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The influence of some trivalent metal cations on the structure and corrosion properties of phosphates

Abstract. This study presents an investigation into the inhibitory properties of phosphate-based systems used for the protection of metals against corrosion. Various types of phosphates are examined, including ortho-, di-, and cyclophosphates, as well as their interactions with metals in aggressive environments. Special attention is given to the influence of iron modifier ions on the efficiency of corrosion inhibition. The potential of modifying phosphate systems to enhance protective properties is analyzed.

The aim of this research is to study and apply complex inhibitors that provide a high level of protection for steel structures against corrosion. The material used for the investigation is carbon structural steel of ordinary quality, compliant with the European standard S235JR (EN 10025-2). Various inorganic phosphate formulations modified with trivalent iron, aluminum, and lanthanum ions were selected as inhibitors.

Shifts in spectral bands were observed, corresponding to the deformation vibrations of hydroxyl groups and Al-O bonds. These shifts suggest interactions between the ionic dopants and the phosphate matrix. Specifically, bands shifted from 735 to 671 cm^{-1}. Similarly, the band associated with Al-O interactions changed from 530 to 555 cm^{-1}, while the HPO_4^- band shifted from 940 to 979 cm^{-1} in the presence of Al^{3+} ions. Deviations in the vibrational frequencies of Al-O and HPO_4^- indicate changes in chemical bonding and increased reactivity of the inhibitors.

Scanning electron microscopy (SEM) provided high-resolution images and elemental composition of corrosion deposits on the steel surface. The method revealed the distribution of elements: oxygen (36.17%), iron (26.79%), phosphorus (4.72%), as well as minor amounts of sodium (0.78%) and aluminum (1.39%) in corrosion products, and allowed localization of protective layers.

The results of this study demonstrate that modifying phosphates with trivalent iron, aluminum, and lanthanum ions leads to significant changes in their inhibitory properties.

Keywords: corrosion, inhibitor, orthophosphates, degree of protection, depth index.

Introduction

The wide variety of available corrosion inhibitors today does not diminish the relevance of developing new formulations with comprehensive protective properties [1], due to the substantial economic losses caused by corrosion processes and the necessity for effective and cost-efficient solutions to protect industrial equipment [2]. Modification of phosphates using cations from the p- and d-block elements represents a promising approach to creating materials with improved inhibitory properties compared to existing analogs [3]. The incorporation of these ions alters the structure and properties of the phosphates, which in turn affects their protective characteristics. This effect is further influenced by factors such as the pH of the medium, as well as the nature and concentration of the phosphate, helping to determine the conditions under which inhibitors are most effective.

Corrosion of metals is a pressing problem in chemical science. Therefore, it is necessary to use inhibitors to protect metals from corrosion. Phosphate-based systems are used as inhibitors. Phosphates are compounds containing phosphorus. However, phosphates have their own peculiarities in their use as inhibitors.

Phosphate systems can be modified to protect against corrosion. Further research is required to optimize the composition and structure of modified phosphate systems and to study their interaction mechanisms with metal surfaces at the molecular level [4].

Modifying the phosphate matrix with p- and d-block metal cations offers a targeted approach to altering their structure and properties. This is primarily due to differences in the size, charge, and chemical reactivity of these ions [5]. The incorporation of metal ions into the phosphate structure leads to variations in physicochemical characteristics, opening up possibilities for the design of functional materials with tailored properties [6].

The relevance of research in this field stems from the urgent need to address corrosion – a significant economic problem for industry, particularly in the metallurgical sector. The search for effective methods to protect equipment from corrosion damage, especially the development of new corrosion inhibitors, remains a priority research area [7-10].

Despite the wide variety of available corrosion inhibitors, optimizing corrosion protection technologies continues to be a topical issue. This is due to the diversity of service environments and operational conditions throughout the equipment life cycle [11-12]. The development of inhibitors with multifunctional action and resistance to changing environmental factors is therefore a key objective.

Current trends in corrosion protection focus on the following key aspects:

- Conducting in-depth analyses of the corrosion behavior of structural materials in various environments (atmosphere, soils, aqueous media) to develop predictive models and methods for forecasting corrosion resistance.
- Developing effective monitoring and diagnostic systems to assess the condition of corrosion protection in metallic structures under real-world operational conditions.
- Carrying out fundamental and applied research in the areas of electrodeposition and chemical-catalytic deposition of metallic and alloy coatings, including the development of nanostructured coatings using electrochemical techniques.
- Study of passivation and behavior of metallic materials in aqueous solutions of containing corrosion inhibitors. Study of adsorption mechanisms of inhibitors on material surfaces using analytical methods.

Particular attention is currently being paid to the development of environmentally friendly corrosion inhibitors [13, 14], including compounds and complexes derived from catalytic production waste and spent catalysts. Promising examples of such inhibitors include organopolymolybdates, aromatic and aliphatic amines, hydrazides of organic acids, and triazoles containing Zn, Ni, Al, Co ions and their respective salts.

