

Mixtures of berberine and dihydroberberine with sulfur-containing compounds as steel corrosion inhibitors in sulfuric acid

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Abstract

The inhibiting effect of berberine, dihydroberberine, phenylthiourea, diphenylthiourea, 2-mercaptobenzothiazole, and sodium diethyldithiocarbamate on the corrosion of low-carbon steel in sulfuric acid has been studied. It has been shown that in the concentration range of 10^{-5} – 10^{-4} mol/L at 25°C, these organic compounds decrease the corrosion rate of steel. Depending on the concentration, the protective efficiency of the compounds ranges within 34–94%. The efficiency decreases with an increase in temperature. The inhibitory effect of some compounds changes to corrosion stimulation at temperatures above 60°C. The compounds studied inhibitor the cathodic evolution of hydrogen, owing to which they reduce the corrosion rate of steel. It has been shown that mixtures of berberine and dihydroberberine with sulfur-containing organic compounds are more efficient as steel corrosion inhibitors. The protective effect of the mixtures on steel corrosion is 85–99.7% at 25°C. It has been found that in the majority of mixtures, the components strengthen the protective effects of each other. It has been shown that in the presence of mixtures in sulfuric acid, the effective activation energy of the corrosion process increases significantly and the capacitance of the double electrical layer decreases. Inhibitor mixtures inhibit both electrode reactions.

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Introduction

Sulfuric acid is commonly used for steel etching in industry because it is cost effective and does not evaporate. Steels are the main structural materials used in various industries. Large amounts of steel are etched with sulfuric acid. Corrosion inhibitors are added to the etching solutions to prevent the metal loss. Efficient acid corrosion inhibitors include pure N, S, O-containing organic compounds or their mixtures [1–6]. Both organic and inorganic components are used in inhibitive mixtures. The components can strengthen or weaken the protective effect of each other. In most cases, mixtures are more efficient than individual compounds [7]. The use of compounds obtained from natural sources as corrosion inhibitors is considered a promising approach [8–14].

Berberine and dihydroberberine have been studied as individual inhibitors in the corrosion of steel in hydrochloric acid [15].

The purpose of this work is to study the protective properties of berberine, dihydroberberine and their mixtures with phenylthiourea, diphenylthiourea, 2-mercaptobenzothiazole and sodium diethyldithiocarbamate in the corrosion of steel in sulfuric acid.

Experimental

Samples were cut from low carbon steel. Their sizes of $0.2 \times 1 \text{ cm}^2$ for impedance measurements, $0.5 \times 1 \text{ cm}^2$ for polarization measurements, and $1 \times 2.5 \text{ cm}^2$ for corrosion tests. Prior to the measurements, the samples were cleaned with emery paper, degreased with alcohol, washed with distilled water, and dried with filter paper. A 0.5 M solution of sulfuric acid was used as the working solution. Mass loss measurements were performed at 25, 40, 60, and 80°C. The studied concentration range of berberine (BBR), dihydroberberine (HBR), phenylthiourea (PTU), diphenylthiourea (DPTU), 2-mectaptobenzithiazole (Kaptax) and sodium diethyldithiocarbamate (DEDTC) was 10^{-5} – 10^{-4} mol/L.

The corrosion rate K was calculated by formula (1):

$$K = \frac{\Delta m}{\tau \cdot S} \quad (1)$$

where Δm is the mass change (grams), τ is the experiment duration (hours), and S is the sample area (m^2).

The efficiency of the additives was estimated by the inhibition coefficient γ (2) and degree of protection Z (3):

$$\gamma = \frac{K_0}{K_i} \quad (2)$$

$$Z = \frac{(K_0 - K_i)}{K_0} \cdot 100\% \quad (3)$$

where K_0 and K_i are the corrosion rates in the uninhibited acid and in the presence of an inhibitor or a mixture, respectively. The mutual effect of mixture components was estimated by the coefficient of mutual effect σ (4) [16]:

$$\sigma = \frac{\gamma_m}{\gamma_1 \cdot \gamma_2} \quad (4)$$

where γ_m , γ_1 and γ_2 are the inhibition coefficients of the mixture and its components, respectively.

Polarization measurements were performed on a PI-50.01 potentiostat (Gomel, Belarus) in a three-electrode temperature-controlled cell at 25°C. A platinum auxiliary

electrode and a saturated silver chloride electrode were used. Potentials E are reported *versus* the latter. Polarization curves were recorded by varying the potential in a stepwise manner from the low value, $E=-0.7$ V, to the high value, $E=-0.3$ V, in 20 mV steps. The current was recorded after keeping the electrode at a certain potential for one minute. Each curve was recorded in triplicate and the results were then averaged.

Capacitance measurements were performed on a Z-500 impedance meter (OJSC Elins, Russia) in a two-electrode cell in a frequency range of 50 mHz–300 kHz at the corrosion potential. A cylindrical platinum electrode was used as the auxiliary electrode. The degree of coverage of the electrode surface was calculated using the formula:

$$\Theta = \frac{(C_0 - C_i)}{C_0} \quad (5)$$

where C_0 and C_i are the double electric layer (DEL) capacitances in the sulfuric acid solution without and with inhibitors, respectively.

