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CORROSION BEHAVIOR OF MILD STEEL IN SODIUM SULFATE SOLUTION IN PRESENCE OF PHOSPHATES OF DIFFERENT COMPOSITION

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ABSTRACT

Corrosion of mild steel in sodium sulfate solutions (1000 mg SO_4^{2-} L^{-1}) in the presence of various phosphate inhibitors was studied gravimetrically. Studies were performed under static conditions at room temperature. The known sodium polyphosphate inhibitor has been found to have low efficacy under these conditions. The inhibitor efficiency of its does not exceed 30.5 % at a concentration of 100 mg P_2O_5 L^{-1}. It has been shown that a more effective inhibitor is calcium-manganese polyphosphate (Ca, Mn)$(PO_3)_2$ obtained on the basis of manganese ore enrichment wastes of the Zhayrem deposit. The inhibitor efficiency of its already at a concentration of 20 mgP_2O_5 L^{-1} is 47.3 %. At a concentration of 100 mgP_2O_5 L^{-1}, the inhibitor efficiency (Ca, Mn)$(PO_3)_2$ is 96.2 %. Electrochemical tests confirmed the results of the gravimetric study. Infrared spectroscopic analysis showed that phosphate protective coatings were formed on the steel surface in solutions of sodium sulfate with additives of calcium-manganese polyphosphate.

Keywords: *corrosion, mild steel, inhibitor, inhibitor efficiency, sodium sulfate solutions.*

INTRODUCTION

Mild steel, due to its high strength, good ductility, and weldability, is the main structural material widely used in various water supply systems. However, due to the low corrosion resistance, such steel requires special corrosion protection measures. The problem of internal corrosion of steel water lines is particularly relevant at present due to the increasing increase in corrosion activators such as sulfates and chlorides in the transported water.

For example, the source of water supply to southern Kazakhstan is the Syrdaria River and its tributaries. At the same time, about 20 million tons of salts are washed off from the fields with collector-drainage return waters to Syrdaria annually. This increases the mineralization of water from 300 - 600 mg L^{-1} in the upper reaches to 3000 mg L^{-1} in the lower reaches of the Ferghana Valley and even more in the area of the river flow. The average content of the main pollutants exceeds the norm by 1.5 - 4.2 times. The main contaminants are sulfate ions, nitrites, and copper cations [1 - 3]. Since in Kazakhstan almost all systems and main water pipelines are made of soft steel and are operated without using any methods to protect the inner surface of pipes from corrosion, the problem of developing methods for corrosion protection of such structures is especially acute.

In the world practice of protecting metals from corrosion, the most expedient from an economic and technological point of view for systems in operation, in particular, steel water pipes, is considered to be a method of protection using corrosion inhibitors, as which phosphate-, silicate-, chromate-, nitrite-, boron-containing compounds are applied from inorganic substances [4 - 7]. Nevertheless, despite the high efficiency, the use of chromates and nitrites does not find practical application due to their high toxicity [6]. The use of silicates is possible only for waters with low mineralization. Moreover, their use can enhance the pH of water [7]. Highly soluble inorganic phosphates of alkali metals, including polymeric ones, have low efficiency at low concentrations, and at high concentrations they can increase metal corrosion rate owing to forming the soluble polyphosphate complexes [8, 9].

In the early 90s the work began to create of new class inhibitors of corrosion metal - sodium silicopolyphosphates modified with non-ferrous metal oxides that showed high anticorrosive properties [10 - 13]. Availability of phosphate and silicate components in the given compounds' structure, each of which has inhibitory properties, as well as the ability to flexibly regulate these substances' properties owing to inserting the various modifying additives into their composition, make it possible to gain new materials with a synergistic effect [10 - 13].

S.N. Narenova et al., G.B. Bekenova et al., V.I. Kapralova et al. described in their works [10 - 13] that glassy polymer sodium phosphates in the range of compositions $1{,}00 < R < 1{,}67$ under static testing conditions in 1 - 15 mg P_2O_5 L^{-1} concentration range accelerate the non-alloy steel's corrosion process. For the same compositions of sodium silicopolyphosphates, the concentration range stimulating the metal corrosion rate is practically absent. Likewise, it was found that inhibitory effect degree depends not just on phosphates concentration in the fluid, but also on the content of silicon dioxide in the glass [13]. Studies have also shown that despite the high inhibitory properties in waters with low salinity, the use of sodium silicopolyphosphates in aqueous media with increased chloride and sulfate ions, which are corrosion stimulants, is inefficient and requires increased consumption of inhibitors [11].

Polyphosphates of alkaline earth metals and *d*-elements, such as zinc and manganese, are more productive in comparison with sodium ones, having an inhibitory effect even in the region of low concentrations, including with the increased content of sulfate ions [13]. However, the given polyphosphates have not found their widespread as metal corrosion inhibitors due to their very low solubility. In addition, these materials were obtained based on technical individual compounds at temperatures of 1000^{o}C and above that causes their high cost [13].

Based on the above, creating the new corrosion inhibitors that comprise d-elements, and in particular, manganese, based on ore dressing waste, and the study of their inhibitory properties, with in sulfate-containing aqueous media, is of certain scientific as well as practical interest.

