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

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Abstract

The effect of Fe(III) salts on the protection of mild steel 08PS in 2 M H_2SO_4 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) has been studied. None of the individual corrosion inhibitors studied can provide efficient protection of steel in H_2SO_4 and H_3PO_4 solutions if significant amounts of Fe(III) salts 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 hexamethylenetetramine as the corrosion inhibitor. The same formulation protects steel in 1 M H_2SO_4+1 M H_3PO_4 (20 and 60°C) with up to 0.10 M Fe(III) cations accumulated in it. Solutions of an H_2SO_4+H_3PO_4 mixture inhibited by the formulation of VNPP-2+KNCS+hexamethylenetetramine can be an alternative to inhibited solutions of individual H_2SO_4 in case of potential accumulation of Fe(III) sulfate in them.

Introduction

Sulfuric acid and its solutions are common technological fluids used at the modern enterprises [1–6]. 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 H_2SO_4 solutions are performed in the presence of corrosion inhibitors (CI), thus minimizing the metal losses caused by its reaction with the acid. The existing range of CIs for steels in acid media, including H_2SO_4 solutions, was reviewed in numerous publications [7–15].

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) sulfate in an H_2SO_4 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:

$$Fe_2(SO_4)_3 + Fe = 3FeSO_4.$$

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 H_2SO_4 solutions that occur under diffusion control [16].

From the practical point of view, it is important to understand how the accumulation of Fe(III) sulfate in H_2SO_4 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.

A promising direction for increasing the efficiency of industrial CIs in H_2SO_4 solutions containing Fe(III) salts involves switching from the individual acid to its mixtures with H_3PO_4. Moreover, for steel protection in solution than comprise acid mixtures, not individual CIs but their mixtures with hexamethylenetetramine (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 [17, 18]. 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 H_2SO_4 + 1 M 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 g·m^{-2}·h^{-1} at $C_{Fe(III)} \leq 0.1$ M [19]. The presence of 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 [20]. It should be noted [19] that the creation of inhibited etching solutions resistant to the accumulation of Fe(III) salts is possible in solutions containing H_3PO_4 alone. However, this way is unreasonable from economical point of view since H_2SO_4 is less expensive than 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 H_2SO_4 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). 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 $H_2SO_4 + H_3PO_4$ solution.

Experimental

The corrosion rate of 08PS 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 H_2SO_4, 2 M H_3PO_4, and the mixture of 1 M H_2SO_4+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 mm×20 mm×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 (H_2SO_4 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. $FeCl_3 \times 6H_2O$ of "pure" grade was used to prepare the Fe(III) chloride solution.

**Results and Discussion**

Sulfuric acid

In background 2 M H_2SO_4 (20°C), the corrosion of 08PS steel occurs at a rate of k_0=9.8 g·m^{-2}·h^{-1} (Figure 1). The presence of 0.05 M $Fe_2(SO_4)_3$ accelerates corrosion by a factor of 1.8, thus the k_0 of steel increases to 8.2 g·m^{-2}·h^{-1} (Figure 1). The increase in corrosion losses due to the presence of the Fe(III) salt in the total corrosion process is 46%.
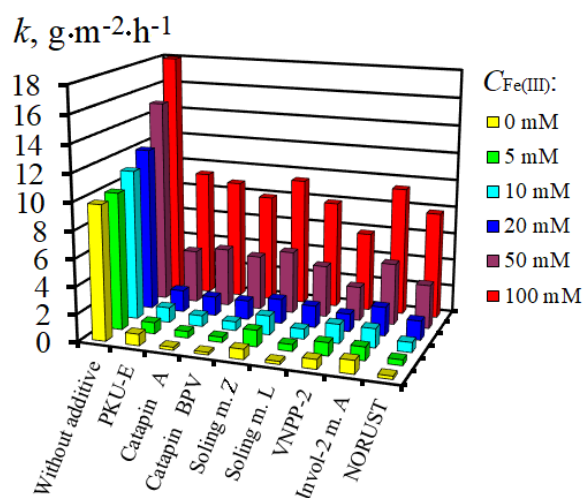
Figure 1. Corrosion rates of 08PS steel in 2 M H_2SO_4 (20°C) containing $Fe_2(SO_4)_3$ in the presence of 2 g·L^{-1} of the inhibitors.

