A study of the H_2SO_4–H_3PO_4–H_2O–Fe(III) system by cyclic voltammetry

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Abstract

The electrochemical behavior of Fe(III) cations on a platinum electrode in the H_2SO_4–H_3PO_4–H_2O, H_2SO_4–H_2O, and H_3PO_4–H_2O systems (25–95°C) has been studied by cyclic voltammetry. The oxidizing ability of the 2.0 M $H_2SO_4 + H_3PO_4$ system containing 0.10 M Fe(III) decreases as the molar fraction of H_3PO_4 increases. The observed effect results from the formation of Fe(III) complexes with phosphate anions that are weaker oxidants than the corresponding hydrate and sulfate complexes. An increase in temperature causes an increase in the oxidizing ability of the H_2SO_4–H_3PO_4–H_2O–Fe(III) system. This effect is most pronounced in the H_3PO_4–H_2O–Fe(III) system. Addition of H_3PO_4 to H_2SO_4 solutions converts Fe(III) cations from hydrate and sulfate complexes to phosphate complexes, which leads to a decrease in their diffusion rate in aqueous acid solutions. The decrease in the diffusion coefficient of Fe(III) cations is about 1/3 of the initial value. A probable reason for efficient protection of low-carbon steel by triazole-based composite inhibitors in H_3PO_4 or its mixtures with H_2SO_4 containing Fe(III), in comparison with similar solutions of H_2SO_4 alone, is that phosphate complexes of Fe(III) are formed in these media. They have a lower oxidizing ability and diffusion rate in comparison with aqueous and sulfate complexes of Fe(III).

Key words: *corrosion inhibitors, cyclic voltammetry, sulfuric acid, phosphoric acid, redox couple Fe(III)/Fe(II), diffusion coefficient.*

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Introduction

Etching of steels in solutions of sulfuric acid is accompanied by accumulation of Fe(III) sulfate due to dissolution of scale and oxidation of Fe(II) salts, that are formed in the reactions of scale and metal with the acid, with atmospheric oxygen [1, 2]. The dissolution of steels in acid solutions containing Fe(III) sulfate occurs not only due to the reaction of iron with an acid:

$$Fe + H_2SO_4 = FeSO_4 + H_2,$$

but also due to the parallel reaction of the metal with a Fe(III) salt:

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

Fe(III) sulfate can considerably decrease the protective effect of metal corrosion inhibitors added to an etching solution [3].

The adverse effect of Fe(III) sulfate on the protective effect of corrosion inhibitors toward mild steels can be overcome by replacing sulfuric acid solutions with $H_2SO_4 + H_3PO_4$ mixtures containing a formulation of IFKhAN-92 (a 1,2,4-triazole derivative), KNCS and urotropine with a 9:1:400 molar ratio of the components. Such inhibited solutions are resistant against the accumulation of Fe(III) salts at H_2SO_4 concentrations up to 60% of the total mass of acids in a solution [4].

Previously, the potentiometry method was used to study an H_2SO_4–H_3PO_4–H_2O system containing Fe(III) and Fe(II) cations [5, 6]. It was shown that a probable reason of the efficient protection of mild steel by triazole-based composite inhibitors in H_3PO_4 or its mixtures with H_2SO_4 containing Fe(III), in comparison with similar solutions of H_2SO_4 alone, is that phosphate complexes of Fe(III) are formed in these media. They have lower oxidizing ability in comparison with aqueous and sulfate Fe(III) complexes.

To reach a more detailed understanding of the processes that occur in the H_2SO_4–H_3PO_4–H_2O–Fe(III) system and to identify the role of H_3PO_4 additives in preserving the protective action of the triazole-based composite inhibitor of in the presence of Fe(III) salts, it seems necessary to study these solutions by the cyclic voltammetry method (CVA). This method is used quite successfully to study solutions containing Fe(III) and Fe(II) cations alone or their mixtures [7–14]. The effect of HF additives on the characteristics of the Fe(III)/Fe(II) redox process in H_3PO_4 solutions was studied by CVA [7]. Addition of hydrogen fluoride reduces the redox potential of the Fe(III)/Fe(II) couple ($E_{\mathrm{Fe(III)/Fe(II)}}$) in this medium. The observed effects result from the binding of Fe cations into fluoride complexes that are characterized by higher stability compared to iron phosphate complexes.