The studies we conducted earlier [14] have shown that by means of acid-thermal processing of waste from gravity enrichment of manganese ore from Zhayrem deposit at 200°C temperature, well-soluble calcium-manganese phosphate products with the main components' molar ratio (in terms of oxides) are formed $CaO:MnO:P_2O_5 = 0.445:0.037:0.518$ and phosphate content in terms of phosphorus pentoxide (73.64 ± 1.33) wt. %. These products have shown rather high inhibitory properties in relation to low-carbon steel in contact with aqueous media with low mineralization and low content of sulfate ions [15].

The purpose of this work was to study mild steel corrosion behavior in sulfate-containing media in the presence of calcium-manganese phosphate obtained based on manganese ore enrichment waste as a corrosion inhibitor.

EXPERIMENTAL

Studies of the inhibitory properties of the phosphate products were carried out by the gravimetric method at room temperature according to the standard procedure [16] on samples in the form of plates 20x50x1 mm in size. Samples were made from mild steel composition: 0.12 % C, 0.2 % Cr, 0.17 % Mn, 0.025 % P, 0.17 % Si, 0.018 % S, 0.25 % Ni, 0.25 % Cu, the rest is iron [17].

Before testing, the surface of the samples was mechanically treated with emery paper number 2000, washed with ethanol, distilled water [18], and dried in a desiccator with calcined calcium chloride to constant weight. For each sample, the initial mass was measured on high-precision scales (with an error of no more than ± 0.1 mg) and the surface area was measured. The tests were carried out on five parallel samples for each inhibitor and each concentration. Statistical processing of the results was carried out using the Ordinary Least squares (OLS) method at a confidence level of 0.90 [16].

As corrosive medium served the model solutions of sodium sulfate with the concentration in terms of SO_4^{2-}-ion 1000 mg L^{-1} with supplements of inhibitors of various compositions and concentrations. The tests were carried out in glass chemical glasses, the volume of the test solutions was 200 cm^3. A well-known corrosion inhibitor $NaPO_3$ (sodium polyphosphate) was used as a comparison inhibitor. After the end of the test, the samples were removed from the solutions, air-dried, and the surface was visually evaluated. After visual evaluation, the corrosion products were removed from the surface with a plastic spatula, the samples were washed with ethanol, distilled water, dried and weighed.

Potentiodynamic polarization studies were carried out using Potentiostat/Galvanostat Model "Palmsens 4" at a scan rate of 1 mV s^{-1} under static conditions. The studies were carried out in a classical three-electrode electrochemical cell at room temperature. A chlorine-silver electrode served as a reference electrode, and a graphite electrode 5 mm in diameter was used as an auxiliary electrode. Mild steel plates with dimensions of 10x10x1 mm served as working electrodes. The working surface area of the samples was 1 cm^2, the rest surface was isolated with epoxy resin [19]. Sodium sulfate solution (1000 mg SO_4^{2-} L^{-1}) with additives of inhibitors of different composition and concentrations was used as electrolyte. At the beginning of the test, the working electrode was immersed in a corrosive medium for 3 hours to obtain a stabilized open circuit potential.

The study of the molecular structure of protective films was carried out by the IR spectroscopic method [20 - 22], by pressing the investigated substance with a sample of potassium bromide. The infrared absorption spectra were recorded on IR-Fourier spectrometer of Nicolet 5700 in the region of 400 cm^{-1} - 3600 cm^{-1}.

RESULTS AND DISCUSSION

The inhibitory properties of the phosphate materials were studied gravimetrically [16] under static conditions at room temperature. The corrosion rate of mild steel was determined by formula (1):

$$V_{corr} = \frac{(m_o - m_{sac})}{S \cdot T}, \frac{mg}{cm^2 day}, \quad (1)$$

where m_o and m_{sac} are the mass of the sample before and after the testing and removal of corrosion products from the surface of the sample, g; S is the surface area, cm^2; T is the time of testing, days. Inhibitor efficiency, IE (%) was calculated by the formula (2):

$$IE = \frac{100\,(V_{corr}^{o} - V_{corr}^{inh})}{V_{corr}^{o}}, \quad (2)$$

where V_{corr}^{o} and V_{corr}^{inh} are the corrosion rate in absence and in presence of inhibitor.

The protection coefficient γ was determined according to (3):

$$\gamma = \frac{(V_{cor}^{o})}{V_{cor}^{inh}} \quad (3)$$

The rate of accumulation of corrosion-salt deposits on the surface of a steel sample was calculated by formula (4):

$$Vdep = \frac{(m_f - m_o)}{S\ T}, \frac{mg}{cm^2 day}, \quad (4)$$

where m_o and m_f are the mass of the sample before and after the testing, g.

