

Research Article

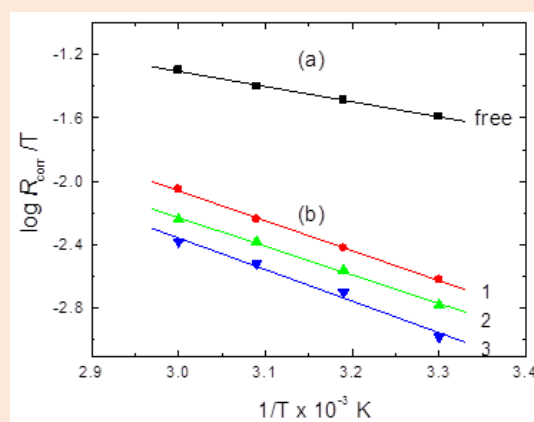
Corrosion Inhibition of Carbon Steel by Some Nonionic Surfactants from Sulfonamide Derivatives in Sulfuric Acid

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Makkah Al-Mukaramah, Saudi Arabia**Abstract**

The effect of three compounds of nonionic surfactants from sulfonamide derivatives on the corrosion of carbon steel Type L-52 in 0.5M H₂SO₄ solution was investigated using chemical methods, e.g. weight loss and electrochemical methods e.g. galvanstatic polarization and electrochemical impedance measurements. The percentage inhibition efficiency was found to increase with increasing concentration of inhibitor, number of ethylene oxide unit and with decreasing temperature. The polarization measurements showed that the studied inhibitors are acting as mixed type. The inhibiting effect of these compounds due to the adsorption of these compounds on the steel surface. The adsorption process was found to obey Temkin adsorption isotherm. The effect of temperature on the rate of corrosion in the absence and presence of these compounds was also, studied. Some activated thermodynamic parameters were computed and discussed. From impedance data it is found that the corrosion of C-steel is controlled by charge transfer at all concentration of inhibitors.

Keywords: Carbon steel, sulfonamide nonionic surfactants, inhibitors, adsorption, thermodynamic parameters.

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The corrosion of carbon steel is a fundamental academic and industrial concern that has received a considerable amount of attention. Acid solutions are widely used for the removal of undesirable scale and rust in several industrial processes. Hydrochloric acid is widely used in the pickling process of steel. The use of inhibitors is one of the most practical methods for protection against corrosion, especially in acid solutions to prevent metal dissolution and acid consumption [1]. Most of the reported acid inhibitors for steel corrosion are synthetic organic compounds containing hetero atoms (nitrogen, sulfur, oxygen and phosphorus), aromatic rings or triple bonds [2–15]. The inhibiting efficiency of organic compounds is strongly dependent on the structure and chemical properties of the layer adsorbed on the metal surface. The strength of adsorbed layer is related to the functional groups connected to aromatic ring [16]. The adsorption of organic compounds depends mainly on the electronic structure of the molecule and that the inhibition efficiency increases with the increase in the number of aromatic rings [17].

The present study is aimed to investigate the effect of three compounds nonionic surfactants from sulfonamide derivatives as inhibitors for the corrosion of carbon steel Type L52 in 0.5M H₂SO₄ solution using weight loss, galvanostatic polarization and electrochemical impedance spectroscopy measurements. The effect of rising

temperature on the corrosion of carbon steel in 0.5M H₂SO₄ containing 500ppm of the inhibitors used was also studied and some thermodynamic parameters were computed.

Experimental Methods

Carbon steel of type L52 was used in this study has the chemical composition C 0.2%, Mn 0.6%, P 0.04 %, Si 0.003% and the remainder iron. Coupons of the steel, with dimensions 3.0 x 2. 0 x 0.1 cm were used for weight-loss measurements. For galvanostatic potentiodynamic polarization and electrochemical impedance spectroscopy measurements, a cylindrical rod embedded in araldite with exposed surface area of 0.36 cm² was used. The electrodes were polished with different emery papers, degreased with acetone and rinsed with distilled water, before inserting in the test solution. For weight loss measurements, Weight-loss measurements were carried out as described elsewhere [18]. The percentage inhibition efficiency (%I.E) and a surface coverage (θ) which represents the part of the metal surface covered by the inhibitor molecules were calculated using the following equations.

$$\% \text{I.E} = \left[1 - \frac{R_{add}}{R_{free}} \right] 100 \quad (1)$$

$$\theta = \left[1 - \frac{R_{add}}{R_{free}} \right] \quad (2)$$

where, R_{add} and R_{free} are the corrosion rates of 1018 C-steel with free and inhibited acid solutions, respectively. The corrosion rate R_{corr} (mg cm⁻² min⁻¹) was calculate from the slopes of the straight line obtained.