Experimental

Solutions were prepared from H_3PO_4 and H_2SO_4 of “chemically pure” grade and distilled water. H_2SO_4 and H_3PO_4 solutions containing Fe(III) salts were obtained by dissolving $Fe(OH)_3$ precipitated with NaOH (“chemically pure” grade) from a $FeCl_3$ solution, in excess H_2SO_4 or H_3PO_4. The Fe(III) chloride solution was prepared from $FeCl_3 \cdot 6H_2O$ (“pure” grade).

CVA studies were performed using an EL-02.061 potentiostat in a thermostated three-electrode glass cell with divided electrode spaces. A platinum wire (area (S) 15.9 mm^2) served as the working electrode. A silver chloride electrode was used as the reference electrode, and a platinum plate ($S = 1.5$ cm^2) was used as the auxiliary electrode. Potential scanning was performed from 1.4 V to 0.0 V, and then from 0.0 V to 1.4 V. The scan rates were 0.02, 0.05, 0.10, 0.20 and 0.40 $V \cdot s^{-1}$. The solutions studied were deaerated with argon both before (for 30 min) and during an experiment. Before an experiment, the working electrode was degreased with acetone, kept for 3 min in concentrated HNO_3, and washed with distilled water. The temperature (t) range of the studied solutions was 25–95°C. The accuracy of temperature control was ±1°C. Electrode potentials are reported *versus* the standard hydrogen electrode.

Experimental results and discussion

The cyclic voltammograms of a Pt electrode in 2.0 M H_2SO_4 + 0.05 M $Fe_2(SO_4)_3$ at 25°C (Figure 1, Table 1) contain two peaks, the maxima of which are located at E = 0.52–0.55 V and 0.75–0.81 V, depending on the potential sweep rate (v). The first cathodic peak corresponds to the reduction of Fe(III) cations on the Pt electrode:

$$Fe^{3+} + e^{-} = Fe^{2+}.$$

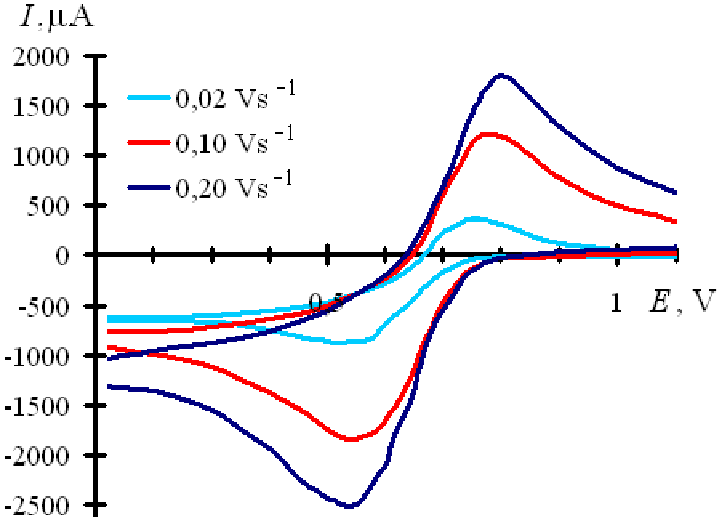

Figure 1. Cyclic voltammograms of platinum electrode in 2.0 M H_2SO_4 + 0.05 M $Fe_2(SO_4)_3$ at different scan rates at 25°C.

Table 1. CVA data of platinum electrode in 2.0 M H_2SO_4 + $Fe_2(SO_4)_3$ at 25°C.

