soluble in water [8]. The qualities of the spacer groups, such as their length, flexibility and chemical structure, have been investigated and it has been shown that the spacer group is of great importance in determining the solution properties of aqueous surfactants [9]. Surfactants, always added to the medium in small quantities, accumulate in special order at the interfaces and modify them by controlling, reducing, or preventing reaction between a substrate and its surroundings. Different surfactant groups have been reported to present corrosion inhibitory potential that depends on the classification of surfactants, the substrate type, inhibitor concentration, inhibitor

structure, acid type and its pH, salt presence, co-surfactant, temperature, and immersion time [9]. The quaternary ammonium surfactants have been reported as excellent corrosion inhibitors for iron and steel in acidic medium [9]. Different adsorption mechanisms for surfactant molecules on the metal surface were found between the Gemini surfactants and the corresponding conventional single-chained surfactants. The adsorption behavior of the Gemini surfactants on surface in acidic medium was found to be affected by (i) length of hydrophobic chains, and (ii) the spacer length of the Gemini surfactants [10]. When a molecule with amphiphilic structure is dissolved in aqueous media, the hydrophobic group distorts the structure of the water, as a result of this distortion, some of the surfactant molecules are expelled to the surfaces of the system with their hydrophobic groups oriented to minimize the contact with the water molecules. Figure 2 shows the behavior of the surfactants in aqueous medium [9].

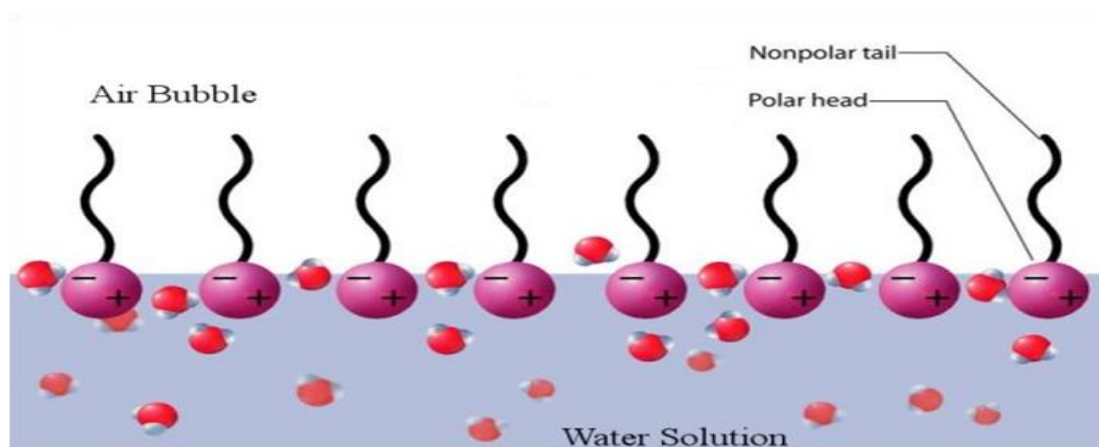


FIGURE 2 Behavior of the surfactants in aqueous medium

When the water surface is or begin to saturate, the overall energy reduction may continue through another mechanism of miscell formation. Organic inhibitors react by adsorption on a metallic surface. Cationic inhibitors (+), like amines, or anionic inhibitors (-), like sulfonates, are preferentially adsorbed depending on the charge of the metal surface (+) or (-). At the

zero point of charge, there is no particular preference for an anodic or cathodic inhibitor. In such a situation, a combination of an inhibitor, which would be strongly adsorbed at more negative potentials along with cathodic protection would provide a greater degree of inhibition than either applying cathodic protection or using inhibitor separately. The formation of a bond between

the metal substrate and the organic inhibitor (chemisorptions) bonds impedes the anodic and cathodic process and protects the metal surface. Consider an inhibitor molecule, e.g. containing a polar amine nitrogen group at the end of a hydrocarbon chain. The active (-NH₂) group contains a pair of unshared electrons which it donates to the metal surface. A chemisorptions bond is, therefore, formed which impedes the electrochemical reaction. The polar amine group displaces water molecules from the surface. On adsorption, most of the metal surface is covered by the adsorbed water molecules. The inhibitors react by replacing water molecules by organic inhibitor molecules. Here n represents the

