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Corrosion inhibition of iron by amphoteric surfactants in hydrochloric acid solutions

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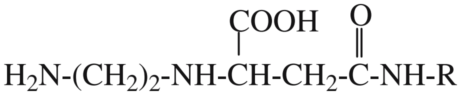
Abstract In the present work the corrosion inhibition of iron in 2 M HCl solution by amphoteric surfactants was studied. The techniques of measurements were (i) weight loss, (ii) linear polarization and (iii) electrochemical impedance spectroscopy. The investigated surfactants have the structure:



$$H_2N\text{-}(CH_2)_2\text{-}NH\text{-}\overset{\overset{COOH}{|}}{CH}\text{-}CH_2\text{-}\overset{\overset{O}{\|}}{C}\text{-}NH\text{-}R$$

where, $R = –C_{14}H_{29}$(I), $–C_{15}H_{31}$(II), $–C_{16}H_{33}$(III), $–C_{17}H_{35}$(IV), $–C_{18}H_{37}$(V). These surfactants have a high inhibitory effect on the corrosion of iron in HCl solution. The inhibiting effect of these surfactants may take place through the blocking effect resulting from their adsorption on the metallic surface and hydrophobic effect. The inhibition efficiency increases according to the order: I < II < III < IV < V. This is due to the increase of the length of the alkyl group (–R) in the surfactants. The adsorption of these surfactants via their adsorption centers on the metallic surface obeyed the Frumkin adsorption isotherm. The presence of these surfactants in the corrosive solution increases the activation energy of the corrosion process with that order of inhibition efficiency.

Introduction

Aqueous solutions of acids are among the most corrosive media. The inhibition of iron corrosion by organic additives has been studied widely [1–9]. A considerable amount of interest has been generated in the study of organic compounds as corrosion inhibitors owing to their usefulness is several industries: during the pickling of metals, cleaning of boilers, acidification of oil wells, etc. [10]. Most acid corrosion inhibitors are nitrogen–sulphur or oxygen containing organic compounds.

Unfortunately, most corrosion inhibitors used in aqueous heating and cooling systems are health hazards [11]. Their toxic properties are caused by the aromatic and N-containing and sulphur containing heterocyclic compounds, which are used widely in pickling processes and in the oil industry.

Research has focused on other types of nontoxic molecules. Amphoteric surfactants which contain both an anionic and cationic moiety in the same molecule have been known for many years. These amphoteric surfactants are of considerable interest, since they combine a positive as well a negative charge in one molecule. These compounds showed a high retarding effect on the corrosion of mild steel [12].

In the present work, the application is studied of amphoteric surfactants as possible corrosion inhibitors of iron in 2 M HCl solution.

Experimental

The working specimens and electrodes were cut from pure iron sheets (99.9% and 0.2 cm thickness). For weight loss measurements specimens of dimensions

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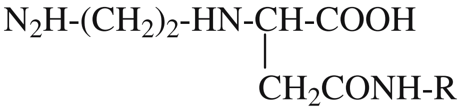
(2.5 × 2.5 × 0.2 cm) were cut from the iron sheets. For electrochemical measurements electrodes of dimensions (0.5 × 0.5 × 0.2 cm) were cut from the iron sheets. For the electrical connection, a copper wire was soldered to the electrode. Just before the use of specimens and electrodes, they were mechanically polished with emery papers of 1/0–4/0 grade and degreased with trichloroethylene.

The surface active agents (SAA) used as inhibitors in this study have the following structure:



$$N_2H\text{-}(CH_2)_2\text{-}HN\text{-}\underset{\displaystyle CH_2CONH\text{-}R}{\underset{|}{CH}}\text{-}COOH$$

where: –R = $C_{14}H_{29}$ (I), $-C_{15}H_{31}$ (II), $-C_{16}H_{33}$ (III), $-C_{17}H_{35}$ (V), $-C_{18}H_{37}$(V).

These surfactants are liquids and for this reason their concentrations are expressed in volume percent (vol.%).

These surfactants are alkyl-*N*(aminoethylene)-β-asparta mates and were prepared as elsewhere described [12].

In this work the corrosion behavior of iron was studied in 2 M HCl solution in the absence and in the presence of different concentrations of the investigated surfactants. In this investigation the techniques of measurements were: (i) weight loss, (ii) linear polarization and (iii) electrochemical impedance spectroscopy (EIS).

In weight loss measurements the iron specimens were weighed before and after the exposure time of 48 h. From these weights the weight loss and corrosion rate were determined.

In electrochemical measurements, the current was derived from a constant current unit (LG Precision Co, Ltd, Korea). The current and potential were measured by digital multimeters (model 1008, Kyoritsu, Japan). A saturated calomel electrode (SCE) and a platinum (Pt) electrode were used as reference and auxiliary electrodes, respectively.