Scan rate (v), V·s^{-1}	**Cathodic peak potential (E_{pc}), V**	**Anodic peak potential (E_{pa}), V**	**Peak potential separation (E_{pa}–E_{pc}), V**	**Half-wave potential ($E_{1/2}$), V**	**Cathodic peak current (I_{pc}), mA**	**Anodic peak current (I_{pa}), mA**	**Peak current ratio (I_{pa}/I_{pc})**
			0.005 M $Fe_2(SO_4)_3$				
0.10	0.61	0.72	0.11	0.67	0.18	0.22	1.22
			0.02 M $Fe_2(SO_4)_3$				
0.10	0.56	0.78	0.22	0.67	0.70	0.63	0.90
			0.05 M $Fe_2(SO_4)_3$				
0.02	0.56	0.76	0.20	0.66	0.80	0.85	1.06
0.05	0.56	0.77	0.22	0.67	1.3	1.3	1.00
0.10	0.55	0.78	0.23	0.67	1.8	1.7	0.94
0.20	0.54	0.79	0.25	0.67	2.5	2.1	0.84
0.40	0.52	0.81	0.29	0.67	3.5	3.0	0.86

The second peak, *i.e.*, the anodic one, corresponds to the oxidation of the Fe(II) cations formed:

$$Fe^{2+} - e^{-} = Fe^{3+}.$$

As v increases, the maximum potential of the cathodic peak (E_{pc}) decreases, while the anodic peak (E_{pa}) grows. The currents in the cathodic and anodic peak maxima (I_{pc} and I_{pa}) also increase with increasing v. The system under study is quasi-equilibrium since the criteria for equilibrium systems are not satisfied for it: $E_{pa} - E_{pc} >> 0.057$ V and $I_{pa}/I_{pc} \neq 1$, but both peaks, the cathodic and anodic ones, are present on the voltammograms [15]. The half-wave potential:

$$E_{1/2} = \frac{E_{pc} + E_{pa}}{2},$$

which is often interpreted as the redox potential of a system, does not depend on v within the measurement accuracy limits. A decrease in the content of Fe(III) in the acid solution does not significantly change the nature of cyclic voltammograms (Figure 2, Table 1), but the values of I_{pc} and I_{pa} decrease.

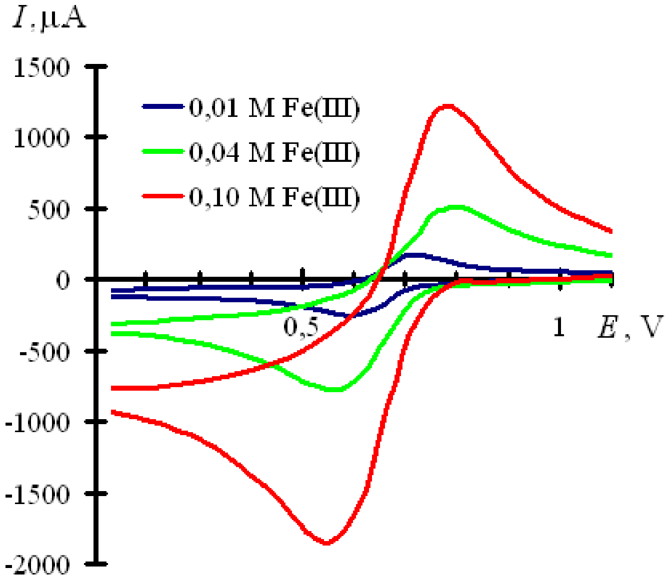

Figure 2. Cyclic voltammograms of platinum electrode in 2.0 M H_2SO_4 + $Fe_2(SO_4)_3$ at different concentration of Fe(III). Scan rate: 0.10 Vs^{-1} at 25°C.

The values of current maxima on the current-potential curves is determined by the Randles-Sevcik equation:

$$I_p = 2.69 \cdot 10^5 z^{1.5} S C D^{0.5} v^{0.5},$$

where $2.69 \cdot 10^5$ is the equation constant at 25°C; z is the number of electrons participating in the redox process ($z = 1$ for the redox pair Fe (III)/Fe (III)); S is the surface area of the electrode (m^2); *C* is the concentration of the electrochemically active substance ($mol \cdot m^{-3}$); *D* is the diffusion coefficient ($m^2 \cdot s^{-1}$); and *v* is the potential sweep rate ($V \cdot s^{-1}$). The Randles–Sevcik equation describes redox processes that occur in equilibrium systems. However, it is noted that it can be used to describe quasi-reversible and even irreversible systems [15]. The criterion for the applicability of this equation for describing the cathode process in this system is that the I_{pc} *vs.* $v^{0.5}$ (Figure 3) and I_{pc} *vs.* $C_{Fe(III)}$ plots (Figure 4) should be linear.

