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SYNTHESIS OF CORROSION INHIBITORS BASED ON (THIO)UREA AND ORTHOPHOSPHORIC ACID

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Abstract

In this article, the optimal conditions for the synthesis of two types of oligomeric corrosion inhibitors, polymethylene diamidophosphate (PDAF-1) and polymethylene thiodiamidophosphate (PTAF-2), based on compounds such as thiourea, urea, and orthophosphoric acid containing nitrogen and phosphorus, as well as their formulas, are determined. At first, (thio)diamidophosphate - PDAF-1 brand corrosion inhibitor was synthesized using urea and orthophosphoric acid in a 2:1 mol ratio at a temperature of 135-140 ºC. The second type of polymethylene thiodiamidophosphate (PTAF-2) corrosion inhibitor was synthesized based on the process of condensation in an aqueous environment at a temperature of 60 ºC, by adding formaldehyde to this obtained compound in a stoichiometric 1:1 mol ratio. The resulting substance is a white solid, non-volatile, the composition of the main component is 98.7%, and other substances - 1.3%. IR-spectra investigated the structure of these two types of corrosion inhibitors. Also, the inhibition efficiency of these corrosion inhibitors was studied by gravimetric and electrochemical methods in corrosive media such as HCl, H_2SO_4 and NaCl=3%. In addition, the factors affecting the inhibition efficiency, such as the pH of the solution, the duration of time, and the concentration of the inhibitor, were also studied. According to the obtained results, the inhibition efficiency of these corrosion inhibitors was between 95.3 and 97.8%. Also, electron microscopy studied and analysed the protection mechanism of corrosion inhibitors on the steel surface.

Keywords: Corrosion inhibitor, Dithioamidophosphates, Formaldehyde, Orthophosphoric acid, Polymethylene thiodiamidophosphate.

Introduction

Protection of metals against corrosion in various corrosive environments[1–3]. A corrosion inhibitor is a compound that is added in low concentrations to a corrosive solution to reduce or minimize the corrosion rate [4-6]. When it comes to the economic damage of this corrosion process, as an example, we can cite the following figures, for example: according to the results of international research conducted by NACE (IMPACT 2016), the annual economic damage of the corrosion process worldwide is 2.5 trillion US. It is concluded that, this figure in each country section, it is about 3.4% of the average gross domestic product (GDP) of each country[7-9]. The results of many years of scientific research carried out by world scientists show that the environment should be taken into account when choosing corrosion inhibitors, and that the use of compounds containing nitrogen and sulfur and substances based on them is more effective for acidic environments[10]. In addition, such as aldehydes, thioaldehydes, including various alkaloids, such as papaverine,

strychnine, quinine, and nicotine, have been proven to be highly effective corrosion inhibitors and meet the requirements for corrosion inhibitors. Many researchers showed that using corrosion inhibitors based on benzoates, nitrites, and inhibitors based on them, as well as chromates and phosphates, have a high inhibition efficiency for alkaline and acidic solutions[11,12]. An anti-corrosion additive is proposed, which is a mixture of orthophosphoric acid, water and a tertiary amine. It has been shown that the synthesized new anti-corrosion composition based on nitrogen- and phosphorus-containing organic compounds, which provides a high protective effect under conditions of sulfide corrosion of steel, amounting to Z = 53.0-80 at low dosages, 9% [13].

The main goal of this work is to study the optimal conditions for the first time synthesis of oligomeric corrosion inhibitors based on urea, thiourea, formaldehyde and orthophosphoric acid, and also to study their inhibition efficiency by gravimetric and electrochemical methods.