The impedance measurements were carried out using an impedance measurements system (1M6 Zahner electrik Meβtechnik, Germany).

Results and discussion

Weight loss measurements

The iron specimens were immersed in the corrosive media (1.0 M HCl solutions) for 48 h. The weight loss of the specimens was determined and from the rate of corrosion, R_w, (mg/cm^2/h) was calculated. Also, the rate of corrosion was calculated as (mpy) using the equation:

$$\text{mpy} = \frac{534W}{DAT} \tag{1}$$

where W = weight loss, mg, D = density, g/cm^2 (7.88 for iron), A = area, square inch, T = exposure time, hours and mpy = mils per year = thousandths of an inch per year.

The data of these measurements are listed in Tables 1–5 for the iron specimens immersed in 2.0 M HCl solution in the absence and in the presence of different concentrations (% v/v) of the investigated surfactants (I–V), respectively at a temperature of 20 °C.

The values of surface coverage, θ, and inhibition efficiency, I%, were calculated from the corrosion rate, R_w, by using the following equations:

$$\theta = 1 - \frac{(R_w)_{inh}}{(R_w)_{free}} \tag{2}$$

$$I\% = \left[1 - \frac{(R_w)_{inh}}{(R_w)_{free}}\right] \times 100 \tag{3}$$

where $(R_w)_{free}$ and $(R_w)_{inh}$ are the corrosion rates of iron in the corrosive media in absence and presence of inhibitor, respectively. The calculated values of θ and

Table 1 Data of weight loss measurements of iron in 2 M HCl in presence of different concentrations of compound (I) at 20 °C

% (v/v)	R_w mg/cm^2/h	R_w mpy	θ	I%
0.000	0.1200	52.464	–	–
0.010	0.0720	31.48	0.40	40
0.025	0.0660	28.860	0.45	45
0.050	0.0450	19.670	0.65	65
0.075	0.0180	7.8700	0.85	85
0.100	0.0108	4.7200	0.91	91

Table 2 Data of weight loss measurements of iron in 2 M HCl in presence of different concentrations of compound (II) at 20 °C

% (v/v)	R_w mg/cm^2/h	R_w mpy	θ	I%
0.000	0.1200	52.464	–	–
0.010	0.0696	30.430	0.420	42.0
0.025	0.0630	27.540	0.475	47.5
0.050	0.0390	17.051	0.675	67.5
0.075	0.0144	6.3000	0.880	88.0
0.100	0.0084	3.6700	0.930	93.0

Table 3 Data of weight loss measurements of iron in 2 M HCl in presence of different concentrations of compound (III) at 20 °C

% (v/v)	R_w mg/cm^2/h	R_w mpy	θ	I%
0.000	0.12	52.464	–	–
0.010	0.0672	29.380	0.44	44
0.025	0.0612	26.760	0.49	49
0.050	0.0360	15.740	0.70	70
0.075	0.0120	5.2500	0.90	90
0.100	0.0060	2.6200	0.95	95

Table 4 Data of weight loss measurements of iron in 2 M HCl in presence of different concentrations of compound (IV) at 20 °C

% (v/v)	R_w mg/cm^2/h	R_w mpy	θ	I%
0.000	0.1200	52.464	–	–
0.010	0.0648	28.330	0.46	46
0.025	0.0576	25.180	0.52	52
0.050	0.0324	14.170	0.73	73
0.075	0.0096	4.2000	0.92	92
0.100	0.0036	1.5700	0.97	97

Table 5 Data of weight loss measurements of iron in 2 M HCl in presence of different concentrations of compound (V) at 20 °C

% (v/v)	R_w mg/cm^2/h	R_w mpy	θ	I%
0.000	0.1200	52.464	–	–
0.010	0.0624	27.280	0.480	48.0
0.025	0.0552	24.130	0.540	54.0
0.050	0.0300	13.120	0.750	75.0
0.075	0.0072	3.1500	0.940	94. 0
0.100	0.0018	0.7900	0.985	98.5

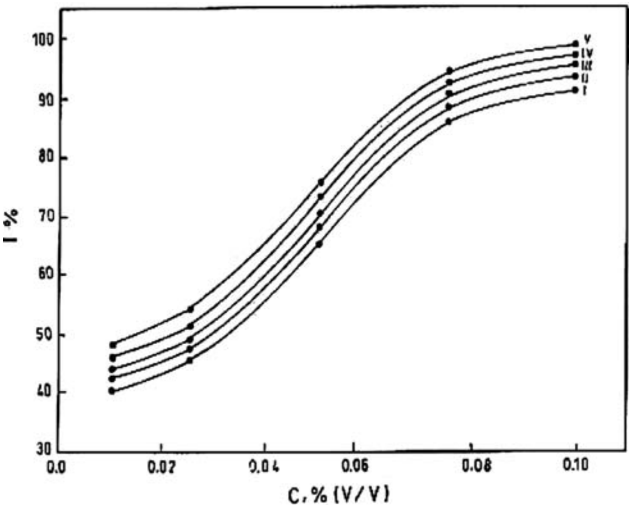


Fig. 1 Variation of inhibition efficiency (I%) as a function of concentration of the surfactants at 20 °C (weight loss measurements)

