Corrosion Inhibition of Carbon Steel in 1M HCl Solution by Schiff Base Compounds Obtained from 1,*3*-Diaminopropane

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Abstract
Two types of Schiff bases were prepared throughout condensation of 1,3-diaminopropane with two different types from Salicyaldehyde and furfuraldehyde. The chemical structures of the prepared Schiff bases were confirmed by using elemental analysis and FT-IR spectra. The synthesized Schiff bases were evaluated as corrosion inhibitors for carbon steel in 1M HCl using different techniques such as weight loss, Potentiodynamic Polarization and Electrochemical Impedance Spectroscopy technique. The corrosion inhibition measurements of these inhibitors showed high protection of the carbon steel alloys against corrosion process in 1M HCl at different concentrations. Both thermodynamic and activation parameters were calculated and discussed. Polarization curves indicated that they are mixed type of the inhibitors. The discussion was correlated the efficient corrosion inhibition of these inhibitors to their chemical structures. The adsorption of Schiff bases obeyed Langmuir adsorption isotherm.

1. INTRODUCTION
An important procedure in regards to the corrosion of metals is the use of organic chemicals to protect the metal surface from aggressive environmental. They do so by adsorption and this is often largely a function of their molecular structure. Those inhibitors containing oxygen, sulpher or nitrogen are generally most effective[1]. Inhibitors are generally used to reduce corrosive attack of the acid solutions on metallic material[2, 3]. Organic inhibitors play significant role in reducing corrosion of industrial metals and alloys in acidic environment encountered during processing of metals and alloys[4, 5]. Heterocyclic compounds with various substituents are considered to be the effective corrosion inhibitors for metals in acid media[6–11]. Schiff bases are important class of ligands in coordination chemistry and find extensive applications in different fields[12].Several Schiff bases have recently been investigated as corrosion inhibitors for various metals

and alloys in acid media[13–18]. On the other hand, the obtained data show that these inhibitors act by adsorption on the surface of the metal/solution interface. This process can be done via: (i) electrostatic attraction between the charged metal and the charged inhibitor molecules, (ii) dipole-type interaction between unshared electron pairs in the inhibitor with the metal, (iii) electrons-interaction with the metal, and (iv) combination of all of the above[19]. If the adsorption process involves overlap of occupied ligand non-bonding orbital with metal empty inner d or f orbital, a coordinate type bond formed and the process is termed chemisorption[18]. This situation can arise in cases where the inhibitor molecules contain lone pairs of electrons, multiple bonds, or conjugated -type bond system[20–23].
 On the other hand, some organic inhibitors could react with metal ions resulting from the corrosion process to form analogous complexes spontaneously

that may decrease or increase the corrosion rate. However, studies about the effect of metal complexes as corrosion inhibitor for steel in acid solution appeared in the literature are extremely limited[24, 25].

The aim of the present work is prepared of Schiff bases then studies the corrosion behavior of carbon steel in 1M hydrochloric acid in absence and presence of a Schiff base.

2. Experimental
2.1. Materials
All chemicals used in this work were laboratory pure. All organic solvents were obtained as pure grade materials. 1,3-diaminopropane (Merck), 2-furancarboxaldehyde and salicyaldehyde from (Sigma Aldrich), and ethanol (Adweic).

2.2. Synthesis of Schiff base
The Schiff base was synthesized by mixing an ethanolic solution of 1,3-diaminopropane (0.5) mol with (1.0) mol of 2- furaldehyde or salicyaldehyde into a two necked round bottomed flask equipped with a magnetic stirrer, condenser and a thermometer. The reaction mixture was heated to 60-70°C for 6h and then the formed precipitate was filtered, washed several times and dried in a desiccators over calcium hydroxide, figure (1). The products were purified and its structure was confirmed by (FTIR) spectroscopy and elemental analysis.

2.3. Characterization of Schiff bases
FT-IR spectra of the superabsorbent composite were recorded on a Nicolet-380 Fourier Transform Infrared Spectrometer in a range of 4000-400 cm^{-1} using KBr pellets.
Elemental analyses were performed on an Elementary Vario EL III Carlo Erba 1108 analyzer.