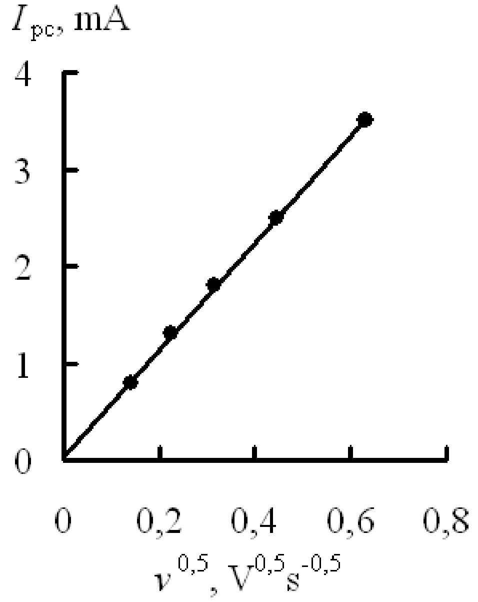

Figure 3. Variation of the cathodic peak current (I_{pc}) vs the square root of scan rate ($v^{0.5}$) for platinum electrode in 2.0 M H_2SO_4 + 0.05 M $Fe_2(SO_4)_3$. t = 25°C.

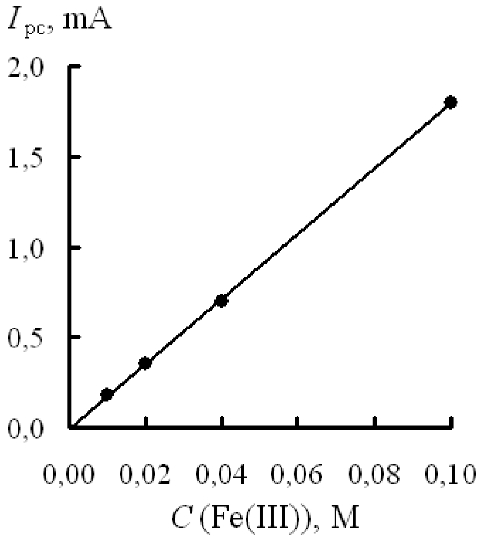

Figure 4. Variation of the cathodic peak current (I_{pc}) vs concentration of $Fe_2(SO_4)_3$ for platinum electrode in 2.0 M H_2SO_4 + $Fe_2(SO_4)_3$. Scan rate: 0.10 $V \cdot s^{-1}$. t = 25°C.

The peaks on the voltammograms of a Pt electrode in $H_2SO_4 + H_3PO_4$ solutions containing 0.10 M Fe(III) are shifted towards lower potentials in comparison with the solution of H_2SO_4 alone, and are characterized by higher non-equilibrium (Figure 5, Table 2). At 25°C, $E_{pa} - E_{pc} = 0.31–0.39$ V in $H_2SO_4 + H_3PO_4$ but as small as 0.23 V in 2.0 M H_2SO_4. Already at 10% H_3PO_4 in the solution, the cathodic current decreases. As the content of H_3PO_4 in the mixture increases, the value of $E_{1/2}$ decreases systematically, indicating a decrease in the oxidative capability of the system. The lowest value $E_{1/2} = 0.41$ V is observed in 2.0 M H_3PO_4, against $E_{1/2} = 0.67$ V in 2.0 M H_2SO_4.