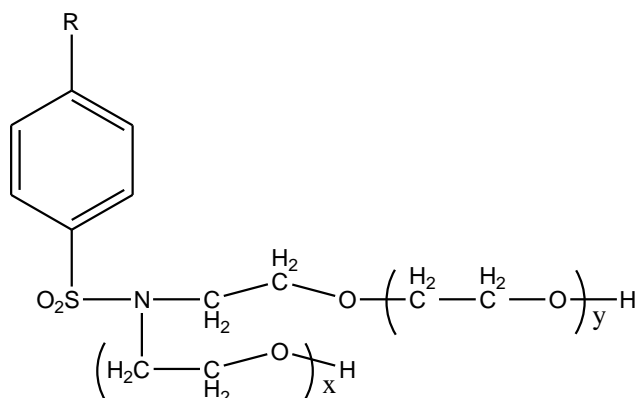
Galvanostatic polarization studies were carried out using a (PS remote) potentiostat with Zm PS6 software for calculation of corrosion parameters. Three compartment cell with a saturated calomel reference electrode was used. The inhibition efficiency IE was calculated using the following equation:

$$\text{IE} = \left[1 - \frac{I_{corr.add}}{I_{corr.free}} \right] 100 \quad (3)$$

where I_{corr. add} and I_{corr. free} are the corrosion current density in free and inhibited acid, respectively.

The electrochemical impedance spectroscopy measurements were carried out at open circuit potential (E_{ocp}) in the frequency range from 10 kHz to 100 mHz with signal amplitude perturbation of 5 mV by using a computer-controlled potentiostat (Auto Lab 30, Metrohm).

Three nonionic surfactants derived from sulfonamide were prepared using a method described elsewhere [19]. They have the general formula:



where, R is C_8H_{17} and $x + y$ is the total number of moles of ethylene oxide added and equal to 5, 7, 9 for compounds I, II and III

Results and Discussion

Weight loss measurements

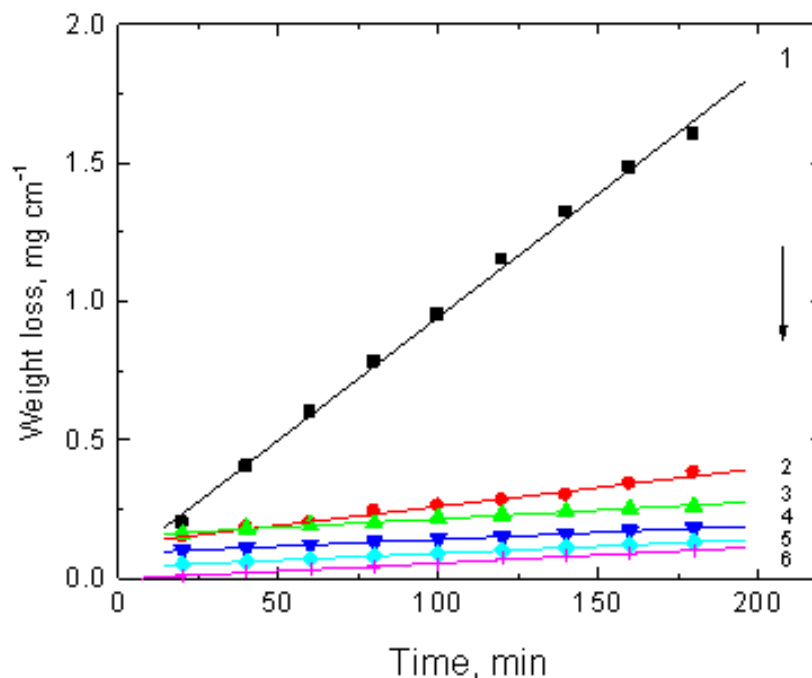
Figure 1 shows the weight loss-time curves for carbon steel in 0.5M H_2SO_4 solution in the absence and presence of different concentrations of compound III as an example of the studied inhibitors. Similar curves were obtained for other two compounds (not shown). As shown from these figures, by increasing the concentration of these compounds, the weight loss of carbon steel samples are decreased. This means that the presence of these compounds retards the corrosion of carbon steel in 0.5M H_2SO_4 or in other words, these compounds act as an inhibitor.

The linear variation of weight loss with time in uninhibited and inhibited 0.5M H_2SO_4 indicates the absence of insoluble surface films during corrosion. In the absence of any surface films, the inhibitors are first adsorbed onto the metal surface and there after impede the corrosion processes [20].