2.4. Weight loss measurements
Weight loss measurements were carried out in a glass cell. The test specimens were used in the form of sheets of dimensions 7×3×0.4 cm, the specimens (its composition: Si, Sn, Mo, Zn, Cu, Ni, Mn, Cr, V, Ti, Al, S, C and Fe of the C-steel alloy are 0.913, 0.019, 0.103, 0.02, 0.135, 0.242, 0.955, 1.09, 0.078, 0.073, 0.570, 0.389, 0.133 and 95.28 respectively) were first mechanically polished by grinding with emery paper from 320 to 1200 grit to obtain a smooth surface degreased with acetone, then washed with double distilled water, and finally dried between two filter papers and weighed. The test specimens were suspended by suitable glass hooks at the edge of the basin, and under the surface of the test solution by about 1cm.
After specified time of immersion specimens were taken out of the test solution, then rinsed with

distilled water, dried as before and weighed again. The average weight loss at a certain time for each set of three samples was taken in mg cm^{-2} and recorded. All experimental were carried out at 25, 40, 55 and 70 ±1°C.

2.5. Electrochemical Techniques
2.5.1. Potentiodynamic Polarization
The electrical circuit used for determining the variation of electrode potential with the electrical current. The potentiodynamic current-potential curves were recorded by changing the electrode potential automatically with a scan rate 2 mV s^{-1} from a low potential of -800 to -300 mV (SCE). Before each run, the working electrode was immersed in the test solution for 30 min to reach steady state. All potentials were measured against SCE. Measurements were obtained using a Voltalab 40 Potentiostat PGZ 301combined with easy corrosion program (Voltamaster 4).

2.5.2. Electrochemical Impedance Spectroscopy (EIS)
Electrochemical impedance were obtained using a Voltalab 40 for all EIS measurements with a frequency range of 100 kHz to 50 mHz with a 4 mV sine wave as the excitation signal at open circuit potential. If the real part is plotted on the X-axis and the imaginary part is plotted on the Y-axis of a chart, we get a Nyquist Plot. The charge transfer resistance values (R_{ct}) was calculated from the difference in impedance at lower and higher frequencies.

3. RESULTS AND DISCUSSION
3.1. Confirmation the chemical structure of the synthesized inhibitors
3.1.1. FTIR spectra
The chemical structures of the prepared Schiff bases (S_1 and S_2) were confirmed by the FTIR spectroscopy and shown in figure (1a, b). The band at 3399 cm^{-1} represented the stretching vibrations of O-H or N-H broad bond. The band at 1624 cm^{-1} was corresponded to the stretching vibration of C=O and that at 1149 cm^{-1} was the vibration of C-N. The band at 2934 and 2842 cm^{-1} were ascribed to the CH$_2$, the band at 1381 cm^{-1} was the absorption of C-O, and that at 1014 cm^{-1} was ascribed to the -CH group. The band at 1501 ascribed to the C=C group. However, the bands at the band at 2920 cm^{-1} was attributed to -CH$_2$.

3.1.2. Elemental analyses
The elemental analyses (measured and calculated), molecular formula and molecular weight of the prepared Schiff bases were listed in table (1). The

analytical data are in good agreement with proposed stoichiometry of the Schiff bases.

3.2. Potentiodynamic polarization

Figures (3, 4) typical Tafel curves obtained for carbon steel in 1 M HCl with and without inhibitors were studies. The values of associated electrochemical parameters, i.e., corrosion potential (E_{corr}), corrosion current density (I_{corr}), cathodic Tafel slopes ($_c$), anodic Tafel slopes ($_a$) and percentage inhibition efficiency ($_p$ %) values were calculated from polarization curves and listed in Table (2). The inhibition efficiency ($_p$ %) was calculated from polarization measurements according to the relation given below:

$$_p \% = [(I_{corr} - I_{inh}) / I_{corr}] \times 100 \qquad (1)$$

Where, I_{corr} and I_{inh} are uninhibited and inhibited corrosion current densities, respectively. They are determined by extrapolation of Tafel lines to the respective corrosion potentials.

Figures (2,3), indicate that both anodic metal dissolution and cathodic reduction reactions were inhibited when the Schiff bases were added to the acid solution and this inhibition was more pronounced with increasing inhibitor concentration.

It was found that, the inhibitors slightly shift the corrosion potential (E_{corr}) in the cathodic direction, and the cathodic Tafel slopes ($_c$) and anodic Tafel slopes ($_a$) values were slightly changed, which confirms that surfactants act as mixed inhibitor. The data show that the corrosion current density (I_{corr}) values decrease with increasing the concentration of surfactant. Therefore, the corrosion inhibition efficiency of this compound depends on its concentration. It is clear that the presence of the inhibitors causes a markedly decrease in the corrosion rate, i.e. shifts the anodic curves to more positive potentials and the cathodic curves to more negative potentials. This may be ascribed to adsorption of the inhibitor over the corroded surface[3, 25]. Therefore, the prepared inhibitors can be classified as mixed-type inhibitor in 1M HCl.

