Effect of Fe(III) salts on the protection of low carbon steel in hydrochloric acid solutions by some corrosion inhibitors*

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Abstract

The effect of Fe(III) salts on the protection of 08PS low carbon steel in 2 M HCl and 2 M H_3PO_4 at 20 and 60°C by corrosion inhibitors (PKU-E, Catapin A, Catapin BPV, Soling m. Z, Soling m. L, VNPP-2, Invol-2 m. A and NORUST CM 150 C) and their mixtures with hexamethylenetetramine (HMT) has been studied. None of the individual and two-component corrosion inhibitors studied can provide efficient protection of steel in HCl and H_3PO_4 solutions if significant amounts of Fe(III) cations are accumulated in them. In H_3PO_4 solutions containing Fe(III) phosphate, efficient protection of steel can be obtained by the formulation comprising $2\ g\cdot L^{-1}$ VNPP-2 + 0.5 mM KNCS + 200 mM HMT as the corrosion inhibitor. The same formulation protects steel in 1 M HCl + 1 M H_3PO_4 (20 and 60°C) with up to 0.10 M Fe(III) cations accumulated in it. Solutions of a HCl + H_3PO_4 mixture inhibited by the formulation of VNPP-2 + KNCS + HMT can be an alternative to inhibited solutions of individual HCl in case of potential accumulation of Fe(III) chloride in them.

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Introduction

Hydrochloric acid and its solutions are common technological fluids used at the modern enterprises [1–7]. These media are often corrosive to the metals in contact with them and cause their serious corrosion damage. Technological processes of cleaning steel products from scale, rust and other mineral deposits with HCl solutions are performed in the presence of corrosion inhibitors (CIs), thus minimizing the metal losses caused by its reaction with the acid. The existing range of CIs for steels in acid media, including HCl solutions, was reviewed in numerous publications [8–16].

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In the course of acid cleaning of the surfaces of steel products from mineral contaminants containing Fe(III) compounds, accumulation of Fe(III) salts in the etching solutions occurs. The presence of Fe(III) chloride in an HCl solution changes its properties significantly. An additional oxidizing agent appears, *i.e.*, the Fe(III) cation, which increases the environment corrosivity to the metal due to the reaction of the Fe(III) salt with iron:

$$\mathrm{Fe} + 2\mathrm{FeCl}_3 = 3\mathrm{FeCl}_2$$

Moreover, it is noted that the presence of Fe(III) salts in acid solutions often makes inhibitor protection of steels in them inefficient. This effect is primarily due to the weak inhibition by inhibitors of the cathodic reduction of Fe(III) cations on steel in solutions of acids that occur under diffusion control [17].

From the practical point of view, it is important to understand how the accumulation of Fe(III) chloride in HCl solutions during various technological operations affects the protection of steels in them by industrial CIs. If such corrosion inhibitors have low efficiency in the media in question, it is necessary to consider the possibility of searching for ways to increase their protective effect.

To improve the protective effect of CIs of steels, it is recommended to add hexamethylenetetramine (HMT) to dilute HCl solutions in case of potential accumulation of $\mathrm{FeCl_3}$ in them [18]. In this regard, it is important to understand how adding HMT to a corrosive environment will affect the effectiveness of inhibitors in slowing down steel corrosion in HCl solutions.