number of molecules which are replaced to accommodate the organic molecule. The hydrocarbon part of the organic inhibitor is oil soluble, hence, it repels water from the metallic surface. It, therefore, provides a barrier which keeps water away and thus prevents corrosion. The hydrocarbon chain attracts the organic molecules and forms an oily layer which prevents corrosion by acting as a barrier against fluids. For instance, Di-ethanolamine effectively inhibits corrosion of carbon steel in petroleum water mixtures. The organic inhibitors are physically adsorbed on the surface [11]. Figure 3 shows a simplified mechanism.

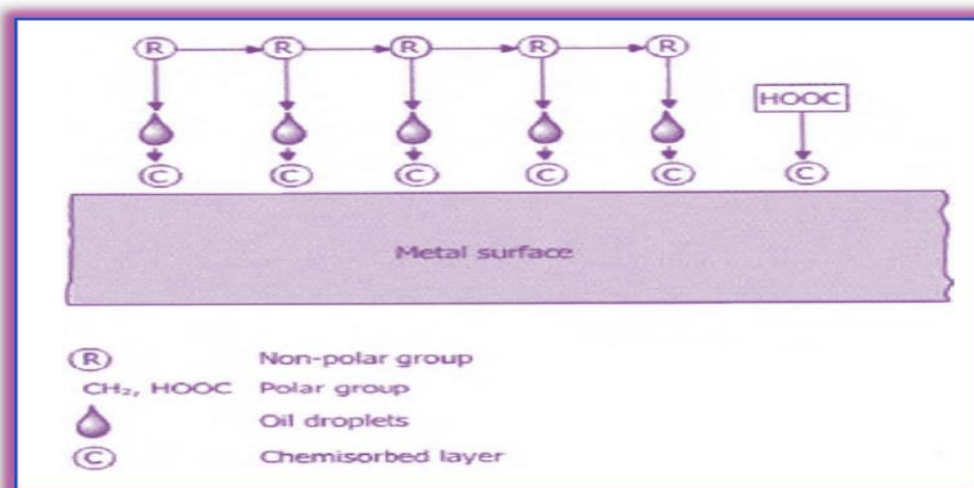


FIGURE 3 The simplified mechanism of organic corrosion inhibition

Experimental part

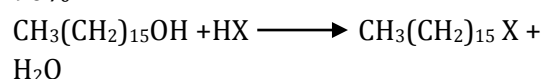
Preparation stage

The corrosion inhibitors (surface active material) were prepared by two steps:

Step (1):

It involved the synthesis of fatty alkyl halides by reaction between one of the fatty alcohol with mineral acid in the presence of a Lewis acid as catalyst and temperature ranging between (55C) for 10 hours, followed by washing with distilled water to get rid of the increase in mineral acid as well as non-reacted catalyst and continuing washing until the

purity of the result was obtained; the result was fatty material dissolved in alcohol by 90%.

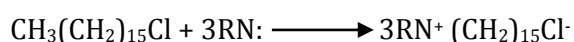


Step (2):

In this step involved the reaction of the prepared fatty alkyl halides from the previous step in various mole ratios with some tertiary amine in the certain mole ratio and carboxylic acid containing a tertiary amino group, and in the presence of catalysts to obtain the corrosion inhibitors. Many of these materials were prepared along with different effectiveness depending on molar ratios of

reactant materials and the nature of the catalyst and reaction time, as follows:

1- Reaction (2 mol) of fatty alkyl halides with (1 mol) of tertiary amines with a temperature ranged from (55 C) for 24 hours gave oily substance (First corrosion inhibitor p1) Alkyl tri methyl ammonium halide with inhibition efficiency by 81.30% in aqueous phase by potentiostate, while in organic phase (bazrcan oil) by weight loss inhibition efficiency was 91.8% at inhibitor concentration of 100 ppm.



