Protective efficiency of expired drug against acid corrosion of carbon steel

L.E. Tsygankova,* V.A. Bryksina, A.A. Uryadnikov and A.E. Abramov

Derzhavin State University, ul. Internatsionalnaya, 33, 392000 Tambov, Russian Federation
**E-mail: vits21@mail.ru*

Abstract

Expired drugs are studied as corrosion inhibitors of metals for more than 10 years. In the present work, the corrosion inhibitory effect of expired drug Omeprazole has been studied against carbon steel corrosion in 1.0 N hydrochloric and sulfuric acid solutions. The concentration of Omeprazole varied within 10–60 mg/L. The methods of gravimetry, potentiodynamic polarization, impedance spectroscopy were used at room temperature. The surface morphology of carbon steel samples after corrosion in the absence and in the presence of Omeprazole has been characterized by scanning electron microscopy. Analysis of the polarization curves in the solutions studied indicates that Omeprazole slows down the anodic and cathodic processes on carbon steel in both media. A decrease in the double-layer capacitance upon the introduction of the inhibitor into the media indicates the adsorption of its components on the metal surface. Impedance spectroscopy data made it possible to determine the adsorption isotherm and calculate the free energy of Omeprazole adsorption. The protective effect of the inhibitor at a concentration of 40 mg/L reaches 90% in both media.

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Introduction

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. 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*. Many expired drugs have similar structures and can be used as corrosion inhibitors for metals. This allows them to be reused instead of destruction.

Use of organic corrosion inhibitors is limited due to increasing environmental legislation throughout the world. Therefore, there is an increasing demand of green corrosion inhibitors that protect metals and alloys at low environmental risks. Expired drugs are the most important alternative candidates for anticorrosion protection of metals. For more than 10 years, drugs are studied as corrosion inhibitors of metals [1–7].

In [1], the inhibitory efficacy of five antibiotics against mild steel corrosion in 0.1, 0.01, and 0.001 N solutions of HNO_3, HCl, and H_2SO_4 was studied by weight loss technique. The authors note that the protective efficacy of various antibiotics varies widely from 17 to 93% depending on the type of acid and its concentration. Unfortunately, they did not indicate the concentration of antibiotics used. The review [2] describes more than 10 drugs used as corrosion inhibitors for mild and carbon steel, aluminum and zinc in solutions of sulfuric and hydrochloric acids. They have hetero-atoms or aromatic ring in their molecular structures. Studies have been carried out by the methods of weight loss, potentiodynamic polarization, electrochemical impedance spectroscopy, linear polarization resistance. The concentration of different drugs varied within 50–900 ppm. The protective effect ranged from 75 to 97%. In all cases, a blocking mechanism of action of inhibitors is adopted, although the apparent activation energy of the corrosion process changes significantly in their presence.

Review [3] provides data on the inhibitory ability of 30 drugs against mild steel corrosion in 1 M HCl solution. A general characteristic of a sufficiently high inhibitory efficacy of drugs in relation to Al, Zn and copper in acidic solution is given. It is noted that the adsorption of drugs on steel in most cases obeys the Langmuir isotherm and only in rare cases the Temkin isotherm. The concentration of inhibitors was 100–600 ppm, their protective effect varied within 80–97%.

Review [4] analyzes eco-friendly corrosion inhibitors, including amino acids, drugs, plant extracts, ionic liquids *etc.* Data are given on the protective effectiveness of a number of drugs in relation to mild steel, aluminum, zinc in solutions of sulfuric, hydrochloric and phosphoric acids. Their rather high protective efficiency is noted.

In [5], the protective efficacy of Sulfadoxine–Pyrimethamine drugs and an industrial inhibitor against corrosion of pipeline steel in petroleum pipeline water was studied. The studies were carried out using the gravimetric method, potentiodynamic polarization and impedance spectroscopy. The authors accepted without evidence the blocking mechanism of action of the inhibitors. The adsorption characteristics of the inhibitors were fitted into Langmuir adsorption isotherm. Both inhibitors showed comparable inhibition efficiencies., close to 80% at a concentration of 0.01 M.