A promising direction for increasing the efficiency of industrial CIs in HCl solutions containing Fe(III) chloride involves switching from the individual acid to its mixtures with $\mathrm{H_3PO_4}$. Moreover, for steel protection in solution than comprise acid mixtures, not individual CIs but their mixtures with HMT and KNCS have to be used. In these acid media, Fe(III) cations are bound into complex compounds with phosphate anions and HMT molecules, thus reducing their oxidizing properties and mobility [19, 20]. As a result, this effect allows some CIs to maintain efficient protection of steels even if Fe(III) salts are accumulated in the corrosive medium. For example, the corrosion rate of steel in the etching solution containing 1 M HCl + 1 M $\mathrm{H_3PO_4}$ ($t \leq 80$°C) with addition of 5 mM IFKhAN-92 + 0.5 mM KCNS + 200 mM HMT does not exceed 3.4 $\mathrm{g \cdot m^{-2} \cdot h^{-1}}$ at $C_{\mathrm{Fe(III)}} \leq 0.1$ M [21]. The presence of $\mathrm{H_3PO_4}$ in the etching solution should also favor the rapid removal of mineral contaminants from the metals, since the dissolution rate of Fe oxides in its presence is high [22, 23]. It should be noted [21] that the creation of inhibited etching solutions resistant to the accumulation of Fe(III) cations is possible in solutions containing $\mathrm{H_3PO_4}$ alone. However, this way is unreasonable from economical point of view since HCl is less expensive than $\mathrm{H_3PO_4}$. The use of acid mixtures would make the etching solution cheaper.

It appears worthwhile to study the effect of Fe(III) salts on the protection of low carbon steel in 2 M HCl by some currently manufactured corrosion inhibitors (Soling m. Z, Soling m. L, VNPP-2, Invol-2 m. A, NORUST CM 150 C, PKU-E) and some inhibitors previously manufactured in the USSR (Catapin A and Catapin BPV). Will be studied as individual CIs and their mixtures with HMT. If the result is unsatisfactory, the possibility of efficient application of the above CIs for steel protection in H_3PO_4 solutions containing $FePO_4$ should be studied. If the result in the H_3PO_4 solution is good, the corresponding CIs will be studied in a HCl + H_3PO_4 solution.

Experimental

The corrosion rate of 08PS low carbon steel (composition, mass%: C – 0.08; Mn – 0.5; Si – 0.11; P – 0.035; S – 0.04; Cr – 0.1; Ni – 0.25; Cu – 0.25; As – 0.08) in 2 M HCl, 2 M H_3PO_4, and the mixture of 1 M HCl + 1 M H_3PO_4 at t=20 and 60°C was determined from the mass loss (≥3 samples per point) of samples sized 50×20×0.5 mm using 50 mL of an acid solution per sample. The duration of the experiments was 2 h. Before the experiments, the samples were cleaned on an abrasive wheel (ISO 9001, grit 60) and degreased with acetone. All the organic CIs tested were added to the media as solutions in ethanol. The resulting concentration of ethanol was 0.24 mol/L.

The efficiency of CIs was estimated as their inhibition coefficients $\gamma=k_0/k_{in}$, where k_0 and k_{in} are the corrosion rates in the background solution and in the solution containing the corresponding additive. In the γ calculations, the acid solution containing only Fe(III) cations in the corresponding concentration as the additive was taken as the background solution.

The solutions were prepared using acids (HCl and H_3PO_4) of "chemically pure" grade and distilled water. Acid solutions containing Fe(III) salts were prepared by the reaction of $Fe(OH)_3$ precipitated by NaOH of "chemically pure" grade from a $FeCl_3$ solution with excess of the corresponding acid. $FeCl_3\times6H_2O$ of "pure" grade was used to prepare the Fe(III) chloride solution.

Results and Discussion

Hydrochloric acid

In background 2 M HCl (20°C), the corrosion of 08PS steel occurs at a rate of k_0=2.5 $g\cdot m^{-2}\cdot h^{-1}$ (Figure 1). The presence of 0.10 M $FeCl_3$ accelerates corrosion by a factor of 3.8, thus the k_0 of steel increases to 6.9 $g\cdot m^{-2}\cdot h^{-1}$ (Figure 1). The increase in corrosion losses due to the presence of the Fe(III) salt in the total corrosion process is 73%.