2- The second corrosion inhibitor (p2) Quaternary Ammonium Salt was prepared from the mixing of (4 mol) alkyl Halide and (1 mol) (Tetra methyl ethylene diamine TMEDA) and the presence of sodium as catalysts at the temperature between (40-60 C) for 4 hours with the inhibition efficiency by 97.56% in aqueous phase by potentiostate, while in organic phase (bazrcan oil), weight loss gave inhibition efficiency was 81.2% at inhibitor concentration of 100 ppm.

3- The last corrosion inhibitor (p3) Dialkyl (Dialkonic acid, di alkyl alkonate) ethylene di ammonium halide was prepared from the raw materials mentioned earlier here, where 4 moles of alkyl halides was used with 1 mole of carboxylic acid containing tertiary amino groups (EDTA) and using hydrocarbon solvents as a medium of reaction at the temperature between 55 C. The inhibition efficiency by 81.79% was obtained in aqueous phase by potentiostate, while in organic phase (bazrcan oil) weight loss gave inhibition efficiency by 83.8% at inhibitor concentration of 100 ppm.

Identification stage

Solubility tests

In this study, the solubility of the prepared corrosion inhibitor compounds was used by using different solvents, as shown in Table 1.

TABLE 1 Solubility test of the prepared compounds

Sample no	Water	Iso Propanol	Ethanol	Toluene	Kerosene	Mestylene	M.E.K
P 1 C ₃₈ H ₇₉ NO ₃	+	+	+	+	+	+	+
P 2 C ₃₈ H ₅₃ N ₂ Cl ₂	+	+	+	+	+	+	+
P 3 C ₄₂ H ₇₈ N O ₈	+	+	+	+	+	+	+

Melting point measurements

We measured the melting points of the raw, intermediate and prepared compounds of the

reaction as shown in Table 2. Melting point measurements were made by the device.

TABLE 2 Melting point of materials prepared

No.	Sample	Melting Pointe °C
1	Long chain alcohol	59.4
2	Long chain alkyl Halide	120
3	Tri Ethanol Amine	17.9
4	Salt of carboxylic acid	238.5
5	P 1	58
7	P 2	55
9	P 3	67

FTIR and $^1\text{H-NMR}$ spectroscopy

FTIR spectrum of compound (p1) showed the appearance of strong absorption bands at $1400\text{-}1450\text{ cm}^{-1}$ belonging to $\nu\text{ R4N}^+$ and other absorption bands, while the spectrum of compound (p3) showed the appearance of strong absorption bands at $1400\text{-}1450\text{ cm}^{-1}$

belonging to $\nu\text{ R4N}^+$ and absorption bands at $1700\text{-}1750\text{ cm}^{-1}$ belonging to $\nu\text{ (C=O carboxylic)}$, and other absorption bands shown in Figures 4, 5 and 6 for compounds (p2&p3)[10]. The $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ of compound (p2) are shown in Figure 6 and 7, respectively.