In [6], the expired desloratidine drug was examined as an inhibitor for carbon steel corrosion in 1 M HCl solution. The potentiodynamic polarization and weight loss techniques were used. Protective efficiency (Z) reaches 92% at a concentration of $19.3 \cdot 10^{-5}$ M. Here, too, without evidence, the blocking mechanism of the inhibitor action is accepted, taking $Z = \theta$, where θ is surface coverage by the inhibitor. It has also been shown that the apparent activation energy E_a increases in the presence of an inhibitor. Adsorption is described using the Langmuir isotherm. $\Delta G^0_{ads} = -41.43$ kJ/mol.

In [7], the corrosion behavior of mild steel in 1 M H_2SO_4 solution in the presence of Pantoprazole drug was investigated using potentiodynamic polarization, quantum chemical calculations, Monte Carlo (MC) and Molecular Dynamic (MD) simulations. The MC and

MD simulations confirm the inhibitor's strong adsorption contact with the metal surface. It appears that the heteroatoms (mostly oxygen and nitrogen) are involved in the adsorption process of the inhibitor based on the adsorption geometries. The creation of a protective anti-corrosion layer on the metal surface is a result of this adsorption affinity. At a concentration of 250 ppm, the protective efficacy of pantoprazole reaches 94%. It acts as a mixed-type inhibitor.

Omeprazole and esOmeprazole drug wastes have been investigated as corrosion inhibitors for C38 steel in 1 M phosphoric acid solution. Their protective efficiency at a concentration of about 10^{-4} M approaches 99% [8, 9].

The aim of this work is to study the inhibitory efficacy of expired drug Omeprazole against carbon steel corrosion in 1 N hydrochloric and sulfuric acid solutions.

Experimental

Electrochemical measurements and corrosion tests were carried out on St3 carbon steel 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 N solutions of HCl and H_2SO_4.

Hydrochloric and sulfuric acids were of "chemically pure" qualifications. The duration of the gravimetric experiments was 24 h. Samples of St3 carbon steel were polished to the 6th grade, degreased with acetone prior to testing.

As an inhibitor, the expired drug Omeprazole (*RS*)-5-methoxy-2-[(4-methoxy-3,5-dimethylpyridin-2-yl)methylsulfinyl]-1*H*-benzimidazole (M=345.417 g/mol) was studied. Its structural formula is shown in Figure 1. The inhibitor concentration was varied within 10–60 mg/L.

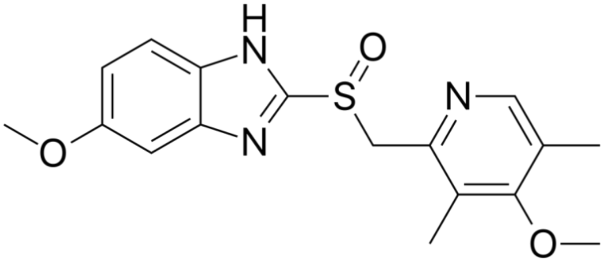

Figure 1. Structural formula of Omeprazole.

Polarization and impedance measurements were carried out in a three-electrode sealed plastic cell after 15 min of keeping the working electrode in the solution. The potentials were measured with respect to the saturated silver/silver chloride electrode and were recalculated to the standard hydrogen scale. The counter electrode is a smooth platinum. Polarization measurements were carried out using an IPC-Pro potentiostat (produced by A.N. Frumkin Institute of Physical Chemistry and Electrochemistry, RAS) by the potentiodynamic method at a potential scan rate of 0.66 mV/s.

Impedance spectra were studied in the frequency range (ω/2π) of 10 kHz–0.05 Hz with an alternating voltage amplitude of 10 mV, using an electrochemical measuring complex from Solartron (UK) consisting of a 1255 SI impedance analyzer and a SI 1287 potentiostat. The results obtained were processed using a program ZView 3.0 that allows calculations for any equivalent circuits with up to 20 elements.

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

$$Z,\% = \frac{K_0 - K_{\text{inh}}}{K_0} \cdot 100 \qquad (1)$$

$$Z,\% = \frac{i_0 - i_{\text{inh}}}{i_0} \cdot 100 \qquad (2)$$

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. Corrosion current densities were calculated by extrapolating the Tafel sections of the polarization curves to the corrosion potential.