In cold 2 M HCl containing no $FeCl_3$, all the CIs studied slow down corrosion of steel by a factor of 10−54. The presence of the Fe(III) salt in the solution significantly decreases the protective effect of all the CIs. In the presence of 0.10 M $FeCl_3$, steel corrosion in HCl solutions containing CIs increases by 21−57 times. All inhibitors, except VNPP-2, stimulate steel corrosion. The increase in corrosion losses due to the presence of 0.10 M $FeCl_3$ in 2 M HCl is 96% in the presence of the PKU-E inhibitor, 98% with Catapin A, 95% with Catapin BPV, 98% with Soling m. Z, 97% with Soling m. L, 98% with VNPP-2, 98% with Invol-2 m. A, and 98% with NORUST CM 150 C. The most effective corrosion inhibitor VNPP-2 slows down steel corrosion by only 1.1 times. The addition of 200 mM HMT to the studied inhibitors somewhat slows down the corrosion of steel, but this effect is insignificant (Figure 2).

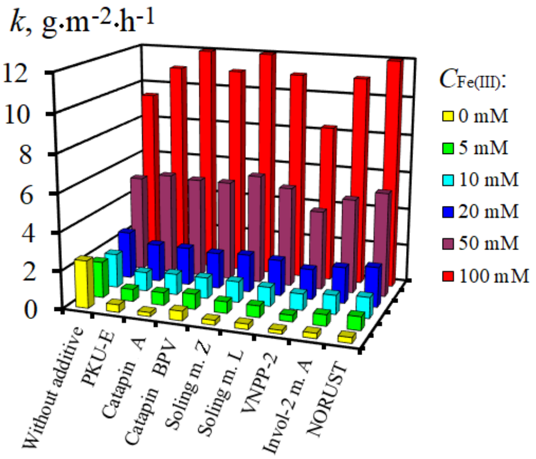

Figure 1. Corrosion rates of 08PS steel in 2 M HCl (20°C) containing $FeCl_3$ in the presence of 2 g·L^{-1} of the inhibitors.

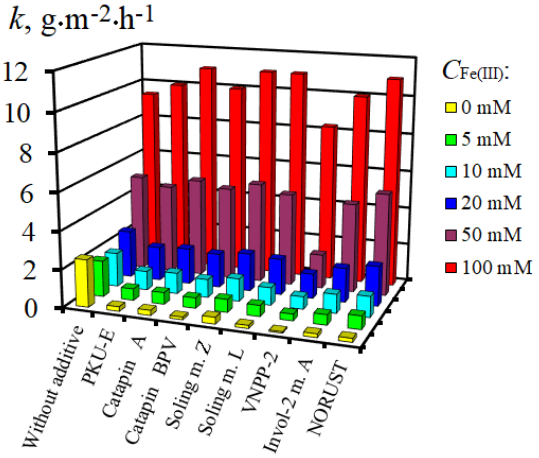

Figure 2. Corrosion rates of 08PS steel in 2 M HCl (20°C) containing $FeCl_3$ in the presence of 2 g·L^{-1} inhibitor + 200 mM HMT.

Increasing the temperature of 2 M HCl to 60°C increases the k of steel in it to 67 g·m^{-2}·h^{-1} (Figure 3). In this environment, addition of 0.10 M $FeCl_3$ increases the corrosion losses by 22 g·m^{-2}·h^{-1}, which is 25% of the total metal losses. The CIs studied inhibit the corrosion of steel in 2 M HCl (60°C) differently. With addition of PKU-E, Soling m. Z, or Invol-2 m. A, the value of k is higher than 1 g·m^{-2}·h^{-1}, while in the case of Catapin A, Catapin BPV, or Soling m. L, VNPP-2, or NORUST CM 150 C, steel corrosion is inhibited to a greater extent and $k<1$ g·m^{-2}·h^{-1}. The presence of the $FeCl_3$ in the solution weakens the effect of all the CIs. The increase in corrosion losses due to the presence of 0.10 M $FeCl_3$ in 2 M HCl (60°C) is 75% with the PKU-E inhibitor, 97% with Catapin A, 96% with Catapin BPV, 95% with Soling m. Z, 95% with Soling m. L, 98% with VNPP-2, 94% with Invol-2 m. A, and 96% NORUST CM 150 C. In general, the corrosion losses of steel in 2 M HCl + 0.10 M $FeCl_3$ in the presence of CIs are high. For the least efficient PKU-E, they amount to 25 $g\cdot m^{-2}\cdot h^{-1}$. VNPP-2 inhibits corrosion most strongly, but even in its presence, k=17 $g\cdot m^{-2}\cdot h^{-1}$.