The inhibition mechanism occurs as follows: due to chemisorption, there is an interaction between the d-electrons of iron atoms. Industrial approaches are important in solving corrosion-related problems.

It is necessary to develop new inhibitor compositions with combining chemical and physical methods [15].

Materials and methods

This study is devoted to investigating the efficiency of complex corrosion inhibitors based on sodium phosphates in aqueous environments. Reagents of analytical grade purity were used for the preparation of phosphate inhibitor solutions. The phosphate compound solutions were prepared using distilled water, with precise weighing of the required amount of each substance to achieve the desired concentration.

The primary components of the inhibitor system included solutions with a concentration of 0.1 mol/L: sodium dihydrogen phosphate (NaH_2PO_4), disodium hydrogen phosphate (Na_2HPO_4), trisodium phosphate (Na_3PO_4), disodium dihydrogen pyrophosphate ($Na_2H_2P_2O_7$), tetrasodium pyrophosphate ($Na_4P_2O_7$), sodium trimetaphosphate ($Na_3P_3O_9$), and sodium hexametaphosphate ($Na_6P_6O_{18}$).

To modify the inhibitor properties and enhance their protective efficiency, trivalent metal ions Al^{3+}, Fe^{3+}, and La^{3+} were used. Solutions of these trivalent ions were prepared by dissolving the respective metal salts. The concentration of each solution was 0.1 mol/L. All solutions were prepared using pure distilled water and filtered prior to use to eliminate possible impurities.

The selection of Al^{3+}, Fe^{3+}, and La^{3+} as modifying ions was based on their structural characteristics and high complexation ability. Trivalent cations exhibit high reactivity, which facilitates their incorporation into the crystalline structure of phosphate compounds. The introduction of a trivalent ion provides charge compensation for the phosphate anion (P^{5+}), stabilizes the phosphate matrix structure, and consequently enhances the protective properties of the resulting inhibitor coating.

Corrosion tests were conducted in accordance with the procedures specified by state standards [16], using steel samples of the following chemical composition (wt.%): Fe – 98.36; C – 0.2; Mn – 0.5; Si – 0.15; P – 0.04; S – 0.05; Cr – 0.3; Ni – 0.2; Cu – 0.2 [12]. This steel grade was selected due to its widespread use in corrosion studies and its relatively simple chemical structure, which facilitates the analysis of its behavior in aggressive environments.

Work method: First, the steel samples were ground and polished to a smooth surface. The grinding was done using abrasive discs with grit sizes of 220, 500 and 1200. Then, they were polished with a polishing paste. After polishing, the samples were degreased using organic solvents (acetone or ethanol). All traces of oil and contaminants were removed. Thus, they were cleaned of impurities. The experiments were conducted at a constant temperature of 25±2°C. The volume of the solution in each test tube or container was 500 ml. All solutions were mixed using magnetic stirrers. The duration of the experiments ranged from 24 to 480 hours.

To evaluate the corrosion behavior of the material, the following methods were applied: gravimetric analysis to determine the corrosion rate based on the mass loss of the specimens; photocolorimetric analysis to determine the concentration of iron ions (Fe^{3+}) in the corrosive medium using the reaction with potassium thiocyanate [17, 18] potentiometric analysis for measuring the pH of aggressive environments using a combined glass electrode and ionomer.

Infrared (IR) spectroscopy for the identification of functional groups and compounds present in the corrosion products were employed in order to analyze the composition and structure of corrosion deposits. The deposit samples were dried and placed into a spectrometer, where the absorption spectrum was measured in the range of 4000–400 cm^{-1}; scanning electron microscopy (SEM) was used to visualize the morphology and microstructure of the corrosion deposits. The samples were examined at high resolution, which allowed the identification of specific features in the shape and distribution of corrosion products on the specimen surfaces.

Quantitative characteristics of corrosion processes were calculated using standard formulas. The uncertainty of measurements was assessed using Student's *t*-test at a confidence level of 0.95 [19].

The phosphorus(V) oxide (P_2O_5) content was determined by a photocolorimetric method based on the formation of a colored phosphovanadomolybdate complex. The optical density of the complex solution was measured using a photocolorimeter. This method allows for the determination of P_2O_5 content in complex matrices. The relative error of the determination was ±1%, and the color stability of the complex was maintained for several hours. The analysis was performed in accordance with standard methodology [20].

Thus, this study is based on the use of modern analytical methods to obtain reliable data on the corrosion behavior of materials in aggressive media and the effectiveness of phosphate inhibitors, enabling precise and objective evaluation of their protective performance.

Results and discussion

Studies of the inhibitory ability of multicomponent orthophosphate systems

The study of the inhibitive efficiency of multicomponent phosphate systems involved the creation of 27 different systems containing phosphate compounds. During the experimental work, potentiometric and photocolorimetric analyses were carried out, enabling the assessment of the concentrations of modifying ions, iron, and phosphate ions in the solution, as well as their influence on corrosion processes. The concentration of modifier ions, iron and phosphate ions in the solution, as well as their effect on corrosion processes.