Materials

The experiments were carried out with samples of carbon steel grade St30 and steel samples of this brand were purchased from "Uzbekistan Metallurgical Combinat" JSC. Water (cooling water in the cooling system of "Mubarak gas processing" and "Shurtan gas processing" plants) was used as a corrosion medium (water composition and properties are as follows: total hardness 6.3 mg-eq/l, total alkalinity 2.08 mg-eq/l, 6.3 mg-eq/l, total alkalinity 2.08 mg-eq/l, Ca^{2+}-4.2 mg-eq/l, Mg^{2+}- mg-eq/l, HCO^{-}_{3}-2.00 mg-eq/l, CO_3^2-0.08 mg-eq/l). Other chemical reagents: hydrochloric acid, sulfuric acid, thiourea, orthophosphoric acid, and formaldehyde were purchased "chemically pure" from “Merit Chemicals” company.

Methods

Polarization curves analysis

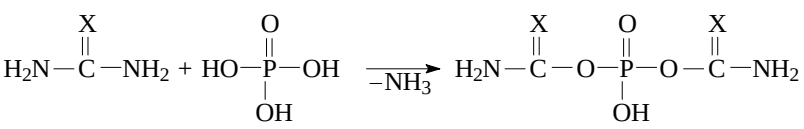
The corrosion-inhibiting properties of an aqueous dispersion of the studied corrosion inhibitor (PDAF-1 and PTAF-2), both with and without additives, were studied by the potentiostatic method on a PI-50-1.1 tool with a PR-8 program, by recording polarization curves on steel electrodes in different corrosion media aqueous, acidic and saline media.

IR analysis

The composition of the inhibitors was investigated using IR-spectra and elemental analysis with Shimadzu IR Tracer-100.

Preparation of polymethylenedi(thio)amidophosphates (PDAF-1 and PTAF-2).

The influence of reaction parameters on the condensation of the interaction of urea and thiourea with orthophosphoric acid and the course of their oligomerization in the mass were studied. It is known that the interaction of urea compounds in a mass depends on their structure, concentration and other factors, consideration of concentration effects in reactions and the influence of reagents on their reactivity. Synthesizing nitrogen- and phosphorus-containing oligomeric corrosion inhibitors without water environment, a reaction first occurs with the formation of (thio)diamidophosphate according to the following scheme.1:

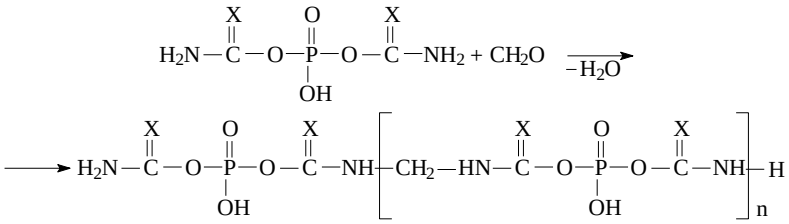


Scheme 1. Synthesis of (thio)diamidophosphate- PDAF-1

Where X–O and S

The ratio of orthophosphoric acid and urea is 1:2. Condensation is carried out in a urea melt at a temperature of 408-413 K.

Next, the calculated amount of formaldehyde was added to the resulting product, and the process of condensation of formaldehyde with di(thio)amidophosphates was carried out at a temperature of 333 K in an aqueous environment. The reaction between formaldehyde and di(thio)amidophosphates can be represented according to the following Scheme 2.



Scheme 2. Synthesis of polymethylenedi(thio)amidophosphates PTAF-2.

Where X – O and S

Corrosion inhibitor obtained on the basis of formaldehyde and diamidophosphates has the following physicochemical properties: white color, solid aggregate, non-volatile, purity 98.7%, and other impurities - 1.3%. However, studies have shown that the temperature regime of synthesis significantly affects their ratio in the final product. In addition to the constant ratio of initial reagents to this synthesis process, the effect of temperature on the process was also studied, where the increase of the reaction temperature from 60 ºC to 100 ºC leads to an increase in the percentage of polymer from 11 to 33%. Also, the continued increase in temperature causes the polymer to achieve homogeneity. In addition, the viscosity of polyamide phosphate increased from 0.06 to 0.13 dl/g[14]. It follows that in order to obtain a high molecular weight polyamide phosphate, the starting reactants should be taken in an equimolar ratio, as shown in Table 1.