3.3. Electrochemical impedance spectroscopy (EIS)

The corrosion behaviors of carbon steel in 1 M HCl in the presence and absence of the synthesized inhibitors were investigated using EIS at 25°C. Figures (5, 6) show that the Nyquist plots of carbon steel in 1 M HCl solution with and without various concentrations of inhibitors. It is clear from these plots that the impedance response of carbon steel has significantly changed after addition of the synthesized inhibitors in the corrosive media. This indicates that the impedance of an inhibited substrate increases with increasing concentration of inhibitor in 1 M HCl.

The charge transfer resistance values (R_{ct}) were calculated from the difference between impedance values at the lower and higher frequencies was suggested[26]. The double layer capacitance (C_{dl}) is obtained from the following equation[27]:

$$C_{dl} = 1 / (2 \; R_{ct} f \; (-Z''_{img})) \qquad (2)$$

Where, f ($-Z''_{img}$) is the frequency at maximum imaginary component of the impedance and R_{ct} is the charge transfer resistances.

The inhibition efficiency percentage ($_Z$ %) for corrosion of carbon steel is calculated from the following equation[27]:

$$_Z \% = \{(R_{ct(inh)} - R_{ct}) / R_{ct(inh)}\} \times 100 \qquad (3)$$

Where, R_{ct} and $R_{ct(inh)}$ are the charge transfer resistance values in the absence and presence of inhibitors for carbon steel in 1M HCl, respectively.

Various impedance parameters such as charge transfer resistance (R_{ct}), double layer capacitances (C_{dl}) and inhibition efficiency ($_Z$ %) were calculated and listed in Table (3). On the other hand, the data in table (3) illustrate the R_{ct} values increase, while C_{dl} values decrease with the inhibitor concentration increment. This increase in the efficiency of inhibition may be due to an increase in the surface coverage of inhibitor.

Decrease in C_{dl}, may be also due to a decrease in local dielectric constant and/or increase in the thickness of the electrical double layer, which suggests that, the inhibitor molecules act by adsorption at the metal/solution interface[15, 18, 26]. Thus, the change in C_{dl} values is a result of the gradual replacement of water molecules by the adsorption of the organic molecules on the metal surface and decreasing the extent of metal dissolution[26, 27]. Results obtained from EIS can be interpreted in terms of the equivalent circuit of the electrical double layer.

3.4. Weight loss measurements
3.4.1. Effect of inhibitor concentration

Weight loss data of carbon steel in 1 M HCl in the absence and presence of different concentrations of inhibitors was calculated and was used to calculate inhibition efficiencies ($_w$%) according to the following equation:

$$_w \% = ((W_{corr} - W_{corr(inh)}) / W_{corr}) \times 100 \qquad (4)$$

Where, W_{corr} and $W_{corr(inh)}$ are the weight loss of carbon steel in the absence and presence of the inhibitors, respectively.

The inhibition efficiency as a function of concentrations of prepared inhibitors at 25°C were calculated and shown in table (4). The results show that the inhibition efficiencies of the prepared

compounds increase with increasing inhibitor concentrations.

The order of the inhibition efficiency from the weight loss measurements are in good agreement with those obtained from the EIS and potentiodynamic polarization methods but with different absolute values, probably due to the different experimental conditions.

3.4.2. Effect of temperature

The effect of temperature on the inhibition efficiency in the temperatures ranges 25-70 °C in 1 M HCl in the absence and presence of 500 ppm concentration of inhibitors (S1 and S2) were studied by weight loss technique. The corrosion parameters such as weight loss, rate of corrosion (K), surface coverage ( ) and inhibition efficiency (w %) for 500 ppm from inhibitors were listed in Table (5). On the other hand, the data in table (5) illustrate that, by increasing the temperature the corrosion rate increases rapidly in absence and presence of inhibitors also. This indicates that, the inhibition efficiency of the inhibitors decrease by increasing temperatures.

3.5. Adsorption isotherm

Compounds inhibit the corrosion of carbon steel by adsorption on the metal–solution interface. The adsorption provides the information about the interaction among the adsorbed molecules themselves as well as their interaction with the electrode surface[28-30]. The degrees of surface coverage ( ) for different concentration of inhibitors in acidic media have been evaluated from weight loss measurements by using the following equation[16, 27, 28, 31]:

$$ = (W_{corr} - W_{corr(inh)}) / W_{corr}$$
$$(5)$$

Where, W_{corr} and $W_{corr(inh)}$ are weight loss of carbon steel in the absence and presence of inhibitor, respectively.

The surface coverage values ( ) were tested graphically for fitting a suitable adsorption isotherm:

$$C/ = C + (1 / K_{ads}) \qquad (6)$$

Where, C is the inhibitor concentration, his fraction of the surface covered, K_{ads} is the equilibrium constant of the inhibitor adsorption process. In these cases, the plots of C/ versus C yield straight lines, clearly proving that the adsorption of the used inhibitors on the carbon steel surface obeys Langmuir isotherm and shown in figures (7, 8).