Electron microscopy (JSM-6390) was used to characterize the surface of the samples after the tests.

Results and Discussion

On Figure 2, the polarization curves measured in 1.0 N HCl solution are shown, and Table 1 shows the kinetic parameters calculated on their base. One can see that Omeprazole causes inhibition of both partial electrode reactions, which increases with an increase in its concentration. The protective effect of the inhibitor increases from 58 to 84% with an increase in its concentration in the range of 10–40 mg/L. Corrosion potential E_{corr} is shifted in the negative direction by 10 mV in the presence of the inhibitor. The Tafel slope coefficients b_a of the anodic polarization curves in the background solution and at the minimum inhibitor concentration are 0.070 V and increase to 0.10 V with increasing inhibitor concentration. The Tafel slopes of the cathodic polarization curves b_c are close to 0.10 V in all solutions.

Table 1. Kinetic parameters of the St3 electrode in 1 M HCl solution in the absence and in the presence of Omeprazole and its protective effect.

Omeprazole concentration, mg/L	$-E_{\text{corr}}$, V	i_{corr}, A/m^2	b_a, V	b_c, V	Z, %
Absent	0.24	1.50	0.070	0.095	–
10	0.25	0.63	0.072	0.095	58
20	0.25	0.51	0.100	0.100	67
40	0.25	0.26	0.100	0.100	84

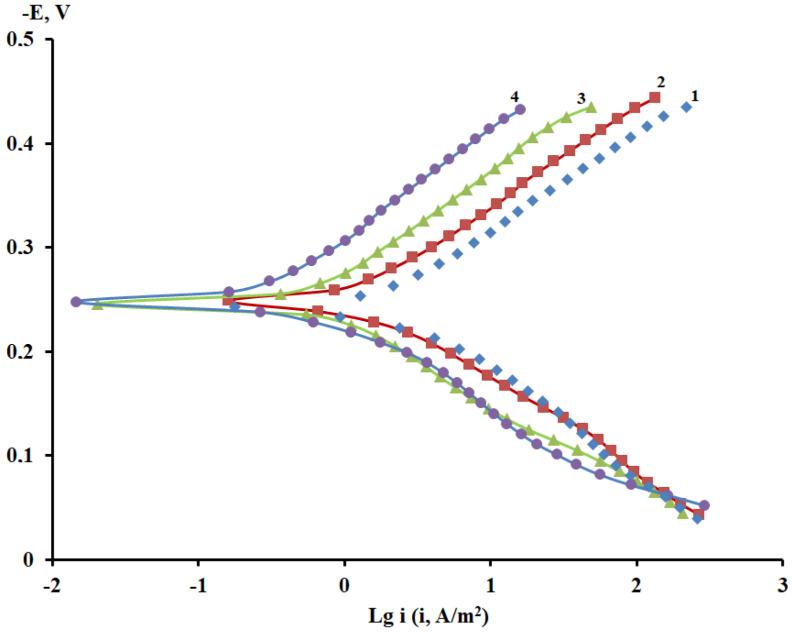

Figure 2. Polarization curves measured on a steel electrode in a solution of 1.0 N HCl in the absence (1) and in the presence of Omeprazole, mg/L: 2 – 10; 3 – 20; 4 – 40.

The polarization curves measured on a steel electrode in a 0.5 M (1.0 N) sulfuric acid solution in the absence and in the presence of Omeprazole additives are characterized by inhibition of both electrode reactions in the inhibited solution. However, the concentration effect of the inhibitor is practically absent (Figure 3). Corrosion potential practically does not change, as does the protective effect of the inhibitor (Table 2). The values of b_a are equal to 0.050 V, $b_c = 0.100–0.115$ V.