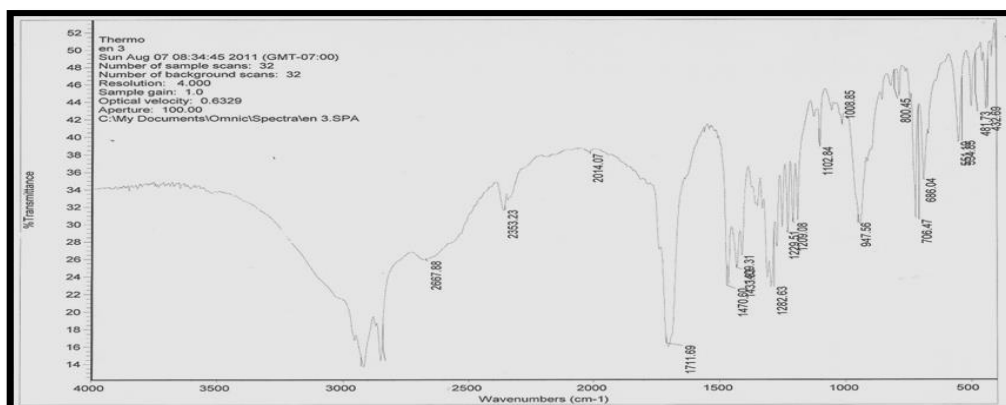


FIGURE 4 Infrared spectrum of the inhibitor p2

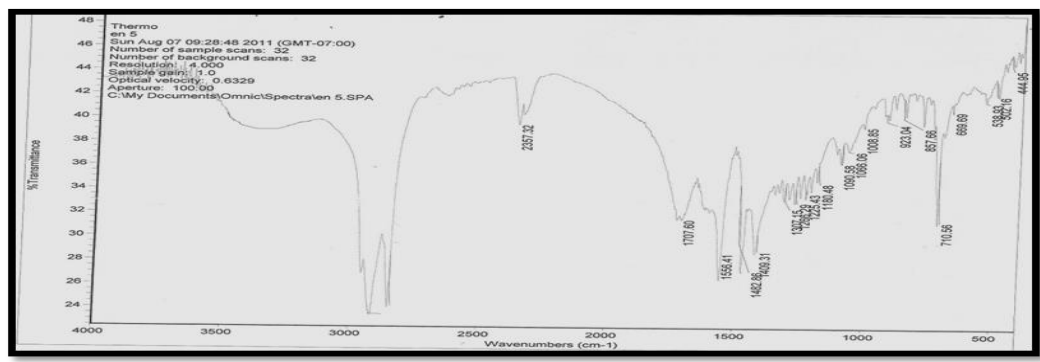


FIGURE 5 Infrared spectrum of the inhibitor p3

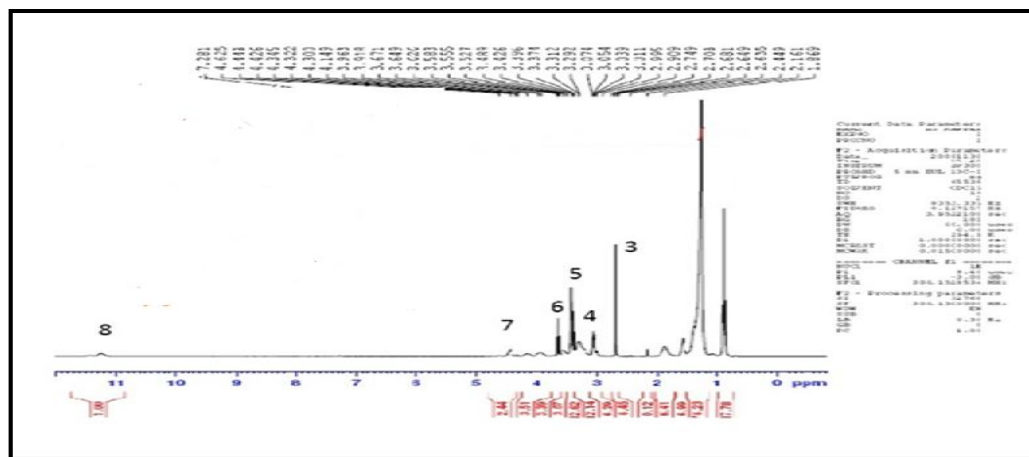


FIGURE 6 $^1\text{H-NMR}$ of synthesized bis quaternary ammonium salt (p2)