3.6. Thermodynamic parameters

Generally, the organic molecules inhibit corrosion by adsorption at the metal-solution interface and the adsorption depends on the molecule's chemical composition, the temperature and the electrochemical potential at the metal–solution interface[26, 29].

Thermodynamic parameters play an important role in understanding the inhibition mechanism. The adsorption heat can be calculated according to the Van't Hoff equation[32, 33]:

$$\ln K_{ads}= (- H_{ads} /RT) + C$$
$$(7)$$

Where, H_{ads} and K_{ads} are the adsorption heat and adsorptive equilibrium constant, respectively and C is constant.

It should be noted that (- H_{ads} /RT) is the slope of the straight line

$\ln K_{ads}$ - 1/T according to Eq (7) and the molecular weight of inhibitor is also a positive constant, so the value of adsorption heat does not change with the unit of adsorptive equilibrium constant.

The straight line

$\ln K_{ads}$ - 1/T was shown in Figures (9, 10).

Because the experiment was carried out at the standard pressure and the solution concentration is so low that it is close to standard condition, the adsorption heat (H_{ads}) can be approximately regarded as the standard adsorption heat under such experimental conditions[34]. The standard adsorption entropy (S_{ads}) was obtained using the thermodynamic basic equation:

$$G_{ads} = H_{ads} - T S_{ads} \qquad (8)$$

The values of G_{ads}, H_{ads} and S_{ads} were calculated and listed in table (6). The positive values of H_{ads} showed that, the adsorption of the inhibitors is an endothermic process[35].

Values of S_{ads} was listed in Table (6) have a positive sign, as was expected, since the endothermic adsorption process is always accompanied by an increase of entropy. This positive value can be explained such that, the adsorption of the prepared compounds from the aqueous solution can be regarded as quasi-substitution process between the organic compound in the aqueous phase and water molecules at the carbon steel surface[36]. This increase of entropy was the driving force of the adsorption of inhibitor onto carbon steel surface[37]. These S_{ads} values indicate that an increase in disordering takes place while going from reactants to the metal / adsorbed species reaction complex.

CONCLUSIONS

The following main conclusions are drawn from the present study:

1. All studied schiff bases are excellent inhibitors and act as mixed type inhibitors for carbon steel corrosion in hydrochloric acid solution. Corrosion inhibition efficiencies are in the order of $(S_1 > S_2)$.
2. Inhibition efficiencies increase by an increase in inhibitor concentration and a decrease in temperature.

3. The adsorption of all additives obeys the Langmuir adsorption isotherm. The negative values of enthalpies identifies that the adsorption is a physical adsorption type.
4. All entropy parameters for adsorption of inhibitors molecules on steel are negative indicating that the entropy of inhibitor

molecules in solution phase is higher than in solid phase.
5. Activation energy increases with addition of inhibitors. It is shown that physisorption occurs in the first stage.
6. The results obtained from different experimental studies are in a good agreement
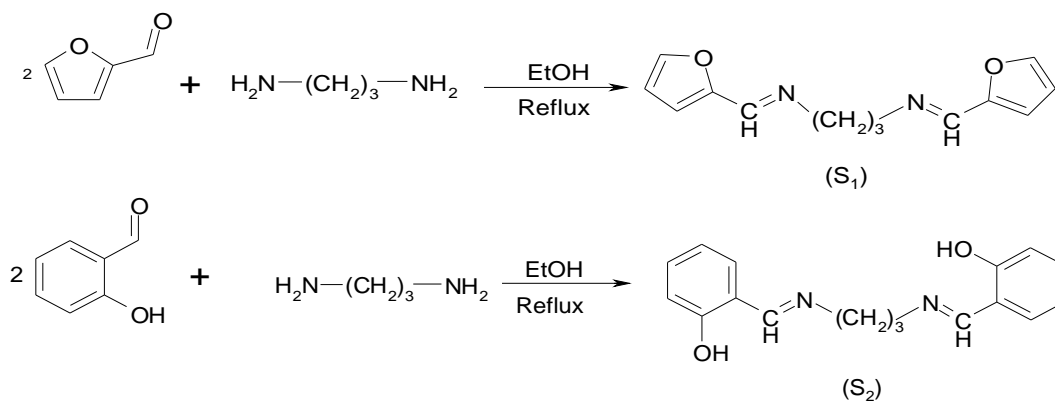