Table 1. The effect of the ratio of initial reagents on the composition of the resulting product. (T=100 ^{0}C, τ= six hours).

Ratio Form.+Aduct. urine	Output, %	$\eta_{пр}$ 0.5 aq. solution. dl/g	Elemental analysis			
			nitrogen		phosphorus	
			Computed	Found	Computed	Found
1:3	67,8	0,075		18,1		18,8
1:2	74,5	0,07		17,9		18,3
1:1	88,3	0,06	18,4	17,4	19,1	19,2
2:1	80,6	0,05		16,9		18,5
3:1	76,4	0,04		17,2		18,4

Among different mole ratios, the highest yield was observed when the mole ratio of the starting materials was 1:1 in Fig 1. The kinetics of formaldehyde consumption and inhibitor formation compared to the initial substances was studied and the decrease of the slope was observed in Fig 2.

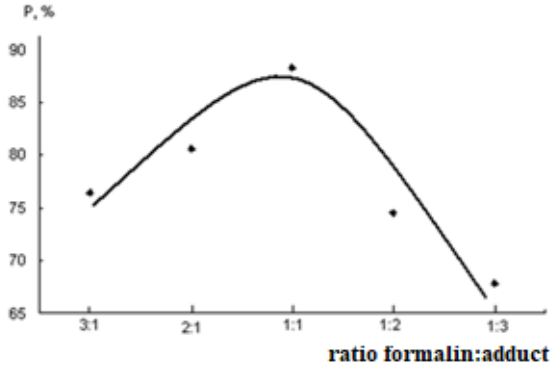
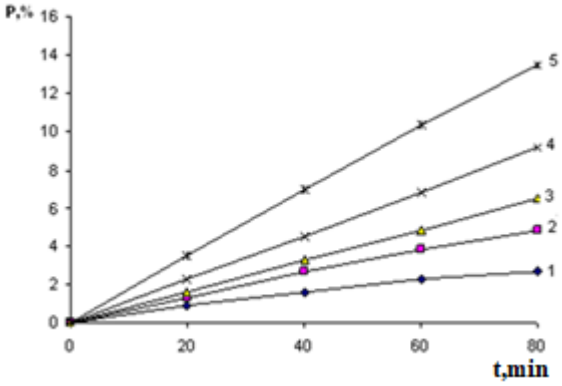


Figure 1. Dependence of polymer yield on the ratio of starting substances. T=373 K, time 6 hours.

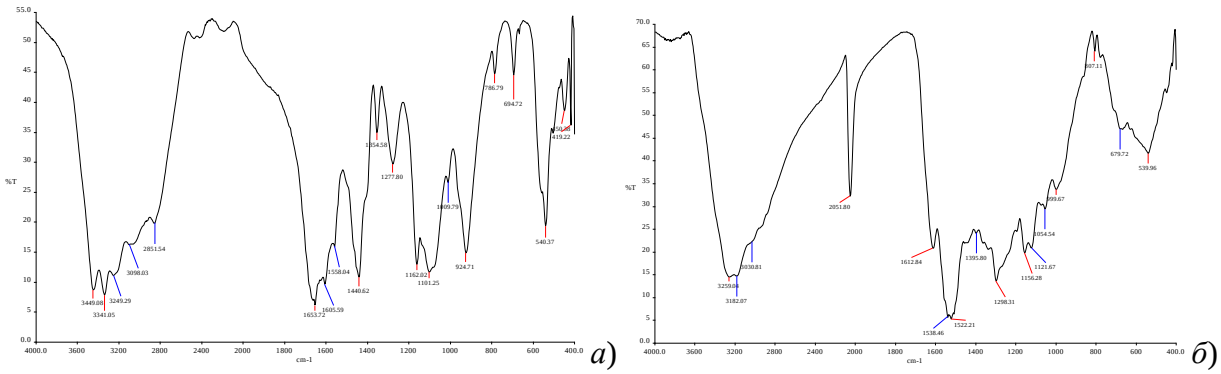


1—1:3; 2—1:2; 3—3:1; 4—2:1; 5—1:1

Figure 2. Kinetic dependence of polycondensation in the Form system: Adduct.urine. in an aquatic environment. (T=373K).

