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MUNDARIJA

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SYNTHESIS AND PROPERTIES OF SOME FIVE-MEMBERED BIAZOCYCLIC DERIVATIVES

Sanalar
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Abstract. During the research, the optimal conditions for the synthesis of 2-hexadecyl-2-imidozoline $C_{20}H_{40}N_2$, 2-octadecyl-2-imidozoline $C_{21}H_{40}N_2$, and 2-octadecyl-2-imidozoline $C_{21}H_{38}N_2$ from the three-acid fraction (TAF) obtained by adsorption of wood chips and extraction in isopropyl alcohol were determined. 98% ethylenediamine was used for the synthesis. TAF is a mixture of palmitic, oleic, and linoleic acids, present in a ratio of 30.3+22.7+42.3% by mass, respectively.

Keywords: *imidazolines, trihydric acid fraction, fatty acids, corrosion inhibitor, diamides, oil production, environmental sustainability*

Annotatsiya. Tadqiqotlar davomida yog‘och qipig‘ini adsorbsiyalab va izopropil spirtida ekstraksiyalab olingan uch kislotali fraksiyadan (FTK) 2-geksadetsil-2-imidozolin $C_{20}H_{40}N_2$, 2-oktadetsil-2-imidozolin $C_{21}H_{40}N_2$ va 2-oktadetsenil-

Usmonova Y.Sh., Kadirov X.I., Nurmanova J.Y., Obidov Sh.B. Synthesis and properties of some five-membered biazocyclic derivatives // Journal of future. 2026. Vol. 2. Iss. 1. pp. 10–19. https://doi.org/10.66960/jof.3093-8899.00015

2-imidozolin $C_{21}H_{38}N_2$ sintez qilishning maqbul sharoitlari aniqlandi. Sintez uchun 98% etilendiamin ishlatilgan. FTK mos ravishda 30,3+22,7+42,3% massa nisbatda bo‘lgan palmitin, olein va linol kislotalarining aralashmasidan iborat.

Kalit so‘zlar: *imidazolinlar, uch atomli kislota fraksiyasi, yog‘ kislotalari, korroziya ingibitori, diamidlar, neft ishlab chiqarish, ekologik barqarorlik*

Аннотация. В ходе исследований были определены оптимальные условия синтеза 2-гексадецил-2-имидозолина $C_{20}H_{40}N_2$, 2-октадецил-2-имидозолина $C_{21}H_{40}N_2$ и 2-октадеценил-2-имидозолина $C_{21}H_{38}N_2$ из трехкислотной фракции (ФТК), полученной адсорбцией древесной стружки и экстракцией в изопропиловом спирте. Для синтеза использовали 98% этилендиамин. ФТК представляет собой смесь пальмитиновой, олеиновой и линолевой кислот, содержащихся в соотношении 30,3+22,7+42,3% масс. соответственно.

Ключевые слова: *имидазолины, трехкислотная фракция, жирные кислоты, ингибитор коррозии, диамиды, нефтедобывающая промышленность, экологическая устойчивость.*

INTRODUCTION

Corrosion is the process and result of the physicochemical interaction of a material with the environment. Corrosion leads to a change in the properties of the metal (mainly in the direction of deterioration of the properties of the structural material), as well as to a change in the properties of the environment or technical system in which this material is a component. Such a definition corresponds to the international standard ISO 8044 [1-3].

Metals and metal alloys are the most important structural materials for many industries. However, they are susceptible to corrosion, i.e., deterioration under the influence of various physicochemical and biological factors. Metals and alloys decompose as a result of environmental influences, which can be chemical, electrochemical, or mechanical. Corrosion can occur due to the presence of aggressive components in technological media, as well as the high intensity of technological operating modes of equipment, such as high pressure, temperature, and the flow rate of the technological medium [4-7]. This leads to the appearance of significant mechanical stresses in the metal, which, together with the high corrosive aggressiveness of the technological media, accelerate the corrosive-mechanical wear of oil production equipment [8-10].

The main cause of corrosion is the thermodynamic instability of the metal in the oxidation process. Instability refers to the process of spontaneous transition of a metal to a more stable state, which occurs with an increase in the oxidation state - the metal forms oxides, bases, or salts [11-12].

