

Protection of stainless steel in sulfuric acid solution containing hydrogen sulfide by inhibitors

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Abstract

The corrosion of chromium-nickel steel 1Kh18N9T in 2 M H₂SO₄ at $t = 20\text{--}100^\circ\text{C}$ was studied using the mass loss method. In addition, steel hydrogenation in the corrosive medium was measured by the vacuum extraction method. IFKhAN-92 (a 1,2,4-triazole derivative) and its combination with KI were studied as corrosion inhibitors. It was shown that 1Kh18N9T steel has low corrosion resistance in H₂SO₄ solutions, particularly at temperatures (t) close to 100°C. The steel corrosion rate is $k = 1.7\text{--}727 \text{ g}/(\text{m}^2\cdot\text{h})$ under the conditions studied and increases with increasing t . Steel hydrogenation occurs at $t = 40\text{--}100^\circ\text{C}$ and is the strongest at 100°C (12 ml/100 g). The presence of 15 mM H₂S in the H₂SO₄ solution slows down steel corrosion ($k = 2.8\text{--}274 \text{ g}/(\text{m}^2\cdot\text{h})$) but intensifies hydrogen absorption. The highest content of hydrogen absorbed in the metal (36 ml/100 g) was observed at $t = 40^\circ\text{C}$ and 1 h exposure of the specimens. It has been shown that, by themselves, 5 mM IFKhAN-92 or 5 mM KI are not suitable for protection of 1Kh18N9T steel in the specified media, since IFKhAN-92 has low efficiency in H₂SO₄ solution without H₂S, whereas, conversely, KI has low efficiency in H₂SO₄ solution containing H₂S. However, their mixture shows versatile protection in these media. The formulation containing 2.5 mM IFKhAN-92 + 2.5 mM KI that suppresses steel corrosion in H₂S-containing medium up to 100°C and totally prevents hydrogen absorption up to 60°C, inclusive, has been recommended for steel protection in H₂SO₄, both in the absence and in the presence of H₂S. This mixture is particularly efficient in solutions without H₂S where it inhibits steel corrosion in the entire t range studied while keeping the content of hydrogen in the metal at metallurgical level.

Key words: acid corrosion, hydrogen sulfide, corrosion inhibitors, stainless steel, hydrogenation, triazoles.

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Introduction

As shown previously [1], inhibitory protection by formulations of a 3-substituted 1,2,4-triazole (IFKhAN-92) and anionic additives (KI, KCNS, or KBr) is an efficient method for corrosion prevention of chromium-nickel stainless steels in H_2SO_4 solutions. The presence of these formulations in an acid solution makes it possible to hinder corrosion of 12Kh18N10T steel in 2 M H_2SO_4 at temperatures up to 100°C to a considerable extent. Of the formulations studied, the maximum protective effect is provided by the equimolar mixture of IFKhAN-92 + KI. The observed effect is based on considerable inhibition of electrode reactions on the metal in a broad range of temperatures t by composite inhibitors.

Steel corrosion in acidic aqueous media is accompanied by hydrogen evolution. It often results in metal hydrogenation that can impair the mechanical properties [2]. A detailed analysis of the regularities of cathodic hydrogen evolution in acidic media and its penetration into metallic iron is given in a review [3].

Identification of the specifics of the effect of organic inhibitors of acid corrosion on steel hydrogenation remains a task of current interest. The most attention is given to studies of inhibitors belonging to quarternary ammonium salts (QAS). For example, A.I. Marshakov *et al.* [4, 5] have shown that, unlike benzonitrile and dibutylsulfoxide, tetrabutylammonium sulfate and tetraethylammonium bromide do not change the mechanism of cathodic hydrogen evolution in acidic sulfate solutions but slow down considerably hydrogen penetration into iron. Later, L.E. Tsygankova *et al.* [6–8] have also shown that Catamin AB (a mixture of alkylbenzyltrimethylammonium chlorides $[C_nH_{2n+1}N^+(CH_3)_2CH_2C_6H_5]Cl^-$, where $n = 10–18$) slows down hydrogen diffusion into steel in acidic sulfate and chloride solutions. Along with corrosion inhibition of high strength steel in solutions of mineral acids, 1,2,4-triazole derivatives and formulations based thereon can slow down hydrogen absorption by the metal, thus preserving its mechanic properties [9]. In this case, the use of triazoles makes it possible to achieve more efficient hindrance of steel hydrogenation in comparison with Catamin AB that is a QAS mixture.

The presence of hydrogenation stimulators in acid solutions can considerably enhance hydrogen absorption by steel, both in case of its corrosion in such background media [2, 10] and in the presence of inhibitors [10]. Hydrogen sulfide that can saturate acid solutions during various production operations is the most hazardous steel hydrogenation stimulator [11–13]. The presence of hydrogen sulfide in hydrochloric acid solution enhances the corrosion of chromium-nickel steel 1Kh18N9T in this solution and causes metal hydrogenation. In the presence of the inhibitors studied (IFKhAN-92 and IFKhAN-92 + urotropine formulation), steel corrosion is intensified and inhibitors are not always able to suppress metal hydrogenation completely [10].

