

Anti-corrosion protection of carbon steel by expired Anaprilin in acidic media

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Abstract

Environmentally friendly corrosion inhibitors, also known as “green” inhibitors, have been used for the past decade in corrosion inhibition experiments. Scientific research is actively conducted on the use of expired medicines as “green” corrosion inhibitors of metals and alloys. In this work, the inhibitory effect of the expired drug Anaprilin against corrosion of carbon steel St3 in 1.0 N solutions of hydrochloric and sulfuric acids was studied. The concentration of the drug varied within 20–80 mg/L. The research was carried out using the methods of gravimetry, potentiodynamic polarization, impedance spectroscopy, and electrochemical diffusion techniques. Gravimetric corrosion tests conducted at room temperature and 80°C showed the protective effectiveness of the drug, reaching 90% in sulfuric acid solution at the highest concentration under study. The analysis of polarization curves in the media studied showed that Anaprilin slows down the anodic process. A decrease in the double layer capacitance upon the introduction of the inhibitor into the media under study indicates the adsorption of its molecules on the metal surface. The impedance spectroscopy data made it possible to determine the adsorption isotherms and calculate the free adsorption energy of Anaprilin.

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Introduction

Since 1977, the first reports about the presence of medicinal drugs in wastewater and groundwater began to appear in media sources. The rapidly increasing rate of population growth has correspondingly led to an increase in the number of pharmaceuticals.

Medicinals can enter the environment from various sources, for example, during the disposal of unused medicines, wastewater from sewage treatment plants, residues and waste from medical institutions, as well as waste from the pharmaceutical industry [1].

Unfortunately, there is still an urgent issue regarding the safe disposal of pharmaceuticals. According to the World Health Organization, pharmaceuticals pose a threat to public health and the environment due to their improper disposal. Thus, pollution of drinking water occurs due to poor-quality operation of landfills, the destruction of bacteria necessary for wastewater treatment, and air pollution is caused by the burning of

pharmaceuticals. The best medical waste disposal system should have a minimum risk assessment for waste management facilities, negligible impact on human health, minimal environmental impact, as well as be cost-effective and easily implemented [2].

The main methods of disposal include encapsulation, inertization, immobilization, landfill disposal, incineration in open containers, chemical decomposition [3].

One of the methods of solving the problem of improper disposal of pharmaceuticals is the possibility of using drugs as metal corrosion inhibitors. This topic has been studied for more than 10 years [4–7].

It is known that the most effective corrosion inhibitors are organic compounds whose molecules contain unsaturated bonds, aromatic rings and heteroatoms such as O, N, S, *etc.* However, due to the stringent requirements of environmental authorities around the world, the use of organic inhibitors is currently limited.

Many expired medicines have a similar structure to organic inhibitors and can be used as metal corrosion inhibitors. This allows them to be reused rather than sent for expensive disposal.

The authors of [4] studied the expired drug Desloratadine as a carbon steel corrosion inhibitor in 1.0 M HCl solution. By the method of potentiodynamic polarization, it is shown that the introduction of Desloratidine into a corrosive medium causes a decrease in the corrosion current. Gravimetric studies have shown that the protective effect of Desloratidine reaches 93% at a concentration of 19.3×10^{-5} M in 24-hour experiments. According to the authors, the drug is adsorbed on the surface of carbon steel in accordance with the Langmuir isotherm, forming a protective film. It is shown that the apparent activation energy E_a of the corrosion process increases in the presence of an inhibitor. This indicates that there is an energetic corrosion mechanism along with a blocking one, which the authors consider to be the only one, assuming the surface coverage with an inhibitor equal to its protective effect.

In [5], the authors studied the expired drug Dulcolax as a carbon steel corrosion inhibitor in 1.0 M HCl solution using chemical analytical and electrochemical methods. The concentration of the drug varied within 100–500 mg/L with a 7-hour duration of the experiment. The magnitude of the protective effect was more than 90% at its highest concentration. Electrochemical impedance spectroscopy data showed that the size of the hodographs increases in proportion to the concentration of the inhibitor. The authors explain this by the predominant mechanism of charge transfer in the corrosive process of steel dissolution.

