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Inhibition Effect of Tetraethanol-β-Octadecenyl Succinylamide in Acidic Corrosion of Carbonized Steel

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Abstract

The influence of tetraethanol-β-octadecenyl succinylamide (TOSA) on the corrosion of steel in H_2S0_4 and HC1 solutions of divers concentrations has been studied using gravimetrical, electrochemical and polarization corrosion test methods. It has been observed that the TOSA behaves as an inhibitor of molecular active cathodic type and has inhibition effect at the beginning stage of corrosion in both acids, but more effectively in H_2S0_4. It is shown that the inhibition effect of the TOSA increases with temperature increasing from 91 % at 20°C to 100 % at 80°C in 0.5 N H_2S0_4 solution and decreases with temperature increasing for higher concentrations of acid solutions. From obtained electrocapillary curves it is evidenced that the TOSA is a primarily additive of molecular type (insignificant shift of the maximum to the positive side on the curves). Moreover, it is surface active compound those superficial strength substantially decreases in H_2S0_4 (30 dyn/cm) and HCl (34.9 dyn/cm) solutions. It has been shown that the TOSA has a high protection effect and high formation rate of adsorption layer on the metal surface in H_2S0_4 (ψ = 96.4 % and τ = 8.0 min) and HC1 (ψ = 91.5 % and τ = 4.0 min).

Introduction

The metal corrosion problem is getting the most urgent actuality due to the constant extending of industrial applications of metal articles and equipment in marine petrol mining, especially in the Caspian marine shelf.

It is known that the use of functional organic inhibitors is a very effective method for anticorrosive protection of metal surfaces contacting with corrosive acid solutions. Early the inhibitor properties of organic nitrogen-containing compounds in the solution of divers mineral acids were studied [1-4].

It was shown that some N-containing organic compounds such as pyridine and its derivatives [5], 4-phenyl thiosemicarbazide [6], alkylene di(tri or tetra)amines [7], aromatic and heterocyclic amines [8], pyrimidine derivatives [9], izomers of aminobenzoic acid [10], and others [11-15] could be used as metal corrosion inhibitors in acid solutions.

The objective of this work is to study mechanism of inhibition effect of tetraethanol-P-octadecenyl succinylamide (TOSA), as corrosion inhibitor of low carbon steel in the mineral acid (HC1 and H_2SO_4) solutions and to determine optimum conditions of their use including effects of temperature and concentrations of studied acids and inhibitor.

Experimental

Materials

The samples of Steel-3 containing 0.28 % C, 0.26 % Si, 0.42 % Mn, 0.024 % P and 0.03 % S before use were ground, refined by ethanol and dried in vacuum exsiccator. The 0.5-5.0 % solutions of chloride and sulfuric acids as a working environment were used. The initial N-containing functional organic compound of TOSA

$$n\text{–}C_{15}H_{29}\text{–}CH{=}CH\text{–}CH_2\text{–}\underset{\displaystyle CH_2\text{–}CON(CH_2CH_{20}H)_2}{\underset{|}{CH}}\text{–}CON(CH_2CH_2OH)_2$$

used as an inhibitor was synthesized by the known method [16].

Measurements

For quantitative assessment of studied inhibitor efficiency the gravimetrical, electrochemical and po-

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larization corrosion test methods were used [17-21]. The gravimetry measurements were carried out at 20-80°C by using planning experimental data method.

The polarization measurements were carried out in the galvanization conditions. The working surface of steel samples was 10 cm^2. The non-working surface was insulated with epoxide resin ED-5. The density of polarized electrodes was measured at 0.001-6.0 mA/cm^2.

The adsorption rate of studied inhibitor on the steel surface was determined by electrochemical polarization method on the base values of measured polarization current in accordance with scheme of the plant presented in the Fig. 1. As a value (θ, mA×h*)* identifying corrosion starting period in no-inhibitor and inhibitor conditions there was used the following equation:

$$\theta = \int_0^{\tau} I(\tau)d\tau \qquad (1)$$

where *I* is a value of polarization current (mA) at given difference of potentials (V_c -V_a = const) and τ is a beginning inhibition time (min).