Figure 1
Synthesis scheme for the preparation of (S$_1$) N,N-bis-(furaldehyde)-1,3-diaminopropane and (S$_2$) N,N-bis-(salicyaldehyde)-1,3-diaminopropane
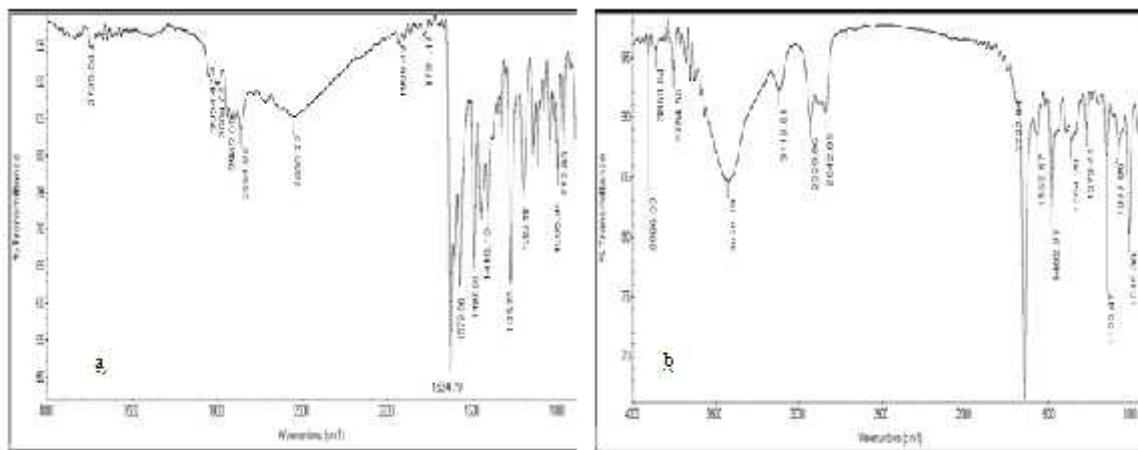

Figure 2
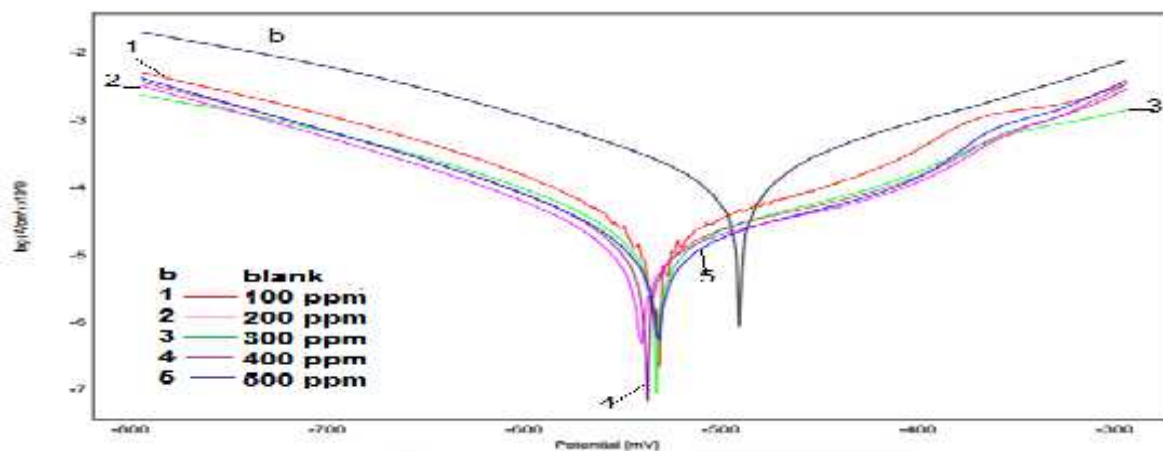FTIR spectrum for a) S$_1$ b) S$_2$