Figure 1 illustrates the dependence of the modifier ion concentration on the content of aluminum, iron, and phosphates when disodium hydrogen phosphate was replaced. In the initial stages of the experiment, a gradual transition of iron from the metal plate into the solution occurred in the form of ions. The amount of iron in the aqueous medium decreases. The concentrations of both modifier ions and phosphate ions decrease [21].

Figure 2 illustrates the dependence of the corrosion rate on the nature of the modifying ions at various component ratios in the system containing disodium hydrogen phosphate. The lowest corrosion rate was observed in the composition with disodium hydrogen phosphate at a 3:1 ratio in the presence of aluminum ions. The protection efficiency reached 79.14%. The inhibitive effect is attributed to the formation of complex aluminum hydroxyphosphate compounds, which create a protective film on the metal surface [22].

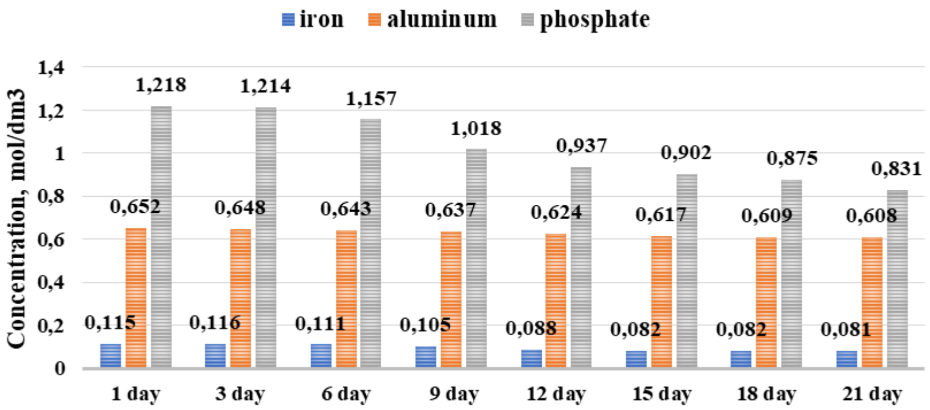

Figure 1 – Dynamics of the concentration of lanthanum, iron, and phosphate ions in the sodium hydrophosphate system

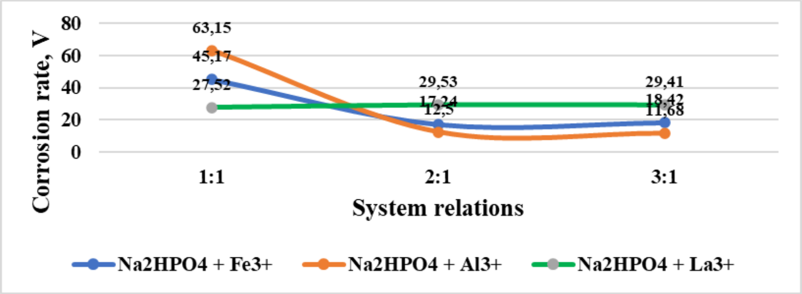

Figure 2 – The effect of the nature of modifier ions on the corrosion rate in the sodium hydrophosphate system of different ratios

Systems containing aluminum exhibited a clear trend: the higher the phosphate content, the lower the corrosion rate. This correlates with the formation of hydroxyphosphate compounds in an alkaline environment. These compounds provide a high degree of corrosion protection, as confirmed by both thermodynamic and kinetic data. The stability of hydroxyphosphate compounds is greatest at pH 7.0, which corresponds with our experimental observations. These compounds possess high reactivity and show little tendency to form undesirable by-products.

Monosodium phosphate, due to its amphoteric properties and its ability to effectively accept and donate protons, becomes more negatively charged. This contributes to a more pronounced shift of pH toward the alkaline range as protons participate in hydrolysis reactions, thereby enhancing its inhibitive effect.

Thus, the findings confirm that hydroxyphosphate compounds formed with aluminum-modifying ions provide the highest level of protection among all orthophosphate-based systems. These results are consistent with the literature and confirm the reliability and effectiveness of these compounds in oxidation and corrosion prevention.

For iron-containing systems, the highest corrosion rate was observed at a 1:1 ratio. At other ratios, the corrosion rate remained approximately the same. In systems with lanthanum ions, the corrosion rate varied depending on concentration. The study revealed that the inhibitive performance of phosphate compounds is also strongly dependent on the pH of the corrosive environment: with increasing pH, the anticorrosive activity of phosphates toward iron and its alloys increases [23].

It is also noteworthy that a comprehensive quantitative assessment of the corrosion process parameters was carried out, including kinetic, chemical, physicochemical, thermodynamic characteristics, and anticorrosion efficiency.