Results and Discussion

Efficiency of individual compounds

Based on the mass loss data, the dependence of the protective effect of the compounds on the corrosion of low carbon steel in 0.5 M sulfuric acid solution *versus* their concentration was obtained. The results are presented in Table 1.

The effect of the inhibitors is insignificant at small concentrations but increases as the concentration increases. At a concentration of 0.1 mmol/L, the Z value of all the inhibitors is above 79%.

In logarithmic coordinates, the plot of $\lg \gamma$ vs. $\lg C$ is almost linear, which indicates that the effect of steel surface blocking by the inhibitor exists, Figure 1.

The effect of temperature on the corrosion rate and protective effect was studied at an inhibitor concentration of 0.1 mmol/L. As the temperature increases, the corrosion rate increases and the protective effect of inhibitors decreases. On increasing the temperature to 80°C, the inhibitory effect of Kaptax, DEDTC and HBR changes to stimulation. The protective effects of the above compounds at 40 and 60°C are 39, 74, 76 and 37, 38, 7%, respectively.

The protective effect of BBR, PTU and DPTU decreases by 10–20% with an increase in temperature from 40 to 80°C. The effective activation energies of the corrosion process in sulfuric acid are presented in Table 2.

The inhibitors (except for DEDTC) insignificantly increase the effective activation energy of the corrosion process (Table 2).

Thus, all the organic compounds studied manifest an inhibitive effect on steel corrosion at temperatures below 60°C.

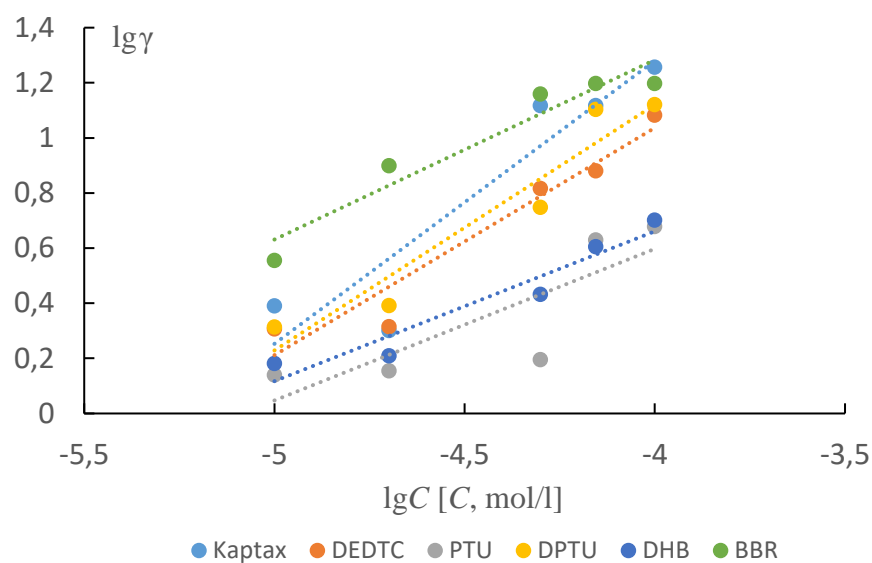


Figure 1. Plot of inhibition coefficient vs. the concentration of organic compounds.

Table 1. Dependence of the protective effect on the inhibitor nature and concentration, $t = 25^\circ\text{C}$.