Figure 3
Potentiodynamic polarization curves for the carbon steel in 1M HCl without
and with Schiff Base (S_1) at 25 ^{o}C
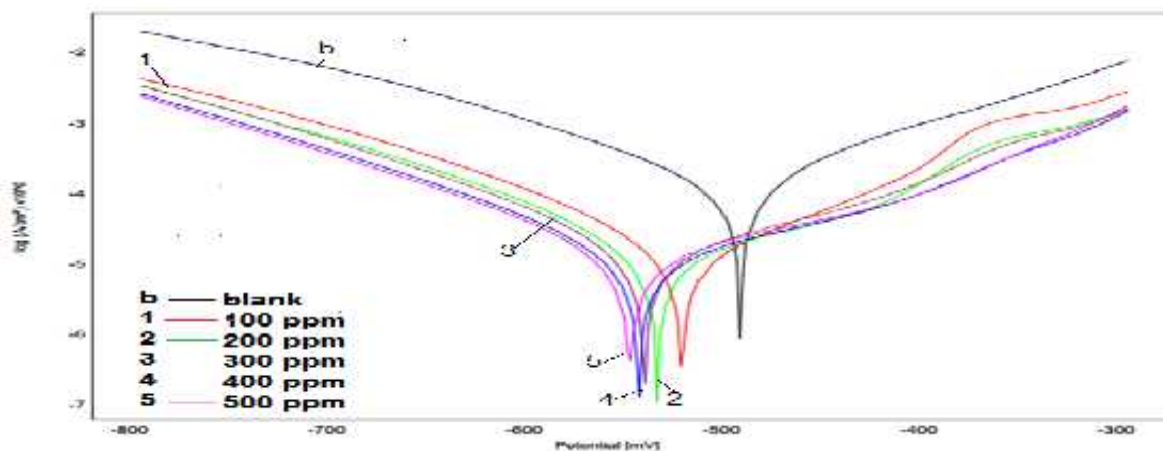

Figure 4
Potentiodynamic polarization curves for the carbon steel in 1M HCl without
and with Schiff Base (S_2) at 25 ^{o}C

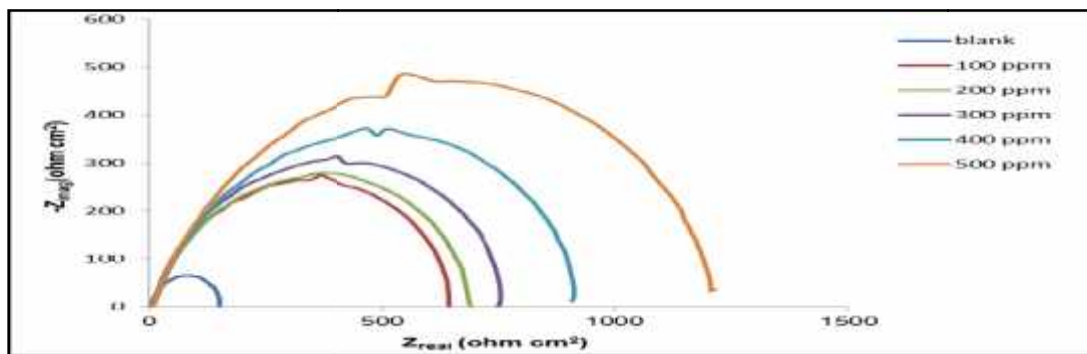**Figure 5**
Nyquist plots for the carbon steel in 1M HCl in the absence and presence of different
concentrations of compound S_1.

Figure 6
Nyquist plots for the carbon steel in 1M HCl in the absence and presence
of different concentrations of compound S_2.

Figure 7
Langmuir isotherm adsorption model on the carbon steel surface of compound S_1
in 1M HCl at different temperatures.

Figure 8
Langmuir isotherm adsorption model on the carbon steel surface of compound S_2
in 1M HCl at different temperatures.

Figure 9
Arrhenius plots (ln k vs. 1/T) for carbon steel dissolution in the absence and the presence of different concentrations
of compound S_1 in 1 M HCl solution.