To study the composition of the corrosion deposits, analysis was performed using an IR spectrometer (Figure 3). For this analysis, only those corrosion deposits exhibiting the highest protective efficiency were selected.

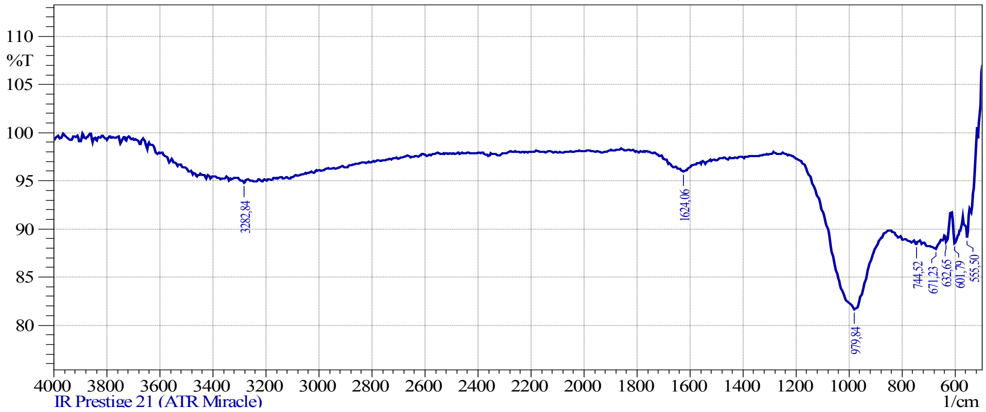

Figure 3 – IR spectrum of corrosion products formed in the system of sodium hydrophosphate with aluminum (ratio 3:1)

Researchers [24] have shown that disodium hydrogen phosphate is characterized by absorption bands in the 1235-1215 cm⁻¹ range, which are attributed to the asymmetric stretching vibrations of phosphorus–oxygen bonds ν(P=O). In the same spectral region, a band corresponding to the symmetric stretching vibrations of the phosphorus–oxygen bonds ν(P=O) is observed, with a maximum absorption at 1130 cm⁻¹.

The bands in the range of 955-940 cm⁻¹ indicate the presence of phosphate residues. These are phosphorus-oxygen-phosphorus bonds ν(P–O–P). In addition, the symmetric vibrations of the phosphate residues are visible at 1130 cm⁻¹.

Analysis of the IR spectra showed that the corrosion spectrum was significantly different from that of sodium hydrogen phosphate. This suggests that new chemical compounds may be formed during the corrosion process.

According to the results, it can be seen that the corrosion process differs depending on the composition of the initial disodium hydrogen phosphate. This can be explained by the complexity of the chemical interactions, and further studies are needed to accurately determine the composition.

The analysis of the corrosion deposits was also conducted using a JOOL JSM-6490 LV low-vacuum scanning electron microscope (manufactured in Japan). The results of this analysis are presented in Figure 4.

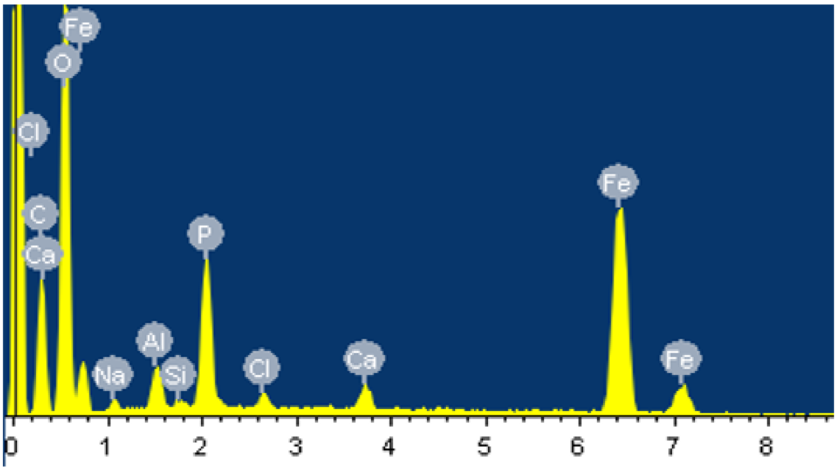

Figure 4 – Elemental composition of corrosion products formed in the system o f sodium hydrophosphate with aluminum (ratio 3:1)

The primary elemental composition of the corrosion deposits formed in the sodium hydrogen phosphate–aluminum system at a 3:1 ratio (Figure 4) is as follows: oxygen (O): 36.17%, iron (Fe): 26.79%, phosphorus (P): 4.72%, sodium (Na): 0.78%, and aluminum (Al): 1.39%. The composition of the corrosion deposits indicates the formation of iron oxides and hydroxides, as well as phosphate compounds, which confirms the effectiveness of this system in corrosion protection.

From these results, it can be seen that phosphate-based inhibitors are more effective.