The calculated values of rate of corrosion, R_{corr} , percentage inhibition efficiency %IE and surface coverage (obtained at different inhibitor concentrations are listed in **Table 1**. Inspection of Table 1 reveals that as the inhibitor concentration is increased, the weight loss decreases while %IE and (increases. This behavior could be attributed to the increased surface coverage due to the increase of the number of adsorbed molecules at the metal surface. At one and the same inhibitors concentration, the percentage of inhibition efficiency decreases in the following sequences: compound III > compound II > compound I.

Effect of temperature

The effect of temperature on the corrosion of carbon steel in 0.5M H_2SO_4 solution devoid of and containing 500 ppm of the inhibitors was investigated in the temperature range of 30-60 °C is using weight loss measurements. Similar curves in Figure 1 were obtained (not shown).

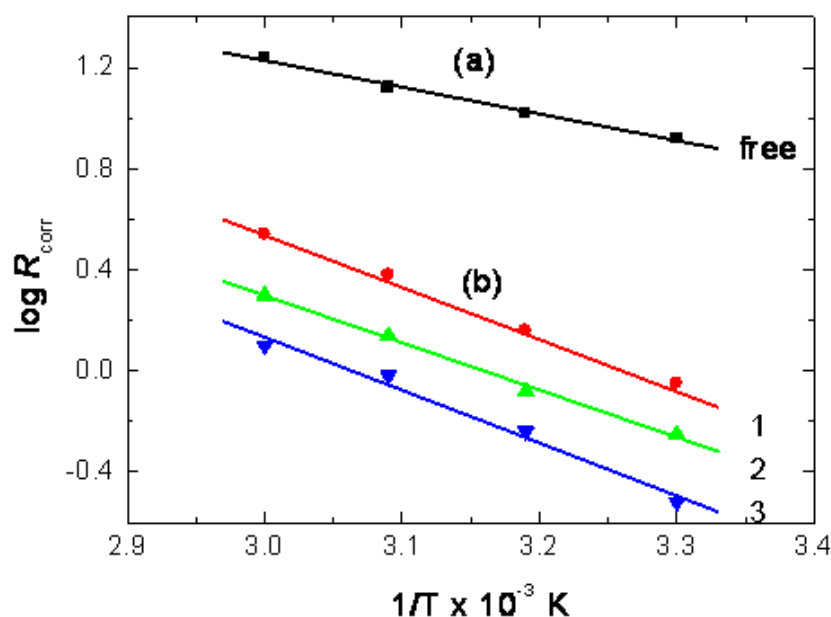


(1) 0.0 ppm, (2) 100 ppm, (3) 200 ppm, (4) 300 ppm, (5) 400 ppm, (6) 500 ppm

Figure 1 Weight loss–time curves for C-steel corrosion in 0.5 M H_2SO_4 in the absence and presence of different concentrations of compound III at 30°C.

Table 1 The effect of increasing inhibitor concentrations on the corrosion of C- steel in 0.5M H₂SO₄ solution using weight loss measurements.

	$R_{\text{corr}} \times 10^{-4} \text{ mg cm}^{-1} \text{ min}^{-1}$	% I.E	θ
1 M HCl + compound I			
0 ppm compound I	9.96	-	-
100 ppm compound I	3.16	68.27	0.683
200 ppm compound I	2.36	76.30	0.763
300 ppm compound I	2.11	78.81	0.788
400 ppm compound I	1.28	87.14	0.871
500 ppm compound I	0.88	91.16	0.912
1 M HCl + compound II			
100 ppm compound II	2.42	75.70	0.757
200 ppm compound II	1.86	81.32	0.813
300 ppm compound II	1.22	87.75	0.877
400 ppm compound II	0.82	91.76	0.918
500 ppm compound II	0.55	94.47	0.945
1 M HCl + compound III			
100 ppm compound III	2.12	78.71	0.787
200 ppm compound III	1.62	83.73	0.837
300 ppm compound III	1.03	89.65	0.896
400 ppm compound III	0.66	93.37	0.934
500 ppm compound III	0.32	96.78	0.968

a) 0.5 MH₂SO₄, b) 0.5 MH₂SO₄ + 500ppm of inhibitor, 1) Compound I, 2) Compound II, 3) Compound III**Figure 2** Arrhenius plot ($\log R_{\text{Corr}}$. And $1/T$) for carbon steel in 0.5 MH₂SO₄ in the absence and presence of 500 ppm of inhibitor

As the temperature increases, the rate of corrosion increases and hence the inhibition efficiency of the additive decreases. This is due to the desorption is aided by increasing the temperature. This behavior proves that the adsorption of inhibitors on C-steel surface occurs through physical adsorption. The activation energy (E_a) of the corrosion process was calculated using Arrhenius' equation [24].

$$\log R_{\text{corr}} = \log A + \frac{E_a}{2.303 RT} \quad (4)$$

Where, record is the rate of corrosion of weight loss, A is Arrhenius constant, R is the gas constant and T is the absolute temperature.