Figure 10
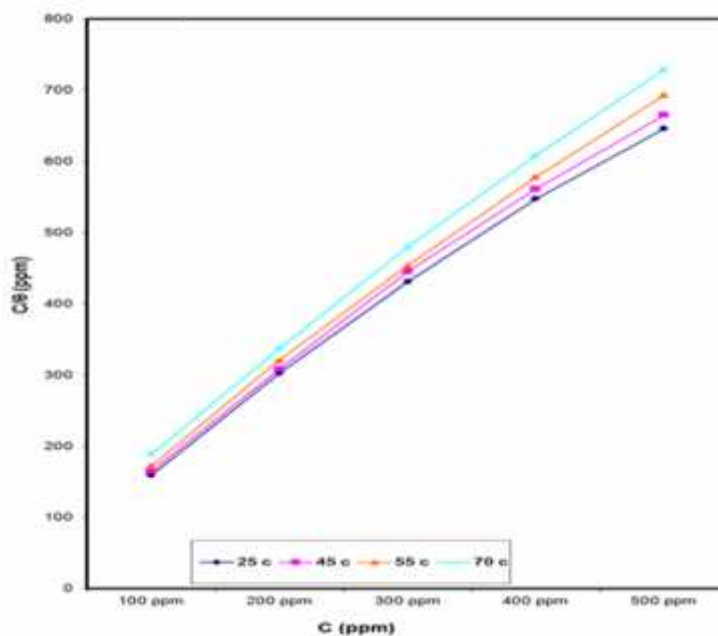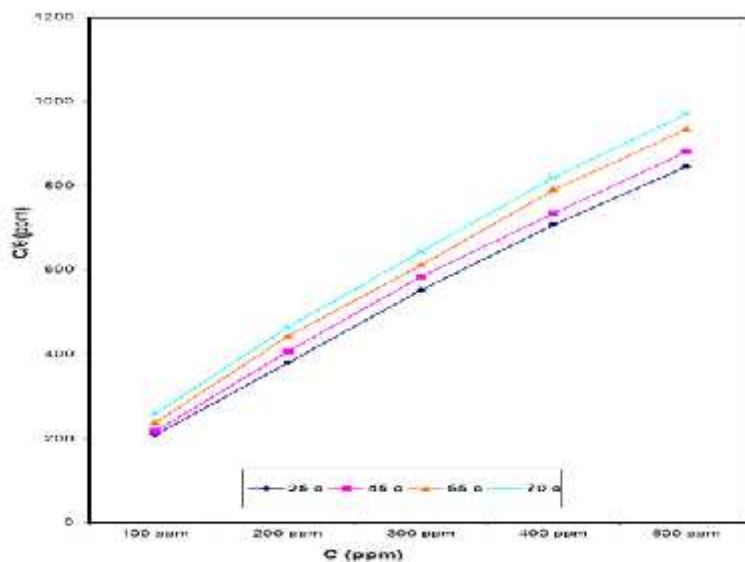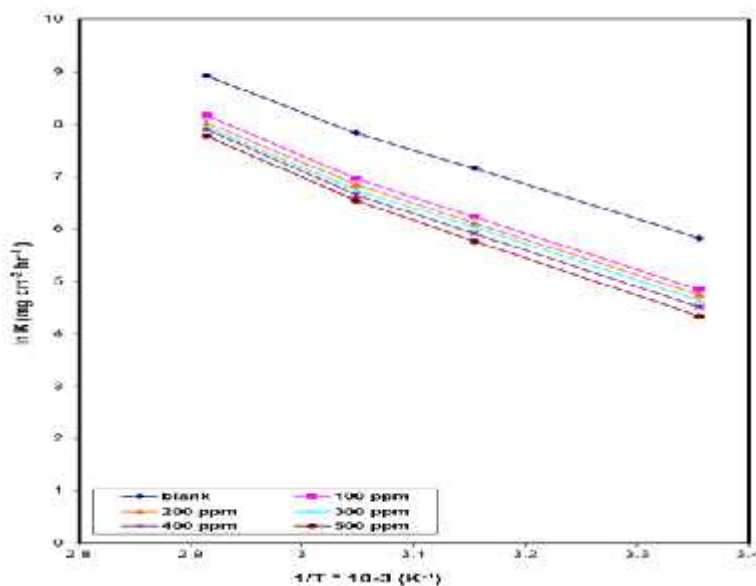Arrhenius plots (ln k vs. 1/T) for carbon steel dissolution in the absence and the presence of different concentrations
of compound S_2 in 1 M HCl solution.
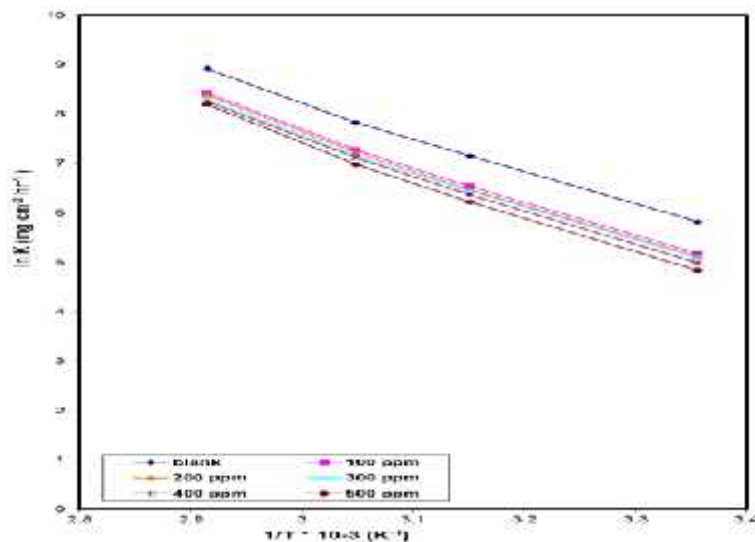
Table 1
Molecular formula, molecular weight and elemental analyses of the prepared Schiff bases (S_1, S_2)