In view of the above considerations, it seems expedient to study the liability of chromium-nickel steel 1Kh18N9T to hydrogenation in H_2SO_4 solutions including those containing H_2S and to estimate the effect of IFKhAN-92 and its equimolar blend with KI on this process.

Experimental procedure

The corrosion rate of foil made of 1Kh18N9T stainless steel (composition, mass%: Fe – 71.92; Cr – 17.57; Ni – 9.16; Ti – 0.83; Si – 0.52) in 2 M H₂SO₄ at $t = 20\text{--}100^\circ\text{C}$ was determined from the mass loss of specimens (no less than 3 specimens per point) sized 20 mm × 10 mm × 0.25 mm, using 50 ml of the acid solution per specimen. The exposure time of the specimens in the solution was 0.25, 0.5, 1, or 2 h. Prior to an experiment, the specimens were cleaned with micron-graded polishing paper M40 and degreased with acetone.

Solutions were prepared from H₂SO₄ of “chemically pure” grade and distilled water. H₂SO₄ solutions were saturated with hydrogen sulfide ($C_{\text{H}_2\text{S}} = 15 \text{ mM}$) obtained from Na₂S (technical grade, PRC standard: GB/T 10500–2000) and 15% H₂SO₄ (“chemically pure” grade). The base concentrations (C) of individual inhibitors and their mixtures amounted to 5.0 mM. A formulation containing 2.5 mM IFKhAN-92 + 2.5 mM urotropine was studied. As shown previously [1], this ratio of components is the best for protection of chromium-nickel stainless steels in H₂SO₄ solutions.

The efficiency of inhibitors was estimated from the inhibition coefficient, $\gamma = k_0/k_{\text{in}}$, where k_0 and k_{in} are the corrosion rates in the background solution and in the solution with the additive being studied, respectively. In the case of inhibited solutions containing hydrogen sulfide, 2 M H₂SO₄ + 15 mM H₂S was considered as the background solution. In order to estimate quantitatively the effect of additives on nitrogen-containing inhibitors, the mutual influence coefficients of the mixture components were calculated [14]:

$$K_m = \frac{\gamma_{\text{mix}}}{\prod_{i=1}^m \gamma_i},$$

where γ_{mix} is the corrosion inhibition coefficient for the inhibitor mixture and $\prod_{i=1}^m \gamma_i$ is the product of the corrosion inhibition coefficients for individual mixture components. If $K_m < 1$, the protection coefficients of the inhibitor components are mutually reduced; at $K_m = 1$, additive behavior is observed; and only at $K_m > 1$ it should be concluded that the components of the inhibitor mixture mutually enhance protection.

The extent of hydrogen absorption by steel 1Kh18N9T was determined by vacuum extraction method. After corrosion tests, the specimens were placed in a vessel, air was evacuated from the vessel to a residual pressure of $1.33 \cdot 10^{-4} \text{ Pa}$, and the vessel was heated to $t = 500^\circ\text{C}$. The amount of hydrogen evolved upon heating the specimens *in vacuo* was estimated from pressure change in 10 min (P_{tot}) measured by a McLeod manometer at a constant volume of the evacuated part of the system. The pressure of evolved hydrogen (P_{H_2}) was calculated from the change in total pressure (P_{tot}) by the formula:

$$P_{\text{H}_2} = P_{\text{tot}} - P_{\text{nat}},$$

where P_{nat} is the correction obtained in the blank test. The volume concentration of hydrogen in steel (ml/100 g of steel) was calculated by the formula:

$$V_{\text{H}_2} = \frac{100KP_{\text{H}_2}}{M},$$

where K is a constant related to the volume of the analytical part of the set-up, and M is the mass of the steel specimen in grams.

The hydrogen content in the metal is given with correction for metallurgical hydrogen, the concentration of which in 1Kh18N9T steel amounts to 2.0 ml/100 g.

Experimental results and discussion

The corrosion of chromium-nickel steel in 2 M H_2SO_4 occurs at rates within $k = 1.7\text{--}727 \text{ g}/(\text{m}^2 \cdot \text{h})$ and accelerates with an increase in t (Figure 1). In fact, according to studies for 0.25 h, an increase in t of the acid by 80°C increases k 97-fold. Corrosion slows down with time at $t \leq 40^\circ\text{C}$, accelerates with time at $t = 60^\circ\text{C}$, and corrosion acceleration changes to corrosion hindrance at $t = 80^\circ\text{C}$. Unfortunately, due to high k values at $t = 80^\circ\text{C}$ and especially at 100°C , we failed to obtain all the values within the selected time periods due to complete dissolution of the specimens. A similar situation is characteristic of inhibited acid solutions if the protective effect of inhibitors is low.