The Study of the inhibitory ability of diphosphate systems

A multicomponent system study was conducted for 18 systems. Figure 5 presents their results.

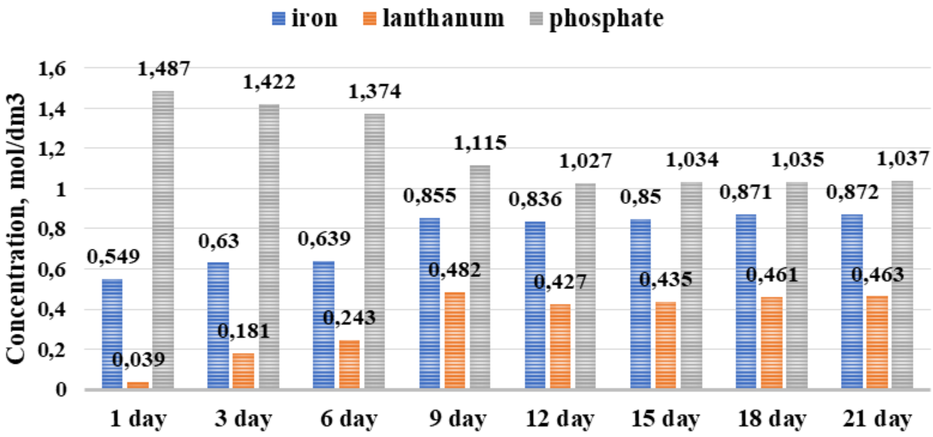

Figure 5 – Dependence of the change in the concentration of the lanthanum, iron, and phosphate modifier ion on time for disodium dihydrogen diphosphate

Nevertheless, the inhibition efficiency of the system reached 8.43%, the highest value among the 18 diphosphate systems studied.

The variation in corrosion rate depending on the component ratios in the disodium dihydrogen diphosphate system with modifier ions is presented in Figure 6. The effect of iron ions on the corrosion rate revealed a complex dependence on phosphate concentration.

A decrease in the corrosion rate is observed with an increase in phosphate concentration at a phosphate ratio of 2:1. This is due to the formation of protective phosphate layers. However, the opposite effect was observed at a ratio of 3:1. This is due to a change in the composition and properties of the resulting compounds.

For the system containing aluminum and lanthanum ions, an increase in corrosion rate was observed with rising phosphate concentrations. This may indicate the formation of soluble complexes or a disruption of the passivating effect of oxide films.

According to the literature [25], a distinctive feature of diphosphate ions in aqueous solutions is their lower stability compared to orthophosphates, which may explain their less pronounced protective effect.

Studies indicate that diphosphates tend to form soluble complex compounds such as Me $[Me_2(P_2O_7)_2]$, which remain stable in mildly acidic environments with pH values ranging from 3 to 5, thereby influencing the corrosion equilibrium. Overall, the presence of diphosphates promotes the formation of stable compounds, which contributes to a decrease in the corrosion rate.

Figure 7 shows a spectrogram of corrosion deposits revealing the presence of bands corresponding to different vibrational modes of the phosphate groups. This is a disodium dihydrogen diphosphate system with a lanthanum ion modifier in a 1:1 ratio.

According to the literature, bands in the region of 1270 cm^{-1} correspond to asymmetric vibrations ν(P=O), while bands in the region of 1092–990 cm^{-1} correspond to symmetric vibrations ν(P=O). Peaks at 884 cm^{-1} and 792–595 cm^{-1} are indicative of asymmetric and symmetric ν(P–O–P) vibrations, respectively. The presence of peaks in the ranges of 1030–980 cm^{-1} and 1620 cm^{-1} on the spectrogram of the corrosion precipitate confirms the formation of unstable compounds.

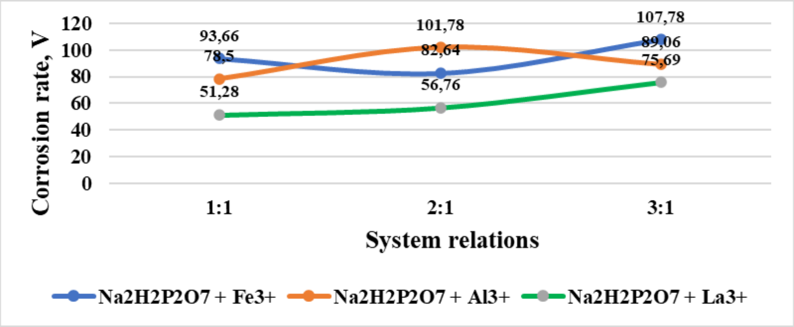

Figure 6 – Dependence of disodium dihydrogen phosphate in different ratios on the nature of the modifier ion