Figure 3 represents a plot of $\log R_{\text{corr}}$ against $1/T$ for uninhibited and inhibited 0.5 M H_2SO_4 containing 500 ppm of the studied compounds. The values of E_a can be obtained from the slope of the straight lines were found to be 20.56 KJ Mol^{-1} in 0.5 M H_2SO_4 and 26.66, 28.32 and 30.73 KJ Mol^{-1} for compound I, II and III, respectively.

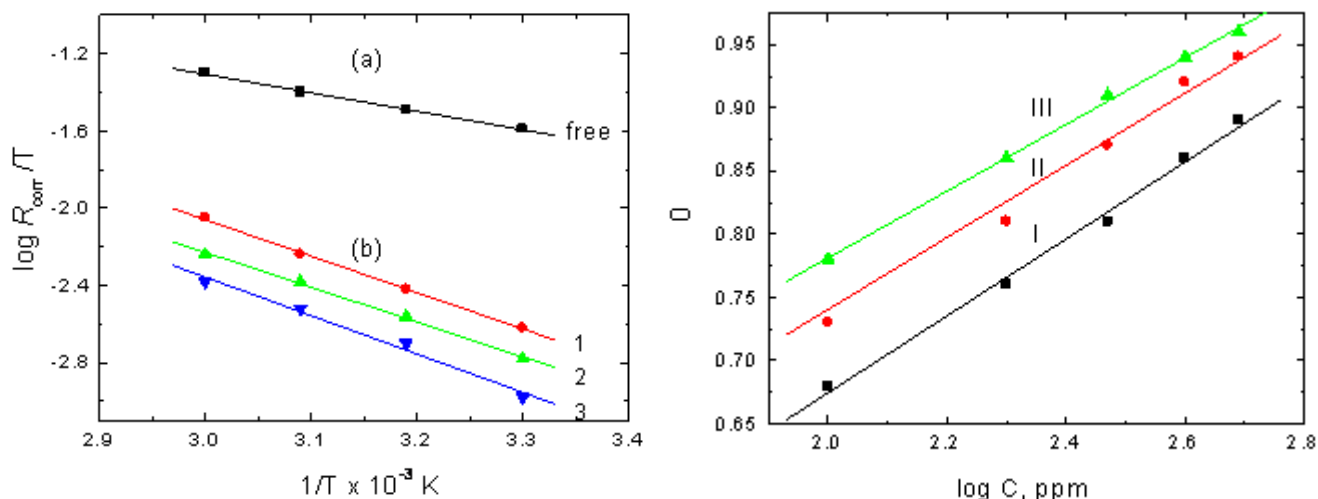
The increase of the activation energy in the presence of inhibitors is attributed to an appreciable decrease in the adsorption process of the inhibitors on the metal surface with increase of temperature and a corresponding increase in the reaction rate because of the greater area of the metal that is exposed to the acid [24]. The entropy of activation (ΔS^*) and the enthalpy of activation (ΔH^*) for dissolution of 1018 carbon steel in 1M HCl in presence of 1000 ppm of each used compound were obtained by applying the transition state equation [15]

$$R_{\text{corr}} = (RT/Nh) \exp (\Delta S^*/R) \exp (-\Delta H^*/RT) \quad (5)$$

Where, N is Avogadro's number, h is Planck's constant. A plot of $\log (R_{\text{corr}}/T)$ vs. $(1/T)$ (**Figure 4**) should give a straight line with a slope of $(-\Delta H^*/2.303 R)$ and an intercept $[\log (R/NH) -\Delta S^o /2.303R]$. The values of ΔH^* obtained from the slope.

On the straight line equal 16.45 KJ Mol^{-1} in 1M HCl and equal 17.83, 18.98 and 20.41 KJ Mol^{-1} in presence of compound I, II and III, respectively. The values of ΔH^* are different for studied compounds which mean that their structure affect the strength of its adsorption on the metal surface. The values of ΔS^* calculated from the intercept of the straight line were found to 302.67 $\text{J Mol}^{-1} \text{K}^{-1}$ in 1M HCl and 323.67, 429.84 and 330.91 $\text{J Mol}^{-1} \text{K}^{-1}$ for compound I, II and III, respectively.