The authors of [6] studied the expired drug Etoricoxib as a carbon steel corrosion inhibitor in 0.5 M H_3PO_4 solution. The data obtained by analyzing the polarization curves indicate a decrease in the corrosion current of steel. The optimal concentration at a temperature of 30°C is 225 mg/L, at a temperature of 60°C - 125 mg/L, which correspond to a protective efficiency of 80.6% and 63.8%, respectively. The authors explain this by a decrease in the adsorption of the inhibitor on the metal surface with increasing temperature. Its adsorption obeys the Langmuir isotherm. The negative value of the free energy of

adsorption indicates that the process is spontaneous. The authors believe that its value of $-0.3265 \text{ kJ}\cdot\text{mol}^{-1}$ is due to the electrostatic interaction between the metal surface and the preparation.

Earlier [7] we studied the protective efficacy of the expired drug Omeprazole in 1.0 N solutions of HCl and H_2SO_4 . The study was carried out using the methods of gravimetry, potentiodynamic polarization and impedance spectroscopy. The morphology of the surface of the steel sample after corrosion tests was evaluated using a scanning electron microscope. The protective effect of Omeprazole reaches 90% at a concentration of 40 mg/L, according to gravimetric tests. Analysis of polarization curves showed that Omeprazole causes inhibition of both partial electrode reactions. It is shown that the adsorption of Omeprazole on the surface of steel obeys the Langmuir isotherm.

The aim of this work is to study the inhibitory effectiveness of expired drug Anaprilin against corrosion of carbon steel in 1.0 N solutions of hydrochloric and sulfuric acids.

Experimental

Electrochemical measurements and corrosion tests were carried out on carbon steel St3 of composition, wt.%: C = 0.2; Mn = 0.5; Si = 0.15; P = 0.04; S = 0.05; Cr = 0.30; Ni = 0.20; Cu = 0.20; Fe = 98.36 in 1.0 N solutions of H_2SO_4 and HCl.

Sulfuric and hydrochloric acids were of “chemically pure” qualifications. The duration of gravimetric tests was 2 and 24 h at room temperature and 0.5 h at 80°C. The remaining tests were carried out at room temperature. The carbon steel St3 samples were polished to grade 6 purity and degreased with acetone before the experiments.

Electrochemical measurements were performed in a three-electrode glass cell after 15 minutes exposure of the working electrode in the solution. Potentials were measured with respect to the saturated silver/silver chloride electrode and recalculated to the standard hydrogen scale. The counter electrode is smooth platinum.

Potentiodynamic polarization measurements were carried out using the IPC-Pro potentiostat (produced by A.N. Frumkin Institute of Physical Chemistry and Electrochemistry, RAS) at a potential scan rate of 0.66 mV/s.

The impedance spectra were studied in the frequency range ($\omega/2\pi$) of 10 kHz–0.05 Hz with an alternating voltage amplitude of 10 mV using an electrochemical measuring complex from Solartron (Great Britain) consisting of an impedance analyzer SI 1255 and a potentiostat SI 1287. The results obtained were processed using a program ZView 3.0 that allows calculations for any equivalent circuits with up to 20 elements. The equivalent circuit used, simulating the behavior of a steel electrode in acid solutions, is shown and described in [7].

The protective effect of the inhibitor was calculated according to the data of gravimetric corrosion tests (1) and polarization measurements (2):

$$Z\% = 100 \cdot (K_0 - K_{\text{Inh}}) / K_0 \quad (1)$$

$$Z\% = 100 \cdot (i_0 - i_{\text{Inh}}) / i_0 \quad (2)$$

where K_0 (i_0) and K_{Inh} (i_{Inh}) are the corrosion rates in the absence and in the presence of an inhibitor in solutions, respectively. The corrosion current densities were calculated by extrapolating the Tafel sections of the polarization curves to the corrosion potential.

The study of the diffusion of hydrogen formed during metal corrosion through a steel membrane was carried out according to the electrochemical diffusion technique described in [8]. The duration of the experiments was 2 h at a corrosion potential. To characterize the effect of the inhibitor on the flow of hydrogen diffusion into the metal, the hydrogen penetration inhibition coefficient γ was used (3):

$$\gamma = i_{\text{H}}^0 / i_{\text{H}} \quad (3)$$

where i_{H}^0 and i_{H} are the hydrogen diffusion currents in the absence and in the presence of an inhibitor in a solution. $\gamma > 1$ corresponds to a deceleration of diffusion, $\gamma < 1$ corresponds to acceleration, and $\gamma = 1$ to the absence of an inhibitor effect.