In cold 2 M H_2SO_4 containing no $Fe_2(SO_4)_3$, all the CIs studied successfully 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. The increase in corrosion losses due to the presence of 0.05 M $Fe_2(SO_4)_3$ in 2 M H_2SO_4 is 91% in the presence of the PKU-E inhibitor, 98% with Catapin A, 98% with Catapin BPV, 92% with Soling m. Z, 97% with Soling m. L, 88% with VNPP-2, 90% with Invol-2 m. A, and 97% with NORUST CM 150 C. Of these, Invol-2 m. A has the smallest efficiency as it slows down steel corrosion only 1.9-fold, while VNPP-2, the most efficient one, slows it down 3.1-fold.

Increasing the temperature of 2 M H_2SO_4 to 60°C increases the k of steel in it to 172 $g \cdot m^{-2} \cdot h^{-1}$ (Figure 2). In this environment, addition of 0.05 M $Fe_2(SO_4)_3$ increases the corrosion losses by 32 $g \cdot m^{-2} \cdot h^{-1}$, which is 16% of the total metal losses. The CIs studied inhibit the corrosion of steel in 2 M H_2SO_4 (60°C) differently. With addition of PKU-E, Soling m. Z, VNPP-2, Invol-2 m. A, or NORUST CM 150 C, the value of k is higher than 5 $g \cdot m^{-2} \cdot h^{-1}$, while in the case of Catapin A, Catapin BPV, or Soling m. L, steel corrosion is inhibited to a greater extent and $k < 5$ $g \cdot m^{-2} \cdot h^{-1}$. The presence of the Fe(III) salt in the solution weakens the effect of all the CIs. The increase in corrosion losses due to the presence of 0.05 M $Fe_2(SO_4)_3$ in 2 M H_2SO_4 (60°C) is 69% with the PKU-E inhibitor, 97% with Catapin A, 94% with Catapin BPV, 59% with Soling m. Z, 87% with Soling m. L, 13% with VNPP-2, 62% with Invol-2 m. A, and 61% NORUST CM 150 C. These effects are weaker than at 20°C. In general, the corrosion losses of steel in 2 M $H_2SO_4 + 0.05$ M $Fe_2(SO_4)_3$ in the presence of CIs are high. For the least efficient Invol-2 m. A, they amount to 34 $g \cdot m^{-2} \cdot h^{-1}$. Soling m. Z inhibits corrosion most strongly, but even in its presence, $k = 13$ $g \cdot m^{-2} \cdot h^{-1}$. At the same time, for PKU-E, Catapin BPV, Soling m. Z, Soling m. L, and NORUST CM 150 C, $\gamma > 10$, which only formally indicates a good inhibition of steel corrosion in the acid in their presence.
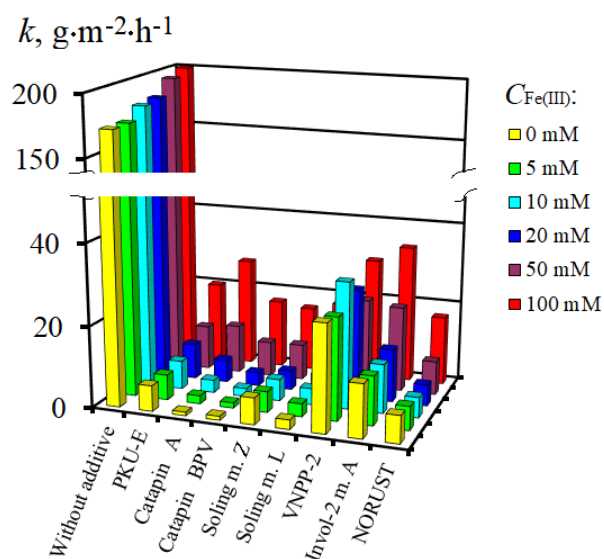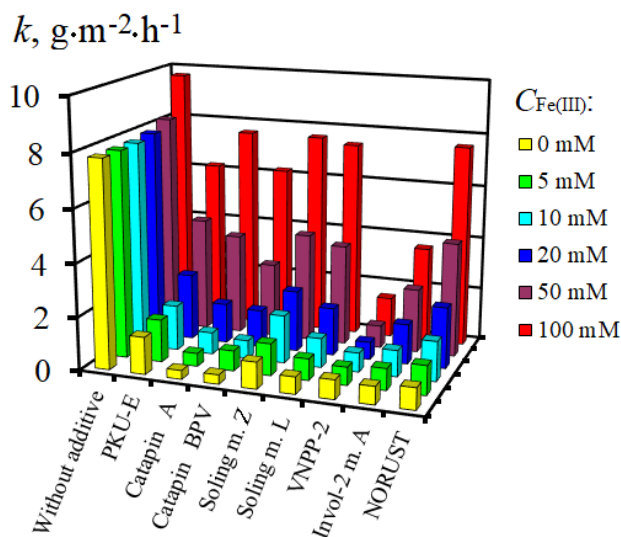

Figure 2. Corrosion rates of 08PS steel in 2 M H_2SO_4 (60°C) containing $Fe_2(SO_4)_3$ in the presence of 2 $g \cdot L^{-1}$ of the inhibitors.