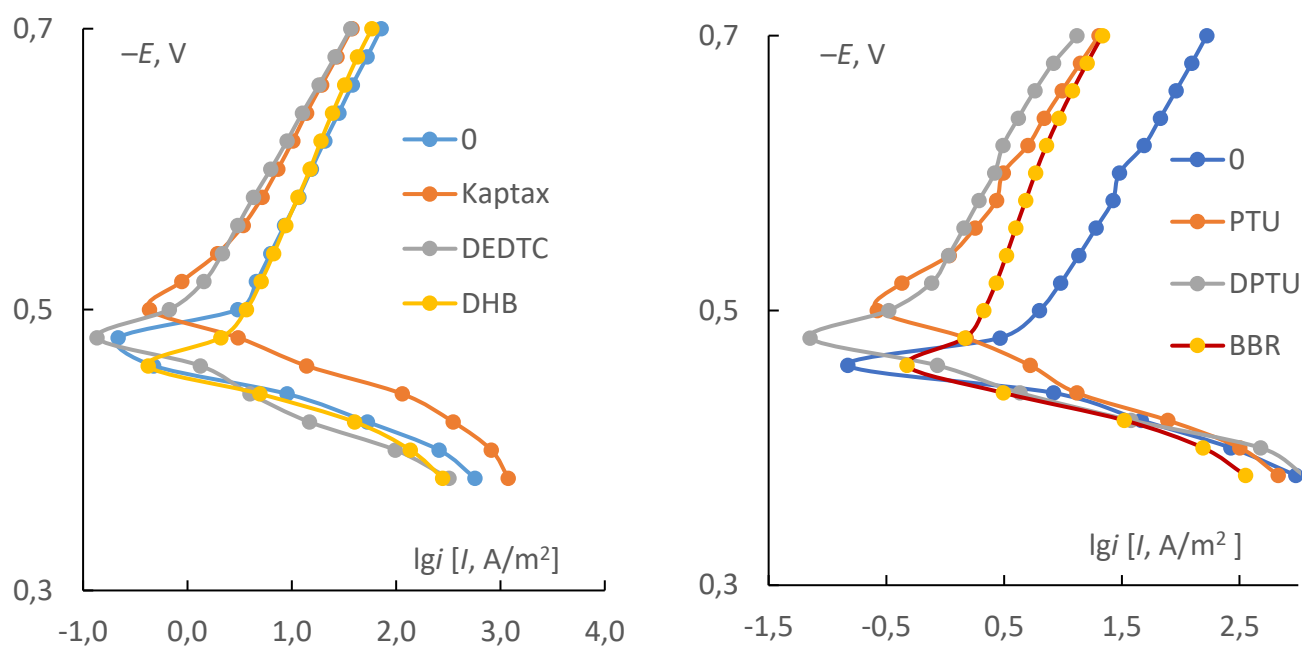
Parameter	Parameter value for inhibitors at concentration C , mmol/L				
	0.01	0.02	0.05	0.07	0.1
HBR					
γ	1.52	1.62	2.71	4.04	5.04
Z , %	34.19	38.57	63.05	75.27	80.17
BBR					
γ	3.60	7.94	14.43	15.77	15.77
Z , %	72.26	87.4	93.07	93.66	93.66
PTU					
γ	1.38	1.43	1.57	4.29	4.77
Z , %	27.60	30.20	36.30	76.69	79.08
DPTU					
γ	2.06	2.47	5.60	12.71	13.21
Z , %	51.45	59.43	82.15	92.29	93.99
Kaptax					
γ	2.46	2.02	13.12	13.12	18.10
Z , %	59.43	50.48	92.38	92.38	94.48

Parameter	Parameter value for inhibitors at concentration C , mmol/L				
	0.01	0.02	0.05	0.07	0.1
DEDTC					
γ	2.0	2.1	6.56	7.61	12.15
Z , %	50.67	52.38	84.76	86.86	91.77

Table 2. Effective activation energies of the corrosion process and correlation coefficients of linear plots.

Parameter	0	HBR	BBR	PTU	DPTU	Kaptax	DEDTC
E_{ef} , kJ/mol	29.8	49.3	40.2	75.6	33.4	34.7	28.1
R , %	95.68	98.28	93.91	95.84	91.54	98.27	96.15

As concerns the effect on the partial electrode reactions, they are cathodic inhibitors since they predominantly inhibit the cathodic hydrogen evolution, Figure 2.

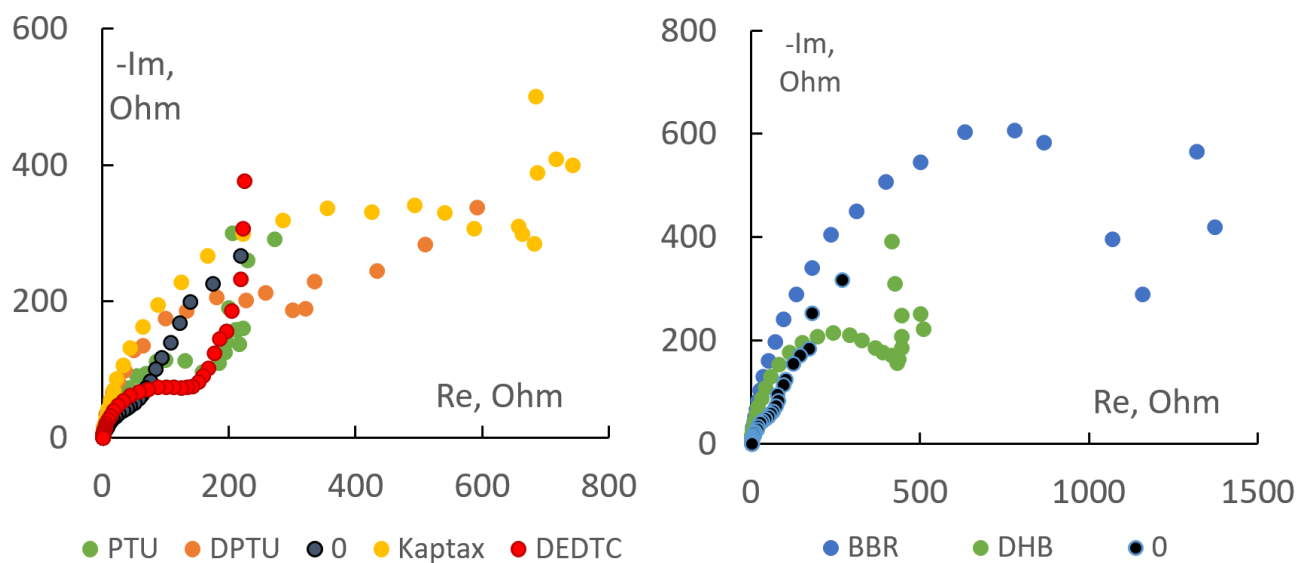
**Figure 2.** Polarization curves of low carbon steel in 0.5 M sulfuric acid without (0) and with additives, $C=0.01$ mmol/L.