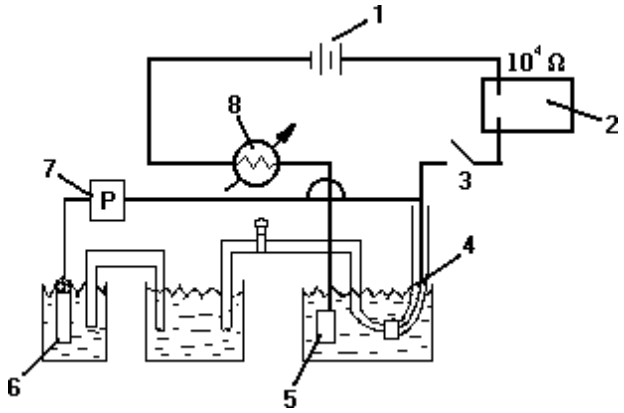

Fig. 1. Sheme of the plant for according of polarity characteristics of steel surface in absence and presence of inhibitors: 1- feed spring , 2 - resistance, 3 - key, 4 - steel eletrode, 5 - additional electrode, 6 - compared calomel electrode, 7 - potentiometer and 8 - milli-ammeter.

As a criterion of inhibitor efficiency in the starting period of corrosion there was used protection effect values (ψ, %) determined by following equation:

$$\psi/100=Q_0\text{-}Q_n/Q_0 \qquad (2)$$

where: Q_0 and Q_n are amounts of electricity between electrodes at given value of potentials during τ time no-inhibitor and inhibitor conditions.

To determine surface activity of studied inhibitor (σ) and state of its molecules on the electrode surface of electrolyte, the electrocapillary curves were recorded with capillary electrometer [22]. The σ value was calculated by following equation:

$$\sigma = g \times r/2[(h_1 - h_2)d_{Hg} - (h_p - h_2)d_p] \qquad (3)$$

where h_1 (30 cm) is a distance between the end of capillary and upper part of Hg (cm), h_2 is a distance between the end of capillary and lower part of Hg (cm), h_p is a height of immersed part of capillary (cm), d_p is a density of solution (g/cm^3), d_{Hg} is a density of Hg (g/cm^3), r is a radius of capillary (μm), g is a gravitational acceleration.

The differential capacity of electrodes was measured with electrical resistor of alternating current (P-568) at 10 kHz frequency where any dispersion was appeared. To determine adsorption characteristics of studied inhibitor the adsorption isotherms were used. Assuming mono-molecular adsorption in calculation of saturation degree (φ) of metal surface with inhibitor, the values of electrode capacity were obtained and following known equation [23] was used:

$$\varphi=(C_0\text{-}C_i)/(C_0\text{-}C_\infty) \qquad (4)$$

where C_0, C_i and C_∞ are capacities of electrode in the blank solution, at given and breaking concentrations of inhibitor.

Results and discussion

The TOSA inhibitor efficiency was determined by study of the influence of following external factors on the surface properties of steel in acid solutions: (1) temperature, (2) concentration of corrosion solution, (3) concentration of inhibitor, and (4) type of corrosion solution, i.e., anionic composition of acid solution. The levels of these factors and the intervals of their variations for studied inhibitor are presented in Table 1.

At constant values of levels of X_1, X_2 and X_4 factors for all inhibitors the levels of X_3 factor were changed in the dependence on the following effective concentrations obtained for TOSA inhibitor: $1.8.10^{-4}$ - $1.8.10^{-3}$ mol/L. After statistical analysis of experimental results for given inhibitor accomplished, the following equation for inhibitor efficiency (ψ) was obtained:

$$\psi_{TOSA} = 68.6\text{-}3.6X_1\text{-}13.5X_2\text{-}13.9X_3 + 5.7X_4\text{-}8.0X_1X_3 + 6.0X_3X_3 \qquad (5)$$

Table 1
The levels of factors and their variation intervals for studied TOSA inhibitor