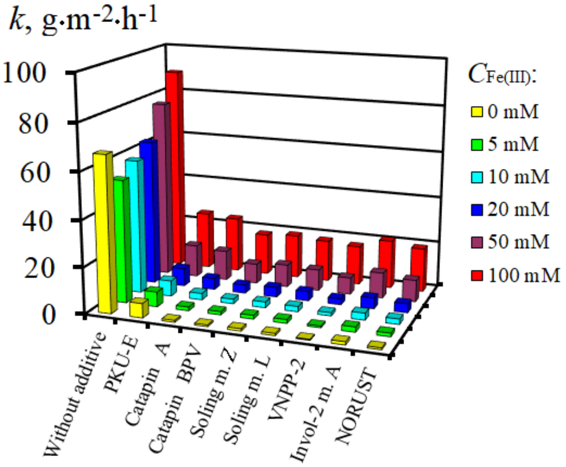

Figure 3. Corrosion rates of 08PS steel in 2 M HCl (60°C) containing $FeCl_3$ in the presence of 2 $g\cdot L^{-1}$ inhibitor.

As in the colder solution, the introduction of HMT additive into the inhibited solutions of 2 M HCl + 0.10 M $FeCl_3$ does not significantly affect the slowing down of steel corrosion (Figure 4). Only in the case of PKU-E, which is the weakest of the inhibitors studied, in the absence of $FeCl_3$ or at low $C_{Fe(III)} \leq 0.02$ M, the addition of HMT significantly improves the protection of steel.

In HCl solutions containing the inhibitors in question, addition of $FeCl_3$ accelerates corrosion. Since the CIs studied do not provide satisfactory protection of 08PS steel in HCl solutions containing $FeCl_3$, the possibility of their application in H_3PO_4 solutions containing $FePO_4$ was investigated.

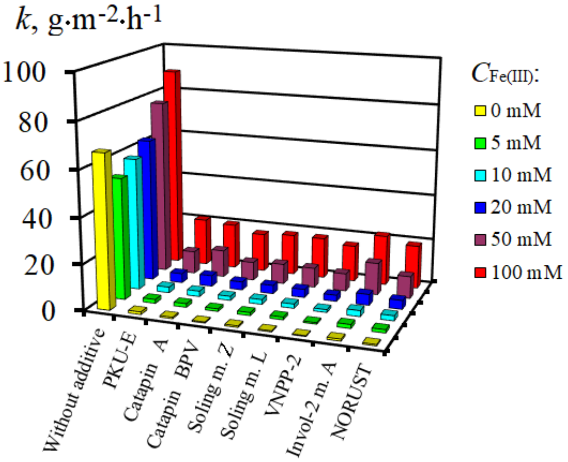

Figure 4. Corrosion rates of 08PS steel in 2 M HCl (60°C) containing $FeCl_3$ in the presence of 2 $g\cdot L^{-1}$ inhibitor + 200 mM HMT.

Phosphoric acid

In comparison with the HCl solution, the *k* value of steel in 2 M H_3PO_4 (20°C) is higher, *i.e.*, 7.8 $g·m^{-2}·h^{-1}$ (Figure 5). In the presence of 0.10 M $FePO_4$, k_0=9.7 $g·m^{-2}·h^{-1}$, the increase in corrosion losses due to the presence of the Fe(III) salt being as small as 20%. In the background 2 M H_3PO_4 solution, weak protection of steel ($\gamma<10$) is provided by PKU-E, Soling m. Z, and NORUST CM 150 C. More efficient inhibitors include Catapin A, Catapin BPV, Soling m. L, VNPP-2, and Invol-2 m. A. The presence of the Fe(III) salt in the solutions reduces the protective effects of all the CIs. The increase in corrosion losses due to the presence of 0.10 M $FePO_4$ in 2 M H_3PO_4 is 77% in the presence of PKU-E, 96% with Catapin A, 94% with Catapin BPV, 87% with Soling m. Z, 91% with Soling m. L, 52% with VNPP-2, 81% with Invol-2 m. A, and 89% with NORUST CM 150 C.