The negative values of ΔS^* in the absence and presence of the inhibitors implies that the activated complex is the rate determining step and represents association rather than dissociation. It is also reveals that an increase in the order takes place in going from reactants to the activated complex.



a) 0.5 MH_2SO_4 , b) 0.5 MH_2SO_4 + 500ppm of inhibitor, 1) Compound I, 2) Compound II, 3) Compound III

Figure 3 Relation between $\text{Log } R_{\text{corr}}/T$ and $1/T$ of carbon steel in 0.5 MH_2SO_4 in the absence and presence of 500 ppm of inhibitor

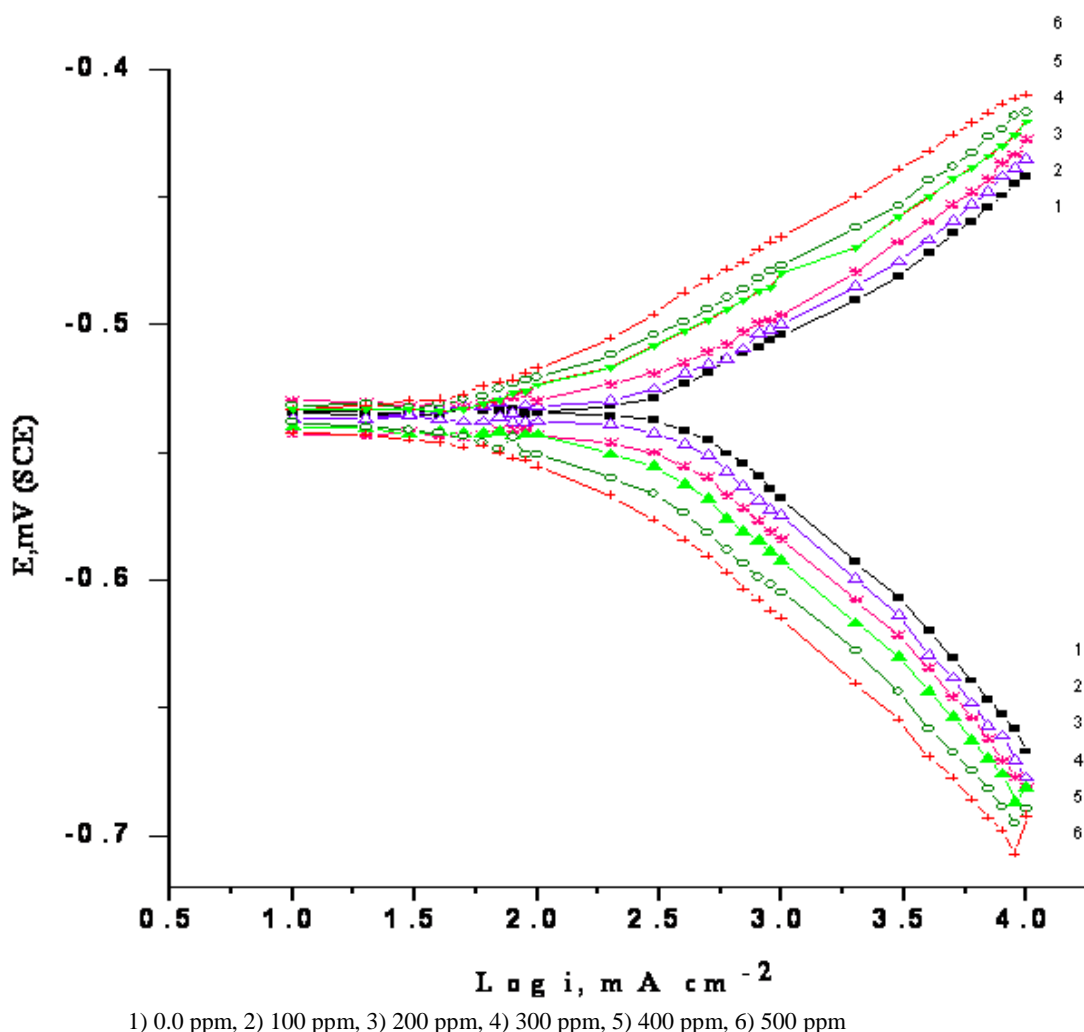


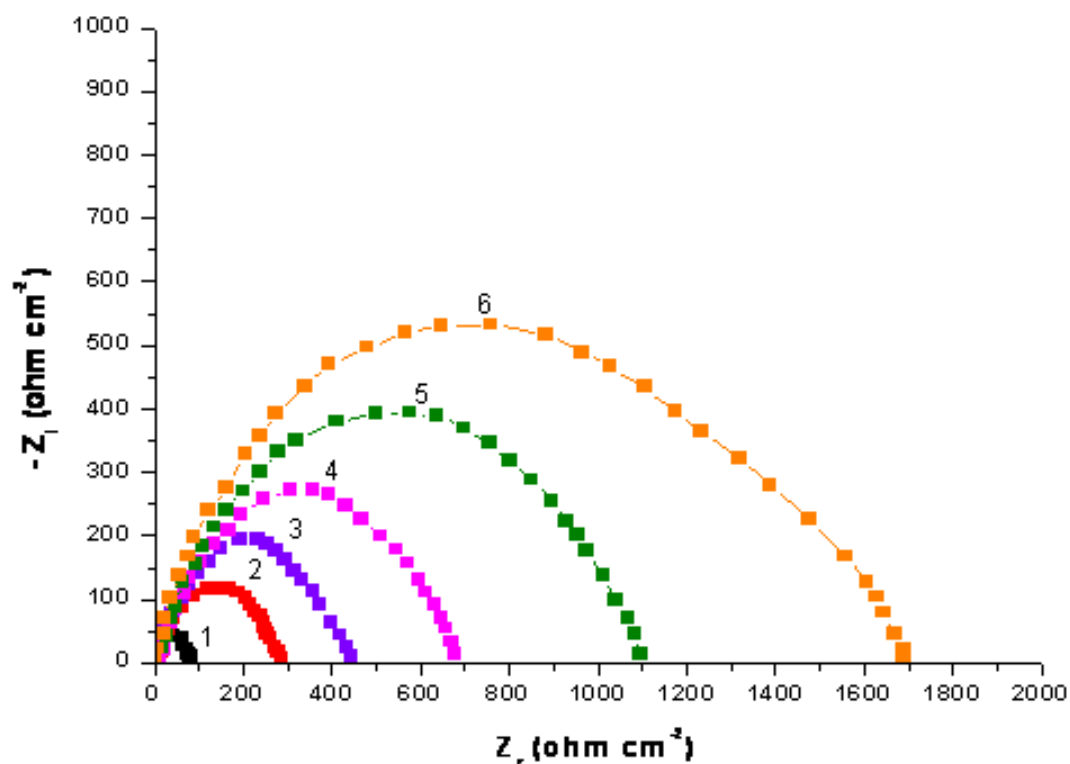
Figure 4 Galvanostatic polarization curves for corrosion of C-steel in 0.5 M H_2SO_4 solution in the absence and presence of different concentrations of compound III.

Galvanostatic polarization studies

Figure 5 shows the galvanostatic polarization curves of C-steel in 0.5M H_2SO_4 solution in the absence and presence of different concentrations of compound III as an example of the studied inhibitors. Similar curves were obtained for other two compounds (not shown).

The corrosion parameters such as, anodic Tafel slope (β_a), cathodic Tafel slope (β_c), corrosion current density (I_{corr}), corrosion potential (E_{corr}), and inhibition efficiency (I.E.) were calculated and given in **Table 2**. The corrosion potential, E_{corr} , is determined by the intersection of the extrapolation of the linear portions of the anodic and cathodic polarization curves. The value of the current at the intersection is the current density, I_{corr} .

Inspection of the data given in table 2, reveals that, as the concentration of the inhibitor increases, the values of BA and BC are approximately constant, suggesting the inhibiting action of these compounds by adsorption at the metal surface according to blocking adsorption mechanism. These molecules should be considered as a mixed type inhibitors. The values of E_{corr} is shifted to more negative potentials, the values of I_{corr} decreases and the values of IE increases indicating the inhibiting effect of these compounds. The inhibition efficiencies of these compounds decrease in the following order: compound III > compound II > compound I which is consistent with that obtained from weight loss measurements.



1) 0.0 ppm, 2) 100 ppm, 3) 200 ppm, 4) 300 ppm, 5) 400 ppm, 6) 500 ppm

Figure 5 The Nyquist plot for corrosion of C-steel in 0.5M H₂SO₄ solution in the absence and presence of different concentrations of compound III.

Table 2 Electrochemical parameters obtained from galvanostatic polarization technique of carbon steel electrode in 0.5 M H₂SO₄ containing different concentrations of sulfonamide nonionic surfactants.