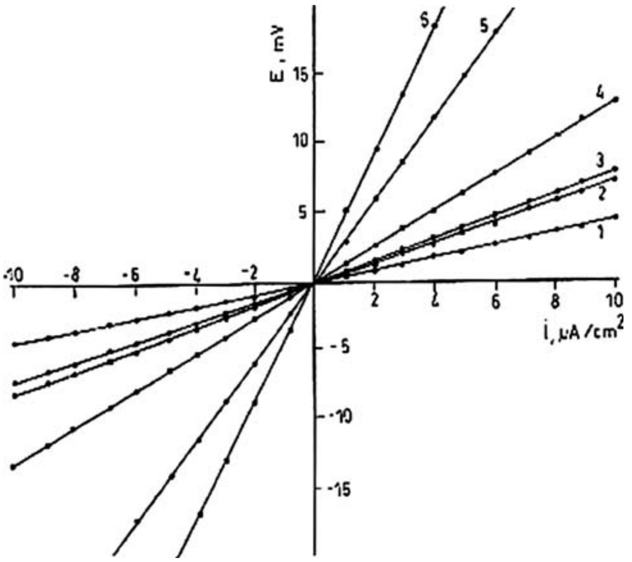


Fig. 2 Potential-current plots for iron in 2 M HCl solutions in absence and in presence of different concentrations of surfactant (I) at 20 °C. 1-blank, 2–0.01, 3–0.025, 4–0.05, 5–0.075 and 6–0.1%

I% are listed in Tables 1–5 for the different concentrations of the investigated inhibitors (I–V), respectively.

Figure 1 represents the variation of inhibition efficiency (I%) as a function of the concentration (% v/v) of the investigated surfactants. The plots of this figure have S-shape, where the inhibition efficiency slightly increases and then greatly increases with the increase of concentration, reaching a limiting value.

The results of Fig. 1 and Tables 1–5 indicate that the investigated surfactants have high inhibiting action on the corrosion of iron in the corrosive media (2 M HCl). The inhibition efficiency of the investigated surfactants increases in the order: I < II < III < IV < V.

Linear polarization measurements

Figure 2 represents the plots of potential, E, versus the current density, i, for the working electrode in pure 2 M HCl solution and in the presence of different volume concentrations of compound (I) at temperature of 20 °C. Similar plots are obtained for the other surfactants (II, III, IV, V) and are not shown. The values of linear polarization resistance, R_p, are deduced from the slopes ($\Delta E/\Delta i$) of straight lines of the plots of Fig. 2 and similar ones and listed, respectively, in Tables 6–10 for the investigated surfactants (I–V). The values of surface overage, θ, and inhibition efficiency, I%, are calculated from ($R_p = (\Delta E/\Delta i)$ by using the following equations:

$$\theta = 1 - \frac{(R_w)_{free}}{(R_w)_{inh}} \quad (4)$$

$$I\% = \left[1 - \frac{(R_w)_{free}}{(R_w)_{inh}}\right] \times 100 \quad (5)$$

where $(R_p)_{free}$ and $(R_p)_{inh}$ are the linear polarization resistances in pure 2 M HCl in absence and in presence of inhibitors, respectively. The calculated values of θ and I% are listed in Tables 6–10 for the different concentrations of the investigated surfactants (I–V), respectively.

Figure 3 represents the plots of inhibition efficiency, I%, versus the concentration (% v/v) of the investigated surfactants. The plots of this figure have S-shape like those obtained by weight loss measurements. The results of these plots and Tables 6–10 indicate that the inhibition efficiency of these surfactants is high and increases in the order: I < II < III < IV < V. This order is the same as that obtained from the weight loss measurements.

Impedance measurements

Figures 4–8 show the impedance diagrams (Nyquist plots) for the working electrode immersed in the corrosive media (2 M HCl) in the absence and in presence of different volume concentrations of the

Table 6 Data of linear polarization measurements of iron in 2 M HCl in presence of different concentrations of compound (I) at 20 °C

% (v/v)	R_p	θ	I%
0.000	450	–	–
0.010	737.70	0.39	39
0.025	803.57	0.44	44
0.050	1285.7	0.65	65
0.075	3000.0	0.85	80
0.100	4500.0	0.90	90

Table 7 Data of linear polarization measurements of iron in 2 M HCl in presence of different concentrations of compound (II) at 20 °C