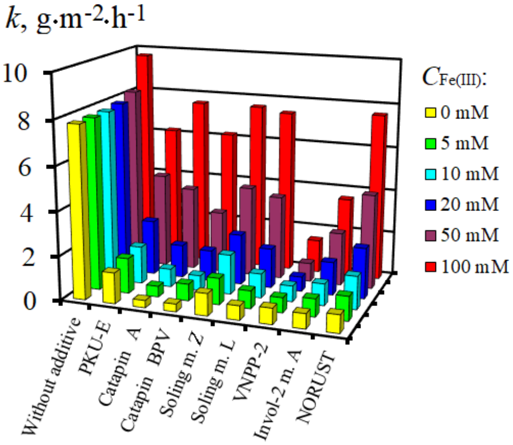

Figure 5. Corrosion rates of 08PS steel in 2 M H_3PO_4 (20°C) containing $FePO_4$ in the presence of 2 $g·L^{-1}$ of the inhibitors.

As in cold HCl, in a solution of H_3PO_4 (20°C) the combined use of the studied inhibitors with HMT does not allow for a significant increase in the protective effect (Figure 6).

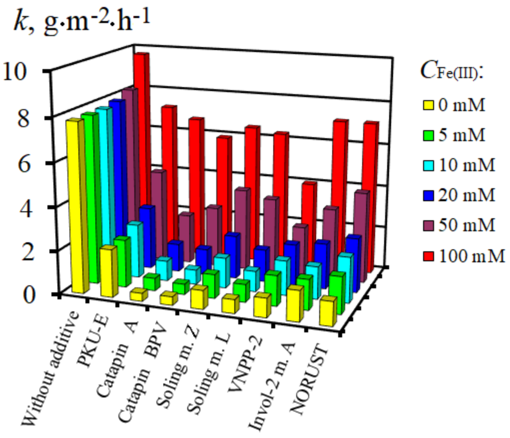

Figure 6. Corrosion rates of 08PS steel in 2 M H_3PO_4 (20°C) containing $FePO_4$ in the presence of 2 $g·L^{-1}$ inhibitor + 200 mM HMT.

In background 2 M H_3PO_4 at 60°C, as one would expect, the k_0 of steel is higher and amounts to 105 $g\cdot m^{-2}\cdot h^{-1}$ (Figure 7). The presence of $FePO_4$ in this medium slows down the corrosion of steel. At C_{FePO_4}=0.10 M, k_0=71 $g\cdot m^{-2}\cdot h^{-1}$. In background 2 M H_3PO_4, PKU-E, Soling m. Z, Soling m. L, VNPP-2, Invol-2 m. A, and NORUST CM 150 C weakly protect steel (γ<10), whereas, in contrast, Catapin A and Catapin BPV are more efficient. The latter two CIs lose their protective effect if $FePO_4$ is present in the acid. The increase in corrosion losses due to the presence of 0.10 M $FePO_4$ in 2 M H_3PO_4 with addition of Catapin A is 95%, with Catapin BPV it is 77%. The *k* value of steel in the presence of Catapin A and Catapin BPV is 22 $g\cdot m^{-2}\cdot h^{-1}$, which is the smallest value. The additional introduction of HMT into the studied inhibited media containing Fe(III), as a rule, worsens the protection of the metal by the inhibitor (Figure 8).