Inhibitor conc., ppm	E, mV (SCE)	I _{corr} , (μA cm ⁻²)	β _a (mV dec ⁻¹)	-β _c (mV dec ⁻¹)	%I.E
0.0 ppm compound I	-527	1.860	173	205	-
100 ppm compound I	-529	0.614	172	207	66.98
200 ppm compound I	-530	0.436	173	206	76.55
300 ppm compound I	-532	0.382	175	208	79.46
400 ppm compound I	-530	0.230	176	209	87.63
500 ppm compound I	-534	0.164	174	210	91.18
100 ppm compound II	-530	0.496	170	208	73.33
200 ppm compound II	-532	0.364	168	209	80.43
300 ppm compound II	-532	0.238	171	208	87.20
400 ppm compound II	-534	0.193	174	210	89.62
500 ppm compound II	-536	0.128	175	212	93.54
100 ppm compound III	-532	0.426	172	209	77.09
200 ppm compound III	-534	0.321	172	208	82.74
300 ppm compound III	-535	0.218	175	205	88.27
400 ppm compound III	-536	0.142	176	207	92.36
500 ppm compound III	-538	0.088	178	205	95.26

Electrochemical impedance spectroscopy (EIS)

Figure 6 shows the EIS curves for C-steel in 0.5M H₂SO₄ solution in the absence and presence of different concentrations of compound III as an example of the studied inhibitors at 30°C after 30 min of immersion. Similar curves were obtained for other two compounds (not shown). It is clear that the Nyquist plots show a single semi-circle showed the single charge transfer process during dissolution which is unaffected by the presence of inhibitor molecules. Deviations from perfect circular shape are often referred to the frequency dispersion of interfacial impedance which arises due to surface roughness, impurities, dislocations, grain boundaries, adsorption of inhibitors, and formation of porous layers and in homogenates of the electrode surface [22–23].

The impedance parameters are analyzed in terms of an equivalent circuit model (**Figure 7**) which includes the solution resistance, R_s , and the double layer capacitance, C_{dl} which is placed in parallel to the charge transfer resistance R_{ct} . The double layer capacitance values were calculated using the following equation

$$C_{dl} = 1 / 2\pi f_{max} R_{ct} \quad (6)$$

where f_{max} is the frequency at which the imaginary component of the impedance ($-Z_{max}$) is maximal. The impedance parameters are given in **Table 3**.

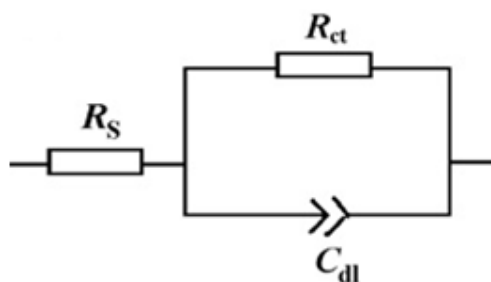
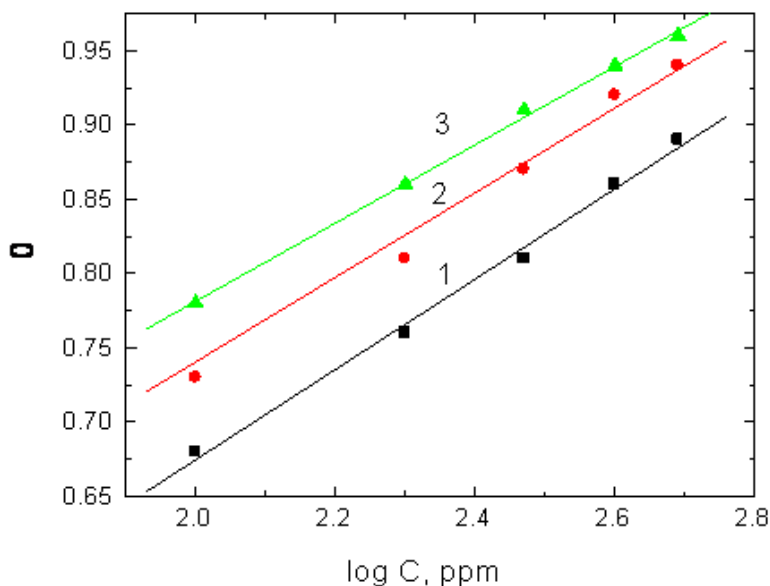


Figure 6 Electrical equivalent circuit (R_s = uncompensated solution resistance, R_{ct} = charge transfer resistance and C_{dl} = double layer capacitance).