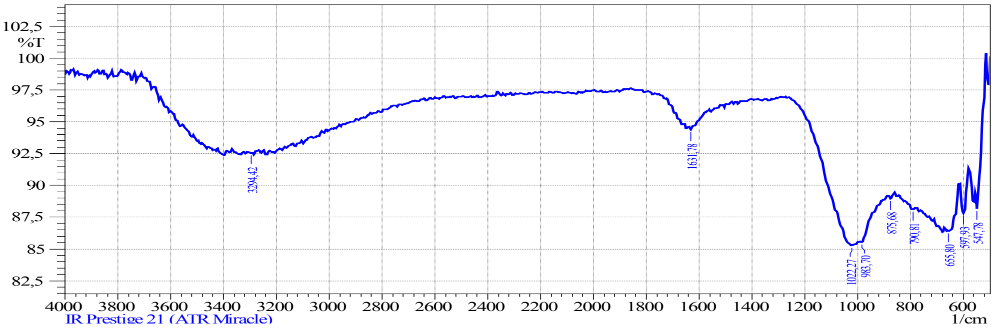

Figure 7 – Spectrogram of corrosion deposition of a composition of disodium dihydrogen diphosphate with lanthanum in a 1:1 ratio

Elemental analysis of the corrosion deposits performed using a scanning electron microscope (Figure 8) revealed that the deposits in this system contain 46.58% oxygen, 13.36% iron, and 7.92% phosphorus, along with trace amounts of sodium (0.44%).

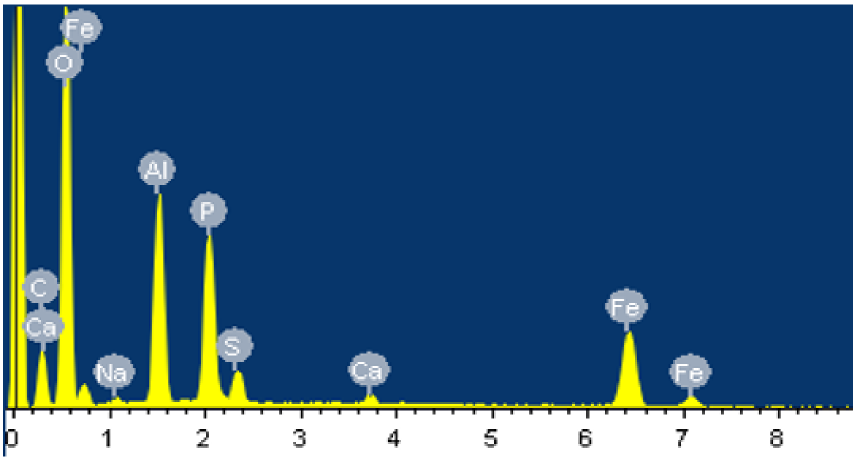

Figure 8 – Elemental composition of corrosion products formed in the disodium hydrophosphate-lanthanum system (1:1 ratio), according to spectroscopy data

The obtained results indicate a complex mechanism of the inhibitory action of diphosphate systems on iron corrosion. Despite the limited stability of the resulting compounds, further research is required to optimize the composition and structure of diphosphate systems in order to enhance their protective properties and stabilize the resulting corrosion deposits.

Studies of the inhibitory ability of cyclophosphate multicomponent systems

In addition to orthophosphate and diphosphate compositions, 18 cyclophosphate systems exhibiting inhibitory activity were also investigated. The most pronounced inhibitory effect was demonstrated by the sodium trimetaphosphate system with the lanthanum ion modifier at a 3:1 ratio (Figure 9). The inhibition efficiency was 45.86%.

The dynamics of ion concentrations (Figure 9) revealed an increase in iron concentration and decrease in the concentration of lanthanum and phosphate ions.

On the ninth day, the concentration of iron and the concentration of lanthanum and phosphate ions continued to decrease. This indicates the formation of complex compounds that slow down the corrosion process.

As in the case of diphosphates, for cyclophosphate systems in the presence of iron ions, an increase in phosphate concentration at a 2:1 ratio leads to a reduction in the corrosion rate. However, at a 3:1 ratio, an increase in the corrosion rate is observed.

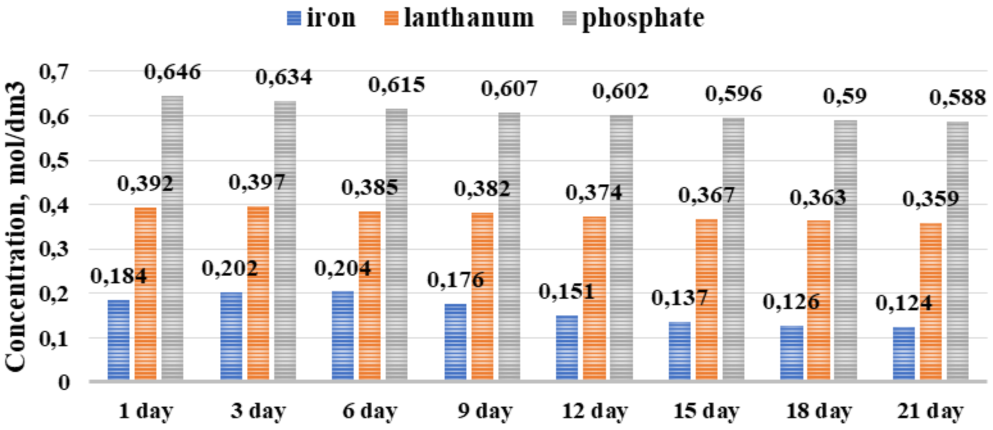

Figure 9 – Dynamics of the concentration of lanthanum, iron, and phosphate ions in the sodium trimetaphosphate system