Factors	Mark of Factors	Levels of Factors			Intervals of Variations
		–I	0	+I	
Temperature of corrosion media (°C)	X_1	20	50	80	30
Concentration of acid (N)	X_2	0.5	2.75	5.0	2.25
Concentration of inhibitor (mg/L)	X_3	100	550	1000	450
Type of acid	X_4	HC1	-	H_2S0_4	-

As is evident from this equation the concentration of inhibitor (X_3) exerts essential influence on the protection properties of the TOSA inhibitor; the increase of its concentration in the electrolyte causes growth of its efficiency. The temperature of corrosion solution (X_1) also exerts essential influence on the inhibitor efficiency.

The increase of temperature exerts negative influence on the protection properties of the TOSA inhibitor. The essential changes for TOSA inhibitor were observed that could be explained by formation of monomolecular film layer of this inhibitor on the surface of steel which was kept on the surface due to the physical and chemical adsorption. The high value of effective energy of activation (E_a) found from the plots of corrosion rate vs. reverse value of temperature (Fig. 2) confirms this fact. The E_a values for studied inhibitor were determined in the various conditions. The obtained results are shown in Table 2.

Table 2
The values of effectivite activation energy (Ea) of steel corrosion in acid and inhibited acid solutions

Acid concentration (N)	E_a (kcal /mol) for corrosion in	
	Acid solution	TOSA (1000 mg/L)
0.5 (H_2S0_4)	11.4	4.6
1.0	12.1	8.2
2.0	11.8	12.0
3.0	12.3	15.1
4.0	11.8	15.9
5.0	12.3	17.1
0.5 (HCl)	13.7	11.9
1.0	14.5	14.8
2.0	14.3	14.2
3.0	14.5	16.5
4.0	14.2	15.1
5.0	13.7	20.1