The expired drug Anaprilin (propranolol) (RS)-1-isopropylamino-3-(1-naphthoxy)-2-propanol hydrochloride was used as an inhibitor. As a medicine, it has antihypertensive, antianginal and antiarrhythmic effects. The structural formula of the drug is shown in Figure 1. The inhibitor concentration was varied within 20–80 mg/L.

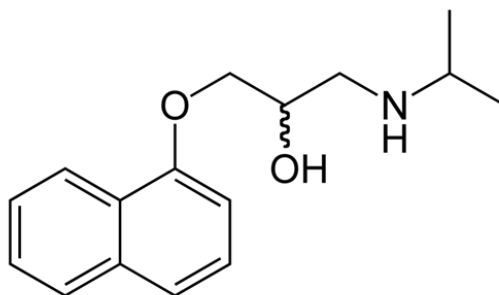


Figure 1. The structural formula of Anaprilin.

Results and Discussion

Acidic solutions are utilized in numerous industrial operations like acid pickling, acid cleaning and acid descaling of metals and their alloys. In this cases, it is necessary to use inhibitors to reduce the rate of dissolution of the metal.

Figure 2 presents the polarization curves obtained in 1.0 N HCl solution, and Table 1 shows the kinetic parameters calculated on their basis.

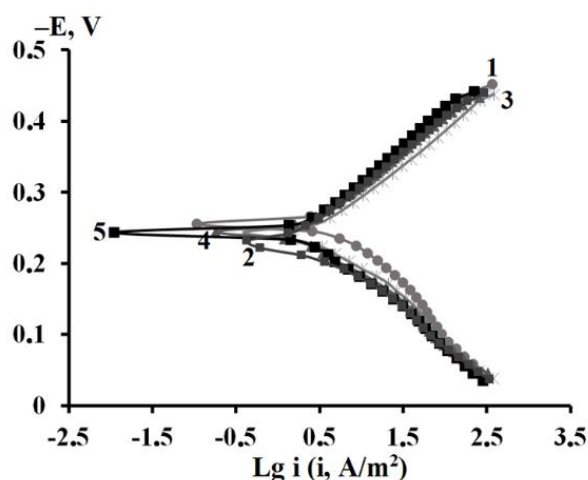


Figure 2. Polarization curves measured on a steel electrode in 1.0 N HCl solution in the absence of (1) and in the presence of Anaprilin, mg/L: 2 – 20, 3 – 40, 4 – 60, 5 – 80.

The corrosion current in the HCl background solution is 3.8 A/m^2 , however, it decreases to 2.2 A/m^2 with the introduction of 20 mg/L inhibitor. With the subsequent increase in inhibitor concentration, a further decrease in corrosion currents is observed, and the maximum Z value is equal to 79% at $c_{\text{Inh}} = 80 \text{ mg/L}$. One can see that Anaprilin causes inhibition of the anodic process. The slope coefficients of the Tafel sections of the anode polarization curves are close to each other. The same pattern is typical for the slope coefficients of linear sections of the cathode polarization curves close to 0.1 V . The corrosion potential shifts by 0.05 V to the positive direction when an inhibitor is introduced.

Table 1. Kinetic parameters of carbon steel electrode in 1.0 N HCl solution in the absence and in the presence of Anaprilin and its protective effect.

c_{Inh} , mg/L	$-E_{\text{corr}}$, V	i_{corr} , A/m ²	b_a , V	b_c , V	Z%
0	0.28	3.8	0.073	0.100	–
20	0.23	2.2	0.086	0.100	42
40	0.23	1.0	0.082	0.105	73
60	0.24	0.9	0.082	0.100	76
80	0.24	0.8	0.081	0.100	79

The polarization curves measured on a steel electrode in 1.0 N H₂SO₄ solution in the absence and in the presence of Anaprilin are shown in Figure 3. When an inhibitor is introduced into the background solution, a slight shift in the corrosion potential is observed in a positive direction. With increasing Anaprilin concentration corrosion currents decrease, which, in turn, causes an increase in protective effectiveness (Table 2). The greatest protective effect of 80% is achieved at the highest concentration. As in hydrochloric acid solution, Anaprilin causes mainly a slowdown in the anodic process.