The inhibitors nearly do not change the polarizability of the cathodic and anodic reactions, as the close values of the Tafel coefficients b_c and b_a demonstrate. In fact, the b_c and b_a values in the pure acid and in the presence of inhibitors amount to 140 ± 10 and 34 ± 5 mV, respectively. Thiourea derivatives and Kaptax decrease the corrosion potential of steel, owing to which apparent stimulation of the anodic reaction occurs, Table 3.

Table 3. Dependence of the corrosion inhibition coefficient on the potential and nature of the additive.

$-E, \text{mV}$	Values of γ for additives					
	HBR	BBR	PTU	DPTU	Kaptax	DEDTC
700	1.23	7.71	8.24	12.69	1.91	1.96
640	1.15	7.25	9.69	16.04	2.04	2.25
480	–	1.99	1.94	41.15	–	–
420	1.33	1.39	0.59	1.22	0.15	3.59
400	1.88	1.73	0.84	0.56	0.32	2.62
380	2.04	2.68	1.41	0.85	0.48	32.07

The Nyquist plots that characterize the frequency dependence of the real and imaginary impedance components are shown in Figure 3.

**Figure 3.** Nyquist plots on steel electrode in sulfuric acid solutions without and with individual compounds. $C=0.01 \text{ mmol/L}$.

In the region of medium and high frequencies, the diagrams have the shape of semicircles. If the solution contains inhibitors, its radius is larger than in the pure sulfuric acid solution. This indicates that the additives manifest an inhibitive effect.

Effect of inhibitor mixtures on steel corrosion

Let us consider the effect of binary mixtures of berberine and dihydroberberine with sulfur-containing organic compounds on the corrosion process. The concentration of the sulfur-containing compound in the mixtures is kept constant at 0.1 mmol/L , while the concentration

of berberine or dihydroberberine is varied from 0.01 to 0.07 mmol/L. The data obtained by the mass loss method are presented in Table 4.

The inhibition coefficients in the presence of inhibitor mixtures in solutions at 25°C are higher than in the presence of the corresponding individual compounds, as shown in Tables 1 and 4. The coefficients of mutual effect of the components are presented in Table 5.

Table 4. Dependence of the inhibition coefficient and degree of protection on the nature and concentration of the components, 25°C.

$C \cdot 10^5$, mol/L	Values of γ and Z for mixtures							
	γ	Z , %	γ	Z , %	γ	Z , %	γ	Z , %
	DEDTC+HBR		DEDTC+BBR		Kaptax+HBR		Kaptax+BBR	
1	93.44	98.93	25.5	96.08	25.5	96.08	64.62	98.45
2	103.54	99.03	100	99.0	44.09	97.73	149.13	99.33
5	123.58	99.19	193	99.48	70.31	98.58	153.05	99.34
7	123.58	99.19	197	99.49	216.79	99.54	323.1	99.69
	PTU+HBR		PTU+BBR		DPTU+HBR		DPTU+BBR	
1	85.44	98.83	50.84	98.04	256.3	99.61	9.63	89.62
2	100.9	99.00	106.6	99.06	128.15	99.22	9.82	89.82
5	100.9	99.00	112.1	99.11	222.13	99.55	26.67	96.25
7	208.25	99.52	93.0	98.93	256.3	99.61	25.57	96.08

Table 5. Dependence of the coefficients of mutual effect on the nature and concentration of the components, 25°C.

$C \cdot 10^5$, mol/L	Values of σ for the mixtures							
	DEDTC with		Kaptax with		PTU with		DPTU with	
	HBR	BBR	HBR	BBR	HBR	BBR	HBR	BBR
1	5.12	0.57	0.92	0.99	13.10	3.32	12.76	0.2
2	5.29	1.05	1.5	1.04	14.51	3.15	5.98	0.09
5	3.78	1.11	1.43	0.57	8.68	1.80	6.21	0.14
7	2.56	1.04	3.0	1.13	12.0	1.37	4.80	0.12

In most cases, the coefficients of mutual effect are larger than one. This indicates that the components mutually enhance the protective effects of each other at this ratio of concentrations. The mixture of BBR and DPTU is an exception: regardless of the

concentration of berberine, the values of σ are less than 1 and mutual weakening of the action of inhibitors occurs. No clear dependence of σ on the concentration of the second component is observed.

As a rule, an increase in the temperature results in a decrease in the efficiency of the mixtures, Table 6.

Table 6. Inhibition coefficients and degree of protection depending on the composition, concentration of the first component of the mixture, and temperature. The concentration of the second component is $C=0.1$ mmol/L.