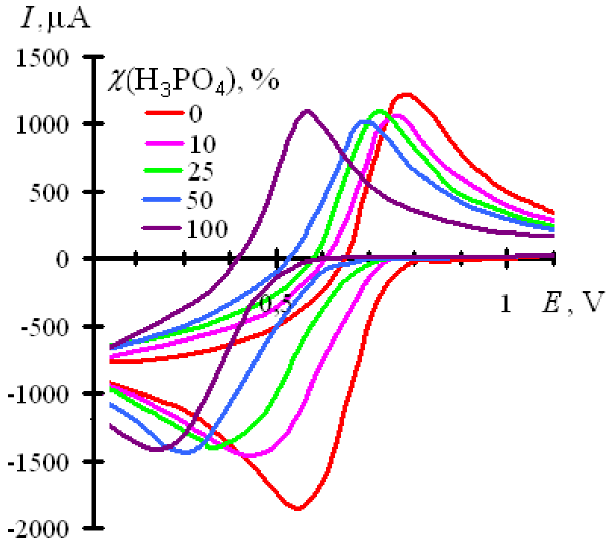

Figure 5. Cyclic voltammograms of platinum electrode in $H_2SO_4 + H_3PO_4$ + 0.10 M Fe(III) (25°C) at different concentration of H_3PO_4. Scan rate: 0.10 $V \cdot s^{-1}$ at 25°C.

Other conditions being equal, an increase in temperature (*t*) results in an increase in the height of the cathodic and anodic peaks on the voltammograms of acid solutions containing Fe(III) (Figure 6, Table 2). An increase in $E_{1/2}$ is observed. The $E_{1/2}$ values in 2.0 M H_3PO_4 + 0.10 M $FePO_4$ are least sensitive to an increase in temperature. Comparison of the $E_{1/2}$ values obtained from these cyclic voltammograms with $E_{Fe(III)/Fe(II)}$ determined in potentiometric studies [5] shows their good agreement. In 2.0 M H_2SO_4 + 0.05 M $Fe_2(SO_4)_3$, $E_{1/2} = 0.67–0.76$ V, $E_{Fe(III)/Fe(II)} = 0.66–0.75$ V; in 2.0 M H_3PO_4 + 0.10 M $FePO_4$, $E_{1/2} = 0.41–0.46$ V, $E_{Fe(III)/Fe(II)} = 0.44–0.46$ V; in 1.0 M H_2SO_4 + 1.0 M H_3PO_4 + 0.025 M $Fe_2(SO_4)_3$ + 0.50 M $FePO_4$, $E_{1/2} = 0.51–0.62$ V, $E_{Fe(III)/Fe(II)} = 0.55–0.60$ V. It should be noted that in [7] larger quantitative differences are observed between the potentials of the Fe(III)/Fe(II) redox pair obtained from the results of parallel measurements by CVA and potentiometry.

Table 2. CVA data of platinum electrode in H_2SO_4 + H_3PO_4 + $Fe_2(SO_4)_3$ + $FePO_4$ system at different temperatures. Scan rate: 0.10 $V \cdot s^{-1}$.

Temp. (*t*), °C	**Cathodic peak potential (E_{pc}), V**	**Anodic peak potential (E_{pa}), V**	**Peak potential separation ($E_{pa}-E_{pc}$), V**	**Half-wave potential ($E_{1/2}$), V**	**Cathodic peak current (I_{pc}), mA**	**Anodic peak current (I_{pa}), mA**	**Peak current ratio (I_{pa}/I_{pc})**
2.0 M H_2SO_4 + 0.05 M $Fe_2(SO_4)_3$							
25	0.55	0.78	0.23	0.67	1.8	1.7	0.94
40	0.63	0.75	0.12	0.69	2.6	2.8	1.08
60	0.68	0.77	0.09	0.73	3.4	3.1	0.91
80	0.70	0.79	0.09	0.75	3.8	3.2	0.84
95	0.71	0.80	0.09	0.76	4.3	4.0	0.93
1.8 M H_2SO_4 + 0.2 M H_3PO_4 + 0.045 M $Fe_2(SO_4)_3$ + 0.01 M $FePO_4$							
25	0.45	0.76	0.31	0.61	1.4	1.4	1.00
1.5 M H_2SO_4 + 0.5 M H_3PO_4 + 0.0375 M $Fe_2(SO_4)_3$ + 0.025 M $FePO_4$							
25	0.36	0.73	0.37	0.55	1.4	1.4	1.00
1.0 M H_2SO_4 + 1.0 M H_3PO_4+ 0.025 M $Fe_2(SO_4)_3$ + 0.50 M $FePO_4$							
25	0.31	0.70	0.39	0.51	1.4	0.87	0.62
40	0.34	0.70	0.36	0.52	1.8	1.4	0.78
60	0.48	0.66	0.18	0.57	2.3	2.5	1.09
80	0.54	0.65	0.11	0.60	3.4	3.5	1.03
95	0.57	0.67	0.10	0.62	3.9	4.2	1.08
2.0 M H_3PO_4 + 0.10 M $FePO_4$							
25	0.25	0.56	0.31	0.41	1.4	0.99	0.71
40	0.28	0.54	0.26	0.41	1.8	1.4	0.78
60	0.35	0.52	0.17	0.44	2.4	2.3	0.96
80	0.38	0.53	0.15	0.46	3.1	2.9	0.94
95	0.39	0.52	0.13	0.46	3.7	3.3	0.89