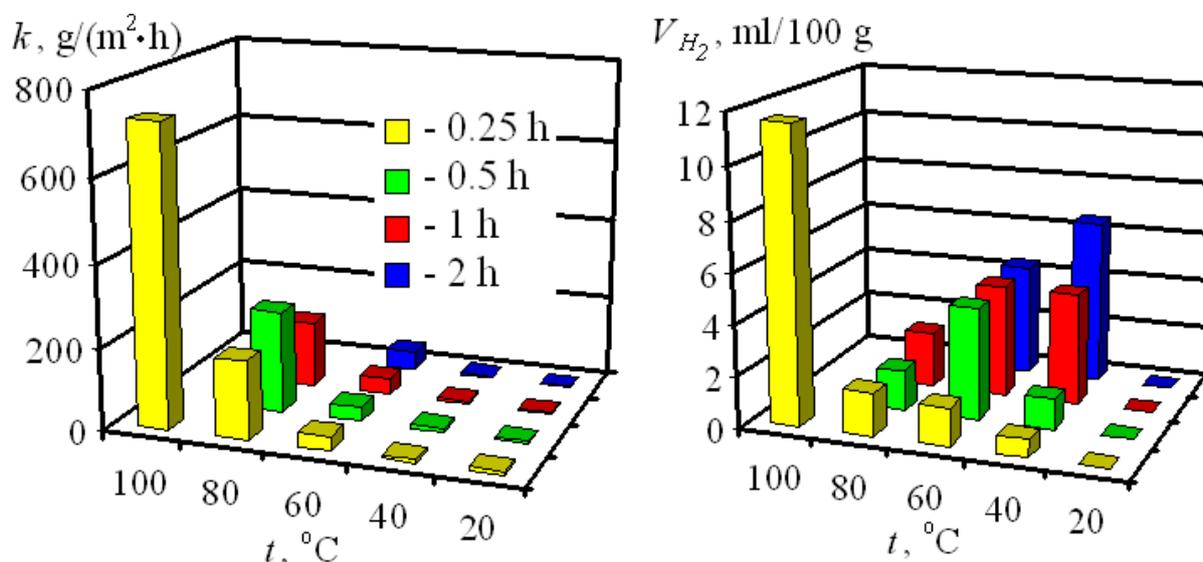


Figure 1. Corrosion rates of steel 1Kh18N9T in 2 M H_2SO_4 and volume of hydrogen absorbed by the metal as a function of specimen exposure time and solution temperature.

At $t = 20^\circ\text{C}$, stainless steel does not undergo hydrogenation even after 2 h exposure to 2 M H_2SO_4 (Figure 1). An increase in t to 40°C results in metal hydrogenation and the hydrogen content in steel at the same exposure time of the specimens in the solution

reaches 6.5 ml/100 g. A further increase in t results in faster steel hydrogenation but the total hydrogen content in the metal becomes lower (4.4 ml/100 g at 60°C and 2.2 ml/100 g at 80°C). At $t = 100^\circ\text{C}$ where the metal corrodes with a maximum k , the hydrogen content in steel reaches a considerable value of 12 ml/100 g even in 0.25 h. The observed complex type of dependence of hydrogen content in stainless steel on the time of specimen exposure to the acid and on t can be explained by competition of two processes: hydrogen evolution on the metal surface with incorporation of a fraction of hydrogen into the metal, and dissolution of a hydrogen-saturated surface layer of the metal. The former process favors hydrogen absorption by the metal, whereas on the contrary, the latter one decreases the hydrogen content in the metal. The combined effect of these processes that are partially independent results in a complex overall picture of metal hydrogenation in the course of time and with an increase in t .

If the acid solution contains 15 mM H_2S , the k of steel ranges within 2.8–274 g/(m²·h). Hydrogen sulfide has a predominantly inhibiting effect on steel corrosion. The decrease in corrosion rate in comparison with H_2S -free solutions can be up to 3.6-fold (Figure 2). It is only in the range of $t = 40\text{--}60^\circ\text{C}$ in the initial time period that corrosion rates of the metal exceed the k values characteristic of 2 M H_2SO_4 in the absence of H_2S . In general, steel corrosion rate in these solutions increases with t increase but slows down with time. According to 0.25 h studies, a t increase by 80°C accelerates steel corrosion 43-fold.