Corrosion inhibitors are widely used in the oil and gas industry to protect various well equipment, pump-compressor pipelines, pipelines, and acid storage tanks. Individual substances, as well as compositions of chemical compounds, can act as inhibitors. When using them, certain technological standards must be observed, including achieving a high degree of protection at the minimum amount of reagent used, maintaining high effectiveness in acidic compositions for at least 30 days [13-15].

At the initial stage of obtaining a complex-acting inhibitory composition, alkylimidazolines with various structures and many production methods were studied as an active base. It is known that the composition of substances and the method of synthesis influence the corrosion-protective properties of the inhibiting composition [16-18]. Alkylimidazolines are thick, viscous, water-insoluble paste-like substances, the high viscosity of which makes it difficult to use them in further research, dosing them at a certain concentration. Therefore, imidazoline samples are used in dilution with solvents with low freezing points in various ratios: the most common solvent among alcohols is methanol, which has the lowest freezing point [19-20].

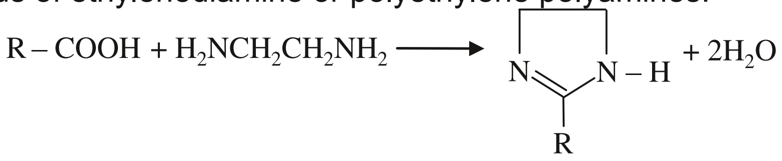
Imidazolines and compositions based on them demonstrate a high protective effect not only against carbon dioxide and hydrogen sulfide corrosion but also in acidic environments [21-23]. Based on this, imidazoline compositions with the addition of surfactants and solvents were tested in aqueous solutions of hydrochloric and sulfamic acids with a mass fraction of 5, 10, 15% for 24 hours at room temperature. The concentration of inhibitor compositions ranged from 0.03 to 1.00% by mass. According to GOST R 9.905-2007 and the technical regulations of companies operating in the oil and gas extraction industry, the permissible limit corrosion rate of St.3 steel under these conditions should be 0.2 g/ (m2·hour) [24-25].

Carboxylic acid diamides are poorly soluble in water and organic solvents, decompose into monoamides under reaction conditions, have low surface activity, and negatively affect the quality of the finished product due to their presence in it [26]. Separation of diamide from the finished product is carried out by the rectification method and requires additional expenses. To achieve economic efficiency in industry, a mixture of fatty acids is used, not pure fatty acids [27-28]. At the same time, the use of a mixture of fatty acids leads to technological problems in determining the optimal reaction conditions and a decrease in the yield of imidazoline, an increase in by-products. Experiments show that the resulting alkylimidazoline and diamide ratios are related to the electrophilicity of the carbonyl group carbon of the acylating agent, i.e., the chain length of the carboxylic acid hydrocarbon radical [29-30].

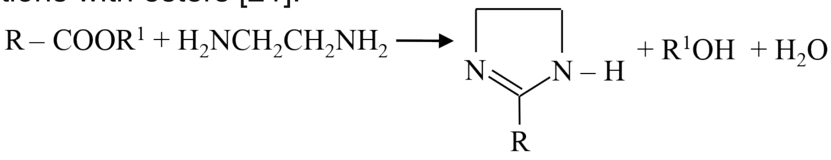
METHODS

In the course of the research, the optimal conditions for the synthesis of 2-hexadecyl-2-imidazoline $C_{20}H_{40}N_2$, 2-octadecyl-2-imidazoline $C_{21}H_{40}N_2$, and 2-octadecyl-2-imidazoline $C_{21}H_{38}N_2$ from the three-acid fraction (TF), obtained by adsorption with wood sawdust and extraction in isopropyl alcohol, were determined. 98% ethylenediamine was used for the synthesis. UVF is a mixture of palmitic, oleic, and linoleic acids and is maintained in a ratio of 30.3+22.7+42.3% by mass, respectively.