Gravimetric tests results are presented in Table 1. It follows from the results that under static tests in sulfate media, the addition of sodium polyphosphate at 1 - 10 mg P_2O_5 L^{-1} concentrations reduces the steel's corrosion rate by 1.2 times, and the inhibitor efficiency is 16.4 % - 19.5 % (Table 1). For 1 - 5 mg P_2O_5 L^{-1} concentration range, the inhibitory properties of the calcium-manganese phosphate product are practically absent (Table 1) at the sulfate ion content of 1000 mg L^{-1}. At 10 mg P_2O_5 L^{-1} concentration, the inhibitor efficiency is 12.7 % with 1.14 protection coefficient. An increase in inhibitors concentration to 20 mg P_2O_5 L^{-1} and above to 50 - 100 mg P_2O_5 L^{-1} weakly affects the properties of sodium polyphosphate, for which the inhibitor efficiency is 21.9; 26.4 and 30.5 %, and protection coefficient is 1.28; 1.36 and 1.44 for concentrations of 20; 50 and 100 mg P_2O_5 L^{-1}, respectively.

For the area of the same concentrations, calcium-manganese phosphate product reduces the corrosion rate by almost two orders of magnitude. At the same time, the inhibitor efficiency is 47.3; 88.7 and 96.2 %, and the protection coefficient is 1.90; 8.84 and 24.3 for 20; 50 and 100 mg P_2O_5 L^{-1} concentrations, accordingly.

It was also found that in sodium sulfate solutions with sodium polyphosphate $NaPO_3 + Na_2SO_4$ additives, the rate of accumulation of corrosion-salt deposits on the surface of a steel sample is 1.2 - 2.2 times less than in sodium sulfate solution without an inhibitor (Table 1), which is probably due to formation of soluble polyphosphate complexes and transition of iron ions into the solution. In sodium sulfate solutions with additives of calcium-manganese phosphate $(Ca, Mn)(PO_3)_2 + Na_2SO_4$, the mass of corrosive depositions formed on

Table 1. Dependence of corrosion indicators of steel samples mild steel on the concentration and inhibitors composition in sulfate media (1000 mg SO_4^{2-} L^{-1}).

Indicator	Inhibitor concentration mg P_2O_5 L^{-1}						
	0.0	1.0	5.0	10.0	20.0	50.0	100.0
			$NaPO_3 + Na_2SO_4$				
Corrosion rate, V_{corr}, mg/cm^{2}day	0.292 ± 0.038	0.235 ± 0.023	0.244 ± 0.033	0.237 ± 0.021	0.228 ± 0.012	0.215 ± 0.013	0.203 ± 0.005
The protection coefficient, γ	0	1.24	1.20	1.23	1.28	1.36	1.44
Inhibitor efficiency, IE, %	0	19.5	16.4	18.8	21.9	26.4	30.5
V_{dep}, mg/cm^{2}day	0.235	0.189	0.107	0.15	0.132	0.163	0.192
			$(Ca,Mn)(PO_3)_2 + Na_2SO_4$				
Corrosion rate V_{cor}, mg/cm^{2}day	0.292 ± 0.038	0.295 ± 0.019	0.288 ± 0.022	0.255 ± 0.017	0.154 ± 0.011	0.033 ± 0.007	0.012 ± 0.003
The protection coefficient, γ	0	0	1.01	1.14	1.90	8.84	24.3
Inhibitor efficiency, IE, %	0	0	1.4	12.7	47.3	88.7	96.2
V_{dep}, mg/cm^{2}day	0.235	0.223	0.234	0.217	0.164	0.049	0.032

steel samples in solutions with inhibitors concentration in 1 - 10 mg P_2O_5 L^{-1} range is comparable to the mass of depositions created on the steel's surface in the control experiment, that is, in the sodium sulfate solution. At 50 and 100 mg P_2O_5 L^{-1} concentrations, the depositions mass diminishes by 4.8 and 7.4 times, consequently (Table 1).

Visual observations established that at presence of calcium-manganese phosphate product at 50 and 100 mg P_2O_5 L^{-1} concentrations in sulfate solution, a thin uniform coating of yellowish-gray color is formed on the steel's surface (Fig.1(a, b)), whereas in the presence of $NaPO_3$ at the same concentrations, steel samples' surface is heterogeneous. It shows anode areas covered with rust-colored iron corrosion products and pure metal areas (Fig. 1(c, d)). That is, in the sulfate solution, $NaPO_3$ addition at 50 - 100 mg P_2O_5 L^{-1} concentrations lead to forming the micro-galvanic elements on the metal surface, which increases corrosion rate and can lead to accelerated growth of ulcerative corrosion that is observed in the experiment.

Infrared spectroscopic analysis of the deposits showed that precipitates containing mainly hydrated iron oxide compounds were formed on the surface of steel samples in a sodium sulfate solution without additives (Fig. 2). This is evidenced by an expanded absorption band in the region of 3200 cm^{-1} - 3400 cm^{-1}, characteristic of valence oscillations of OH groups of hydrated iron oxide compounds of the FeOOH type [20, 21].