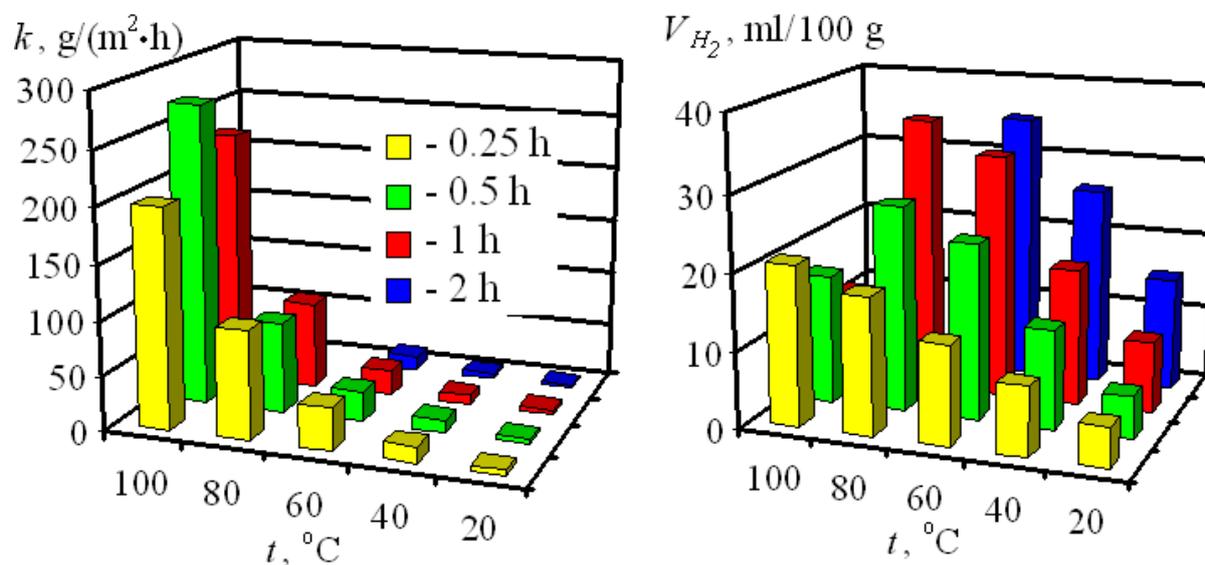


Figure 2. Corrosion rates of steel 1Kh18N9T in 2 M H_2SO_4 + 15 mM H_2S and volume of hydrogen absorbed by the metal as a function of specimen exposure time and solution temperature.

At $t = 20^\circ\text{C}$, stainless steel is hydrogenated considerably in 2 M H_2SO_4 + 15 mM H_2S even after 0.25 h of specimen exposure to the acid. Increasing the time of specimen exposure to the corrosive medium to 2 h increases the hydrogen content in the metal 2.9-fold. Increasing the t of the solution increases the hydrogen content in steel. The maximum

metal hydrogenation is observed upon 0.25 h exposure of specimens to the solution at $t = 100^\circ\text{C}$. At longer exposures, it is observed at $t = 80^\circ\text{C}$ and may reach 36 ml/100 g of steel, which is 3 times higher than in the H_2S -free solution. At $t = 20\text{--}80^\circ\text{C}$, longer exposures of specimens to the corrosive medium enhance steel hydrogenation that decreases only at $t = 100^\circ\text{C}$. This is probably due to fast steel dissolution and removal of the hydrogenated steel layer.

IFKhAN-92 by itself slows down steel corrosion only slightly in the entire range of acid solution temperatures ($\gamma \leq 5.8$) and γ decreases with time (Figure 3, Table 1). Despite this, addition of 5.0 mM IFKhAN-92 totally suppresses steel hydrogenation at $t \leq 60^\circ\text{C}$. Beginning from $t = 80^\circ\text{C}$, the presence of hydrogen is detected in steel. It reaches 7.2 ml/100 g at 100°C .

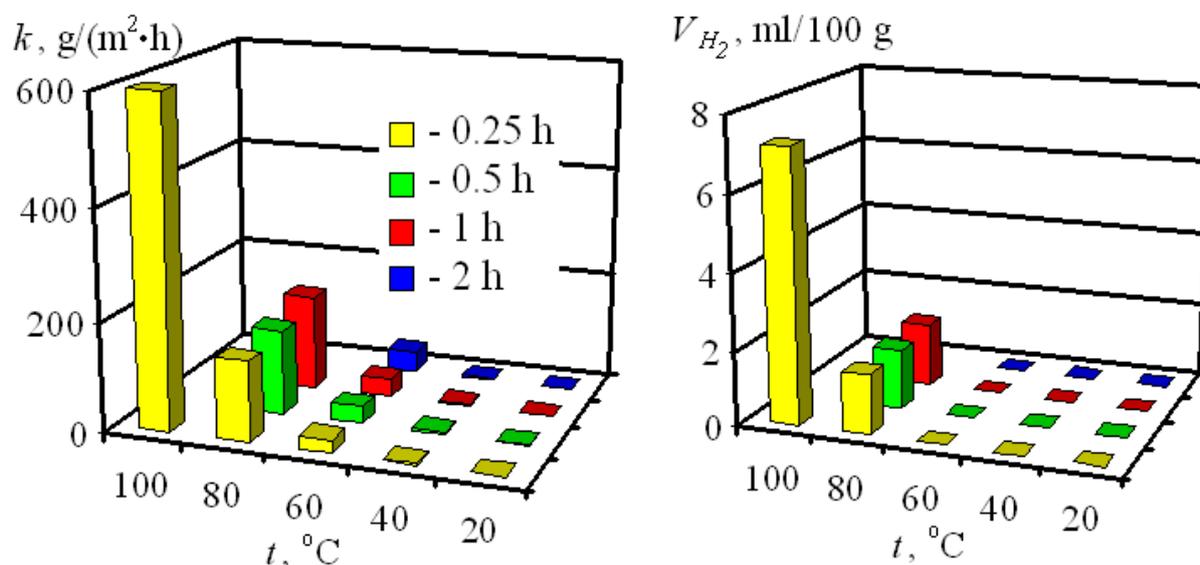


Figure 3. Corrosion rates of steel 1Kh18N9T in 2 M H_2SO_4 containing 5 mM IFKhAN-92 and volume of hydrogen absorbed by the metal as a function of specimen exposure time and solution temperature.