% (v/v)	R_p	θ	I%
0.000	450	–	–
0.010	0769.23	0.415	41.5
0.025	0833.33	0.460	46.0
0.050	1363.64	0.670	67.0
0.075	4090.00	0.890	89.0
0.100	6000.00	0.925	29.5

Table 8 Data of linear polarization measurements of iron in 2 M HCl in presence of different concentrations of compound (III) at 20 °C

% (v/v)	R_p	θ	I%
0.000	450	–	–
0.010	796.46	0.435	43.5
0.025	873.79	0.485	48.5
0.050	1475.4	0.695	69.5
0.075	5000.0	0.910	91.0
0.100	10000	0.955	95.5

Table 9 Data of linear polarization measurements of iron in 2 M HCl in presence of different concentrations of compound (IV) at 20 °C

% (v/v)	R_p	θ	I%
0.000	450	–	–
0.010	0825.69	0.455	45.5
0.025	0918.37	0.510	51.0
0.050	1607.14	0.720	72.0
0.075	6428.57	0.930	93.0
0.100	18000.0	0.975	97.5

Table 10 Data of linear polarization measurements of iron in 2 M HCl in presence of different concentrations of compound (V) at 20 °C

% (v/v)	R_p	θ	I%
0.000	450	–	–
0.010	0873.79	0.485	48.5
0.025	1000.00	0.550	55.0
0.050	1875.00	0.760	70.0
0.075	9000.00	0.950	95.0
0.100	45000.0	0.990	99.0

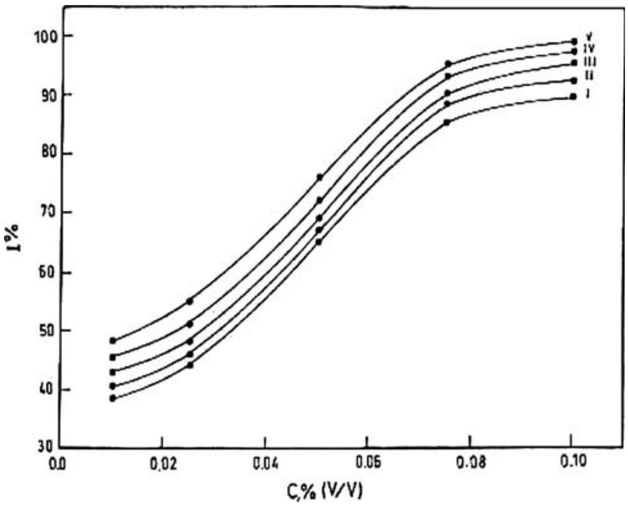


Fig. 3 Variation of inhibition efficiency (I%) as a function of concentration of the surfactants at 20 °C (linear polarization measurements)

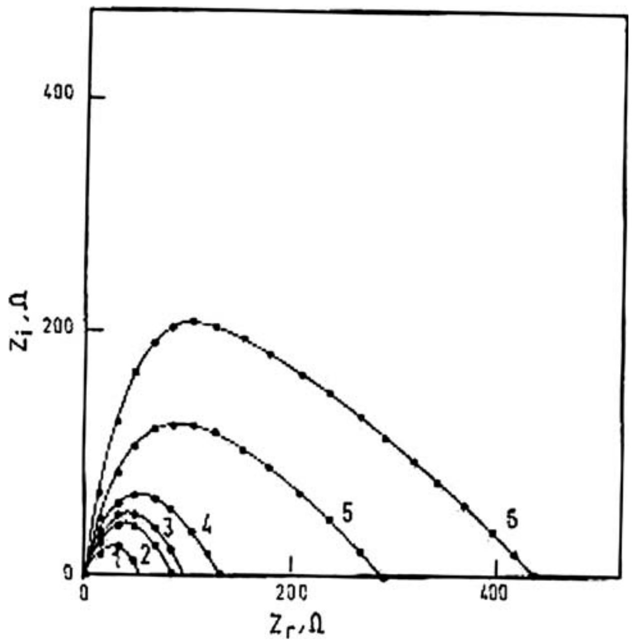


Fig. 4 Nyquist plots for iron in 2 M HCl in presence of different concentrations of surfactant (I) at 20 °C. 1-blank, 2–0.01, 3–0.025, 4–0.05, 5–0.075 and 6–0.1%