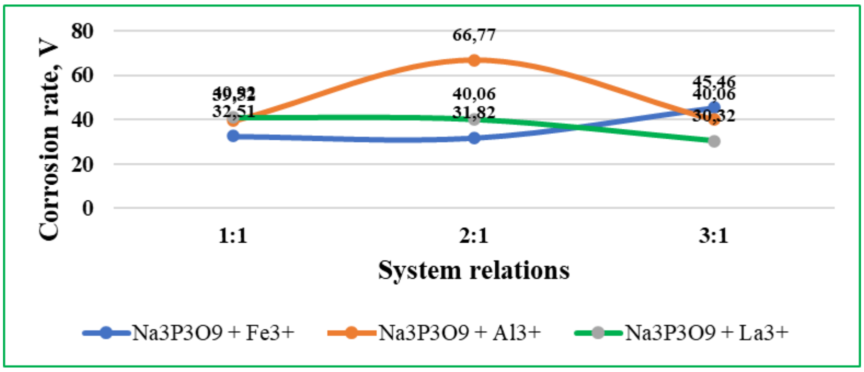

Figure 10 – Dependence of the corrosion rate on the nature of the modifier ion at different ratios for sodium trimetahosphate

For the system containing aluminum ions, an inverse dependence is observed, while for the system with lanthanum, a gradual decrease in the corrosion rate is recorded with an increase in phosphate concentration.

The analysis of the IR spectrum of corrosion products for the sodium trimetaphosphate system with lanthanum does not correspond to the previously described spectra, suggesting the formation of a new compound responsible for creating a protective film.

Figure 12 shows the analysis of corrosion deposits using a scanning electron microscope. The results revealed O_2 (36.97%), Fe (28.68%), and P (5.24%), as well as small amounts of Na and C.

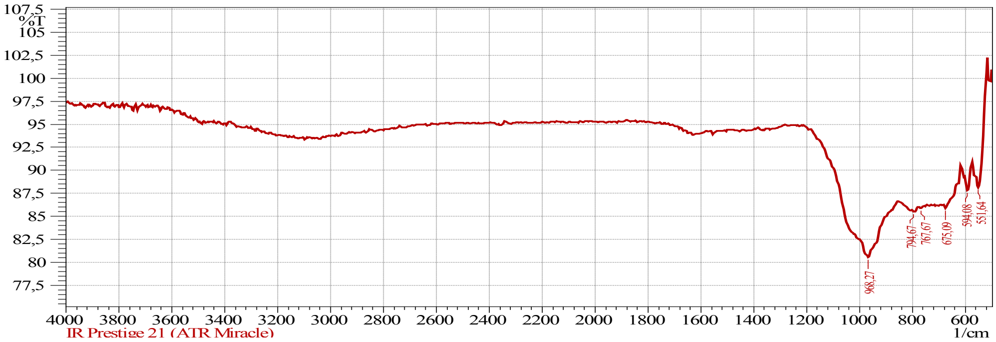

Figure 11 – IR spectrum of corrosion products formed in the system of sodium trimetaphosphate with lanthanum (ratio 3:1)

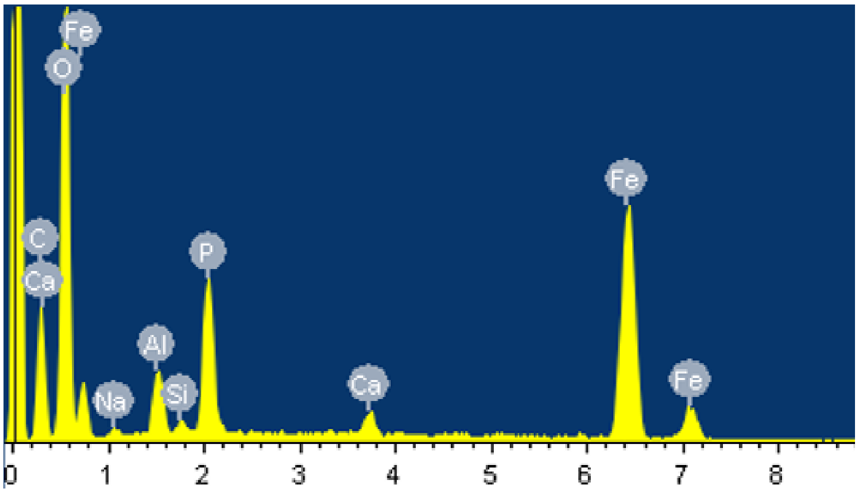

Figure 12 – Elemental composition of corrosion products formed in the system of sodium trimetaphosphate with lanthanum (ratio 3:1), according to spectroscopy data

It should be noted that the inhibitory capacity of phosphates is observed only up to a certain concentration, beyond which their activity begins to decline. Specifically, for Na_2HPO_4, the maximum protective effect is achieved at a concentration of 0.05 mol/dm^3.