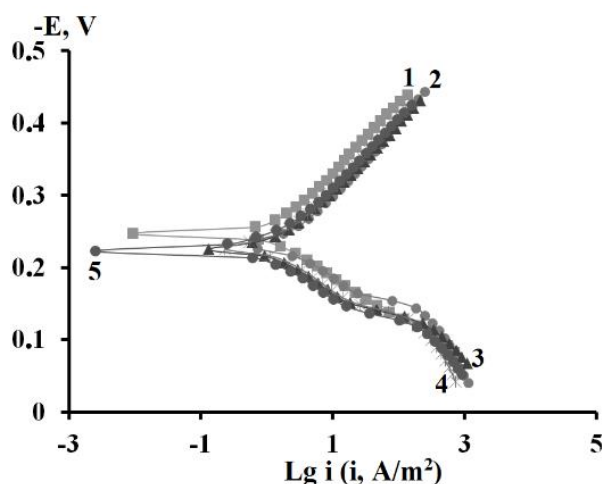


Figure 3. Polarization curves measured on a steel electrode in 1.0 N H₂SO₄ solution in the absence of (1) and in the presence of Anaprilin, mg/L: 2 – 20, 3 – 40, 4 – 60, 5 – 80.

Table 2. Kinetic parameters of carbon steel electrode in 1.0 N H₂SO₄ solution in the absence and in the presence of Anaprilin and its protective effect.

c_{Inh} , mg/L	$-E_{\text{corr}}$, V	i_{corr} , A/m ²	b_a , V	b_c , V	Z%
0	0.25	1.36	0.054	0.100	–
20	0.23	1.09	0.056	0.105	20
40	0.23	0.55	0.055	0.110	60
60	0.23	0.58	0.063	0.105	78
80	0.23	0.27	0.055	0.105	80

Acid etching or descaling can be carried out in acid solutions for either a long or shorter time, as well as at elevated temperatures. Accordingly, the protective effectiveness of Anaprilin was studied during gravimetric tests of steel in the acid solutions under study at room temperature with different durations of the experiment and at 80°C (Tables 3–5).

The results of 2-hour corrosion tests of carbon steel in the acid solutions studied and the values of the protective effect of the inhibitor are shown in Table 3.

The protective effect of the inhibitor increases with an increase in the concentration of Anaprilin in HCl and H₂SO₄ solutions. Already at a concentration of 20 mg/L in hydrochloric acid solution, Z is 52%, while in an environment with sulfuric acid, the protective effect is 61%. A further increase in the concentration leads to values of Z = 65% and Z = 75% in the solutions of hydrochloric and sulfuric acids, respectively.

Table 3. Steel corrosion rate in HCl and H₂SO₄ solutions and protective effect of Anaprilin according to the gravimetric corrosion tests. Exposure time is 2 h at room temperature.

<i>c</i> _{Inh} , mg/L	<i>K</i> , g/m ² ·h		<i>Z</i> %	
	HCl	H ₂ SO ₄	HCl	H ₂ SO ₄
0	5.623	9.847	–	–
20	2.698	3.886	52	61
40	2.223	2.752	60	72
60	2.106	2.348	63	76
80	1.966	2.412	65	75

The increase in exposure time to 24 hours (Table 4) practically did not change the protective effectiveness of Anaprilin in hydrochloric acid solution, even led to its decrease at the lowest concentrations, while in H₂SO₄ solution, on the contrary, the *Z* values increased at all concentrations of the inhibitor.

Table 4. Steel corrosion rate in HCl and H₂SO₄ solutions and protective effect of Anaprilin according to the gravimetric corrosion tests. Exposure time is 24 h, at room temperature.

<i>c</i> _{Inh} , mg/L	<i>K</i> , g/m ² ·h		<i>Z</i> %	
	HCl	H ₂ SO ₄	HCl	H ₂ SO ₄
0	1.651	9.523	–	–
20	1.117	3.667	32	62
40	0.885	1.561	46	84
60	0.629	1.335	62	86
80	0.572	0.759	65	92

Corrosion tests at 80°C showed that the protective effectiveness of Anaprilin in HCl solution is slightly higher than at room temperature, and in a sulfuric acid medium, on the contrary, lower (Table 5). The *Z* values are close in both solutions at the maximum inhibitor concentration, amounting to 72 and 74%.