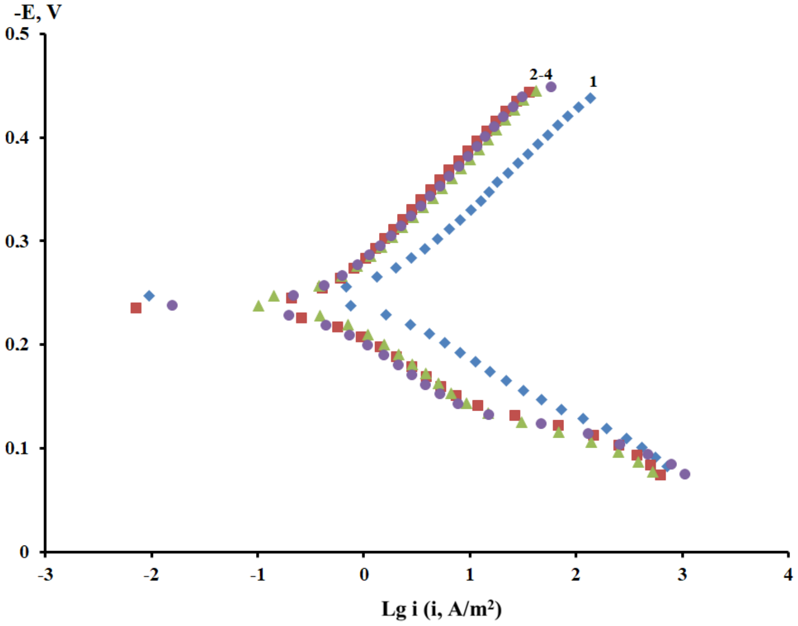

Figure 3. Polarization curves measured on a steel electrode in a solution of 1.0 N H_2SO_4 in the absence (1) and in the presence of Omeprazole, mg/L: 2 – 10; 3 – 20; 4 – 40.

Table 2. Kinetic parameters of the St3 electrode in 1.0 N H_2SO_4 solution in the absence and in the presence of Omeprazole and its protective effect.

Omeprazole concentration, mg/L	$-E_{corr}$, V	i_{corr}, A/m^2	b_a, V	b_c, V	Z, %
Absent	0.25	1.26	0.050	0.100	–
10	0.24	0.10	0.050	0.115	78
20	0.25	0.10	0.050	0.115	78
40	0.24	0.10	0.050	0.115	78

The results of gravimetric corrosion tests of steel in the studied acid solutions and the magnitude of the protective effect of the inhibitor are shown in Table 3. The protective effect of the inhibitor increases with its concentration in both media. A comparison of the *Z* values calculated from the data of gravimetric and polarization measurements shows a qualitative agreement. Some discrepancies in the values are explained by the different duration of the tests: in the first case, the exposure time was 24 hours, in the second one – no more than half an hour.

Table 3. Steel corrosion rate in HCl and H_2SO_4 solutions and protective effect of Omeprazole according to the gravimetric corrosion tests.

C_{inh}, mg/L	K, g/(m^2·h)		Z_{inh}, %	
	HCl	H_2SO_4	HCl	H_2SO_4
0	2.6626	9.5156	–	–
10	0.2580	2.6181	90	72
20	0.2284	1.2429	91	87
40	0.2168	0.9347	92	90
60	0.2713	0.4649	90	95

The protective effect of Omeprazole in HCl solution is slightly higher than in sulfuric acid medium. So, already at a concentration of 10 mg/L, the value of *Z* reaches 90%, while in the H_2SO_4 solution it is only 72%. But at C_{inh}=40 mg/L, the *Z* values in both media are close to 90%.

SEM images of the sample surface after corrosion tests in inhibited and non-inhibited sulfuric acid solutions are shown in Figure 4. The state of the metal surface after exposure to the inhibited solution indicates a good protective efficiency of the inhibitor. A similar picture is typical for 1.0 N HCl solution.