The bands in area 1430 cm^{-1}, 875 cm^{-1} and 712 cm^{-1} indicate the existence of calcite-type carbonate compounds, and the bands in 1020 cm^{-1} - 1144 cm^{-1} and 460 cm^{-1} area show the presence of sulfate ions [20, 21]. The data are consistent with the authors' test results that showed that the primary corrosion products on mild steel formed in H_2SO_4 solutions under static conditions at 25°C in pH 0.08 - 1 1.5 range are located in the most active areas from the point of view of corrosion impact and contain $FeSO_4.5H_2O$ [23].

The different nature of the corrosion depositions formed on the steel surface in (Ca, Mn) $(PO_3)_2$ + Na_2SO_4 solutions (Fig. 3(a, b)) is confirmed not only by visual observations, but also by IR spectra, on which there are no characteristic absorption bands of sulfate and carbonate groups. Instead, a wide band with high intensity in 970 cm^{-1} - 1080 cm^{-1} area and a band in 547 cm^{-1} area have been observed, characteristic of valence fluctuations of PO_4^{3-} phosphate ion. Alongside, there have been marked the bands in 3300 cm^{-1} areas (valence vibrations of OH-groups) and 1650 cm^{-1} (deformation vibrations of H_2O), indicating the availability of structurally bound water, possibly in the form of HPO_4^{2-}.

On IR spectra of depositions created in sodium sulfate solution with sodium polyphosphate additives presence, a ledge, and a small absorption band of the symmetrical valence oscillation of POP-bond in 750 cm^{-1} areas have been observed, and the oscillation band of PO_2 groups - 1080 cm^{-1} is clearly expressed as well (Fig. 4(a, b)). The given ledges and bands, along with the band in the region of 2300 $см^{-1}$ (Fig. 4(b)), indicate the existence of diphosphate ion - $H_2P_2O_7^{2-}$, which confirms the assumption related to creation of diphosphate complexes.

The data of gravimetric tests are confirmed by electrochemical studies. Fig. 5 shows changes in the corrosion potential of the working electrode for 1 hour when various inhibitors are added at 100 mg P_2O_5 L^{-1} concentration to the sodium sulfate solution.

As can be seen from Fig. 5, the working electrode's corrosion potential in the sodium sulfate solution without adding the inhibitor during the first 15 minutes sharply shifts towards the negative value, and then gradually stabilizes. When sodium polyphosphate is supplemented to the sulfate solution, the working electrode's corrosion potential also shifts towards negative values, which indicates the formation of films on steel with weak

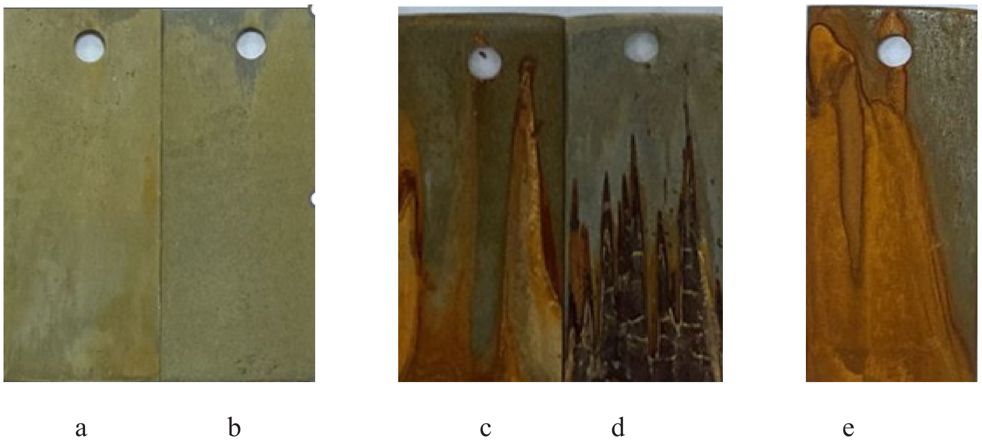


Fig. 1. Steel samples after testing in sodium sulfate solution with additives (Ca, Mn) $(PO_3)_2$: (a) - 50 mg P_2O_5 L^{-1}; (b) - 100 mg P_2O_5 L^{-1}; $NaPO_3$; (c) - 50 mg P_2O_5 L^{-1}; (d) - 100 mg P_2O_5 L^{-1}; (e) - 1000 mg SO_4^{2-} L^{-1}, control.