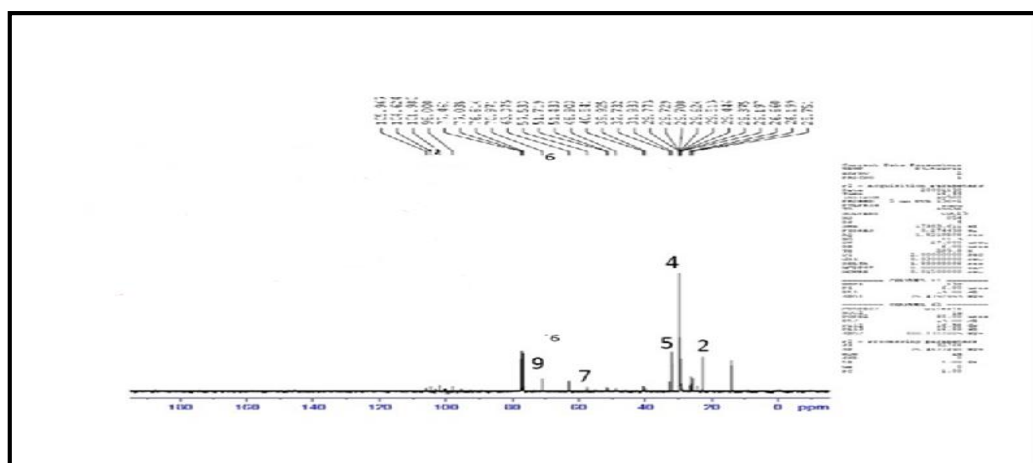


FIGURE 7 ^{13}C -NMR of synthesized bis quaternary ammonium salt (p2)

Corrosion measurements

Weight Loss Measurements (in acidic medium)

The circular low carbon steel sheet of 2.5 cm x 0.02 cm were abraded with a series of emery paper (grade 320-500-800) and then washed with distilled water and acetone. After weighting accurately, the specimens were immersed at inclined position in beakers which contained 50 mL of studied sulfuric acid concentration with different concentrations of the prepared inhibitors.

All the aggressive acid solutions (with or without inhibitors presence) were open to air. After a specific time, the specimens were taken out, washed, dried and weighted accurately.

Weight Loss Measurements (in Crude oil)

The same procedure for weight loss measurement that applied in acidic medium was also carried out with crude oil obtained from Bazrcan oil field (well No.1011). The weight loss of low carbon steel sheets for both media (sulfuric acid and crude oil) was obtained at the lab temperature. The corrosion rate ($\text{mg}\cdot\text{cm}^{-2}\cdot\text{min}^{-1}$), and percentage protection (inhibition efficiency P%) were calculated.

The material composition employed for the present work was characterized with two different instrumentations (Atomic Absorption Spectrophotometer and SpectroMaXx/ stationary metal analyser) to specify its actual metallurgical type (in percent) as a specimen characterization as shown in Table 3 below.

TABLE 3 The specimen characterization

C%	Si%	Mn%	P%	S%	Cr%	Mo%	Ni%
0.187	0.311	1.03	0.007	0,011	0.004	0.002	0.029
Al%	Co%	Cu%	V%	W%	Pb%	Sn%	As%
0.037	0.001	0.003	0.0005	0.0222	<0.0032	0.0040	0.0084
Zn%	Bi%	Ca%	Ce%	B%	Zn%	La%	Fe%
0.0019	<0.0039	0.00046	<0.0030	0.00074	<0.0020	<0.001	98.3

Measuring of open circuit potential

To determine the open circuit potential of the specimens, the specimens were immersed in the acidic solution prepared in the lab to reach the steady state between the specimen's material and electrolytic solution. The change

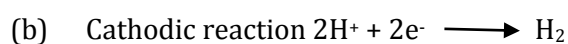
in potential according to the current was determined within 5 min., and time step was equal to 60 sec. In the base of the Tafel extrapolation, under the theorem of mixed potential, to determine the corrosion rate and values of potential from cathodic and anodic

polarization are considered. After determining the open circuit potential, low current from cathodic was passed through the specimens by reducing magnitude of variable resistance gradually. Then, the working electrode potential was measured against the current. By potentiostat device, final results, i.e., the value of current density, were obtained using the prepared inhibitors p1, p2 and p3, analyzed as current-potential data by using potentiostat device software.

Results and discussion

The concern for corrosion by sulfuric acid has increased in the oil and gas industry of the transformation of H_2S , SO_x produced during oil extraction and refining into concentrated sulfuric acid where the burning of fuel is a major cause of global warming or the stability of the global climate.

The most widely used alloy for handling sulfuric acid is carbon steel. When carbon steel contacts dilute sulfuric acid, an immediate attack on the metal takes place with the formation of hydrogen gas and ferrous ions, as shown in reactions (1) and (2).



The rate of corrosion of carbon steel in dilute acids depends strongly on steel chemical compositions, especially the carbon content and the reasons for these unusual experimental results are not clear [11].