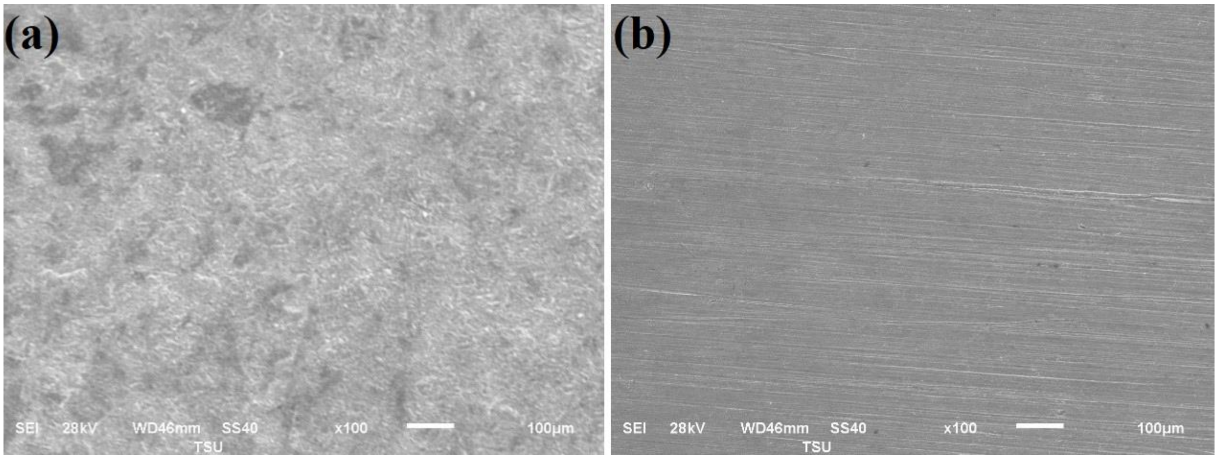

Figure 4. SEM image of the surface of carbon steel after 24 h of immersion in 1.0 N H_2SO_4 solution in the absence (a) and in the presence (b) of 60 mg/L Omeprazole.

The protective effectiveness of the inhibitor studied in HCl and H_2SO_4 solutions is confirmed by the impedance measurement results (Figures 5 and 6).

Figure 5 shows the hodographs measured in 1.0 N HCl solution. As can be seen, the diameter of the arcs increases with an increase in the inhibitor concentration, which indicates an increase in the total resistance in the system and a decrease in the corrosion rate. This is consistent with the results obtained from polarization curves and gravimetric tests.

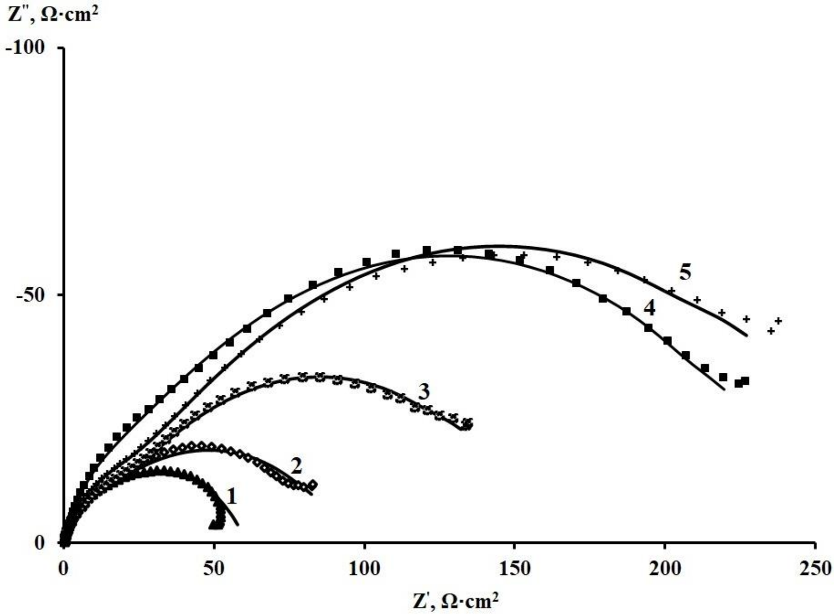

Figure 5. Nyquist diagram for steel electrode in 1.0 N HCl solution at corrosion potential in the absence (1) and in the presence of Omeprazole, mg/L: 2 – 10; 3 – 20; 4 – 30; 5 – 40.

The diameter of semicircles in a Nyquist diagram measured in H_2SO_4 solution increases with increasing the inhibitor concentration (Figure 6). This indicates an increase in its effectiveness in reducing the corrosion rate of steel, as in a hydrochloric acid solution. The impedance data indicate the presence of a concentration dependence of the protective effectiveness of the inhibitor, which was observed in the gravimetric tests, but was absent in polarization measurements in a sulfuric acid solution. It is possible that in a sulfuric acid medium, a higher sensitivity of the impedance spectroscopy method to changes in the concentration of Omeprazole is manifested in comparison with polarization measurements.