$t, ^\circ\text{C}$	Parameter	Values of the parameters at the concentration of the first component in the mixture, mmol/L:							
		0.01	0.02	0.05	0.07	0.01	0.02	0.05	0.07
		BBR+PTU				BBR+DPTU			
40	γ	92.8	5.72	49.9	4.31	100	148	168	185
	Z, %	98.9	82.52	97.99	76.83	99	99.32	99.4	99.46
60	γ	6.77	20.37	13.17	20	3.16	21.9	37.57	39.75
	Z, %	85.23	95.09	92.41	95	68.43	95.44	97.34	97.48
80	γ	16.35	15.94	56.51	34.36	1.72	8.32	12.37	61.69
	Z, %	93.88	93.73	98.23	97.09	41.78	87.98	91.92	98.38
		BBR+Kaptax				BBR+DEDTC			
40	γ	20.84	22.98	24.78	29.34	21.85	28	34.46	49.78
	Z, %	95.20	95.65	95.96	96.59	95.42	96.42	97.06	97.99
60	γ	0.87	22.85	79.23	135.83	1.81	6.39	23.89	62.49
	Z, %	—	95.62	98.73	99.26	44.99	84.37	95.81	98.40
80	γ	1.28	6.23	32.81	40.19	1.17	1.0	1.38	1.47
	Z, %	22.07	83.95	96.95	97.51	14.63	—	27.69	32.23
		HBR+PTU				HBR+DPTU			
40	γ	46.65	40.63	43.43	25.19	21.20	33.09	18.2	7.13
	Z, %	97.86	97.54	97.69	96.03	95.28	96.98	94.51	85.99
60	γ	12.02	50.87	19.08	99.2	28.96	29.24	32.74	28.14
	Z, %	91.68	98.03	94.76	98.99	96.55	96.58	96.95	96.45
80	γ	2.11	1.98	5.29	16.7	1.16	1.29	2.53	2.07
	Z, %	52.53	49.64	81.12	94.01	13.68	22.51	60.57	51.68

$t, ^\circ\text{C}$	Parameter	Values of the parameters at the concentration of the first component in the mixture, mmol/L:							
		0.01	0.02	0.05	0.07	0.01	0.02	0.05	0.07
		HBR+Kaptax				HBR+DEDTC			
40	γ	3.68	4.36	5.23	5.74	2.41	3.34	3.25	4.53
	Z, %	72.86	77.09	81.22	82.57	58.55	70.11	69.21	77.92
60	γ	7.82	7.99	13.73	10.44	0.93	0.86	1.08	1.45
	Z, %	87.22	87.48	92.71	90.43	–	–	7.8	31.38
80	γ	1.44	1.83	7.24	6.24	1.06	1.10	1.14	1.23
	Z, %	30.44	45.5	86.19	83.69	6.28	9.29	12.47	19.23

The temperature dependence of the efficiency of the BBR+DPTU mixture is somewhat different. At 40 and 60°C, BBR at almost all concentrations has a higher inhibition coefficient than at 25°C.

In most cases, the effective activation energy of the corrosion process in the presence of the mixtures in the sulfuric acid solution is higher than that of the individual inhibitors, see Tables 7 and 2.

Table 7. Effect of the nature and composition of the inhibitive mixtures on the effective activation energy of the corrosion process.

C, mmol/L	Values of E_{act} (kJ/mol) for the mixtures							
	BBR with				HBR with			
	Kaptax	DEDTC	PTU	DPTU	Kaptax	DEDTC	PTU	DPTU
0.01	142.7	127.4	47.5	89.2	108.6	78.0	127.1	153.2
0.02	118.2	144.2	35.5	68.3	98.2	80.9	119.8	140.9
0.05	98.3	147.6	40.6	69.6	89.9	86.1	97.4	133.9
0.07	56.0	127.6	31.7	44.3	108.3	91.2	88.7	133.8

This agrees with the larger protective efficiency of the mixtures in comparison with the individual inhibitors.

The effect of the mixtures on the partial electrode reactions also differs. As an example, Figure 4 shows the polarization curves of steel in a solution of mixtures of berberine with sulfur-containing compounds.

Almost irrespective of the concentration of BBR, the inhibitor mixtures can be attributed to mixed-type inhibitors since they slow down both electrode reactions.

The Nyquist plots for the inhibitor mixtures are presented in Figure 5.

Like with individual inhibitors, the semicircle radius for the mixtures is larger than in the pure sulfuric acid solution. The impedance plots of the individual inhibitors and mixtures are satisfactorily described by the equivalent circuit shown in Figure 5, where R_1 is the electrolyte resistance, C is the DEL capacitance, and CPE is the constant phase element. The DEL capacitances and degrees of surface coverage with the inhibitors and their mixtures are presented in Table 8.