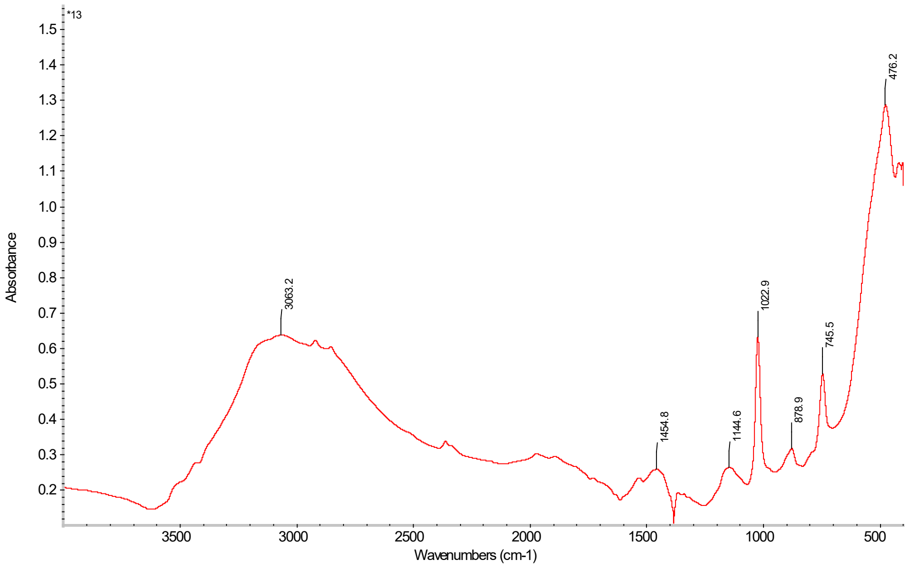
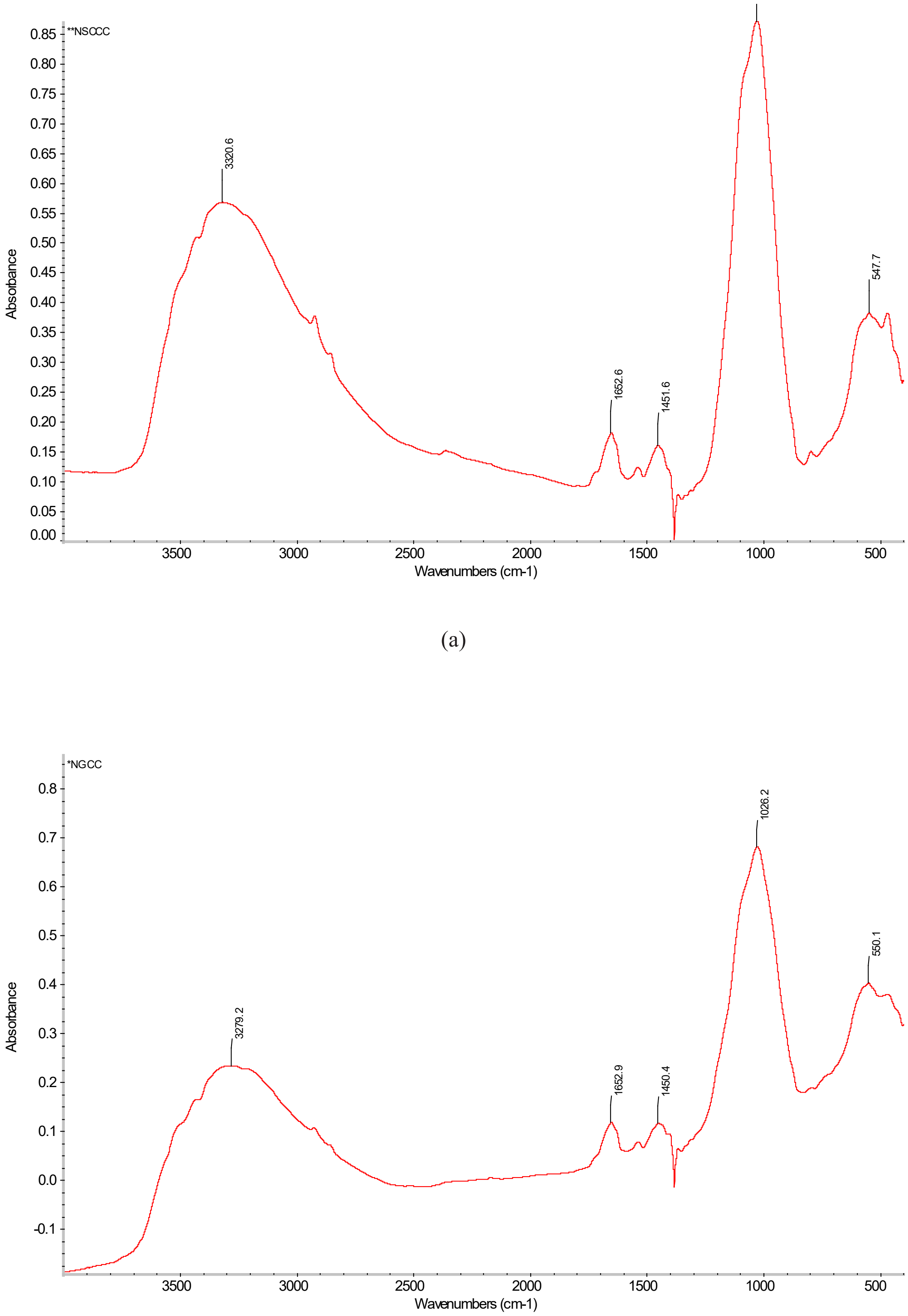