Conversely, addition of IFKhAN-92 to H_2S -containing solutions efficiently slows down steel corrosion 11–144 fold (Figure 4). In this case, coefficient γ increases with t increase up to 100°C , as it is typical of high-temperature corrosion inhibitors [15]. The high efficiency of IFKhAN-92 in this medium results from its joint effect with H_2S on the corrosion process. Previously we noted that the protective effect of IFKhAN-92 in acid media can be improved considerably by hydrophobic sulfur-containing additives, including sodium sulfide that is converted to H_2S in acidic media [16]. Though steel corrosion is efficiently hindered under these conditions, it is noted that metal hydrogenation occurs starting from 60°C and increases with t rise and in the course of time. The maximum hydrogen content in steel is observed at 100°C and amounts to 11 ml/100 g.

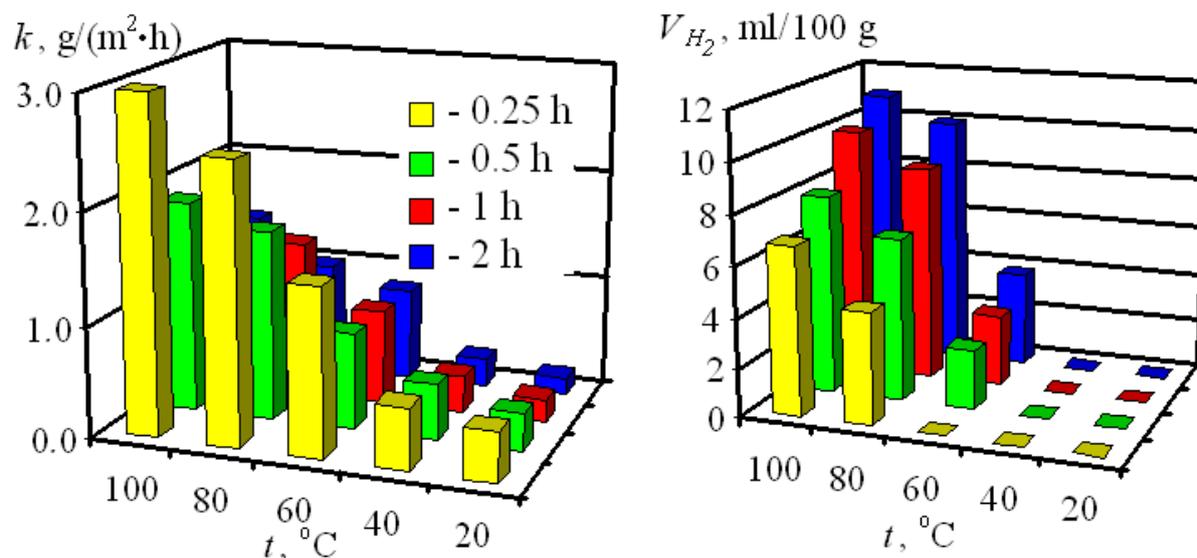


Figure 4. Corrosion rates of steel 1Kh18N9T in 2 M H_2SO_4 + 15 mM H_2S containing 5 mM IFKhAN-92 and volume of hydrogen absorbed by the metal as a function of specimen exposure time and solution temperature.

KI itself is efficient as a steel corrosion inhibitor in H_2SO_4 solutions (Figure 5a). At all t values studied, it slows down corrosion 15–404 fold, and the maximum k in the presence of this compound is no higher than 1.8 $\text{g}/(\text{m}^2\cdot\text{h})$. The temperature maximum of efficiency of this additive is not reached at least up to 100°C , which also characterizes it as a high-temperature corrosion inhibitor of 1Kh18N9T steel in H_2SO_4 solution. The presence of KI slows down steel corrosion with time, whereas 5.0 mM KI also completely suppresses steel hydrogenation in the entire t range studied.