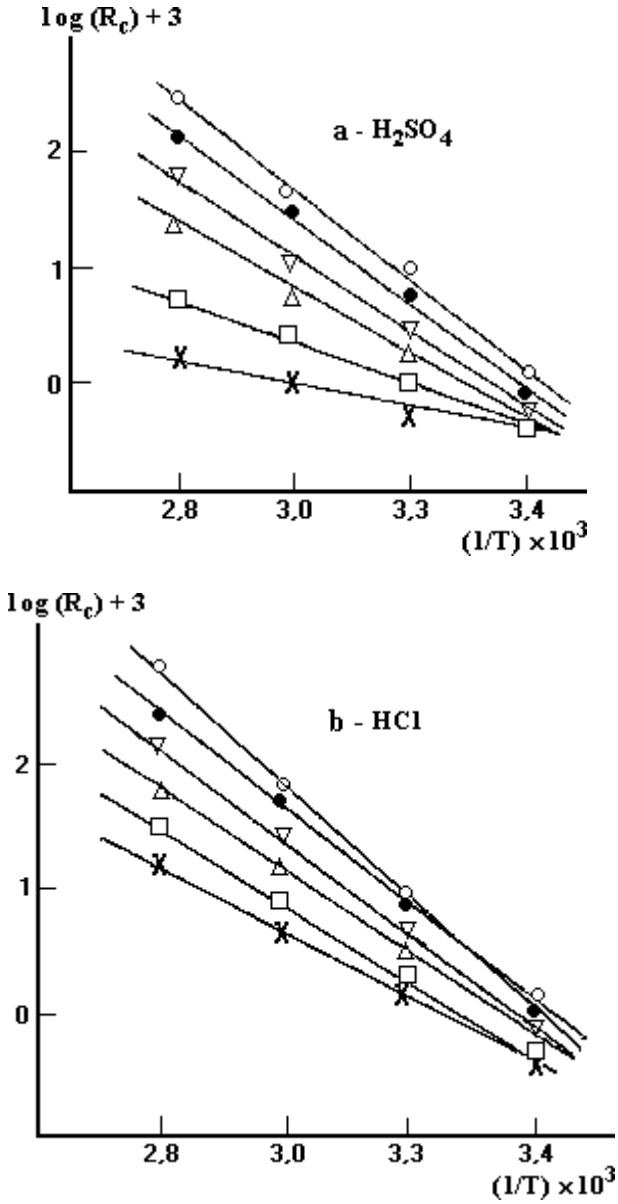

Fig. 2. Arrhenius plots of corrosion rate log (R_c) vs. 1/T to determine effective activation energy (E_a) at divers concentrations of acid solutions: (-o-) 0.5 N, (-•-) 1.0 N, (-Δ-) 2.0 N, (-∇-) 3.0 N, (-[]-) 4.0 N and (-×-) 5.0 N;

The influence of temperature factor is correlated with the following experimental data: Inhibition effect increases with temperature increasing from 91 % at 20°C to 100 % at 80°C in the 0.5 N H_2S0_4 solution. The inhibition effect decreases with temperature increasing for higher concentrations of acid solution (Table 3). It is shown that the inhibitor efficiency decreases with solution corrosiveness increasing. The TOSA inhibitor is more efficient in sulfuric acid solutions in comparison with chloride acid, and shows more stable protection properties at 20°C.

Table 3
Dependence of the efficiency of TOSA inhibitor (ψ) on temperature and concentrations of inhibitor, H_2S0_4 and HCl acid solutions

Acid concentration (N)		ψ (%)											
		Concentration of inhibitor [TOSA] x 10^3 (mol/L) at											
		20°C			40 °C			60 °C			80 °C		
		0.18	0.92	1.8	0.18	0.92	1.8	0.18	0.92	1.8	0.18	0.92	1.8
(H_2S0_4)	0.5	75.5	82.6	91.3	78.5	85.5	94.3	81.3	88.4	69.7	77.0	58.3	100
	1.0	73.2	81.4	91.4	75.1	82.8	93.3	76.9	97.1	85.8	85.7	74.4	97.0
	2.0	67.8	78.5	91.7	67.2	77.9	91.0	66.6	84.3	53.8	72.9	42.4	89.8
	3.0	62.7	78.5	91.7	67.2	72.7	88.9	56.7	91.4	66.8	80.0	55.4	82.9
	4.0	57.7	73.0	92.1	52.5	67.8	86.9	46.9	84.6	62.2	73.2	50.8	76.1
	5.0	52.5	70.2	92.3	44.7	62.5	84.5	37.1	95.1	81.3	83.7	69.9	69.1
(HCl)	0.5	64.1	71.1	79.9	67.1	74.1	82.9	69.9	78.8	41.7	67.4	30.3	88.7
	1.0	61.8	70.0	80.0	63.7	71.4	81.9	65.5	86.5	57.0	75.1	45.6	85.6
	2.0	56.4	67.1	80.3	55.8	66.5	79.6	55.2	77.2	54.8	65.8	43.4	78.4
	3.0	51.3	64.3	80.4	48.3	61.4	77.5	45.3	90.4	76.9	79.0	65.5	71.5
	4.0	46.3	61.6	80.8	41.1	56.4	75.5	35.5	65.9	29.3	54.5	17.9	64.7
	5.0	41.1	58.8	80.9	33.3	51.1	73.1	25.7	76.6	47.0	65.2	35.6	57.7

Afterwards, the surface activity of the inhibitor and the state of its molecules in electrolyte at electrode layer were studied. For this purpose the electrocapillary curves were recorded, those results of which are given in Fig. 3. The comparative analysis of these curves and curves of initial solutions shows that the studied inhibitor is a surface active compound those superficial strength substantially decreases a in H_2S0_4 and HCl solutions. The difference of superficial strength (Δσ) for the TOSA close to the corrosion φ-potentials of Fe has following values: 30 and 34.9 dyn/cm in 0.5 N H_2S0_4 and HC1 solutions.