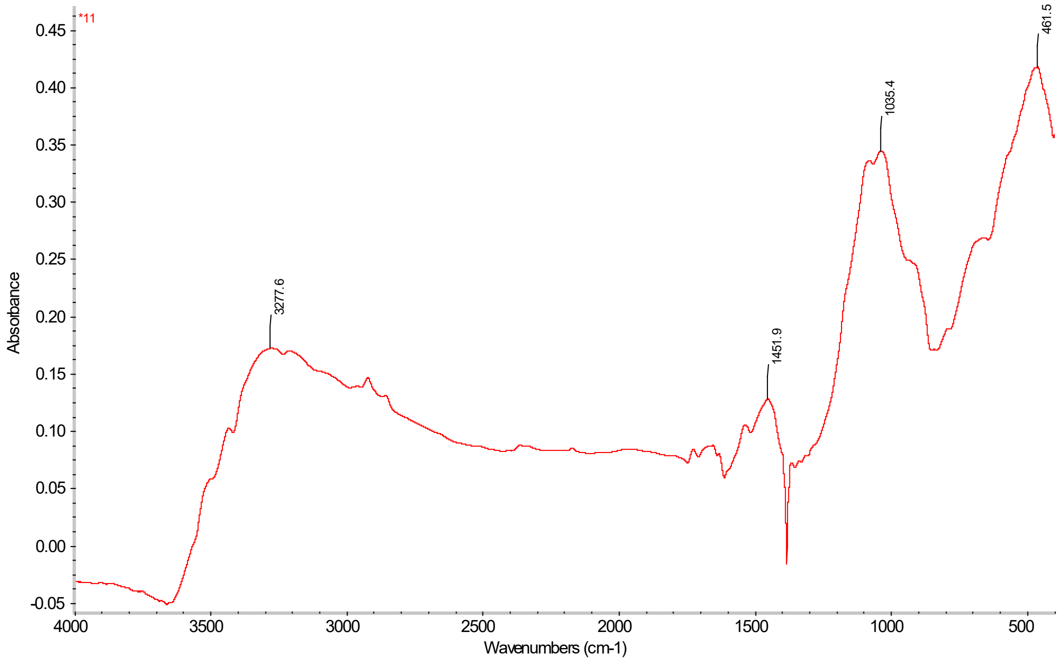
Fig. 2. IRS of corrosion depositions on mild steel in the sodium sulfate solution 1000 mg SO_4^{2-} L^{-1}.



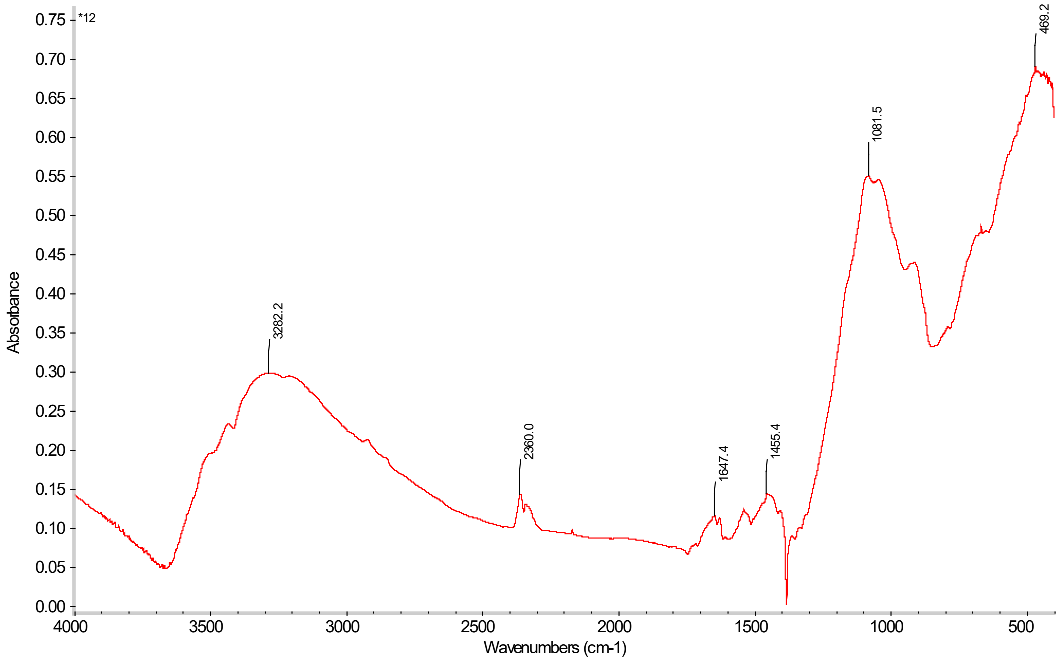
(a)

(b)

Fig. 3. IRS of corrosion depositions on mild steel in the sodium sulfate solution with additives (Ca, Mn) $(PO_3)_2$: (a) - 50 mgP_2O_5 L^{-1}; (b) - 100 mgP_2O_5 L^{-1}.



(a)



(b)

Fig. 4. IRS of corrosion depositions on mild steel in sodium sulfate solution with additives $NaPO_3$: (a) - 50 mgP_2O_5 L^{-1}; (b) - 100 mgP_2O_5 L^{-1}.