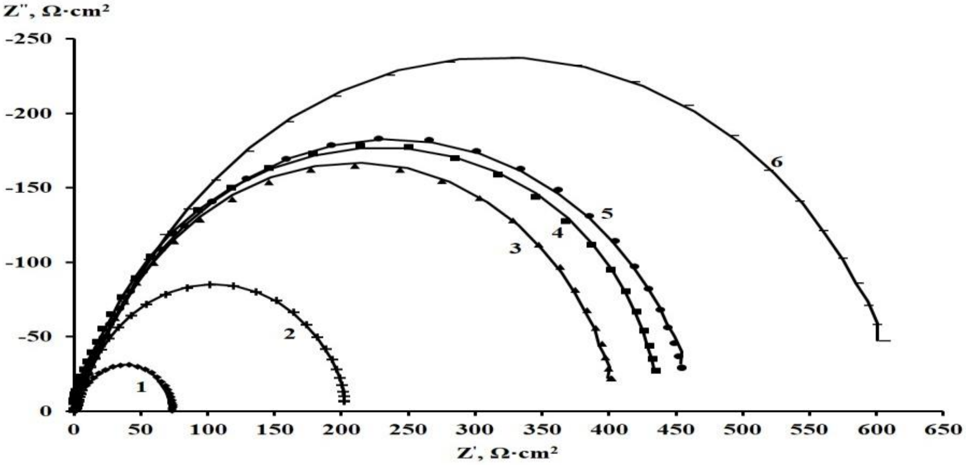

Figure 6. Nyquist diagram for steel electrode in 1.0 N H_2SO_4 solution at corrosion potential in the absence (1) and in the presence of Omeprazole, mg/L: 2 – 10; 3 – 20; 4 – 30; 5 – 40, 6 – 60.

To process the experimental data of the electrochemical impedance, the equivalent electrical circuit shown in Figure 7 has been considered. In it, R_s is the resistance of the solution, R_1 and R_2 are the charge transfer resistance of the anodic and cathodic partial electrode reactions, respectively, C_{dl} is the double layer capacitance, C_a and R_a are the capacitance and transfer resistance of the intermediate adsorbed particles formed during the anodic ionization of steel in acid media, $Z(d)$ is Warburg diffusion impedance.

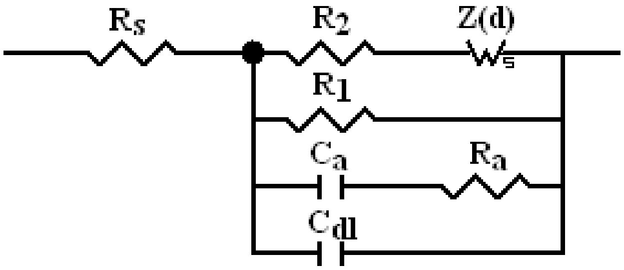

Figure 7. Equivalent circuit simulating the behavior of a steel electrode in acid solutions.

Table 4 shows the data of impedance spectroscopy for carbon steel obtained in 1.0 N H_2SO_4 solution without and with tested Omeprazole concentrations.

Table 4. Numerical values of elements of the equivalent circuit at E_{corr} of the steel electrode in 1.0 N H_2SO_4 solution without and with tested concentration of Omeprazole.

Parameter	**Background**	**C_{inh}, mg/L**			
		10	**20**	**40**	**60**
R_s, $\Omega\cdot cm^2$	0.76	1.35	1.35	1.90	2.05
C_{dl}, $\mu F/cm^2$	58.52	23.02	13.38	11.11	9.12
R_1, $\Omega\cdot cm^2$	71.36	204.1	405.4	465.3	620.1
R_2, $\Omega\cdot cm^2$	51.97	35.89	82.06	60.06	97.12
C_a, $\mu F/cm^2$	39.1	11.3	4.56	4.02	2.88
$Z(d)-R$, $\Omega\cdot cm^2$	132120	24946	41198	34664	39794
$Z(d)-P$	0.91	0.80	0.83	0.79	0.78
$Z(d)-T$, s	12.78	1.99	1.86	2.07	2.08
R_a, $\Omega\cdot cm^2$	5.14	8.72	28.10	21.47	32.68