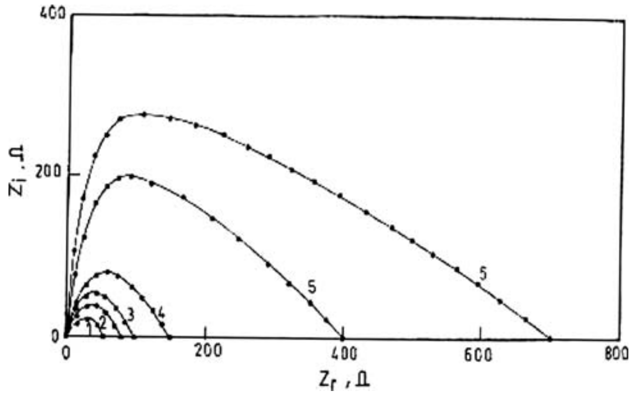


Fig. 5 Nyquist plots for iron in 2 M HCl in presence of different concentrations of surfactant (II) at 20 °C. 1-blank, 2–0.01, 3–0.025, 4–0.05, 5–0.075 and 6–0.1%

investigated surfactants (I–V), respectively, at a temperature of 20 °C. These impedance diagrams are not perfect semicircles which has been attributed to frequency dispersion [13, 14]. The charge transfer resistance (R_t) values are calculated from the difference in impedance at lower and higher frequencies, as previously suggested [15]. These R_t values are listed in Tables 11–15 for the investigated surfactants (I–V), respectively.

The double layer capacitance, C_{dI}, was determined from frequency, f, at which Z_i was maximum, using the relationship.

$$f\left(-Z_{i,\max}\right) = \left(2\pi C_{\mathrm{dl}} R_{\mathrm{t}}\right)^{-1} \quad (6)$$

The inhibition efficiency, I%, of the working electrode is calculated from the charge transfer resistance, R_t, using the equation:

$$\mathrm{I}\% = \frac{R_{\mathrm{tcorr}}^{-1} - R_{\mathrm{tcorr(inh)}}^{-1}}{R_{\mathrm{tcorr}}^{-1}} \times 100 \quad (7)$$

where $R_{\mathrm{t\ corr}}$ and $R_{\mathrm{t\ corr\ (inh)}}$ are the charge transfer resistance values in the absence and in the presence of inhibitor, respectively. The impedance parameters derived from these investigations are given in Tables 11–15; the results of these tables indicate that as surfactant concentration increases the values of R_t and I% increase, but the values of C_{dl} decrease. This indicates the occurrence of adsorption of surfactants on the electrode surface.

Figure 9 represents the variation of inhibition efficiency, I%, as a function of inhibitor concentration. The plots of this figure have S-shape like those obtained from weight loss and linear polarization measurements. The inhibition efficiency, I% of the investigated surfactants increases in the order: I < II < III < IV < V. This order is the same as that previously obtained by weight loss and linear polarization measurements.

The relation θ and log (C), where C is the inhibitor concentration, has the character of an S-shaped adsorption isotherm, Fig. 10. This result shows the investigated surfactants were adsorbed on the iron surface according to the Frumkin isotherm:

$$\theta(1-\theta)^{-1} \exp\left(-fC\right) = KC \quad (8)$$

where K is the equilibrium constant of the adsorption process, f is a function of adsorption energy, and θ is the surface coverage.

Figure 11 shows Arrhenius plots of the corrosion rate (mpy) in 2 M HCl solution in absence and in presence of 0.05% of the investigated surfactants. The apparent activation energies could be determined from Eq. 9:

$$R_{\mathrm{w}} = K \exp\left(-E_{\mathrm{a}}/RT\right) \quad (9)$$

where E_a is the apparent corrosion activation energy. The calculated values of E_a are: 38.18, 41.04, 43.90, 46.77, 49.63 and 52.50 kJ/mol in pure HCl and in the presence of 0.05% of surfactants (I–V), respectively.

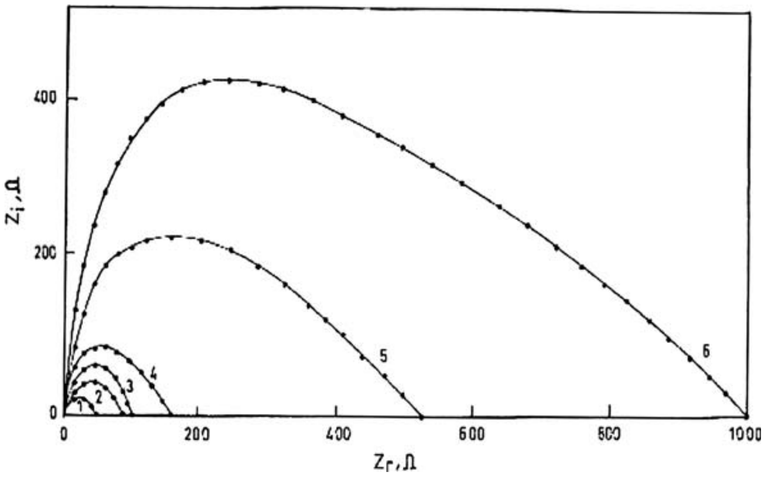


Fig. 6 Nyquist plots for iron in 2 M HCl in presence of different concentrations of surfactant (III) at 20 °C. 1-blank, 2–0.01, 3–0.025, 4–0.05, 5–0.075 and 6–0.1%