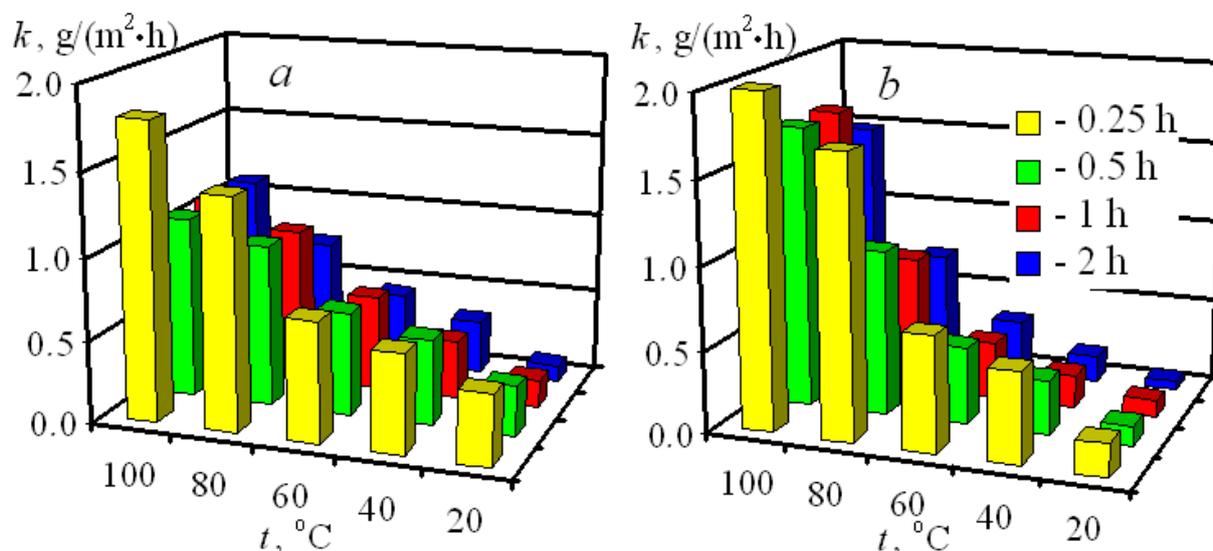


Figure 5. Corrosion rates of steel 1Kh18N9T in 2 M H_2SO_4 containing 5 mM KI (a) and 2.5 mM IFKhAN-92 + 2.5 mM KI (b) as a function of specimen exposure time and solution temperature.

Table 1. Corrosion inhibition coefficients (γ) of steel 1Kh18N9T in 2 M H₂SO₄ provided by inhibitors at various temperatures and exposure times.

Inhibitor	τ , h	γ values at various temperatures, °C				
		20	40	60	80	100
Without H ₂ S						
5 mM IFKhAN-92	0.25	5.8	2.6	1.5	1.3	1.2
	0.5	5.8	2.4	1.2	1.6	–
	1.0	3.4	2.1	1.2	0.93	–
	2.0	2.1	1.4	1.2	–	–
5 mM KI	0.25	17	15	49	136	404
	0.5	17	16	60	252	–
	1.0	16	22	71	171	–
	2.0	17	18	104	–	–
2.5 mM IFKhAN-92 + 2.5 mM KI	0.25	38	16	51	112	364
	0.5	43	26	81	247	–
	1.0	28	39	117	196	–
	2.0	28	38	142	–	–
15 mM H ₂ S						
5 mM IFKhAN-92	0.25	11	27	26	40	67
	0.5	14	22	32	48	144
	1.0	20	26	27	56	144
	2.0	18	18	15	–	–
5 mM KI	0.25	0.98	1.4	2.2	4.7	1.1
	0.5	1.0	1.1	1.4	2.0	1.7
	1.0	1.1	0.98	0.88	0.86	1.7
	2.0	1.0	0.72	0.72	–	–
2.5 mM IFKhAN-92 + 2.5 mM KI	0.25	16	44	98	83	59
	0.5	9.4	21	49	59	137
	1.0	15	24	40	61	154
	2.0	15	17	30	–	–

Addition of 15 mM H₂S to 2 M H₂SO₄ + 5 mM KI solution can accelerate steel corrosion up to 144-fold (Figure 6). In this case KI, which efficiently suppresses steel corrosion and hydrogenation in H₂SO₄ itself, loses its protective properties and the maximum decrease in the k of steel provided by it becomes $\gamma \leq 4.7$. The γ values observed in H₂SO₄ solutions without H₂S are 11–367 times higher than in its presence. Steel hydrogenation is also observed. It increases with time and with t increase up to 80°C. In this case, the maximum hydrogen content of steel can be comparable to that in the background solutions, namely, 38 ml/100 g. Apparently, the insignificant decrease in hydrogen content in steel at 100°C is also due to its dissolution and removal of the hydrogenated steel layer.