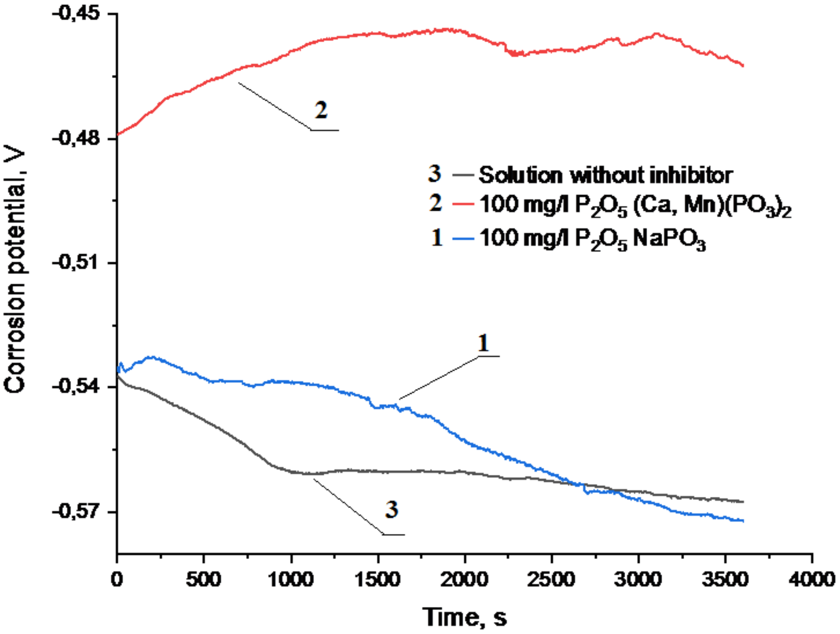


Fig. 5. Dynamics of the change in corrosion potential of the working electrode in sodium sulfate solution in various inhibitors presence.

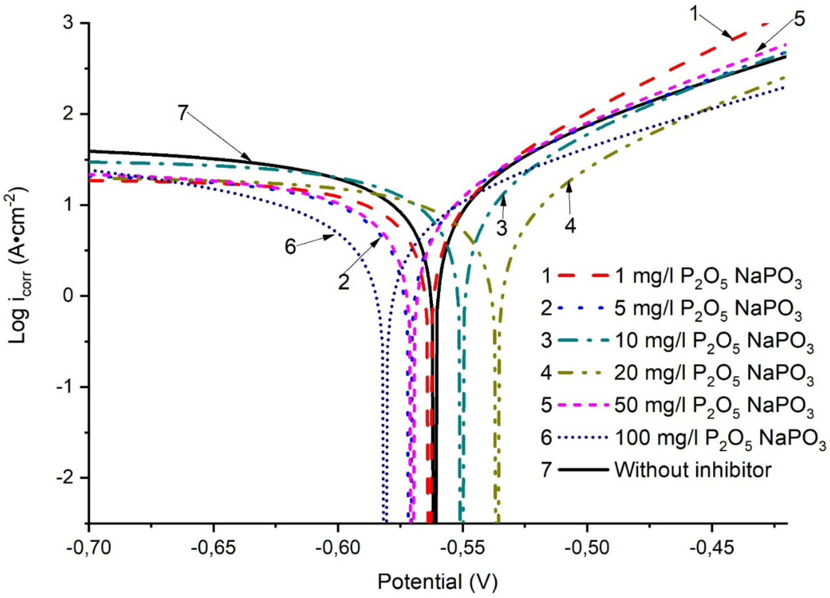


Fig. 6. Potenciodynamic polarization curves taken for mild steel in sodium sulfate solution with sodium polyphosphate additives at various concentrations.

protective properties.

When the calcium - manganese phosphate is added to the sodium sulfate solution, the electrode potential increases towards positive values (Fig. 5). Apparently, this is due to forming the strong layers with high protective properties on the steel surface.

These results are confirmed by the analysis of polarization curves taken in the potency dynamic mode (Figs. 6, 7) for steel samples in sodium sulfate solutions with additives of sodium polyphosphate and calcium-manganese phosphate at various concentrations.

It has been shown that when the concentration of

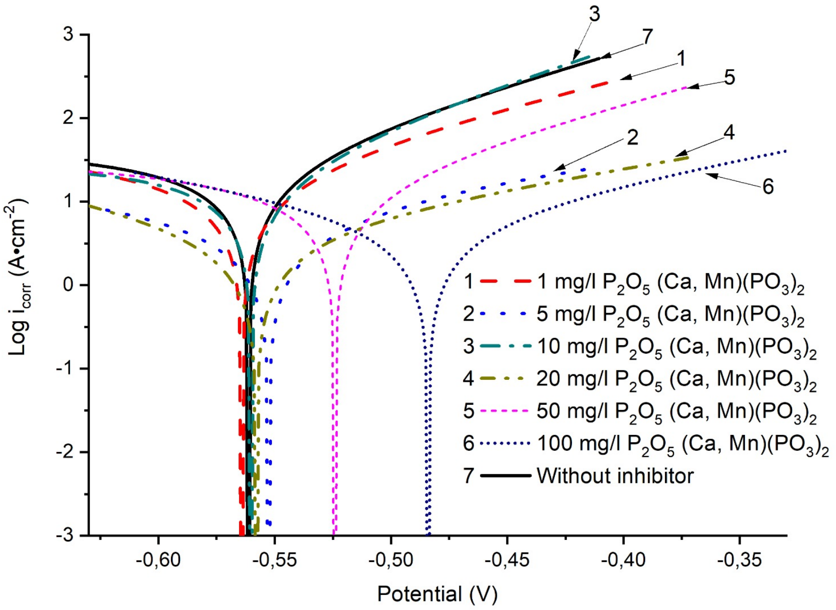


Fig. 7. Potentiodynamic polarization curves taken for mild steel in sodium sulfate solution with calcium-manganese phosphate product additives at various concentrations.