The shift of the maximum on the observed electrocapillary curves allows to get to understanding type of adsorption and orientation of inhibitor molecules in the Hg-solution interface. The TOSA inhibitor is primary the additive of molecular type showing an insignificant shift of the maximum to the positive potentials on the curves (Fig. 3). It is known that the shift of maximum can be caused by polar organic compounds as well as by additives of ionic type [24]. The analogous specific adsorption of some N-containing compounds noted for Fe corrosion in acid solutions was observed by Hackerman and Hurd [25].

The φ - scale potentials allow to use surface activity data obtained on the Hg for interpretation of adsorption properties on the Fe. The obtained adsorption characteristics of surface activity (σ) and inhibition rates of corrosion (γ) for the TOSA inhibitor in acid solutions are: σ = 396 dyn/cm, Δσ- = 30.0 dyn/cm and γ = 11.6 in 0.5 N H_2S0_4 solution (φ_{Hg} = -0.1 V), σ = 392 dyn/cm, Δσ = 34.9 dyn/cm and γ = 5.0 in 0.5 N HCl solution (φ_{Hg} = -0.2 V).

As is evidenced from these data there is a parallelism between adsorption of the TOSA inhibitor on the Hg and its protection effect on Fe in H_2S0_4 (γ = 11.6) and in HCl (γ = 5.0).

Then, the inhibition effect of the TOSA at the beginning stage of steel corrosion was studied. The necessity of this study is conditioned by known fact