IR **spectrum** analysis

The structure of the synthesized corrosion inhibitors was studied and analyzed by IR spectrum analysis of its components and results are presented in Fig 3.



а) *б)*

Figure 3. IR spectrum of (a) polymethylene diamidophosphate and (b) polymethylene thiodiamidophosphate

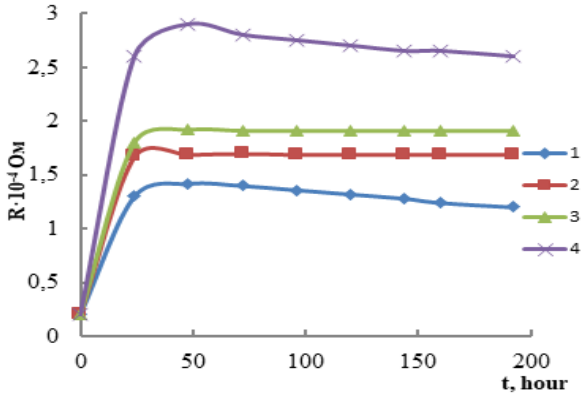
The IR spectra from Figs 3 a & 3b of the starting reagents and the resulting compound contain bands in the region of 3449, 3341 and 3182 cm^{-1}, corresponding to free hydroxyl groups. The structure of primary and secondary amides and thioamide compounds is characterized by the occurrence of valence vibrations in the IR spectrum regions at 1395, 1522, 1605 and 3449, 3259, 3249, 3098 cm^{-1}, respectively. In this case, the valence bands of C=S and C=O groups appear in the region of 1612, 1653 and 1680 cm^{-1}, and in the regions of 2851 and 1440 cm^{-1} . We observed the allowed resonances of CH- and CH_2- groups. Unbonded and bonded P=O phosphorus-oxygen bonds were observed to appear in the regions of 1277, 1298, and 924 cm^{-1}.

Results and Discussion

Studying physicochemical and inhibitory properties of synthesized oligomeric corrosion inhibitors

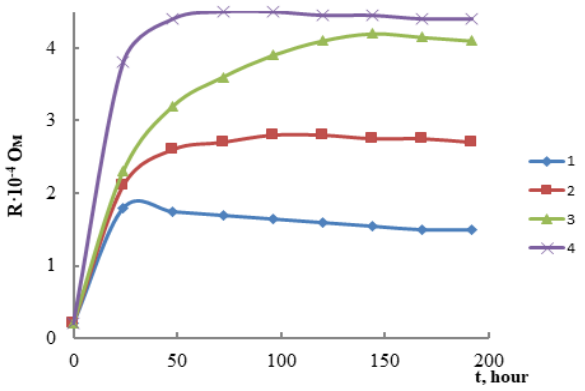
The corrosion-inhibiting properties of an aqueous dispersion of the corrosion inhibitor under study

(PTAF-2), both with and without additives, were studied by the potentiostatic method on a PI-50-1.1 device with a PR-8 program, by recording polarization curves on steel electrodes in aqueous water, acidic and saline environments. The initial aqueous dispersion of the test oligomer was purified by dialysis[15,16]. One of the accurate and at the same time fast methods for studying the anti-corrosion properties of inhibitors is the experimental study of polarization curves and polarization resistance of a steel electrode or probe in aqueous, saline and acidic environments. Figs 4-6 show the results of measuring the polarization resistance of a steel probe in various environments, as well as in the presence of inhibitors.