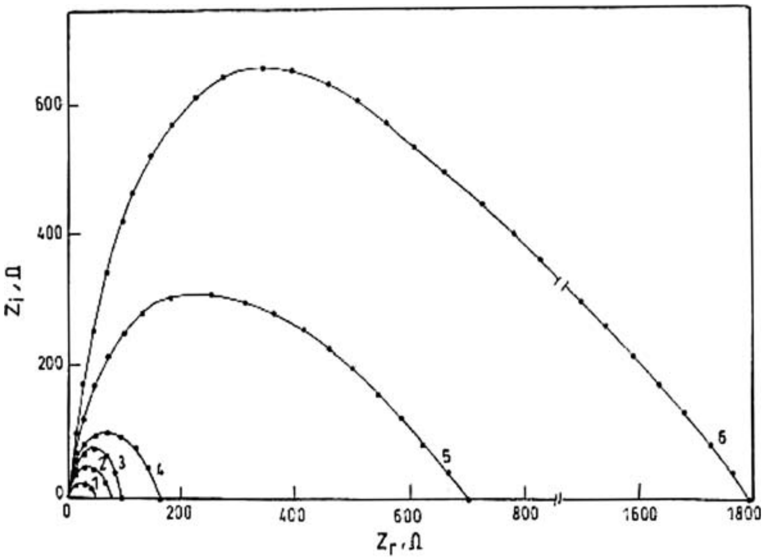


Fig. 7 Nyquist plots for iron in 2 M HCl in presence of different concentrations of surfactant (IV) at 20 °C. 1-blank, 2–0.01, 3–0.025, 4–0.05, 5–0.075 and 6–0.1%

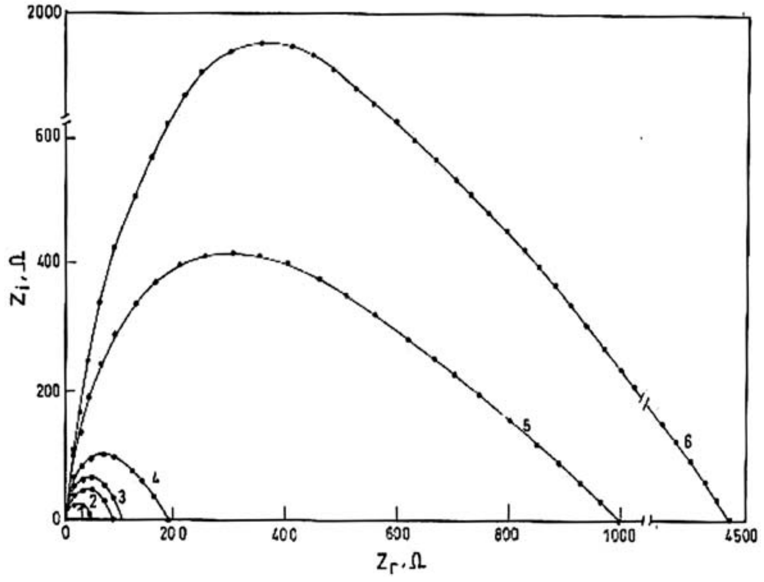


Fig. 8 Nyquist plots for iron in 2 M HCl in presence of different concentrations of surfactant (V) at 20 °C. 1-blank, 2–0.01, 3–0.025, 4–0.05, 5–0.075 and 6–0.1%

Table 11 Data of impedance measurements of iron in 2 M HCl in presence of different concentrations of compound (I) at 20 °C

% (v/v)	R_t	C_{dl}	θ	I%
0.000	45	1422.22	–	–
0.010	075.63	568.88	0.405	40.5
0.025	079.65	512.73	0.435	43.5
0.050	125.00	204.80	0.640	64.0
0.075	281.25	40.38	0.840	84.0
0.100	473.68	14.26	0.905	90.5

Table 12 Data of impedance measurements of iron in 2 M HCl in presence of different concentrations of compound (II) at 20 °C

% (v/v)	R_t	C_{dl}	θ	I%
0.000	45	1422.22	–	–
0.010	078.26	523.28	0.425	42.5
0.025	084.91	442.90	0.47	47.0
0.050	140.63	163.26	0.68	68.0
0.075	391.3	20.93	0.885	88.5
0.100	692.3.0	6.68	0.935	93.5

Table 13 Data of impedance measurements of iron in 2 M HCl in presence of different concentrations of compound (III) at 20 °C

% (v/v)	R_t	C_{dl}	θ	I%
0.000	45	1422.22	–	–
0.010	081.08	487.73	0.445	44.5
0.025	089.11	403.99	0.495	49.5
0.050	152.54	138.50	0.705	70.5
0.075	529.41	011.44	0.915	91.5
0.100	1000.0	3.2	0.955	95.5

Table 14 Data of impedance measurements of iron in 2 M HCl in presence of different concentrations of compound (IV) at 20 °C