The simplest and most accurate method of estimating the corrosion rate is weight loss analysis. A weighted sample (coupon) of low carbon steel under consideration is introduced into the corrosive environment, and later removed after a reasonable time interval. The coupon is then cleaned of all corrosion products(s) and is reweighted. The circular low carbon steel sheets were studied in different concentrations (50 ppm, 75 ppm,

and 100 ppm) of the synthesized inhibitors (p1, p2, and p3). The corrosion rate ($mg.cm^{-2} min^{-1}$) was calculated from the following equation:

$$R_{corr} = W / S.t$$

The surface coverage (θ), percentage protection efficiency was calculated as follows:

$$\theta = [wt\ loss\ (uninh) - wt\ loss\ (inh)] / wt\ loss\ (uninh.)$$

$$P\% = [[wt\ loss\ (uninh) - wt\ loss\ (inh)] / wt\ loss\ (uninh.)] \times 100.$$

Also, the percentage protection efficiency (p%) can be calculated from the corrosion rate data of the tested low carbon steel without $R_{corr.}(uninh.)$ or with inhibitor presence $R_{corr.}(inh.)$ at a specific concentration and temperature by applying the followed equation:

$$P\% = [[R_{corr}\ (uninh) - R_{corr.}(inh)] / R_{corr.}(uninh.)] \times 100. [12]$$

The obtained results from the weight loss for low carbon steel specimens in wet crude oil media (Bazrgan) proved the inhibition efficiency for synthesized inhibitors (p1, p2, and p3) from 81.2 to 91.8% at 100 ppm after studying the optimum the concentrations.

Results of polarization curves

E_{corr}, i_{corr}, and P% Results

A positive value of (P%) indicates the inhibition of corrosion by the added inhibitors (p1, p2, and p3) while a negative value of (P%) implies corrosion simulation or corrosion acceleration.

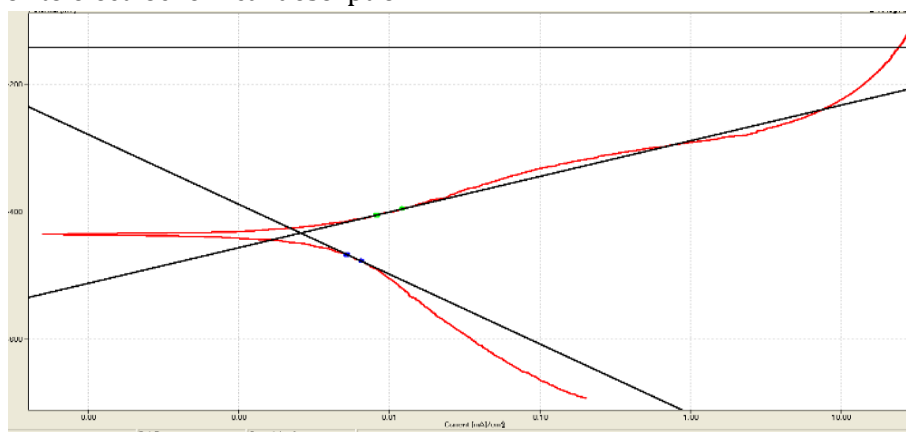
For the best compound (p3) in aqueous phase, the results indicated the (P%) change from 50% to 97.5% with 0.01N to 0.05 N of sulfuric acid with concentration of 50 ppm to 100 ppm, as shown in Table 4.

TABLE 4 Values of the open circuit potential (OCP), the corrosion potential (E_{corr}), the corrosion current densities (I_{corr}) p%, for low carbon steel compound p3

Condition	Conc.	Temp. K	OCP, V	E, V	$I, A/cm^2 (x10^{-6})$	W.L. g/m ² /d	Penetration mm.y	P%	θ
H ₂ SO ₄ ONLY	0.05N	338	-0.539	-0.5391	120.40	31.02	1.40	-	-
P3+0.05N H ₂ SO ₄	50ppm	338	-0.501	-0.4805	35.54	8.89	41.30	50.335	0.503
P3+0.01N H ₂ SO ₄	75ppm	338	-0.477	-0.4765	24.25	6.06	28.1	89.67	0.896
P3+0.01N H ₂ SO ₄	100ppm	338	-0.488	-0.4332	15.86	2.708	18.9	97.56	0.975

The obtained results indicated that the variation of the Tafel slopes and the corresponding transfer coefficients could be interpreted in terms of the variation in the nature of the rate-determining step from charge transfer process to either chemical-desorption or to electrochemical desorption.