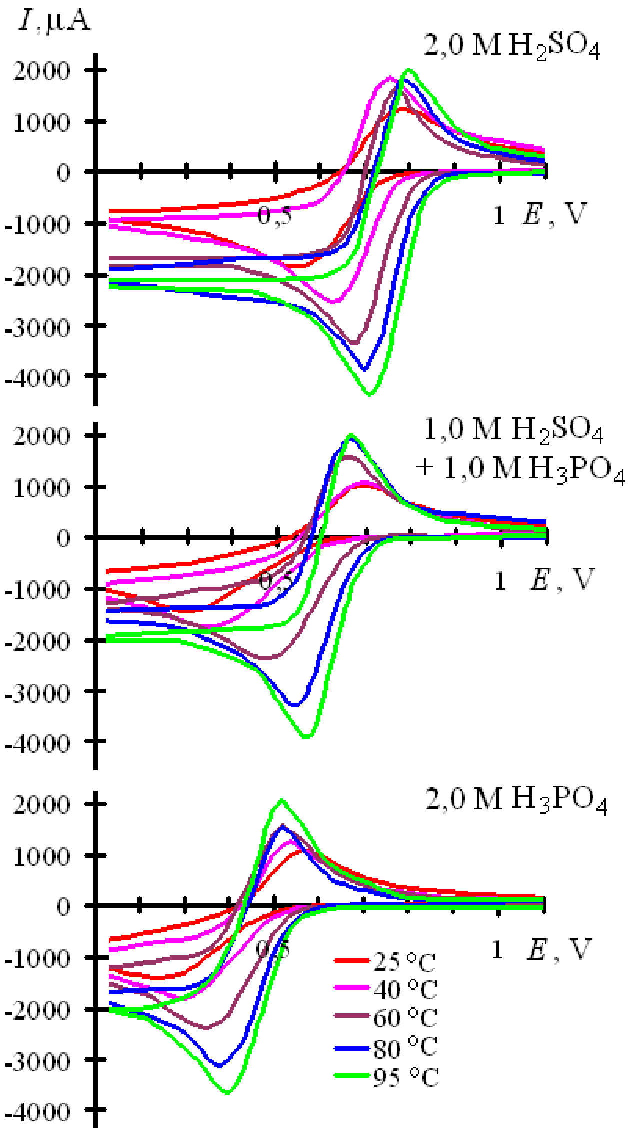

Figure 6. Cyclic voltammograms of platinum electrode in H_2SO_4 + H_3PO_4 + 0.10 M Fe(III) at different temperatures. Scan rate: 0.10 $V \cdot s^{-1}$.

It appears important to estimate the effect of H_3PO_4 addition on the diffusion rate of Fe(III) cations in the media being studied. The results that we presented above show the possibility of using the Randles-Sevcik equation for describing the cathodic process in the systems we are studying. The I_{pc} values obtained experimentally allowed us to calculate the $D_{Fe\ (III)}$ parameter by the Randles-Sevcik equation (Figure 7). The values of the diffusion coefficient of Fe(III) cations show that already at 10% H_3PO_4 in the system, it decreases by about 1/3 compared to the value observed in H_2SO_4. The average $D_{Fe(III)}$ value that we obtained in 2.0 M H_3PO_4 is 110 $\mu m^2 \cdot s^{-1}$, which is close to $D_{Fe(III)}$ = 120 $\mu m^2 \cdot s^{-1}$ in 1.0 M H_3PO_4 (20°C) according to [7].