A comparison of the protective effects calculated from the data of corrosion and polarization tests shows qualitative agreement, and some discrepancies in numerical values are obviously due to the different duration of the experiment. In contrast to longer gravimetric tests, polarization measurements last 15–20 minutes. To resolve the issue of the mechanism of the inhibitory action, the apparent activation energy *E*_a of the corrosion process in the solutions studied in the absence and presence of the inhibitor was calculated using the Arrhenius Equation:

$$\ln(K_2/K_1) = \left[E_a (T_2 - T_1) / RT_1 \cdot T_2 \right] \quad (4)$$

where K_1 and K_2 are the corrosion rates of steel at temperatures of 293 K and 353 K, respectively, $T_1 = 293$ K, $T_2 = 353$ K. The E_a values obtained are shown in Table 6. It follows that in media inhibited, the activation energy of the process differs from its value in background solutions, although slightly. If these values coincide, it could be argued that there is a blocking mechanism of action of the inhibitor. The slight difference observed allows for a mixed mechanism: blocking along with energy one.

Table 5. Steel corrosion rate in HCl and H₂SO₄ solutions and protective effect of Anaprilin according to the gravimetric corrosion tests at 80°C. Exposure time is 0.5 h.

c_{Inh} , mg/L	K , g/m ² ·h		$Z\%$	
	HCl	H ₂ SO ₄	HCl	H ₂ SO ₄
0	271.5	330.9	–	–
20	108.1	192.3	60	42
40	95.2	118.6	65	64
60	78.1	77.4	71	77
80	75.9	86.7	72	74

Table 6. The values of the apparent (effective) activation energy of the steel corrosion process in 1.0 N HCl and H₂SO₄ solutions.

c_{Inh} , mg/L	$E_{a, \text{eff}}$, kJ/mol	
	HCl	H ₂ SO ₄
0	55.5	50.3
20	52.9	55.9
40	53.8	53.9
60	51.7	50.0
80	52.3	51.3

It is known that the aggressiveness of acidic media consists not only in the loss of metal mass, but also in the penetration of hydrogen deep into the metal, which subsequently causes its embrittlement. Therefore, an important property of the inhibitor is its ability to reduce the diffusion of hydrogen into the metal. Anaprilin, starting from a concentration of 20 mg/L and at all subsequent concentrations, reduces the diffusion current of hydrogen i_H through a steel membrane in the solutions of hydrochloric and sulfuric acids studied. At the same time, the inhibition coefficient of hydrogen diffusion γ increases (Table 7).

Table 7. Effect of Anaprilin on the hydrogen diffusion current i_H through a steel membrane in 1.0 N HCl and H₂SO₄ solutions and its inhibition coefficient γ .

c_{Inh} , mg/L	HCl		H ₂ SO ₄	
	i_H , A/m ²	γ	i_H , A/m ²	γ
0	0.09	–	0.76	–
20	0.76	1.20	0.55	1.39
40	0.69	1.30	0.69	1.10
60	0.62	1.50	0.65	1.12
80	0.47	2.00	0.58	1.31

The protective effectiveness of the inhibitor studied in 1.0 N HCl and H₂SO₄ solutions is confirmed by the results of impedance measurements (Figure 4 and 5).

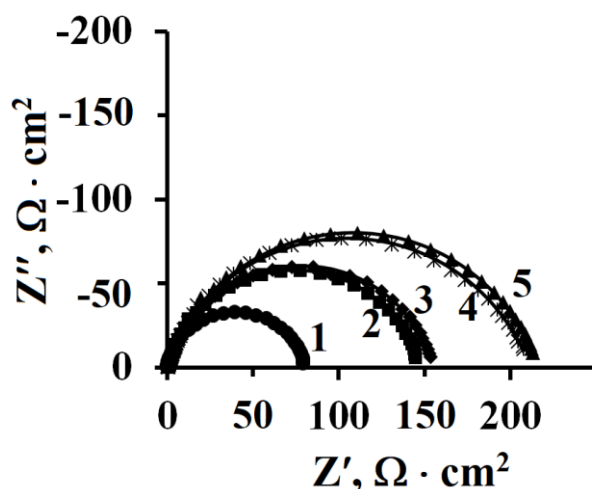


Figure 4. Nyquist diagram of a steel electrode in 1.0 N HCl solution at E_{corr} in the absence (1) and in the presence of Anaprilin, mg/L: 2 – 20, 3 – 40, 4 – 60, 5 – 80. The dots correspond to the experimental data, whereas the solid lines correspond to the impedance spectra fitted using the equivalent circuit.