Table 2. Indicators of corrosion resistance of steel when exposed in sodium sulfate solutions with additives of various inhibitors according to the electrochemical tests results.

Corrosive environment	Inhibitor concentration, mg P_2O_5 L^{-1}	Potential, V	Current density, μA cm^{-2}	Corrosion rate, mm/year	Inhibitor efficiency, IE, %
Solution Na_2SO_4 (1000 mgSO_4^{2-}•L^{-1})	0.0	-0.561	34.6	0.403	0.0
Na_2SO_4 + (Mn, Ca) $(PO_3)_2$	1.0	-0.564	23.5	0.274	32.0
	5.0	-0.553	27.5	0.320	20.6
	10.0	-0.550	15.5	0.180	55.3
	20.0	-0.538	19.8	0.231	42.7
	50.0	-0.524	4.5	0.053	86.8
	100.0	-0.484	6.2	0.071	82.4
Na_2SO_4 + $NaPO_3$	1.0	-0.563	18.5	0.216	46.4
	5.0	-0.551	21.0	0.245	39.2
	10.0	-0.548	29.9	0.348	13.6
	20.0	-0.536	19.2	0.224	44.4
	50.0	-0.560	20.2	0.235	41.7
	100.0	-0.581	11.7	0.136	66.2

calcium manganese phosphate increases from 1 mg P_2O_5 L^{-1} to 100 mg P_2O_5 L^{-1}, the corrosion potential shifts towards a positive value from -0.564 V to -0.484 V. The corrosion current density decreases from 23.5 μA cm^{-2} to 6.2 μA cm^{-2}, which corresponds to the inhibitor efficiency of 82.4 % for a concentration of 100 mg P_2O_5 L^{-1} (Fig. 7, Table 2) and confirms the results of gravimetric tests.

The inhibitor efficiency for sodium polyphosphate in sulfate solutions based on electrochemical test data weakly depends on the inhibitor concentration. Corrosion potential shifts towards positive values by only 0.25 V. Minimum corrosion current of 11.7 μA

cm^{-2} is observed at 100 mg P_2O_5 L^{-1} polyphosphate concentration, providing 66.2 % inhibitor efficiency.

The tests conducted showed the possibility of applying the calcium-manganese phosphate product obtained on the basis of waste of manganese ore enrichment from Zhayrem deposit for anticorrosive water treatment. At that, in our opinion, the most efficient use of this product can be predicted to protect the inner surface of reclamation systems of agricultural water supply. Since the given product is in fact a liquid phosphorus fertilizer that comprises the trace element manganese.

CONCLUSIONS

- A protective effect of a calcium-manganese phosphate synthesized based on tailings of enrichment of manganese ore of the Zhayrem deposit on corrosion of mild steel in sodium sulfate solutions was found.
- It was shown that calcium-manganese phosphate in 20 - 100 mg P_2O_5 L^{-1} concentration range lessens the steel's corrosion rate by almost two orders of magnitude as compared with metal's corrosion rate in the sodium sulfate solution (1000 mg SO_4^{2-}) without additives. At the same time, the inhibitor efficiency is 47.3 %; 88.7 % and 96.2 %, and the protection coefficient is 1.90; 8.84 and 24.3 for concentrations 20; 50 and 100 mg P_2O_5 L^{-1} correspondingly.
- For comparison, sodium polyphosphate in concentrations of 1 - 10 mg P_2O_5 L^{-1} reduces the corrosion rate of steel by 1.2 times with an inhibitor efficiency of 16.4 % - 19.5 %. Increasing the concentration of $NaPO_3$ to 100 mg P_2O_5 L^{-1} has little effect on its inhibitory effect. Inhibitor efficiency of sodium polyphosphate for concentrations of 20; 50 and 100 mg P_2O_5 L^{-1} is 21.9 %; 26.4 % and 30.5 %, and the protection coefficient is 1.28; 1.36 and 1.44, respectively.
- Electrochemical studies showed that when the concentration of the calcium-manganese phosphate inhibitor increases from 1 to 100 mg P_2O_5 L^{-1}, the corrosion potential shifts towards a positive value, and the corrosion current density decreases by 5.7 times at an inhibitor concentration of 100 mg P_2O_5 L^{-1}. This indicates a high efficacy of the inhibitory effect of calcium manganese phosphate as a corrosion inhibitor of mild steel in contact with sodium sulfate solutions.

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