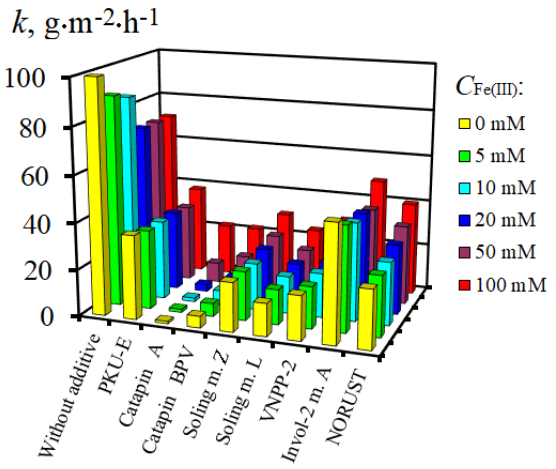

Figure 7. Corrosion rates of 08PS steel in 2 M H_3PO_4 (60°C) containing $FePO_4$ in the presence of 2 $g\cdot L^{-1}$ of the inhibitors.

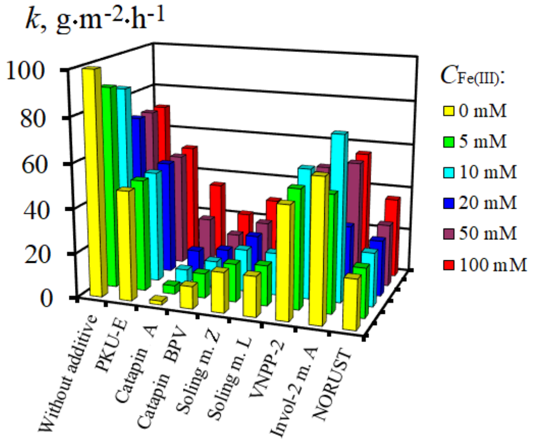

Figure 8. Corrosion rates of 08PS steel in 2 M H_3PO_4 (60°C) containing $FePO_4$ in the presence of 2 $g\cdot L^{-1}$ inhibitor + 200 mM HMT.

Since individual CIs and their mixtures with HMT do not provide satisfactory protection of 08 PS steel in 2 M H_3PO_4 containing $FePO_4$, we studied three-component formulations that additionally contained 0.5 mM KNCS + 200 mM HMT. As we noted above, similar mixtures based on the IFKhAN-92 inhibitor can significantly slow down the corrosion of low carbon steel in a medium containing H_3PO_4, even in the presence of Fe(III) salts.

In cold 2 M H_3PO_4 containing no $FePO_4$, all the three-component CI formulations successfully slow down the corrosion of steel, reducing it by a factor of 10–65 (Figure 9). The presence of the Fe(III) salt in the solution reduces the protective effect of all the composite CIs. With the exception of the PKU-E + KNCS + HMT and Soling m. Z + KNCS + HMT combinations, rather low k values of steel are observed in the presence of the composite CIs studied. The increase in corrosion losses due to the presence of 0.10 M $FePO_4$ in 2 M H_3PO_4 with addition of KNCS + HMT formulation with the PKU-E inhibitor is 95%, with Catapin A it is 71%, with Catapin BPV – 33%, with Soling m. Z – 91%, with Soling m. L – 81%, with VNPP-2 – 71%, with Invol-2 m. A – 89%, and with NORUST CM 150 C – 77%.

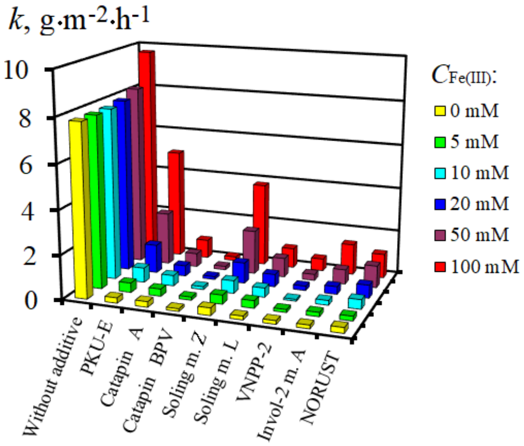

Figure 9. Corrosion rates of 08PS steel in 2 M H_3PO_4 (20°C) containing $FePO_4$ in the presence of 2 g·L^{-1} of the inhibitors + 0.5 mM KNCS + 200 mM HMT.