One of the important methods for the synthesis of alkylimidazolines is the synthesis of fatty acids of ethylenediamine or polyethylene polyamines:



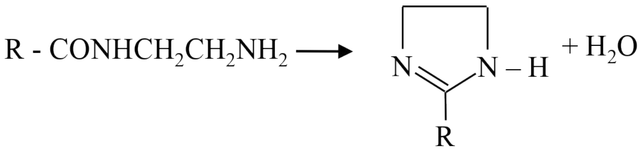
or their reactions with esters [21]:



The process takes place in two stages. Step 1: Formation of fatty acid with ethylenediamine to form acid amidoamines:

$$R-COOH + H_2NCH_2CH_2NH_2 \longrightarrow R\text{ - }CONHCH_2CH_2NH_2 + H_2O$$

Stage 2: heterocyclization of acid amidoamine with the formation of alkylimidazoline derivatives:

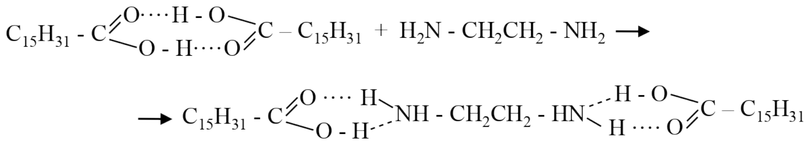


The secondary products of the process are diamides:

$$R\text{ - }CONHCH_2CH_2NH_2 + R\text{ - }COOH \longrightarrow R\text{ - }CONHCH_2CH_2NHCO\text{ - }R + H_2O$$

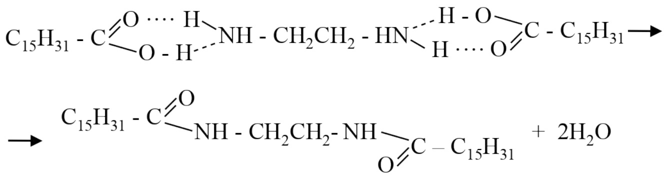
Three different reactions were carried out to determine the optimal conditions for the heterocyclization reaction. The sequence of the experiment: a three-neck flask was equipped with a thermometer, a Wurtz nozzle with a Liebig cooler, and a mechanical stirrer; thermal treatment of the reaction mass was carried out using an electric heater. 100 g of UCP and 100 g of ethylenediamine were placed in the reactor and heated to 40°C, and the mixer was activated during the dissolution of UCP. With intensive mixing and a gradual increase in temperature, condensation of water vapor was observed in the refrigerator at 130°C, and the condensate was collected in the collector. With an increase in temperature above 200°C, an excess of ethylenediamine was also distilled, and at a temperature above 250°C, the reaction water of heterocyclization was distilled. Heat treatment was carried out at 270°C for 1 hour. Then the process is carried out in a vacuum, and the temperature is 180 mm Hg. During 1 hour, complete heterocyclization was carried out, and the remaining water was separated.

During the research, the mechanism of chemical transformations was investigated, and pure fatty acid - palmitic acid - was used as an acylating reagent in the experiments. It has been established that with an increase in the temperature of the reaction mixture, fatty acids react not only in a mono-, but also in a dimeric state and form three-molecular complexes in the acid-amino-acid system through hydrogen bonds according to the following scheme:

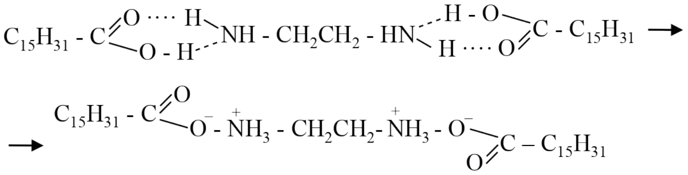


(1)

Three-molecular complexes form diamides (2), diammonium salts (3), and acid amide monohydrates (4) with a stable structure, possessing ion-exchange activity at 130 °C:



(2)



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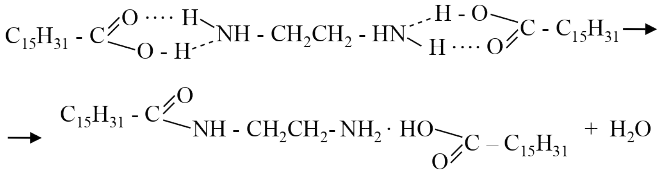
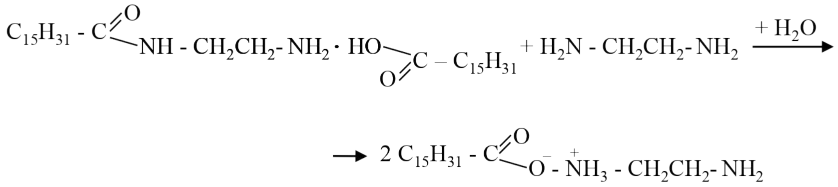


$$C_{15}H_{31}-C\genfrac{}{}{0pt}{}{O\cdots H}{O-H\cdots}NH-CH_2CH_2-HN\genfrac{}{}{0pt}{}{\cdots H-O}{H\cdots O}C-C_{15}H_{31}\rightarrow$$

$$\rightarrow C_{15}H_{31}-\overset{O}{\overset{\|}{C}}-NH-CH_2CH_2-NH_2\cdot HO-\underset{O}{\underset{\|}{C}}-C_{15}H_{31}+H_2O$$