% (v/v)	R_t	C_{dl}	θ	I%
0.000	45	1422.22	–	–
0.010	84.00	453.52	0.465	46.5
0.025	92.78	369.98	0.515	51.5
0.050	163.64	119.56	0.725	72.5
0.075	692.31	6.68	0.935	93.5
0.100	1800.0	0.9877	0.975	97.5

Table 15 Data of impedance measurements of iron in 2 M HCl in presence of different concentrations of compound (V) at 20 °C

% (v/v)	R_t	C_{dl}	θ	I%
0.000	45	1422.22	–	–
0.010	088.23	411.07	0.490	49.0
0.025	102.27	307.57	0.560	56.0
0.050	191.49	87.26	0.765	76.5
0.075	1000.00	3.20	0.955	95.5
0.100	4500.00	0.1580	0.990	99.0

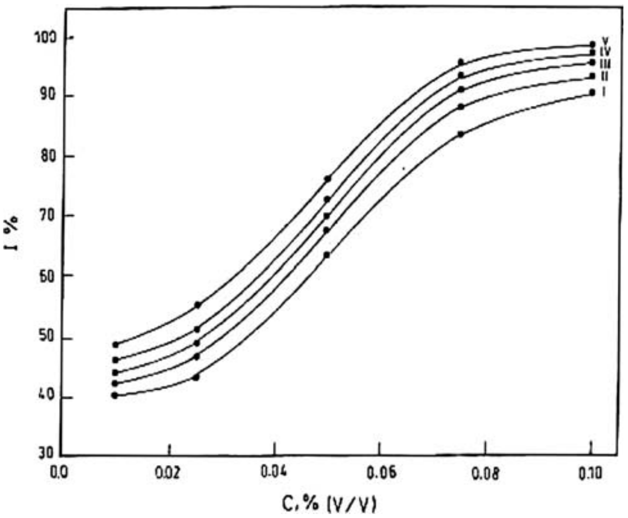


Fig. 9 Variation of inhibition efficiency (I%) as a function of surfactants concentration at 20 °C (impedance measurements)

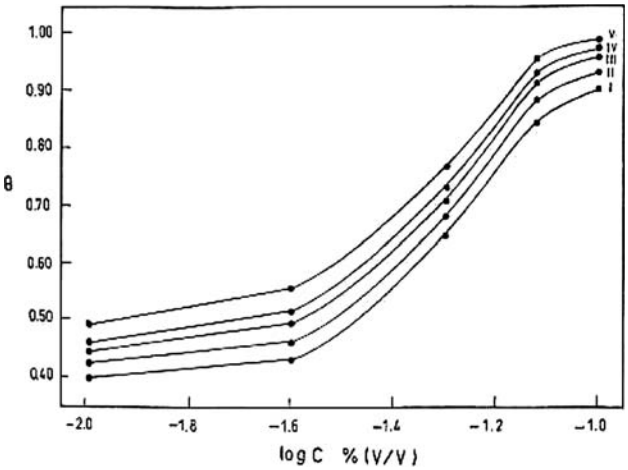
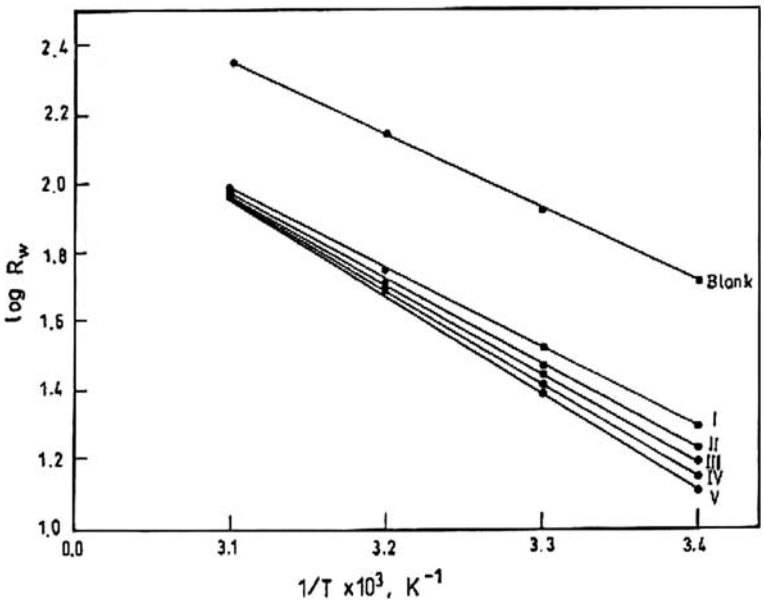


Fig. 10 Frumkin's adsorption isotherms for surfactants on iron surface at 20 °C (weight loss data)

Ideally, a corrosion inhibitor is a substance that increases the activation energy of corrosion process. This is clear from the values of E_a in pure acid and in the presence of inhibitors. This indicates that the energy barrier for the corrosion reaction increases in the presence of these additives. This means that by addition of inhibitor in to the acid solution, the corrosion reaction will be further pushed to a surface site, and is characterized by higher values of E_a, indicating that iron corrosion occurs at the uncovered part of the surface. The value of E_a for the investigated inhibitors increases in the order: I < II < III < IV < V. This order is the same as that of the inhibition efficiency, I%, of the investigated surfactants.