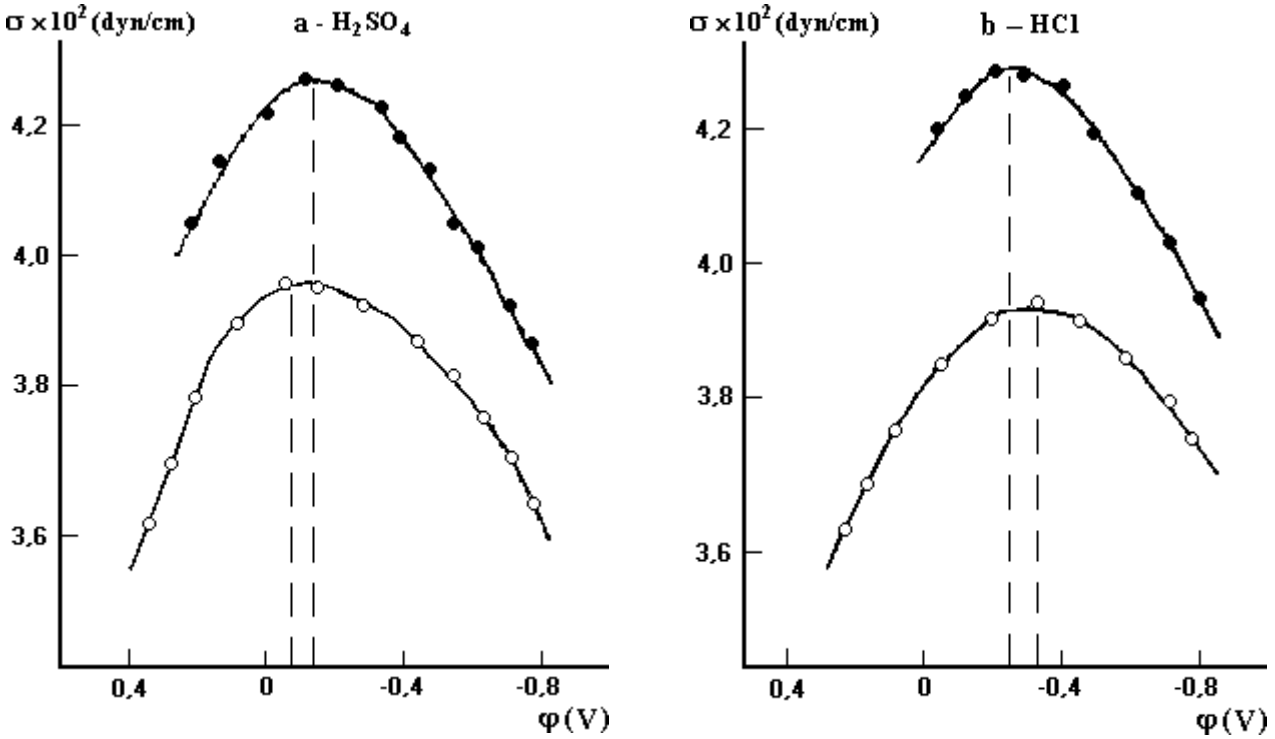

Fig. 3. Electrocapillary curves of the TOSA inhibitor in (a) 0.5 N H_2S0_4 and (b) 0.5 N HCl solutions at [TOSA] = 0.0 (-•-) and $3.7.10^{-3}$ mol/L (-o-).

that the most extent of corrosion destruction processes is noted exactly at the beginning stage of contact between metal surface and corrosive media.

The obtained experimental results are presented in Table 4. It is shown that the TOSA inhibitor have the most protection effects and higher formation rate of adsorption layer on the metal surface in H_2S0_4 (ψ = 96.4 % and τ = 8.0 min) and in HC1 (ψ = 91.5 % and τ = 4.0 min). As is obvious from the data of Table 5 the low concentration of TOSA inhibitor in 0.5 N H_2S0_4 does not provide stable inhibition at the beginning stage of corrosion. However, increase in TOSA concentration ($9.2.10^{-4}$ mol/L) provide 91.8 % protection during 20 min. Further increase in concentration (to $3.7.10^{-3}$ mol/L) causes a substantial decreases in formation time of the adsorption layer (to 8 min) and increases in the protection effect (95.4 %). The transfer from sulfuric acid to HCl also considerably decreases the formation time of the adsorption layer. The high adsorption of surface active organic compound on the steel electrode surface can be explained by specific adsorption power of Cl^- anions with negative charge increasing of electrode surface.

The increase in H_2S0_4 concentration from 1.0 N to

Table 4

Adsorption rate (θ) and protection effect (ψ and τ) of inhibitor at its divers concentrations

[TOSA] × 10^3 (mol/L)	0.5N H_2SO_4 solution			0.5N HC1 solution		
	θ (max h)	ψ (%)	τ (min)	θ (max h)	ψ (%)	τ (min)
0.55	221.3	75.2	-	50.4	70.2	6.0
0.92	114.9	87.1	20	28.6	83.0	6.0
1.80	77.0	91.4	13	18.2	89.2	4.0
3.70	40.9	95.4	8	14.4	91.5	4.0
9.20	36.6	95.9	8	-	-	-
00.00	893.2	-	-	169.2	-	-

2.0 N does not change protection efficiency of inhibitor and has rather high value (Table 5). The protection properties of inhibitor change no either in the high concentration of HC1 and the electrochemical corrosion rate has a tendency to decrease. However,

Table 5

Adsorption rate (θ) and protection effect (ψ and τ) of TOSA inhibitor at divers concentrations of acids solution

Concentration of acids		[TOSA]×10^3 (3.7mol/L)		
(N)	θ (mA×h)	θ (mA×h)	ψ (%)	τ (min)
H_2S0_4				
1.0	920	49.3	94.6	13
2.0	1572	92.4	94.1	17
3.0	1774	140.0	92.1	9-30
4.0	1977	201.0	89.9	12-21
5.0	1959	261.0	86.7	-
HCl				
1.0	283	39.9	86.0	12
2.0	315	45.4	85.5	15
3.0	406	42.6	89.5	18
4.0	523	55.5	89.4	21
5.0	594	82.3	86.1	16

the increase in HCl concentration from 1.0 N to higher ones increases τ value where the stable inhibition is nearly in three time as much (τ = 19.5 min in 1.0 N HCl). At the same time it the inhibitor decreases in protection efficiency of (ψ = 75.4 % in 5.0 N HCl, Table 5).

The results of this study show that the inhibitor of molecular active type, as the TOSA one, has high inhibition effect at the beginning stage of protection layer formation.