From Table 4 it follows that with increasing concentration of Omeprazole the charge transfer resistance in the anodic reaction R_1 increases with a less significant increase in the charge transfer resistance of the cathodic reaction R_2. As the inhibitor concentration increases, a decrease in the electric double layer capacitance C_{dl} takes place. This indicates an adsorption of the inhibitor on the electrode surface. Similar dependences of R_1, R_2, and C_{dl} on the concentration of Omeprazole are observed for a steel electrode at the corrosion potential and in 1 N HCl solution (Table 5). The results obtained allow to calculate electrode surface coverage θ with the inhibitor according to the formula [10]:

$$\theta = \frac{C_0 - C}{C_0 - C_1} \tag{3}$$

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 inhibitor particles, respectively.

The calculation of C_1 is performed according to the dependence $C_{dl}=C_{dl}(1/C_{inh})$ for H_2SO_4 and HCl solutions (Figure 8).

The value of C_1 calculated according to Figure 8 is equal to 7 $\mu F/cm^2$ in H_2SO_4 solution and 6 $\mu F/cm^2$ in HCl solution. The corresponding coverages θ of the electrode surface with Omeprazole at different concentrations in 1.0 N H_2SO_4 and 1.0 N HCl solutions are presented in Table 6.

Table 5. Numerical values of elements of the equivalent circuit at E_{corr} of the steel electrode in 1.0 N HCl solution without and with tested concentrations of Omeprazole.

Parameter	**Background**	**Omeprazole, mg/L**			
		10	**20**	**30**	**40**
R_s, $\Omega\cdot cm^2$	0.49	0.49	0.47	1.0	0.49
C_{dl}, $\mu F/cm^2$	100.55	56.07	34.61	19.20	20.68
R_1, $\Omega\cdot cm^2$	60.41	93.98	158.7	250.5	276.0
R_2, $\Omega\cdot cm^2$	14.39	10.13	16.47	22.91	21.00
C_a, $\mu F/cm^2$	39.1	21.05	19.77	10.07	10.88
$Z(d)-R$, $\Omega\cdot cm^2$	2968	1524	1100	8879	2758
$Z(d)-P$	0.59	0.50	0.54	0.56	0.54
$Z(d)-T$, s	33.92	31.62	23.28	12.01	22.19
R_a, $\Omega\cdot cm^2$	8.70	17.34	20.69	8.85	32.19

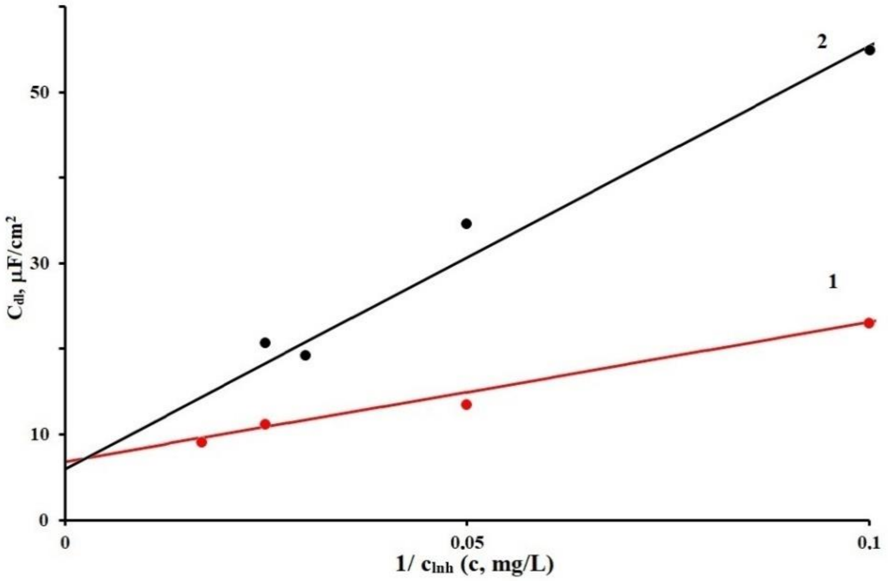

Figure 8. The dependence of C_{dl} ($\mu F/cm^2$) on $1/C_{Inh}$ in 1.0 N H_2SO_4 (1) and 1.0 N HCl (2) solutions.