Fig. 11 Arrhenius plots of weight loss measurements for iron in 2 M HCl in absence and in presence of 0.05% of surfactants



The inhibition efficiency of the organic compounds depends on many factors including the number of adsorption sites and their charge density, molecular size, heat of hydrogenation, mode of interaction with the metal surface and formation of metallic complexes [16]. The inhibition of a corrosion process by addition of surfactants may take place through two effects, namely blocking and hydrophobic effects. The former occurs when the inhibitor molecule is adsorbed on the metal surface through the adsorption centers in the molecules. In the investigated surfactants there are more than one N-atom and O-atom which can act as adsorption centers. The second effect of inhibitors is hydrophobing the interface by surface active compounds to inhibit charge and mass transfer. Surfactants have a characteristic structure consisting of a structural group that has very little attraction for the solvent, known as a hydrophobic group, together with a polar head. When the surfactant is dissolved in water, the presence of the hydrophobic group in the solvent causes a distortion of the solvent liquid structure, increasing the free energy of the system. As compromise, the surfactant concentrates at the interface because there the thermodynamically best arrangement is possible. At the metal solution interface the surfactant molecules orientate their polar heads toward the metal and are adsorbed on its surface. The hydrophobic part is oriented away into the solution, repelling thus, the aqueous fluid. Under such conditions a diffusion barrier to chemical and/or electrochemical attack of the solution on the metal surface is established.

The inhibiting effect increases with an increase of surfactant concentration [17]. At low surfactant concentration the adsorption takes place by horizontal binding to hydrophobic region. This adsorption is a competitive one because the inhibitor displaces progressively the water molecules and other ions adsorbed. When surfactant concentration increases, a perpendicular adsorption takes place as a result of an inter-hydrophobic chain interaction [18].

Figure 12 represents the variation of inhibition efficiency, I%, with the number of carbon atoms in

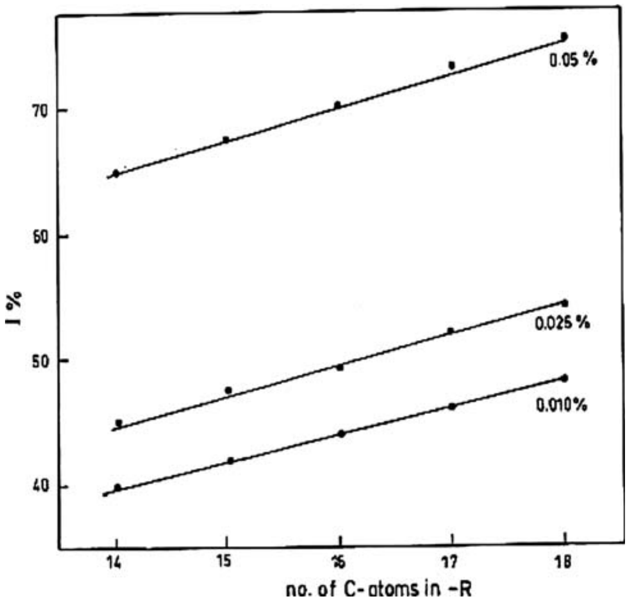


Fig. 12 Variation of inhibition efficiency (I%) as a function of the number of carbon atoms in the alkyl group (–R) in presence of different concentrations of the surfactants in 2 M HCl solutions at 20 °C

the alkyl group (–R), at different concentrations of surfactants. These alkyl groups have electron repelling properties, which increase with the increase of the number of $-CH_2$ groups in –R. This increases the electron density on the adsorption centers of the surfactant molecules. On the other hand the hydrophobic interaction increases with the increase of the length of –R [17, 19, 20].

Conclusion

1. The investigated surfactants are nontoxic and have a high inhibitory effect on the corrosion iron in HCl solutions.
2. The inhibition of the corrosion process of iron by addition of surfactants may take place through two effects, namely the blocking and hydrophobic effects. The former occurs by the adsorption of inhibitor molecules via adsorption centers on the metallic surface. The second effect of inhibitors is hydrophobing the interface by surface active compounds to inhibit charge and mass transfer.
3. The adsorption of surfactants on the metal obeys the Frumkin adsorption isotherm.
4. The presence of inhibitors increases the activation energy of the corrosion process.
5. The state of the adsorption centers renders these surfactants highly effective even in presence of low concentrations of them.

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