It is well known that the adsorption of the organic polar compounds on the metal-solution interface is a necessary condition of their inhibition effect in metal corrosion processes [24]. The surface of metal (Fe) electrode has a positive charge in H_2S0_4 solutions. However, the electrode potential has slightly negative charge in accordance with φ-scale in the case of HC1 solutions [26]. Taking into consideration the observed fact that the inhibition effect of N-containing compound such as the TOSA, reduce the adsorption efficiency of its molecules on the metal-electrolyte interface, the field of adsorption potentials of the inhibitors and the interfacial interactions with metal surface excite interest to further thorough investigation.

The plots of differential capacity vs. adsorption potential for divers TOSA inhibitor concentrations in H_2S0_4 and HCl solutions are presented in Fig.4. As is evident from Fig.4 data the capacity is approximately up to 24.0 mkf/cm^2 in the blank solution (0.5 N H_2S0_4 and 0.5 N HCl) while this parameter is substantially decreased by addition of inhibitor to the studied system. The minimum value of capacity in H_2S0_4 solution is somewhat lower than in HCl solution and adsorption potential field is shifted to the negative side. The field of inhibitor adsorption in HCl solution is extended in the anode field and the desorption come at -0.05 V potential against -0.2 V for H_2S0_4 solution. This fact can be explained by the well known additional specific adsorption effect of surface active chlor-anions in HCl solutions [27,28].

The adsorption parameters of inhibitor layer were calculated on the base of capacity measurements. The values of adsorption properties of studied inhibitor, such as adsorption equilibrium constant (K_a) and attraction constant (A), were calculated by following known equation [24]:

$$K_aI = \varphi/ (1- \varphi) \exp [-2A\varphi] \qquad (6)$$

where I is a concentration of TOSA initiator (mol/L) and φ is a saturation degree of electrode surface (%) which is determined by following equation:

$$\varphi = (1/f) \ln[(1 + K_{a(max)}I)/(1 + K_{a(min)}I)] \quad (7)$$

where f is a factor associated with a heterogeneous surface, $K_{a(max)}$ and $K_{a(min)}$ are adsorption equilibrium constants corresponding to the maximum and minimum values of adsorption energy respectively.

The obtained TOSA adsorption parameters have the following values: K_a = 13.6 and A = 4.4 in 0.5 N H_2S0_4 solution and K_a = 3384 and A = 0.36 in 0.5 N HC1 solution. As is evidenced from these values, the attraction constant A has positive values in both acid solutions that is associated with intermolecular attraction force of adsorption molecules of the inhibitor. The TOSA inhibitor forms more compact (φ = 98 %) and dense (A = 4.4) surface film in H_2S0_4 and ensures high inhibition effect on corrosion intensity of the steel with temperature increase.