In H_2SO_4 solutions containing the inhibitors in question, addition of $Fe_2(SO_4)_3$ accelerates corrosion. Despite the fact that some of them have high values of $\gamma > 10$, the observed metal losses remain high. Since the CIs studied do not provide satisfactory protection of 08PS steel in H_2SO_4 solutions containing $Fe_2(SO_4)_3$, the possibility of their application in H_3PO_4 solutions containing $FePO_4$ was investigated.

Phosphoric acid

In comparison with the H_2SO_4 solution, the k value of steel in 2 M H_3PO_4 (20°C) is slightly smaller, *i.e.*, 7.8 g·m^{-2}·h^{-1} (Figure 3). 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 3. 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.

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·m^{-2}·h^{-1} (Figure 4). 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·m^{-2}·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 ($\gamma<10$), 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 $g·m^{-2}·h^{-1}$, which is the smallest value.
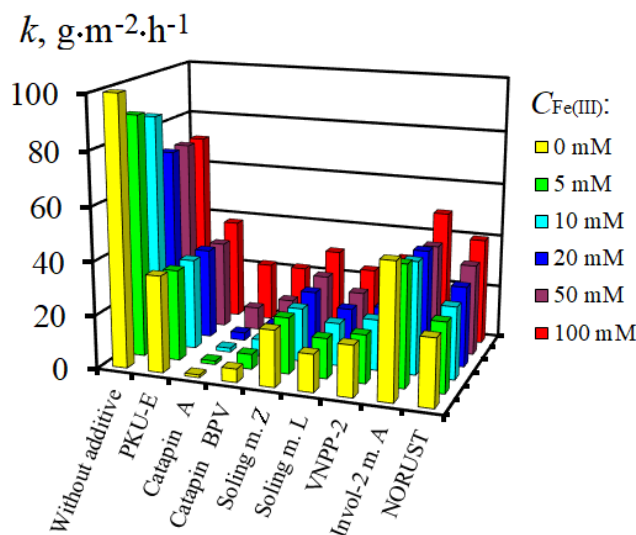
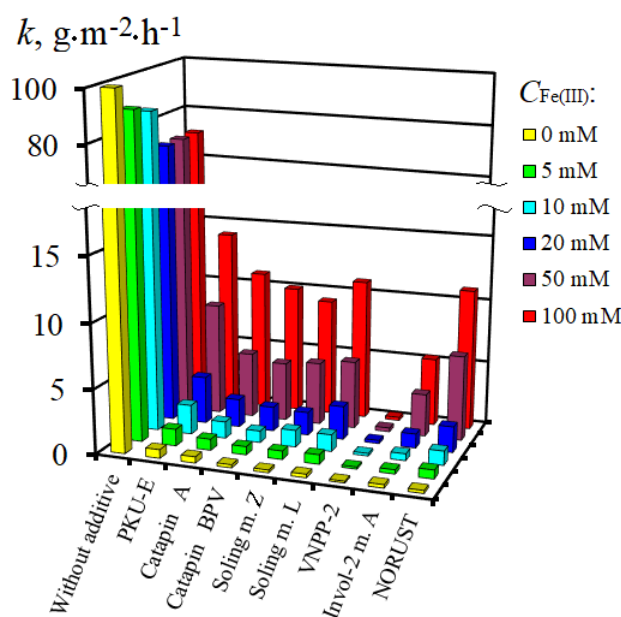**Figure 4.** Corrosion rates of 08PS steel in 2 M H_3PO_4 (60°C) containing $FePO_4$ in the presence of 2 $g·L^{-1}$ of the inhibitors.

Since individual CIs do not provide satisfactory protection of 08PS steel in 2 M H_3PO_4 containing $FePO_4$, we studied their 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 5). 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 – 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%.
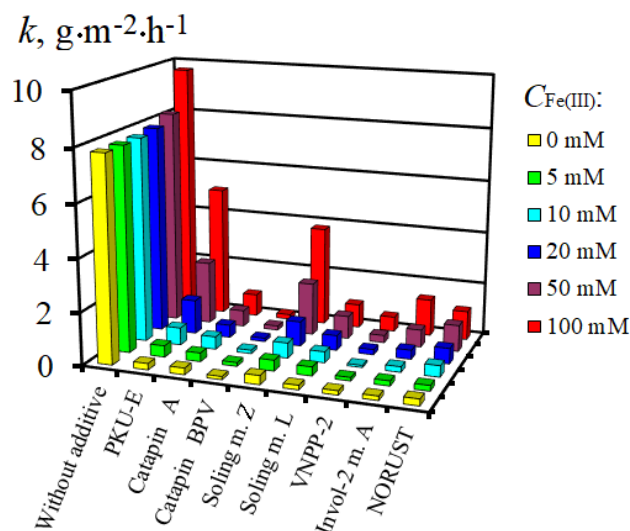
Figure 5. Corrosion rates of 08PS steel in 2 M H_3PO_4 (20°C) containing $FePO_4$ in the presence of 2 g·L^{-1} of the inhibitors+0.5 mM KNCS+200 mM HMT.