1) Compound I, 2) Compound II, 3) Compound III

Figure 7 Temkin adsorption isotherm plotted as θ versus $\log C$ of inhibitors for corrosion of C-Steel in 0.5 M H₂SO₄ at 30°C

Table 3 EIS data of C-steel in 2M HCl and in the absence and presence of different concentrations of investigated inhibitors at 25 °C

Inhibitor System	Conc. (ppm)	R_s ($\Omega \text{ cm}^2$)	R_{ct} ($\Omega \text{ cm}^2$)	C_{dl} ($\mu\text{F cm}^{-2}$)	%I.E _(b)
Blank	0.5M H ₂ SO ₄	2.14	80	183	-----
	100	2.03	197	181	63.20
	200	1.96	355	173	77.50
Compound I	300	1.75	554	149	85.60
	400	1.54	817	126	90.20
	500	1.27	1060	101	92.00
	100	1.97	273	179	70.70
	200	1.69	410	171	80.50
Compound II	300	1.53	615	139	86.90
	400	1.22	900	119	91.00
	500	1.09	1220	97	93.40
	100	1.88	308	173	74.20
	200	1.46	452	166	82.23
Compound III	300	1.18	679	131	88.20
	400	1.11	1101	113	92.75
	500	1.03	1691	91	95.80

Inspection of Table 3, it is clear that as the concentration of inhibitors increases the values of R_{ct} increases and the C_{dl} value decreases. This is due to the gradual replacement of water molecules by the adsorption of the inhibitor molecules on the metal surface, decreasing the extent of dissolution reaction. The higher (R_{ct}) values, are generally associated with slower corroding system [24]. At one and the same inhibitor concentration, the order of inhibition efficiency obtained from EIS measurements decreases in the following order: III > II > I. The inhibition efficiency obtained from EIS are in good agreement with those obtained from weight loss and galvanostatic polarization techniques. But yielded different absolute values, probably due to the different experimental conditions

Adsorption isotherms and inhibition mechanism

Nonionic surfactant molecules inhibit the corrosion process by the adsorption on metal surface. Theoretically, the adsorption process can be regarded as a single substitutional process in which an inhibitor molecule (Inh.) in the aqueous phase substitutes an "x" number of water molecules adsorbed on the metal surface [25].



Where x is known as the size ratio and simply equals the number of adsorbed water molecules replaced by a single inhibitor molecule. The adsorption depends on the structure of the inhibitor, the type of the metal and the nature of its surface, the nature of the corrosion medium and its pH value, the temperature, and the electrochemical potential of the metal-solution interface. Also, the adsorption provides information about the interaction among the adsorbed molecules themselves as well as their interaction with the metal surface. Actually an adsorbed molecule may make the surface more difficult or less difficult for another molecule to become attached to a neighboring site and multilayer adsorption may take place. There may be more or less than one inhibitor molecule per surface site. Finally, various surface sites could have varying degrees of activation. For these reasons a number of mathematical adsorption isotherm expressions have been developed to take into consideration some of non-ideal effects.

Attempts were made to fit (values to various isotherms including Frumkin, Langmuir, Temkin and Freundlich isotherms. By far the results were best fitted by Temkin adsorption isotherm. Temkin adsorption isotherm could be represented using the following equation [26, 27]:

$$\exp(f\theta) = K_{\text{ads}} C \quad (8)$$

Where K_{ads} is the equilibrium constant of the adsorption, C is the inhibitors concentration in the bulk of the solution and f is the interaction parameter which depends on the intermolecular interaction in the adsorption layer and the heterogeneity of the surface.

Plotting against $\log C$ (Figure 7) Gave a straight lines in all compounds was obtained. This indicates that the adsorption of sulfonamide nonionic surfactants at the carbon steel surface follows Temkin adsorption isotherm. This isotherm is applied for ideal case of physical and chemical adsorption on a smooth with no interaction between the adsorbed molecules.

It is obvious that, the values of IE increased with an increase in the number of ethylene oxide unit. These finding could be explained on the bases of the fact that, an increase in concentration of the inhibitor would result in a lowering of the interfacial tension at the metal surface. This lowering in the interfacial tension is thought to a decrease of the bulk concentration of the inhibitor and an increase in its concentration at the metal surface.

Conclusions

1. Nonionic surfactants from sulfonamide are good inhibitors for corrosion of carbon steel in 0.5M H_2SO_4 solution.
2. The inhibition efficiency was found to increase with increasing the inhibitor concentrations, number of ethylene oxide unit and/or decreasing the temperature.
3. The inhibiting effect of these compounds was explained due to the adsorption of their compounds on the steel surface.
4. The adsorption process follows Temkin adsorption isotherm.

The inhibition efficiency obtained from different techniques showed a good agreement.

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