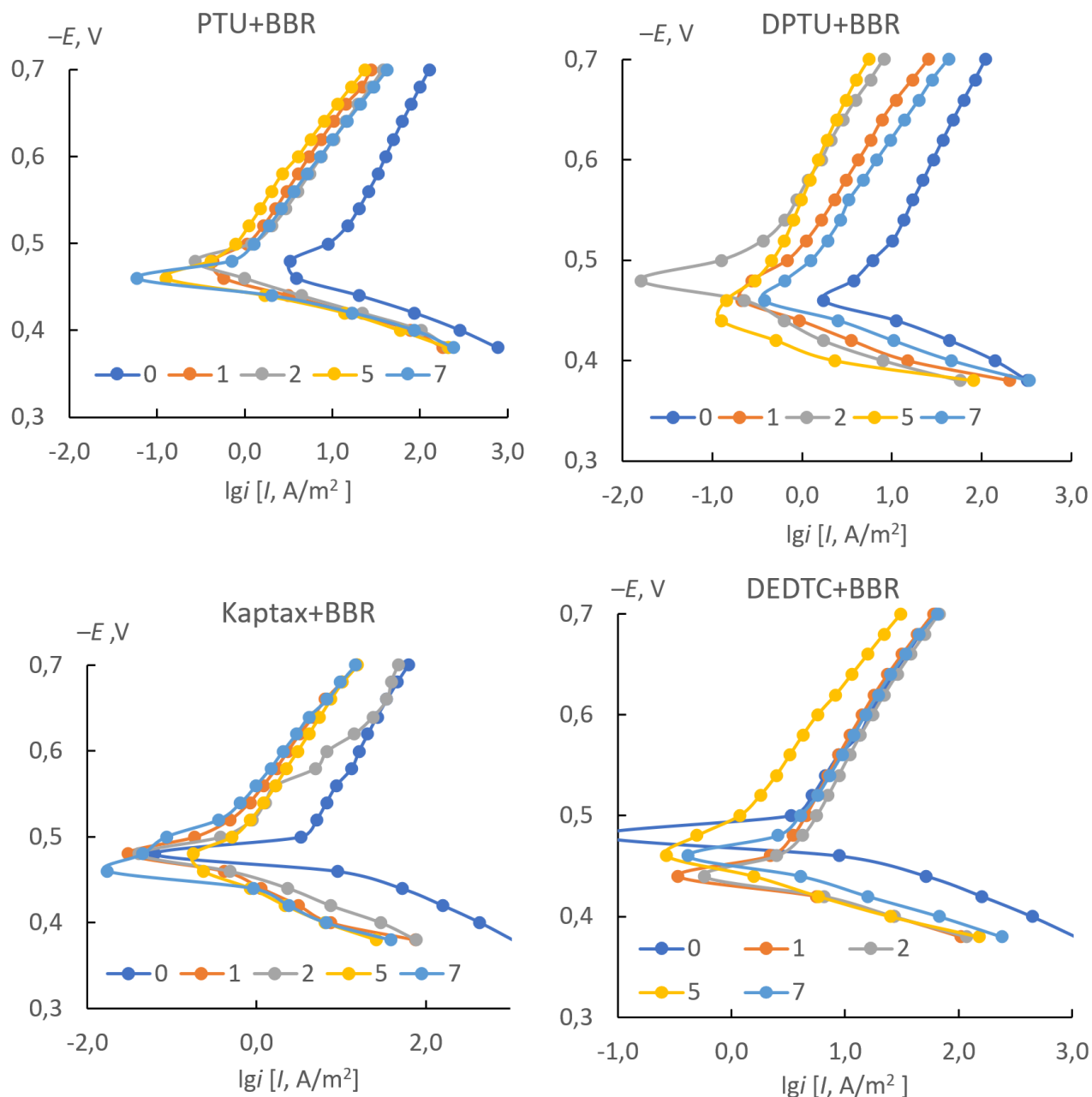


Figure 4. Polarization curves of steel in 0.5 M in sulfuric acid without (0) and in the presence of mixtures of BBR with sulfur-containing organic compounds. BBR concentration, mmol/L: 0.07 (1), 0.05 (2), 0.02 (5), 0.1 (7). The concentration of the second component is 0.1 mmol/L.

Table 8. Dependence of capacitance and degree of surface coverage on the nature and concentration of one of the mixture components. $C_0=4.2 \cdot 10^{-5}$ F. The concentration of the individual additives is 0.1 mmol/L.

Additive	$C \cdot 10^5, F$	Θ	Additive	$C \cdot 10^5, F$	Θ
BBR	1.2	0.71	Kaptax	1.18	0.48
DEDTC	1.1	0.74	PP4	1.24	0.71
DPTU	1.14	0.73	PTU	1.88	0.55
BBR+DEDTC			BBR+Kaptax		
1*	4.1	0.023	1	1.4	0.67
2	2.18	0.52	2	1.29	0.69
5	2.1	0.53	5	1.6	0.62
7	2	0.52	7	1.8	0.57
BBR+DPTU			BBR+PTU		
1	1.22	0.709	1	1.8	0.57
2	1.73	0.59	2	2.3	0.45
5	1.16	0.72	5	2.3	0.45
7	1.4	0.67	7	1.8	0.57
HBR+DEDTC			HBR+Kaptax		
1	4.0	0.048	1	2.8	0.33
2	3.6	0.14	2	2.7	0.36
5	3.46	0.176	5	2.78	0.34
7	3.46	0.176	7	1.3	0.69
HBR+DPTU			HBR+PTU		
1	1.3	0.69	1	1.6	0.62
2	0.63	0.85	2	2.99	0.29
5	1.35	0.68	5	2.0	0.52
7	0.75	0.82	7	1.8	0.57

*Note: Numbers 1, 2, 5 and 7 correspond to the concentrations of the second mixture component: 0.01, 0.02, 0.05 and 0.07 mmol/L, respectively.