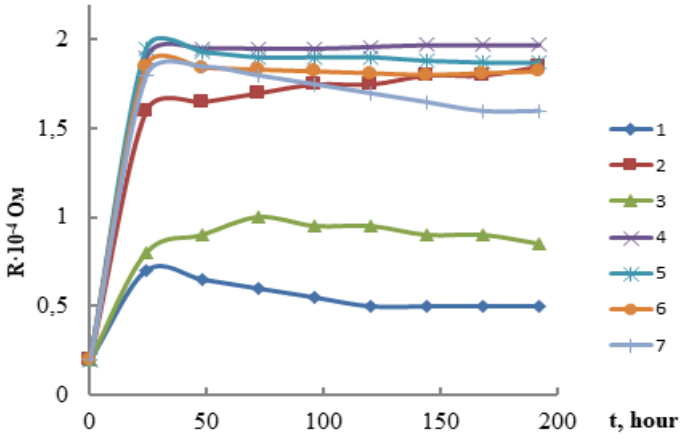
1– H_2O; 2–C=0,001%; 3– C=0,01%; 4–C=0,1%

Figure 4. Change in the polarization resistance of a steel electrode in an aqueous environment in the presence of an inhibitor PTAF-2



l. NaCl-C=3%; 2-C=0,001%; 3-C=0,01%; 4-C=0,1%

Figure 5. Change in polarization resistance in a salt environment in the presence of the PTAF-2 inhibitor



1-HCl, C=5%; 2- H_3PO_4 (extraction) and PTAF-2 C = 0.001%; 3- H_3PO_4 (term), PTAF-2 C=0.001%; 4- HCl, C=5% with PTAF-2 C=0.001%; 5- HC1, C = 5% with PTAF-2 C = 0.001%; 6- HC1, C=5% with PTAF-2 C=0.1%; 7- HC1, C=5% with PTAF-2 C=1%.

Figure 6. Change in polarization resistance in an acidic environment in the presence of the PTAF-2 inhibitor.

The results of calculating the braking coefficient (g) and protection level (Z) values are presented

in Tables 2-3, from which it can be seen that the most significant results were obtained in the presence of 0.1 and 0.01% solutions of the corrosion inhibitor obtained by ammonium dihydrogen phosphate with formaldehyde (IR- 5).

The work uses the method of alternative research of graphical processing of corrosion curves. For these purposes, polyamides, phenol-formaldehyde resins, and other polymers are quite acceptable. The traditional way of graphically processing polymerization curves to determine the corrosion rate is to extrapolate in semilogarithmic coordinates the rectilinear sections of the cathode and anodic branches until their mutual intersection. However, despite the obvious simplicity of the method, its practical use is often associated with a number of complications. The construction of tangents itself is quite subjective, and for a more reliable extrapolation an additional accurate determination of the corrosion potential is necessary.

The inhibitory properties of the composition of the oligomeric corrosion inhibitor (PTAF-2) synthesized by us are presented in Table 2.

Table 2. Protective properties of the composition (PTAF-2) in relation to steel in an aqueous environment.

With inhibitor mg/l	Protective effect, %, at temperature K		
	293	313	333
100	94,6	91,3	89,2
200	95,1	93,0	90,4
300	96,6	94,1	92,0
400	97,8	96,4	95,3

The research results indicated that at a composition concentration (PTAF-2) of 100 mg and a temperature of 293-333 K; the protective effect is 89.2-94.6%, depending on the composition ratio. With an increase in the inhibitor concentration (PTAF-2) from 100 to 400 mghl-1, the protective effect against corrosion increases and reaches 95.3-97.8% at 293-333 K. An important property of inhibitors is the preservation of the protective effect at elevated temperatures. From the data in Table 2, it can be seen that the protective effect decreases with increasing temperature (from 293 to 333 K), but still remains significant. The results obtained confirm the possibility of using the proposed composition (PTAF-2) as a metal corrosion inhibitor in relation to aqueous and saline environments. Table 3,2 shows data on the effectiveness of the corrosion inhibitor in aqueous and saline environments. To identify the process of concentration-dependent corrosion inhibition, steel plates were immersed in a potential measuring cell containing aqueous solutions of the test oligomer. Experimental data on studying the corrosion rate of steel plates in aqueous dispersions (PTAF-2) both with and without additives showed that when the concentration changes, the stationary potential of the electrode shifts to the positive region, due to the formation of a barrier type of corrosion protection. This effect increases significantly with increasing concentration of the oligomeric inhibitor in the aqueous dispersion[17].