Figure 4 shows the hodographs measured in 1.0 N HCl solution at E_{corr} . As can be seen, the diameter of the arcs increases with an increase in the concentration of the inhibitor, which indicates an increase in the overall resistance in the system and a decrease in the corrosion rate. The diameter of the semicircles in the Nyquist diagrams measured in H₂SO₄ solution also increases with increasing inhibitor concentration (Figure 5), as in HCl solution. This indicates a decrease in the rate of corrosion of steel.

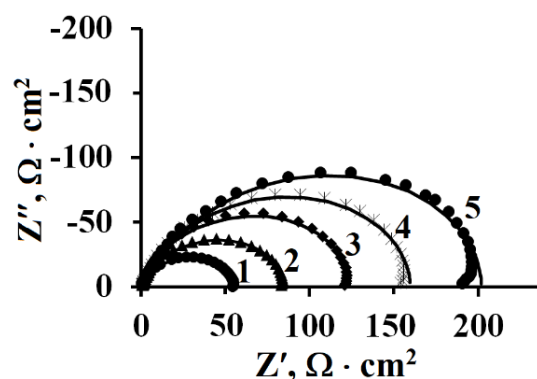


Figure 5. Nyquist diagram of a steel electrode in 1.0 N H_2SO_4 solution at E_{corr} in the absence of (1) and in the presence of Anaprilin, mg/L: 2 – 20, 3 – 40, 4 – 60, 5 – 80. The dots correspond to the experimental data, whereas the solid lines correspond to the impedance spectra fitted using the equivalent circuit.

The analysis of Nyquist diagrams was carried out according to the methodology and using an equivalent circuit described in [7]. The hodographs shown in Figures 4 and 5 correspond to an increase in the charge transfer resistance in the anode reaction when an inhibitor is introduced and its concentration increases. This is consistent with the deceleration of the anode reaction by an inhibitor in the solutions studied, also shown on the basis of the polarization curves. The capacitance of the electrical double layer C_{dl} decreases with the introduction of an inhibitor and an increase in its concentration, indicating its adsorption on the surface of the steel electrode (Table 8). This makes it possible to calculate the steel surface coverage θ with an inhibitor according to the Formula 5:

$$\theta = (C_0 - C) / (C_0 - C_1) \quad (5)$$

where C_0 , C and C_1 are the capacitances of the electric double layer in the solution without addition of an inhibitor, with that and with the maximum coverage of the electrode surface with the adsorbed inhibitor particles, respectively.

The calculation of C_1 was based on the dependence $C_{\text{dl}} = C_{\text{dl}}(1/c_{\text{Inh}})$ for HCl and H_2SO_4 solutions [7]. The values obtained for solutions of 1.0 N hydrochloric and sulfuric acids are equal to 28 and 22 $\mu\text{F}/\text{cm}^2$, respectively. The steel surface coverage with Anaprilin, depending on its concentration in the media under study, is shown in Table 8.

Table 8. The C_{dl} values and electrode surface coverage θ with Anaprilin in H_2SO_4 and HCl solutions.

c_{Inh} , mg/L	HCl		H_2SO_4	
	C_{dl} , $\mu\text{F}/\text{cm}^2$	θ	C_{dl} , $\mu\text{F}/\text{cm}^2$	θ
0	62.1	–	76.9	–
20	35.5	0.78	52.1	0.16

c_{Inh} , mg/L	HCl		H ₂ SO ₄	
	C_{dl} , $\mu\text{F}/\text{cm}^2$	θ	C_{dl} , $\mu\text{F}/\text{cm}^2$	θ
40	34.6	0.81	32.2	0.43
60	32.9	0.86	29.9	0.59
80	30.2	0.94	15.7	0.86

To select an isotherm corresponding to the data given in Table 8, it was checked their correspondence to the Temkin isotherm $Bc = \exp(f \cdot \theta)$, the Frumkin isotherm $Bc = [\theta/(1-\theta)] \exp(-2a\theta)$, and the Langmuir isotherm $c/\theta = 1/B + c$, where f is the factor of the energy inhomogeneity of the surface, B is the constant of adsorption equilibrium, a is the attraction constant characterizing the interaction between adsorbed particles, c is inhibitor concentration. For this, the graphical dependences of θ vs. $(\ln c)$, $\ln[c(1-\theta)/\theta]$ vs. θ and c/θ vs. c , corresponding to the Temkin, Frumkin and Langmuir isotherms, respectively, were considered (Figure 6).