As one could have expected, in the presence of individual organic compounds and their mixtures, the DEL capacitance is smaller than in the pure acid solution. In most cases, it varies gradually with a change in the concentrations of the mixture components. At the same time, the mixtures sometimes exhibit higher capacitance values and, hence, lower degrees of surface coverage with inhibitors in comparison with the pure additives. It is possible that in the case of joint adsorption of the mixture components, effects (*e.g.*, steric) that affect the

DEL structure exist. Anyway, the surface blocking effects do exist. The inhibitors and their mixtures slow down corrosion by blocking a fraction of the surface and increasing the activation energy of the corrosion process.

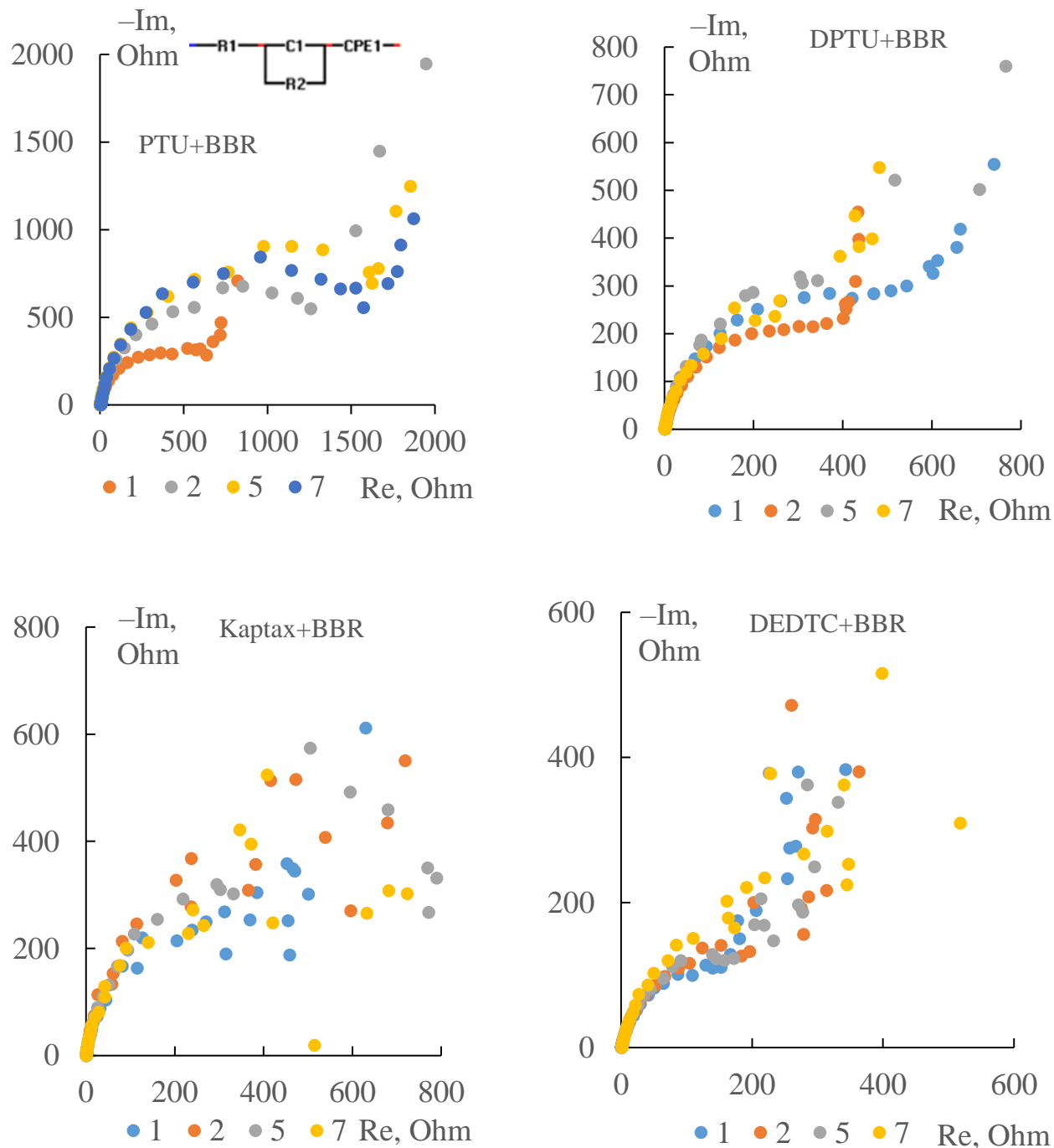


Figure 5.1. Nyquist plots on steel electrode in sulfuric acid solutions in the absence (0) and in the presence of mixtures of compounds. The concentration of the sulfur-containing compound is 0.1 mmol/L; the concentrations of BBR and HBR are (mmol/L): 0.01 (1), 0.02 (2), 0.05 (5) and 0.07 (7).