Polarization measurements give reason to believe that the aqueous dispersion of the oligomer under study, as a surfactant, is initially adsorbed on the surface of the steel electrode with the subsequent formation of a film that prevents further corrosive destruction of the metal.

As a result, both the hydrogen ion discharge reactions (increased hydrogen evolution overvoltage) and the ionization reactions (anodic dissolution) of iron slow down. In this case, it is necessary to assume that the dipoles of the surfactant are located with negative ends towards the solution, which contributes to a shift of the potential of zero charge and, consequently, the stationary potential in the positive direction. The shift of the points of zero charge in the positive direction is accompanied by a simultaneous increase in the overvoltage of hydrogen evolution and a slowdown in the corrosion destruction reaction. Along with this, it should be noted that the nature of the adsorption of the inhibitor on the surface of the steel electrode, the effectiveness of its action, as well as whether the inhibitors belong to the cathodic and anodic types depend not only on its nature, but also to a large extent on the potential of the medium. Judging by the steady-state potential, with the addition of an oligomeric inhibitor based on (PTAF-2), the degree of protection increases significantly. Moreover, more effective protection against corrosion occurs in the presence of a 0.1% solution of PTAF-2, the protective coefficient of which passes through a maximum[18].

Table 3. Protective properties of corrosion inhibitor oligomer in aqueous and salt media at 293 K

Sample name	C, conc.%	Environment	Braking coefficient,γ	Degree of protection, Z
PTAF-2	0,1	Water	26,7	98,2
PTAF-2	0,01	Water	19,6	94,6
PTAF-2	0,001	Water	14,3	91,5
PTAF-2	0,1	NaCl=3%	19,5	96,7
PTAF-2	0,01	NaCl=3%	15,8	93,3
PTAF-2	0,001	NaCl=3%	10,4	89,9

The mechanism of action of this oligomeric corrosion inhibitor is determined mainly by the transition of the surface-protected metal to a stable surface film state with the participation of particles of fine additives. However, the action of inhibitors in this case is more complex than in film formation, and is also associated with the nature of adsorption of surfactant ions. For example, with a positive surface charge of the electron relative to the solution, an oligomeric inhibitor, which is anions, will be adsorbed on it, while with a negative surface charge, the inhibitor, which is undissociated molecules[19].

CONLUSION

In present research, synthesis of the new oligomer-type of corrosion inhibitor containing nitrogen and phosphorus based on (thio)urea and orthophosphoric acid, and formaldehyde also its various properties were studied. The new oligomer-type of corrosion inhibitors were synthesized and its structure was confirmed by the IR spectroscopy. According to the obtained results, the valence vibration of amides and thioamides appeared at 1395, 1522, 1605 and 3449, 3259 3249, 3098 cm^{-1}, the appearance of bands in the regions of 1612, 1653, and bands in the region of 1680 cm^{-1} indicates related groups C=S and C=O, and in the regions of 2851 and 1440 cm^{-1}, observed the CH– and CH_2– groups. Free and bound P=O groups appear in the valence vibration of 1277, 1298, and 924 cm^{-1}. The surface morphology of this modification was studied by the electron microscopy. In addition, optimum conditions for the synthesis of corrosion inhibitors, molar ratios of starting materials: (thio)urea and orthophosphoric acid 1:2, formaldehyde and di(thio)amidophosphates in

a 1:1 molar ratio, and reaction temperatures of 408-413 K and 333 K were determined. The structure of both synthesized corrosion inhibitors was determined using IR spectra.

Acknowledgment

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Authors' Declaration

- Conflicts of Interest: None.
- We hereby confirm that all the figures and tables in the manuscript are ours.
- Ethical Clearance: The project was approved by the local ethical committee in University of Baghdad.

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