The obtained values of the cathodic β_c and anodic β_a Tafel slopes and the cathodic α_c and anodic α_a of transfer coefficients differed after synthesizing new compounds that indicated their effects on the metal dissolution and subsequently the whole corrosion process.

**FIGURE 8** Tafel curve for p3= $92.25 - 2.58 / 92.25 = 97.56\%$

$E_{\text{corr}} = -433 \text{ mV}$
 $I_{\text{corr}} = 2.58 \mu\text{A}/\text{cm}^2$
Wt loss rate = 27.084 MPY

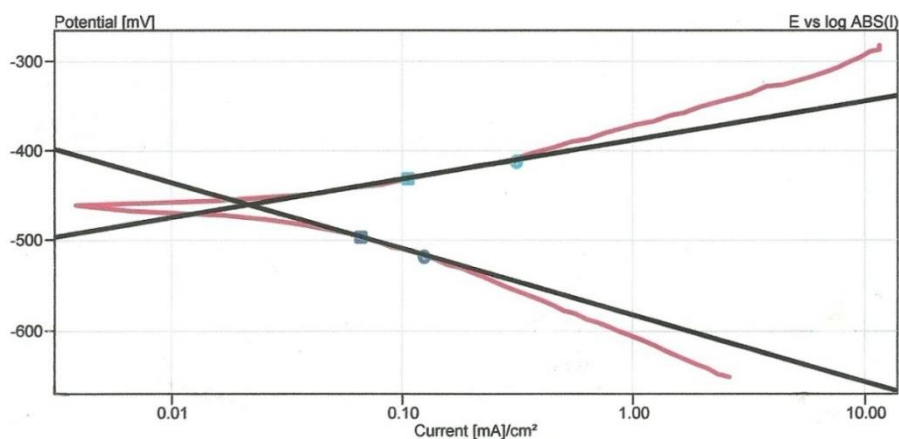


FIGURE 9 Tafel curve for $p_2=100x (92.25-17.25)/92.25 =81.79\%$

$E_{corr} = -460.4 \text{ mV}$
 $I_{corr} = 17.25 \mu\text{A}/\text{cm}^2$
 $W.LOSS = 26.076 \text{ mpy}$

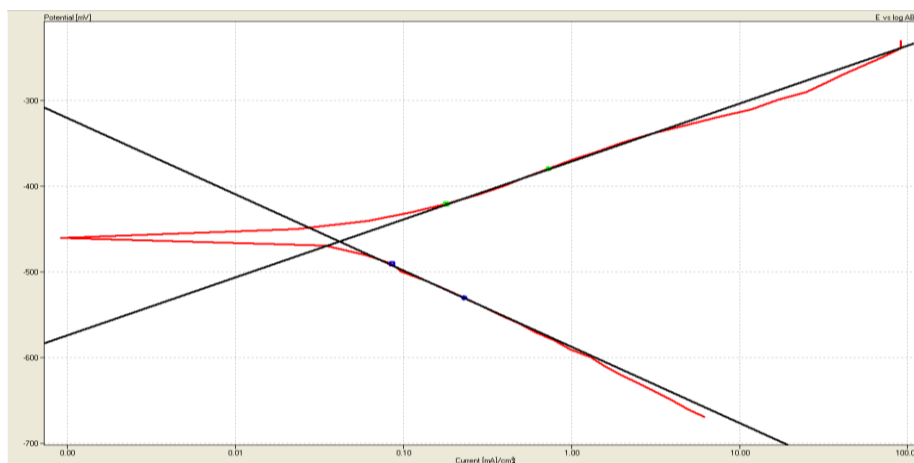


FIGURE 10 Tafel curve for $p_1=100x (92.25-16.72)/92.25=81.30\%$

$E_{corr} = -464.2 \text{ mV}$
 $I_{corr} = 41.72 \mu\text{A}/\text{cm}^2$
 $Wt \text{ loss rate} = 23.133 \text{ MPY}$

P1, P2, and P3 compounds were adsorbed through the electrostatic interactions between the positively charged nitrogen atom and the negatively charged metal surface. Thus, the better performance of the tested compounds in acidic solutions can be related to the specific adsorption of anion having a smaller degree of hydration, such as chloride ions, which is expected to be more pronounced. Being specifically adsorbed, they created an excess

negative charge toward the solution and favor more adsorption of the cations[13].