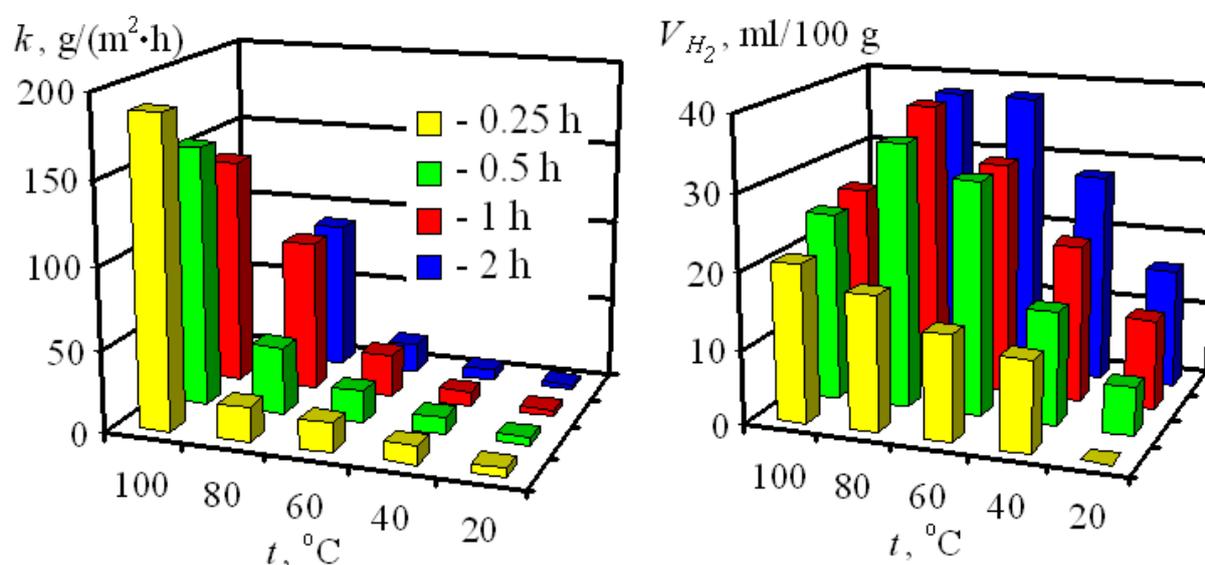


Figure 6. Corrosion rates of steel 1Kh18N9T in 2 M H₂SO₄ + 15 mM H₂S containing 5 mM KI and volume of hydrogen absorbed by the metal as a function of specimen exposure time and solution temperature.

The protective effect of an equimolar mixture of IFKhAN-92 and KI (5.0 mM) in 2 M H₂SO₄ at $t \leq 60^\circ\text{C}$ exceeds those of its components, *i.e.*, KI and particularly IFKhAN-92 (Figure 5b). KI is somewhat more efficient at higher t . In fact, the k of steel in a solution inhibited by 5.0 mM of the mixed inhibitor at $t = 100^\circ\text{C}$ is 1.1–1.5 times higher than that in the acid containing 5.0 mM KI. However, corrosion is hindered 16–364 fold in the presence of the mixed inhibitor ($t = 20\text{--}100^\circ\text{C}$) and $k \leq 2 \text{ g}/(\text{m}^2 \cdot \text{h})$. The inhibitor also hinders steel corrosion in time. The growth of the γ coefficient of the formulation up to 100°C allows us to consider it as a high-temperature inhibitor. The mixture components predominantly manifest an antagonism of action, though it becomes weaker with time (Table 2). In many respects, the antagonism of action of the components of this inhibitor mixture in H₂SO₄ without H₂S is determined by the high inhibitive effect of one of its components, namely KI. The hydrogen content in all steel samples exposed to the acid solution with addition of 2.5 mM IFKhAN-92 + 2.5 mM KI remains at metallurgical level.

Table 2. Mutual influence coefficients (K_m) of inhibitor mixtures 2.5 mM IFKhAN-92 + 2.5 mM KI at various temperatures and exposure times.

τ , h	K_m values at various temperatures, °C				
	20	40	60	80	100
Without H ₂ S					
0.25	0.39	0.41	0.69	0.63	0.75
0.5	0.44	0.68	1.1	0.61	–
1.0	0.51	0.84	1.4	1.2	–
2.0	0.78	1.5	1.1	–	–
15 mM H ₂ S					
0.25	1.3	1.2	1.7	0.43	0.80
0.5	0.67	0.87	1.1	0.61	0.56
1.0	0.68	1.2	1.7	1.3	0.63
2.0	0.85	1.3	2.8	–	–

The protective effect of 2.5 mM IFKhAN-92 + 2.5 mM KI in 2 M H₂SO₄ + 15 mM H₂S is comparable to that of 5 mM IFKhAN-92 alone and is much higher than that of 5 mM KI alone (Figure 7). The mixed inhibitor slows down steel corrosion 9.4–154 fold, while the maximum k value is ≤ 3.4 g/(m²·h), which is somewhat higher than that in the solution without H₂S. The mixture components mostly manifest antagonism of action at $t = 20, 80$ and 100°C and synergism with low K_m values at $t = 40$ and 60°C (Table 2). Unlike in the acid solutions containing no H₂S, the antagonism of action of the components of this inhibitor mixture is mainly determined by the high inhibitive effect of IFKhAN-92, rather than KI, in this medium. The hydrogen content in steel specimens exposed to 2 M H₂SO₄ + 15 mM H₂S + 2.5 mM IFKhAN-92 + 2.5 mM KI at $t \leq 60^\circ\text{C}$ remains at metallurgical level. Some hydrogenation of specimens is only observed at higher solution temperatures, with a maximum of 9.3 ml/100 g of the metal at 100°C . It should be noted that it is the smallest value of maximum hydrogenation that we observed in hydrogen sulfide-containing media. It is 3.9 times smaller than the value observed in the background solution, 4.1 times smaller than the value characteristic of the solution inhibited with 5 mM KI, and 1.2 times better than the value obtained in the medium inhibited with 5 mM IFKhAN-92. In this case, unlike in 2 M H₂SO₄ + 15 mM H₂S + 5 mM IFKhAN-92, the absence of hydrogenation is observed up to 60°C rather than up to 40°C .