Diphosphate systems are characterized by weaker protective activity, which is attributed to their lower stability in aqueous solutions and their tendency to form soluble protonated complexes that promote metal dissolution.

Diphosphates and cyclophosphates, unlike orthophosphates, have a stimulating effect on the corrosion process. Among cyclophosphate systems, trimetaphosphates have the highest protective properties. These systems reduce the corrosion rate. They are able to form polyphosphate compounds that inhibit the diffusion of oxygen to the metal surface. Aluminum, lanthanum, and iron ions also significantly enhance the protective functions of phosphate systems. The difference in the protective activity of lanthanum, iron, and aluminum ions is explained by the peculiarities of their structure and electronic configuration. The effect of these ions on the pH increases the thermodynamic stability of the system. Prevents the diffusion of corrosive agents. Promotes the formation of protective hydroxyphosphate compounds. Modification with aluminum ions has protective properties in phosphate systems. Aluminum ions react quickly and form stable complexes with hydroxyphosphates. This contributes to the formation of a strong protective film on the metal surface. The results obtained indicate that multicomponent phosphate systems are effective in the production of corrosion inhibitors. Phosphate systems are effective inhibitors of metal corrosion. They are environmentally friendly and affordable. However, they have limitations in certain situations. By modifying phosphate systems, their protective properties can be increased.

Conclusion

Regularities based on experimental research. Analysis of the obtained data allows the formulation of the following conclusions:

1. Influence of the nature of phosphate anions on the pH of the medium and the kinetics of corrosion processes. The investigation of the effect of various phosphate anions on the corrosion behavior of metals revealed the following descending order of inhibitory activity, as determined by the degree of pH shift and the reduction in corrosion rate:

$$Na_2HPO_4 > Na_3P_3O_9 > Na_6P_6O_{18} >$$
$$> NaH_2PO_4 > Na_4P_2O_7 > Na_3PO_4$$

Results from potentiometric, photocolorimetric, and kinetic studies demonstrate that hydroorthophosphate anions, which exhibit amphoteric properties, are the most effective in shifting the pH of the medium toward the alkaline range. This effect is attributed to the ability of these anions to undergo protonation and deprotonation. In the mildly alkaline medium (pH 7.46-9.23) created by hydroorthophosphate, intensified interaction with the metal surface is observed, which in turn contributes to the deceleration of the corrosion process.

2. Influence of phosphate concentration on corrosion rate. A correlation between the inhibitory efficiency of phosphates and their concentration in solution has been established. The highest protective efficiency is exhibited by systems containing Na_2HPO_4. Phosphate anions of higher order (e.g., diphosphates and hexaphosphates) demonstrate weaker inhibitory properties. It is assumed that this is due to the formation of excessive phosphate deposits on the metal surface, leading to the development of a non-uniform and brittle protective film. Increasing phosphate concentration may promote decomposition of system components and disturb the acid-base equilibrium, thereby negatively impacting corrosion processes.

3. Influence of the nature of modifier ions on the corrosion rate. Studies have shown that systems modified with aluminum ions exhibit the greatest inhibitory effect. The descending order of inhibitory activity of the modifier ions is as follows:

$$Al^{3+}>Fe^{3+}>La^{3+}$$

The best protective performance was demonstrated by multicomponent systems modified with aluminum ions. This is attributed to the high reactivity of Al^{3+} ions and their ability to form stable complexes that stabilize the protective films.

4. Advantages of hydroxyphosphate systems modified with aluminum ions.Systems modified with aluminum ions are characterized by several advantages, including the absence of undesirable side reactions, which increases their safety and effectiveness as corrosion inhibitors. The shift of the pH into the mildly alkaline range (approximately 7.5-9.0) enhances the corrosion resistance of metals and alloys, thereby reinforcing the inhibitory properties of phosphate systems. The obtained data indicate a high degree of material protection, as confirmed by low corrosion depth indicators (level 4), and underscore the effectiveness of these systems in preventing corrosion processes.

Systems modified with aluminum ions are characterized by the absence of adverse side reactions. They increase the corrosion resistance of metals and alloys in alkaline pH environments (approximately 7.5-9.0). At the same time, the inhibitory properties of phosphate systems are enhanced. The results obtained indicate a high level of material protection.

Acknowledgments

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Conflict of interest

The authors declare that they have no conflicts of interest.

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