Scanning Electron Microscopy (SEM)

The SEM micrograph of the corroded carbon steel in 0.01N H₂SO₄ in the presence and absence of (P2) are shown in Figure (11 A–B). In Figure 11A (absence of inhibitor) of bare carbon steel surface, notches and defects were observed, while a layer of closely packed film

was obtained in Figure 11 B, and the surface was free from pits and it was smooth. It was concluded from Figure 11 A-B that corrosion does not occur in presence of (P2) and hence corrosion was inhibited strongly when the

inhibitor was present in the solution, then scanning electron microscopy observation confirmed the existence of adsorbed protective film on the carbon steel surface.

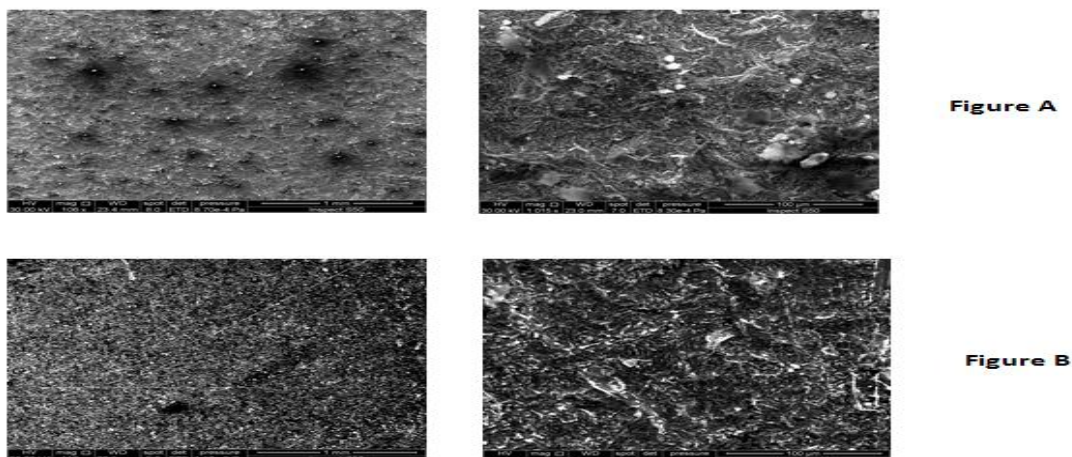


FIGURE 11 Surface characterization by SEM for carbon steel in 0.1N H₂SO₄

- (A) in the absence of inhibitor P2
(B) in the presence of inhibitor P2

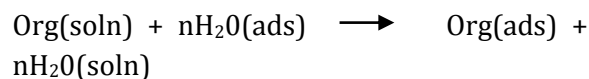
Conclusion

All prepared compounds in the present work had the ability to be miscible with water and hydrocarbon solvents, demonstrating that the material prepared is a surface-active compound, characterized by a length of chain on the one hand and its proximity to the two parties, one hydrophilic and the other hydrophobic.

The variety of melting points of the raw materials (long chain alcohols and long chain alkyl halides) from the melting point of the product compounds was meant to complete the chemical reaction and prepare the corrosion inhibitors. The mechanism of corrosion inhibitors is as follows: When polar nitrogen groups attach to a hydrocarbon chain donate electrons to the metal surface, a strong chemisorptive relationship is produced. The strength of protection is determined by this relationship.

The hydrocarbon component of the inhibitor is oil soluble and water resistant; the long hydrocarbon chain orients towards the solution and forms a hydrophobic network

(repels water from the metal surface). Organic molecules take the place of water molecules that have been desorbed:



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