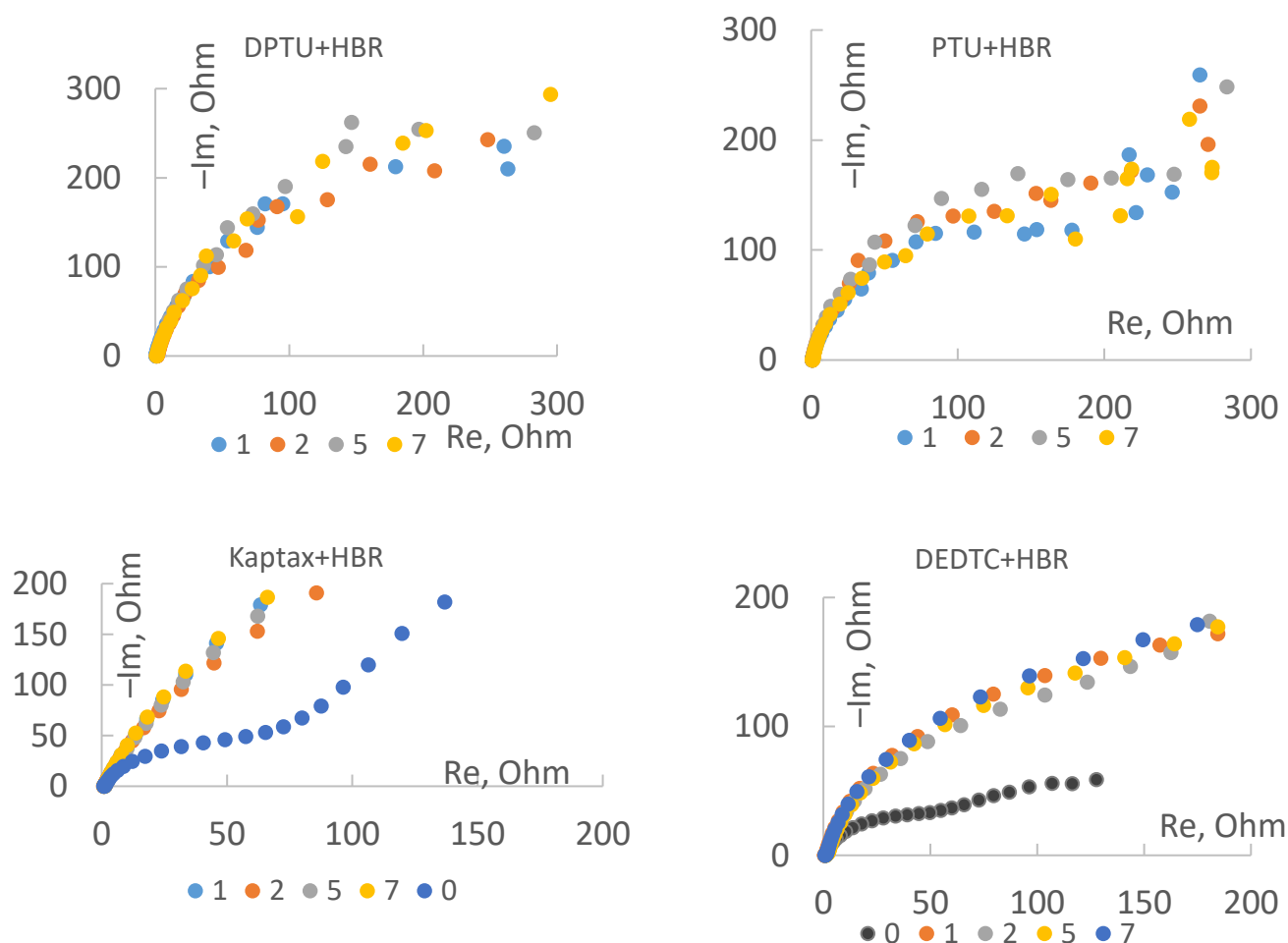


Figure 5.2. Nyquist plots on steel electrode in sulfuric acid solutions in the absence (0) and in the presence of mixtures of compounds. The concentration of the sulfur-containing compound is 0.1 mmol/L; the concentrations of BBR and HBR are (mmol/L): 0.01 (1), 0.02 (2), 0.05 (5) and 0.07 (7).

Conclusions

1. The compounds studied are corrosion inhibitors of steel in sulfuric acid solutions.
2. Mixtures of berberine and dihydroberberine with sulfur-containing additives are more efficient as inhibitors of acid corrosion of steel in comparison with their components.
3. In the berberine-sodium diethyldithiocarbamate mixture, the inhibitors mutually weaken the protective effects of each other, while in the other mixtures, the components enhance the effects of each other.
4. The individual inhibitors are predominantly cathodic type additives that hinder hydrogen evolution. The mixtures affect both partial reactions of the corrosion process.
5. Both the individual inhibitors and inhibitor mixtures have an activation-blocking mechanism of action.

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