Samples	M wt (g/mol)	M Formula	C %		H %		N %	
			Cal.	found	Cal.	found	Cal.	found
S_1	230	$C_{13}H_{14}N_2O_2$	67.83	67.79	6.09	6.07	12.17	12.06
S_2	282	$C_{17}H_{18}N_2O_2$	72.34	72.29	6.38	6.34	9.92	9.84

Table 2
Polarization parameters of the inhibitors S_1 and S_2 at temperature 25 °C

Inhibitor	Conc. (ppm)	E_{corr} mV (vs. SCE)	I_{corr} mA cm^{-2}	R_p K cm^2	a mV dec^{-1}	c mV dec^{-1}	p %
Blank	0.0	-501.6	0.1756	0.17	124.1	-131.2	0.0
	100	-557.7	0.0661	0.67	134.4	-121.5	62.2
	200	-557.7	0.0602	0.75	136.2	-121.5	65.7
S_1	300	-558.4	0.0598	0.76	135.7	-119.0	66.6
	400	-550.5	0.0341	1.18	128.7	-118.7	80.5
	500	-549.7	0.0294	1.46	126.6	-119.1	83.3
	100	-544.8	0.0767	0.66	134.5	-139.0	56.3
	200	-550.9	0.0472	0.95	141.5	-125.9	73.0
S_2	300	-557.9	0.0431	0.96	134.5	-122.4	75.4
	400	-555.1	0.0429	0.98	136.6	-120.8	75.5
	500	-553.3	0.0403	1.01	136.0	-119.7	77.1

Table 3
EIS parameters for corrosion of carbon steel in 1M HCl in the absence and presence of different concentrations of Inhibitors S_1 and S_2 at temperature 25 °C

Inhibitor	Conc., ppm	R_s cm^2	R_{ct} K cm^2	C_{dl} µF cm^{-2}	, %
Blank	0.0	4.425	0.1406	80.56	0.0
S_1	100	8.452	0.6428	49.51	78.1
	200	8.577	0.6854	46.44	79.5
	300	8.891	0.758	41.98	81.45
	400	11.29	0.918	38.83	84.68
	500	13.05	1.218	36.55	88.45
S_2	100	8.359	0.6357	50.10	77
	200	8.962	0.8429	47.29	83.3
	300	9.397	0.8695	43.24	83.8
	400	9.544	0.8744	41.50	84
	500	9.592	0.9114	39.11	84.5

Table 4
Surface coverage and percentage inhibition efficiency for carbon steel in 1M HCl in the absence and presence of different concentrations of Inhibitors S_1 and S_2 at temperature 25 °C

Inhibitor	Conc., ppm	Weight loss, mg	K, mg/cm^2.h		w %
Blank	0.0	0.115	338.8	----	----
S_1	100	0.043	126.7	0.6261	62.6
	200	0.039	115.2	0.6609	66.1
	300	0.035	103.1	0.6957	69.6
	400	0.031	91.3	0.7304	73.0
	500	0.026	78.6	0.7739	77.4
S_2	100	0.048	141.4	0.5826	58.3
	200	0.043	129.6	0.6261	62.6
	300	0.036	106.1	0.6870	68.7
	400	0.033	97.2	0.7131	71.3
	500	0.029	85.4	0.7478	74.8

Table 5
Effect of temperature on carbon steel corrosion in absence and presence of 500 ppm of S_1 and S_2 inhibitors concentration in 1M HCl

Samples	Temp., °C	Weight loss, mg	K, mg/cm^2.h		w, %
Blank	25	0.115	318.17	--	--
	40	0.435	1281.536		
	55	0.848	2498.258		
	70	2.543	7491.828		
S_2	25	0.026	78.621	0.7739	77.4
	40	0.108	129.627	0.7517	75.2
	55	0.234	315.228	0.7241	72.4
	70	0.799	518.506	0.6858	68.6
S_2	25	0.029	85.445	0.7478	74.8
	40	0.119	359.436	0.7264	72.6
	55	0.293	863.197	0.6545	65.5
	70	1.026	3025.625	0.5965	59.7

Table 6
Thermodynamic parameters of adsorption on carbon steel surface in 1M HCl containing different concentrations of compounds at different temperatures

Inhibitor	Temp., °C	$K_{ads} \times 10^{-3}$ M^{-1}	G_{ads} kJ mol^{-1}	H_{ads} J mol^{-1}	S_{ads} J mol^{-1} K^{-1}
S$_1$	25	11.945	-33.25		111.65
	40	11.562	-35.37	61.94	111.43
	55	11.543	-36.46		111.35
	70	10.087	-37.74		110.22
S$_2$	25	10.560	-32.91		110.62
	40	9.060	-34.71	60.94	109.34
	55	7.621	-35.33		107.89
	70	6.347	-36.42		106.37

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