It turned out that the best fitting of the data to the linear dependence corresponds to the Langmuir isotherm in the case of HCl solution, and to the Frumkin isotherm in the H₂SO₄ medium (Figure 6). Table 9 shows the numerical values of the approximation reliability calculated using the least squares method.

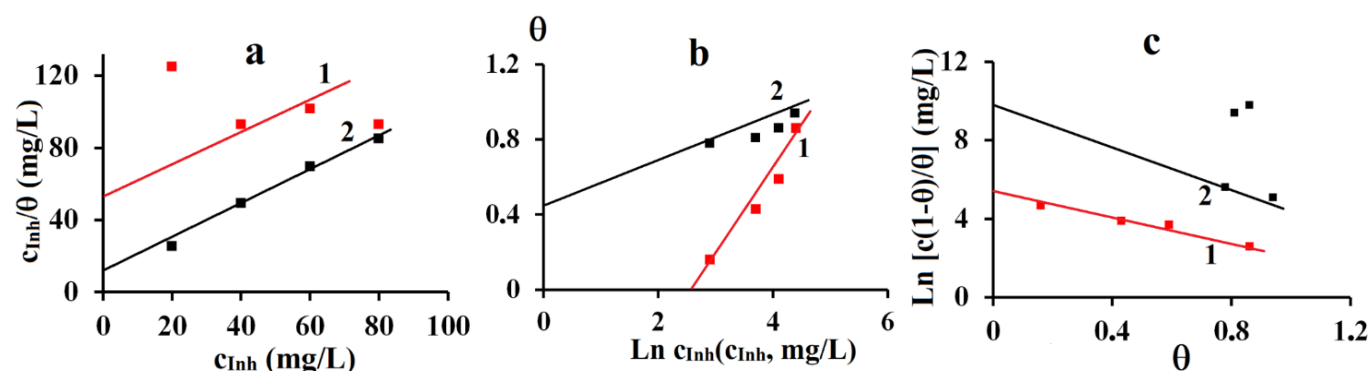


Figure 6. Adsorption isotherms for St3 steel in 1.0 N H₂SO₄ (1) and 1.0 N HCl (2) solutions with Anaprilin.

Table 9. Indicators of the accuracy of the approximation.

Isotherm	Accuracy of the approximation, R^2	
	HCl	H ₂ SO ₄
Langmuir	0.991	0.556
Temkin	0.815	0.949
Frumkin	0.069	0.973

In the case of hydrochloric acid, the segment cut off on the vertical axis of Figure 6a makes it possible to calculate the adsorption equilibrium constant B , which turned out to be 0.125 L/mg. In the H_2SO_4 solution, the segment cut off on the vertical axis of Figure 6c, equal to $-\ln B$ in accordance with the logarithmic form of the Frumkin equation $\ln[c(1-\theta)/\theta] = -\ln B - 2a\theta$, also allows us to calculate the constant B , which is equal to 0.005 L/mg.

For a known value of B , the free adsorption energy $-\Delta G_{\text{ads}}^{\circ}$ is calculated by the formula:

$$-\Delta G_{\text{ads}}^{\circ} = RT \ln(B \cdot 10^6) \quad (6)$$

where 10^6 is the concentration of water in solution, mg/L.

The values of $-\Delta G_{\text{ads}}^{\circ}$ at a temperature of 298 K in 1.0 N HCl and H_2SO_4 solutions amount to 29.1 kJ/mol and 21.1 kJ/mol, respectively. It can be assumed that the adsorption of Anaprilin is of a physical nature.

Conclusions

By means of gravimetry, potentiodynamic polarization, impedance spectroscopy and electrochemical diffusion techniques, the protective effectiveness of the expired drug Anaprilin against corrosion of carbon steel in 1.0 N solutions of sulfuric and hydrochloric acids has been studied.

The magnitude of the protective effect of Anaprilin reaches 92% at a concentration of 80 mg/L in a solution of sulfuric acid at 24 hours of exposure, while in a solution of hydrochloric acid $Z = 65\%$. Analysis of the polarization curves showed that Anaprilin causes inhibition of the anode process in both media. Impedance spectroscopy data show a decrease in the capacitance of the double electric layer with an increase in the concentration of the drug. This made it possible to calculate the steel surface coverage with an inhibitor, determine the type of adsorption isotherm and calculate the free adsorption energy of Anaprilin.

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