In background 2 M H_3PO_4 (60°C), all the three-component CI formulations successfully slow down steel corrosion, reducing it by a factor of 160–810 (Figure 10). The presence of the Fe(III) salt in the solution significantly reduces the protective effect of the composite CIs, except for the VNPP-2 + KNCS + HMT formulation. The increase in corrosion losses due to the presence of 0.10 M $FePO_4$ in 2 M H_3PO_4 with addition of the KNCS + HMT formulation with the PKU-E inhibitor is 95%, with Catapin A – 96%, with Catapin BPV – 98%, with Soling m. Z – 98%, with Soling m. L – 98%, with VNPP–2 – 48%, with Invol–2 m. A – 95%, and with NORUST CM 150 C – 98%.

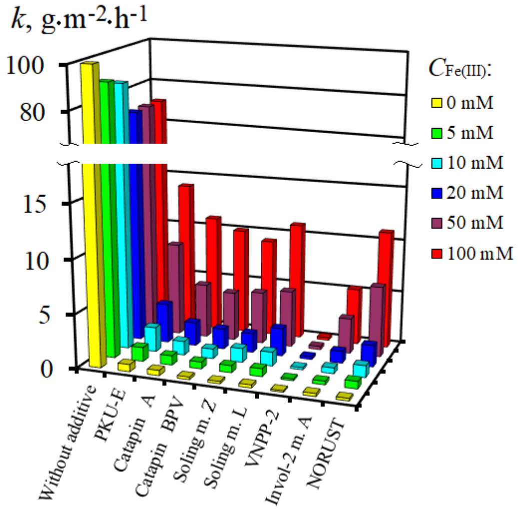

Figure 10. Corrosion rates of 08PS steel in 2 M H_3PO_4 (60°C) containing $FePO_4$ in the presence of 2 $g \cdot L^{-1}$ of the inhibitors + 0.5 mM KNCS + 200 mM HMT.

The VNPP-2 + KNCS + HMT formulation for which $k \leq 0.25$ $g \cdot m^{-2} \cdot h^{-1}$, which is a promising result for further studies, was selected from the CI mixtures studied. Along with it, we studied the formulation comprising Invol-2 m. A + KNCS + HMT, for which we tried to obtain a satisfactory effect by increasing the content of the components.

Mixture of hydrochloric and phosphoric acids

In the 1 M HCl + 1 M H_3PO_4 background solution, the k values of steel are 4.0 and 54 g $g \cdot m^{-2} \cdot h^{-1}$ at 20 and 60°C, respectively (Table 1). Addition of Fe(III) salts to this medium accelerates corrosion of steel. At 20°C in the presence of 0.10 M Fe(III), the increase in corrosion losses is 4.2 $g \cdot m^{-2} \cdot h^{-1}$, or 51% of the total corrosion rate. At 60°C in the presence of 0.10 M Fe(III), the increase in corrosion losses is 29 $g \cdot m^{-2} \cdot h^{-1}$, or 35% of the total corrosion rate. Inhibition of this medium with the VNPP-2 + KNCS + HMT formulation slows down the corrosion of steel to 0.13–0.35 $g \cdot m^{-2} \cdot h^{-1}$ at 20°C and to 0.10–1.5 $g \cdot m^{-2} \cdot h^{-1}$ at 60°C. At 20°C, the increase in corrosion losses due to the presence of 0.10 M Fe(III) is 0.22 $g \cdot m^{-2} \cdot h^{-1}$ or 63%, while at 60°C it is 1.4 $g \cdot m^{-2} \cdot h^{-1}$, or 93%. In contrast, under the same conditions, the Invol-2 m. A + KNCS + HMT formulations are inferior in protective effect to the VNPP-2 + KNCS + HMT formulation.

Table 1. Corrosion rates of 08PS steel (*k*, g·m^{-2}·h^{-1}) in 1 M HCl + 1 M H_3PO_4 containing Fe(III) salts.