The TOSA adsorption ability conforms to its effi-

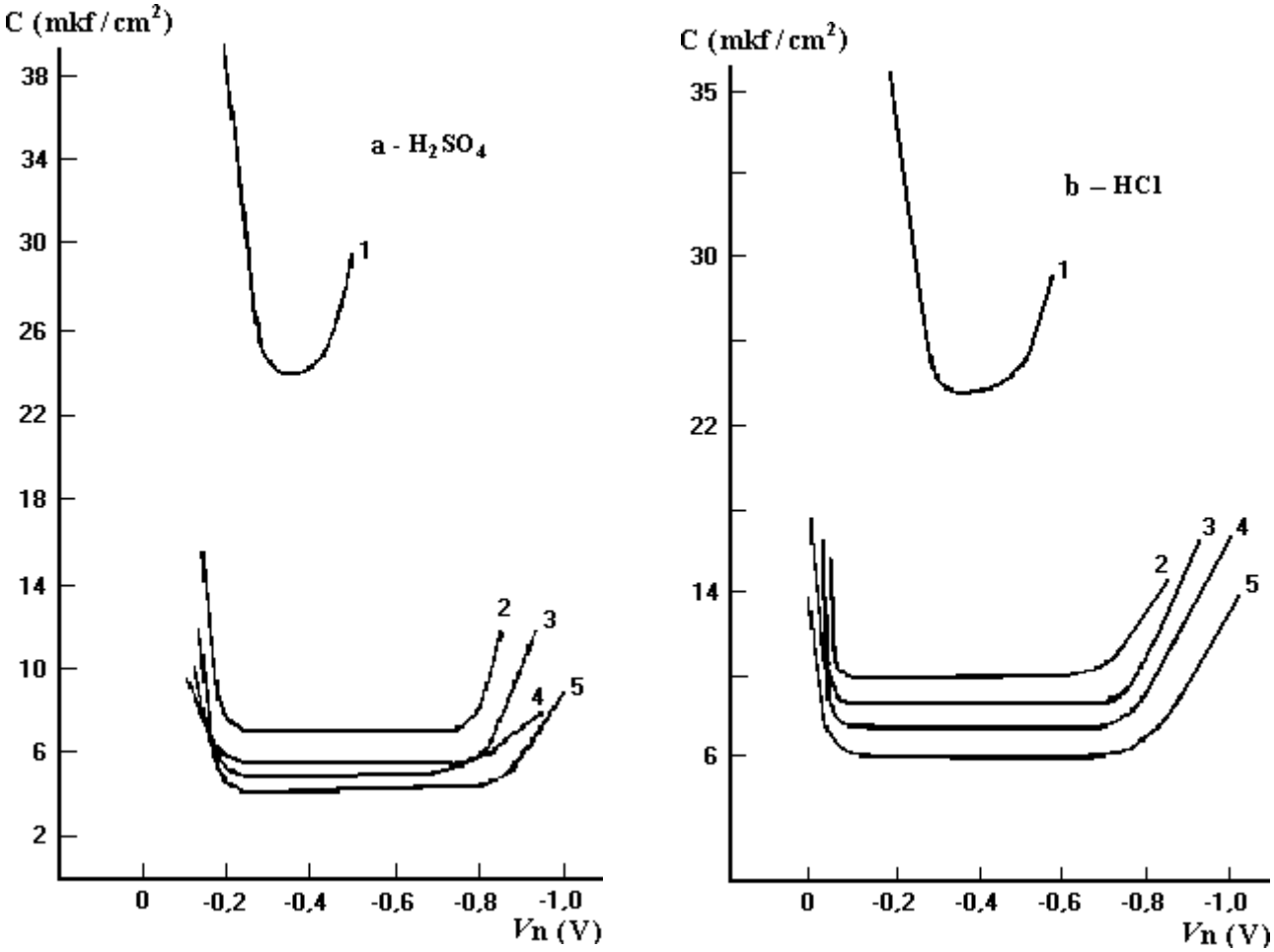

Fig. 4. Plots of differential capacity (C) of steel vs. adsorption potential (V) for divers inhibitor concentrations in (a) 0.5N H_2S0_4 and (b) 0.5 N HC1 solutions: (1) in no-inhibitor, conditions [TOSA].10^3 = 0.55 mol/L (2), 0.92 mol/L (3), 1.8 mol/L (4) and 3.7 mol/L (5).

ciency of anti-corrosion protection in corrosion processes. The TOSA as a molecular additive showing higher effects of protection considerably reduce the capacity of double layer on the steel surface.

Conclusion

Surface active functional N-containing compound of TOSA is effective cathodic inhibitor of carbonized steel corrosion in H_2S0_4 and HCl solutions, being more efficient in H_2S0_4.

TOSA inhibitor shows the most protection effects and higher formation rate of adsorption layer on the metal surface in H_2S0_4 (ψ = 96.4% and τ = 8.0 min) and in HC1 (ψ = 91.5 % and τ = 4.0 min).

TOSA inhibitor enhances protection properties of adsorption layer with increase in the concentrations of both acids and inhibitors in corrosive solution.

TOSA inhibitor forms more compact (φ = 98 %) and dense (A = 4.4) surface film in H_2S0_4 and ensures higher inhibition effect on steel corrosion with temperature increasing; the protective effect of the TOSA inhibitor increases with temperature increasing that is correlated with chemical nature of its adsorption ability on the steel surface.

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