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 6). 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 6. Corrosion rates of 08PS steel in 2 M H_3PO_4 (60°C) containing $FePO_4$ in the presence of 2 g·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·m^{-2}·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 sulfuric and phosphoric acids

In the 1 M H$_2$SO$_4$+1 M H$_3$PO$_4$ background solution, the k values of steel are 8.5 and 143 g·m^{-2}·h^{-1} at 20 and 60°C, respectively (Table 1). Addition of Fe(III) salts to this medium at 20°C accelerates corrosion, while at 60°C, in contrast, the latter is hindered. At 20°C in the presence of 0.10 M Fe(III), the increase in corrosion losses is 4.5 g·m^{-2}·h^{-1}, or 35% of the total corrosion rate. In contrast, at 60°C, the corrosion decreases by 12 g·m^{-2}·h^{-1}. Inhibition of this medium with the VNPP-2+KNCS+HMT formulation slows down the corrosion of steel to 0.05–0.76 g·m^{-2}·h^{-1} at 20°C and to 0.11–0.71 g·m^{-2}·h^{-1} at 60°C. At 20°C, the increase in corrosion losses due to the presence of 0.10 M Fe(III) is 0.71 g·m^{-2}·h^{-1} or 93%, while at 60°C it is 0.60 g·m^{-2}·h^{-1}, or 85%. In contrast, under the same conditions, the Invol-2 m. A+KNCS+HMT formulations are inferior in protective effect to the Invol-2 m. A+KNCS+HMT formulation.

Table 1. Corrosion rates of 08PS steel (k, g·m^{-2}·h^{-1}) in 1 M H$_2$SO$_4$+1 M H$_3$PO$_4$ containing Fe(III) salts.

Temperature	$C_{Fe(III)}$, mM					
	0	5	10	20	50	100
Without additives						
20°C	8.5	8.5	8.7	10	11	13
60°C	143	139	138	136	131	131
2 g·L^{-1} Invol-2 m. A+0.5 mM KNCS+200 mM HMT						
20°C	0.10	0.10	0.10	0.20	0.59	2.0
60°C	0.52	0.59	0.91	1.3	2.8	8.4
4 g·L^{-1} Invol-2 m. A+1.0 mM KNCS+200 mM HMT						
60°C	1.0	1.0	0.86	1.9	2.9	5.2
2 g·L^{-1} VNPP-2+0.5 mM KNCS+200 mM HMT						
20°C	0.05	0.01	0.08	0.15	0.36	0.76
60°C	0.11	0.10	0.25	0.23	0.29	0.71

The efficiency of the CIs studied in the inhibition of mild steel corrosion in cold H$_2$SO$_4$ solution differs. On transition to hot H$_2$SO$_4$, the PKU-E, Soling m. Z, VNPP-2, Invol-2 m. A, and NORUST CM 150 C agents poorly inhibit corrosion. In H$_3$PO$_4$ 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 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 H_2SO_4 in the system. At $t \leq 60°C$ in 1 M H_2SO_4+1 M H_3PO_4 containing 2 $g \cdot L^{-1}$ VNPP-2+0.5 mM KNCS+200 mM HMT, the corrosion rate of 08PS steel does not exceed 0.76 $g \cdot m^{-2} \cdot h^{-1}$, which is an outstanding result.

A new inhibited acid formulation based on H_2SO_4 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 [17]. Moreover, the addition of HMT further binds Fe(III) cations into more complex compounds, thus enhancing the effect of H_3PO_4 [18]. By analogy with the IFKhAN-92 inhibitor [19], 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 nine CIs studied can provide efficient protection of low-carbon steel in H_2SO_4 solutions 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 H_2SO_4+H_3PO_4 (20 and 60°C) containing Fe(III) salts. This solution can be an alternative to inhibited solutions of individual H_2SO_4 in case of potential accumulation of Fe(III) sulfate.

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