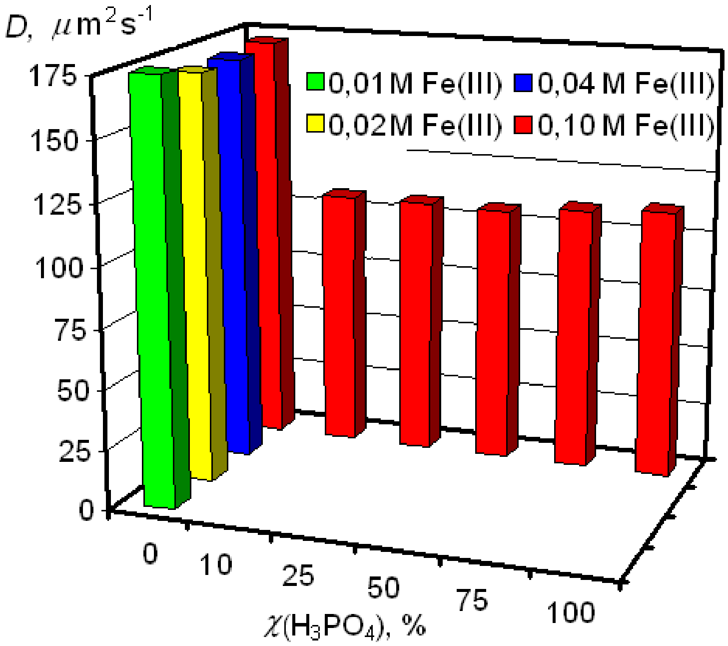

Figure 7. Effect of H_3PO_4 and Fe(III) concentrations in a H_2SO_4 + H_3PO_4 + Fe(III) system on the diffusion coefficient of the Fe(III) cation. t = 25°C.

Thus, the use of CVA to study H_2SO_4 + H_3PO_4 mixtures containing Fe(III) allowed us to find additional reasons of the preservation of the protective effect of the IFKhAN-92 based composite inhibitor in the corrosion of steels in these media. Phosphate ions bind the strong oxidant additionally present in the solution, *i.e.*, Fe(III) cations, into strong complexes. These phosphate complexes have lower oxidizing ability and mobility than aqueous and sulfate complexes of Fe(III). Addition of H_3PO_4 affects the thermodynamic properties of the corrosive environment by reducing its oxidative capability. This additive also changes the kinetic parameters of the reduction of Fe(III) cations on steel that occurs in diffusion mode [16]. A decrease in the diffusion rate of Fe(III) cations should slow down their reduction. Apparently, such a complex effect should be sufficient to enable the composite inhibitor based on IFKhAN-92 not only to slow down the reduction of protons but also suppress the reduction of Fe(III) cations. At the same time, the IFKhAN-92 inhibitor itself plays an important role in the observed effect. This compound can form monomolecular and polymolecular protective layers on the surface of metals in acid solutions. These layers are strongly bound to the metal surface by chemical bonds [17, 18], which allows the maximum protective effect to be provided in such strongly corrosive environments as acids.

Conclusions

1. The oxidizing capability of the H_2SO_4–H_3PO_4–H_2O system containing Fe(III) and Fe(II) decreases as the relative content of H_3PO_4 increases. The observed effect results from the formation of Fe(III) complexes with phosphate anions that are weaker oxidants than the corresponding hydrate and sulfate complexes.

2. Addition of H_3PO_4 to H_2SO_4 solutions converts Fe(III) cations from hydrate and sulfate complexes to phosphate complexes, which leads to a decrease in their diffusion rate in aqueous acid solutions. The decrease in the diffusion coefficient of Fe(III) cations is about 1/3 of the initial value.
3. The oxidizing ability of Fe(III) phosphate complexes is lower than that of its aqueous and sulfate complexes. These effects explain the efficient protection of low-carbon steel by composite inhibitors based on triazoles in H_3PO_4 or in its mixtures with H_2SO_4 containing Fe(III) in comparison with similar solutions of H_2SO_4 alone.

Acknowledgments

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