Our study allowed us to find an interesting phenomenon. Addition of a small amount of H₂S to the H₂SO₄ solution drastically changes the behavior of corrosion inhibitors. KI that efficiently protects steel in the absence of H₂S nearly loses this property if H₂S is present. Conversely, the substituted triazole acquires a high protective effect in the H₂S-

containing medium. From this perspective, the composite inhibitor that retains the properties of KI in H_2SO_4 and the properties of IFKhAN-92 in $H_2SO_4 + H_2S$ appears interesting. It is efficient in both media and allows steel hydrogenation to be minimized in these media.

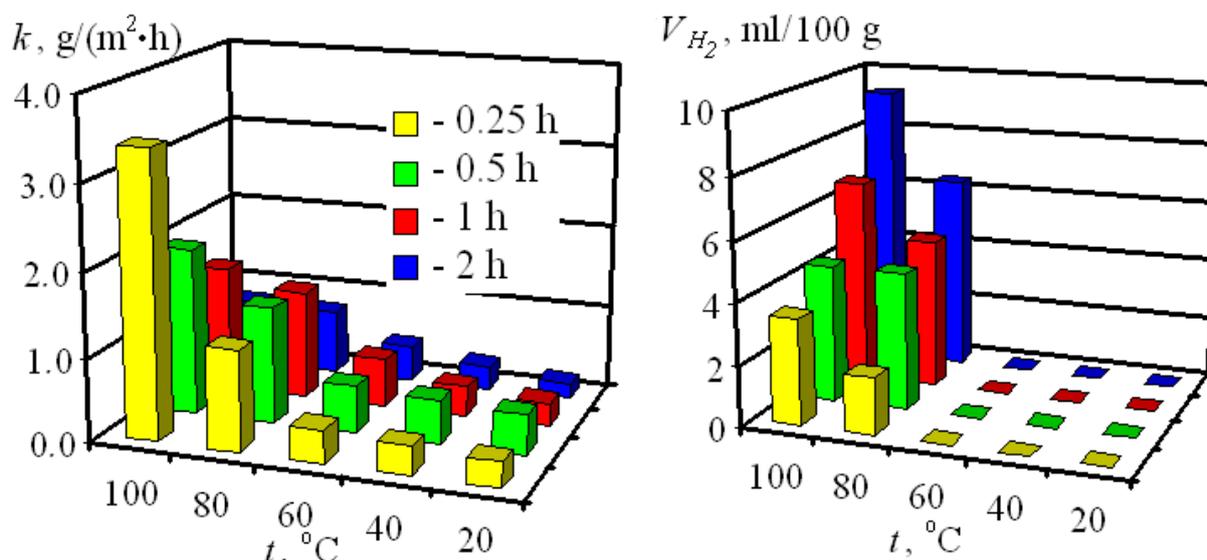


Figure 7. Corrosion rates of steel 1Kh18N9T in 2 M $H_2SO_4 + 15$ mM H_2S containing 2.5 mM IFKhAN-92 + 2.5 mM KI and volume of hydrogen absorbed by the metal as a function of specimen exposure time and solution temperature.

Conclusions

1. The corrosion rates of chromium-nickel steel 1Kh18N9T in 2 M H_2SO_4 in the temperature range of $t = 20–100^\circ C$ vary within 1.7–727 $g/(m^2 \cdot h)$ and increase with an increase in t . Hydrogenation of steel occurs at $t = 40–100^\circ C$ and is the strongest at $100^\circ C$ (12 ml /100 g).
2. Addition of 15 mM H_2S to 2 M H_2SO_4 solution ($t = 20–100^\circ C$) mostly slows down the corrosion of chromium-nickel steel 1Kh18N9T to rates within 2.8–274 $g/(m^2 \cdot h)$. In this case, enhancement of steel hydrogenation to 36 ml/100 g was noted.
3. The formulation containing 2.5 mM IFKhAN-92 + 2.5 mM KI can be used for protection of chromium-nickel steel in H_2SO_4 solutions, both in the absence and in the presence of H_2S . It considerably inhibits the metal corrosion in the H_2S -containing medium up to $100^\circ C$ and completely prevents steel hydrogenation up to $60^\circ C$. This mixture is particularly efficient in solutions without H_2S , where it inhibits steel corrosion in the entire temperature range studied while keeping the content of hydrogen in the metal at metallurgical level.

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