Temperature	**$C_{Fe(III)}$, mM**					
	0	**5**	**10**	**20**	**50**	**100**
Without additive						
20°C	4.0	3.6	3.1	3.9	5.1	8.2
60°C	54	50	48	52	70	83
2 g/L Invol-2 m. A + 200 mM HMT + 0.5 mM KNCS						
20°C	0.20	0.35	0.49	0.55	1.6	4.4
60°C	0.64	1.0	1.2	2.2	4.2	6.0
4 g/L Invol-2 m. A + 200 mM HMT + 1.0 mM KNCS						
60°C	0.53	1.1	1.3	2.2	3.4	6.6
2 g/L VNPP-2 + 200 mM HMT + 0.5 mM KNCS						
20°C	0.13	0.15	0.15	0.21	0.30	0.35
60°C	0.10	0.05	0.08	0.15	0.75	1.5

The efficiency of the CIs studied in the inhibition of low carbon steel corrosion in cold HCl solution differs. On transition to hot HCl, the PKU-E agent poorly inhibit corrosion. In H_3PO_4 solution at 20°C, PKU-E, Soling m. Z, NORUST CM 150 C poorly inhibit corrosion, while at 60°C, Soling m. L, VNPP-2, and Invol-2 m. A also join this list. For all the CIs that demonstrate high protective effects in acids that do not contain Fe(III) salts, the presence of such salts deteriorates the protective effect significantly. At a high content of Fe(III) salts in the acids, protection is almost lost and the corrosion rate of the metal becomes high.

The absence of a positive effect of HMT on slowing down the corrosion of steel by the studied inhibitors was unexpected, since this is the only additive described in the literature [18] that allowed nitrogen-containing inhibitors to maintain their protective effect on steels when $FeCl_3$ salt accumulated in the HCl solution. This difference is due to the fact that the authors [18] studied dilute acid solutions, while we studied more concentrated environments.

The formulation of VNPP-2 + KNCS + HMT provides good protection of steel in H_3PO_4 even if up to 0.1 M $FePO_4$ is accumulated in it. This effect is important because it persists when a substantial fraction of H_3PO_4 is replaced by HCl in the system. At $t \leq 60$°C in 1 M HCl + 1 M H_3PO_4 containing 2 g·L^{-1} VNPP-2 + 0.5 mM KNCS + 200 mM HMT, the corrosion rate of 08 PS steel does not exceed 1.5 g·m^{-2}·h^{-1}, which is an outstanding result.

A new inhibited acid formulation based on HCl and H_3PO_4 with low corrosivity against low carbon steels, which is resistant to the accumulation of Fe(III) salts, has been developed.

Efficient inhibition of steel corrosion in this environment is achieved by the complex action of the components it comprises. Phosphoric acid binds Fe(III) cations into complex compounds, reducing their oxidizing ability and mobility in the corrosive environment [19]. Moreover, the addition of HMT further binds Fe(III) cations into more complex compounds, thus enhancing the effect of H_3PO_4 [20]. By analogy with the IFKhAN-92 inhibitor [21], it can be assumed that the formulation of VNPP-2 + KNCS + HMT should form a protective film on steel, which can efficiently slow down the reduction of Fe(III) cations, thus preventing corrosion.

Conclusions

1. None of the eight CIs studied can provide efficient protection of low carbon steel in HCl solutions (20 and 60°C) if significant amounts of Fe(III) salts are accumulated in the medium.
2. In H_3PO_4 solutions (20 and 60°C) containing $FePO_4$, efficient steel protection can be obtained by the formulation comprising VNPP-2 + KNCS + HMT as the CI.
3. The formulation comprising 2 $g \cdot L^{-1}$ VNPP-2 + 0.5 mM KNCS + 200 mM HMT allows steel to be protected in the mixture of HCl + H_3PO_4 (20 and 60°C) containing Fe(III) salts. This solution can be an alternative to inhibited solutions of individual HCl in case of potential accumulation of Fe(III) chloride.

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