Table 6. The values of the electrode surface coverage with Omeprazole in H_2SO_4 and HCl solutions.

C_{inh}, **mg/L**	**10**	**20**	**30**	**40**	**60**
θ in H_2SO_4	0.59	0.87	–	0.92	0.96
θ in HCl	0.47	0.70	0.86	0.84	–

To select an isotherm corresponding to the data given in Table 6, it was checked their correspondence to the Temkin isotherm $Bc=\exp(f\theta)$, 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 graphic 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.

It turned out that the best fitting of the data to the linear dependence corresponds to the Langmuir isotherm (Figure 9). The segment cut off on the vertical axis of Figure 9 makes it possible to calculate the adsorption equilibrium constant B. The value of B is 0.14 L/mg in the H_2SO_4 solution and 0.09 L/mg in the HCl solution.

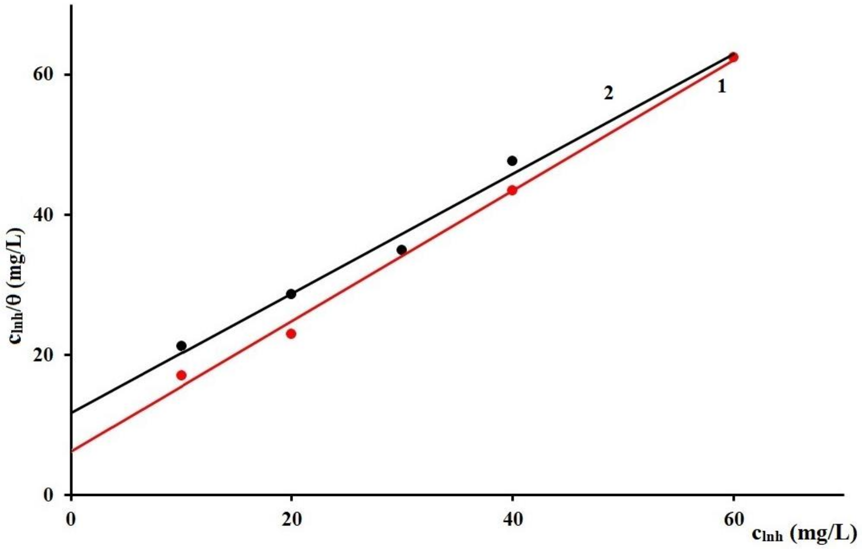

Figure 9. Dependence c/θ *vs.* c for 1.0 N H_2SO_4 (1) and 1.0 N HCl (2) solutions with Omeprazole.

The value of the adsorption equilibrium constant B enables us to calculate the value of free adsorption energy according to the equation:

$$-\Delta G^0_{\text{ads}} = RT\ln\left(B\cdot 10^6\right),$$

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

The value of $-\Delta G^0_{\text{ads}}$ at a temperature of 298 K in 1.0 N H_2SO_4 and 1.0 N HCl is characterized by the values of 29.3 and 28.3 kJ/mol, respectively. There is practically no effect of the medium on ΔG^0_{ads}.

It should be borne in mind that the adsorption of inhibitor particles occurs by displacing adsorbed water molecules from the steel surface, since iron (steel) is a hydrophilic metal. Therefore, the calculated value actually represents a change in free energy due to the energy spent on the displacement of water molecules and the release of energy directly due to adsorption. Therefore, the real value of ΔG^0_{ads} must include the energy expended to displace adsorbed water molecules. Naturally, it should be noticeably higher than the obtained value, which indicates the chemisorption of the inhibitor on the steel surface.

Conclusions

The protective effectiveness of the expired drug Omeprazole against corrosion of carbon steel in 1 N solutions of sulfuric and hydrochloric acids was studied using gravimetry, potentiodynamic polarization, and impedance spectroscopy methods. The magnitude of 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.

Impedance spectroscopy data on the effect of Omeprazole concentration on the electric double layer capacitance made it possible to calculate the surface coverage of the steel electrode, determine the adsorption isotherm, and calculate the free energy of Omeprazole adsorption.

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