(4)

An excess of ethylenediamine provides a strongly basic medium for the reaction, and under such conditions, a proton shift occurs in three-molecular complexes, forming salts with a binary structure:

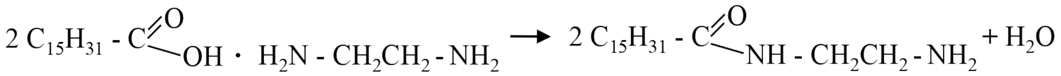


$$C_{15}H_{31}-\overset{O}{\overset{\|}{C}}-NH-CH_2CH_2-NH_2\cdot HO-\underset{O}{\underset{\|}{C}}-C_{15}H_{31}+H_2N-CH_2CH_2-NH_2\xrightarrow{+H_2O}$$

$$\rightarrow 2\,C_{15}H_{31}-\overset{O}{\overset{\|}{C}}-\overset{-}{O}-\overset{+}{N}H_3-CH_2CH_2-NH_2$$

(5)

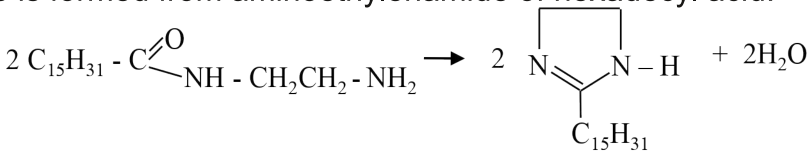
At the same time, with an increase in temperature by 130 °C, internal diamidation of ammonium salts or hydrates also occurs:



$$2\,C_{15}H_{31}-\overset{O}{\overset{\|}{C}}-OH\cdot H_2N-CH_2CH_2-NH_2\rightarrow 2\,C_{15}H_{31}-\overset{O}{\overset{\|}{C}}-NH-CH_2CH_2-NH_2+H_2O$$

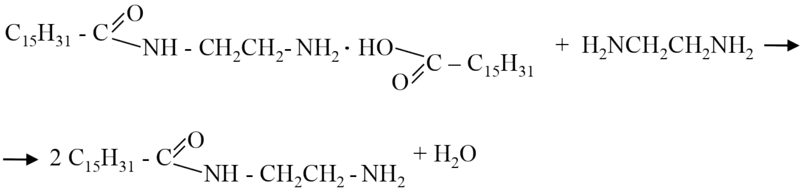
(6)

This reaction, accompanied by the transition of the binary complex to diamidoamine, is of great importance in the formation of alkylimidozolines, and during the second stage of thermal treatment of the reaction, 2-hexadecyl-2-imidozoline is formed from aminoethylenamide of hexadecyl acid:



(7)

Hexadecylic acid aminoethylenamide, in turn, can also be formed by amidation from an acid-amino-acid three-molecular complex:

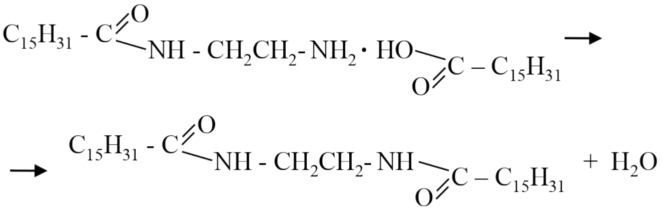


$$C_{15}H_{31}-\overset{O}{\overset{\|}{C}}-NH-CH_2CH_2-NH_2\cdot HO-\underset{O}{\underset{\|}{C}}-C_{15}H_{31}+H_2NCH_2CH_2NH_2\rightarrow$$

$$\rightarrow 2\,C_{15}H_{31}-\overset{O}{\overset{\|}{C}}-NH-CH_2CH_2-NH_2+H_2O$$

(8)

Aminoethylenamide of hexadecylic acid forms diamides in the secondary direction:



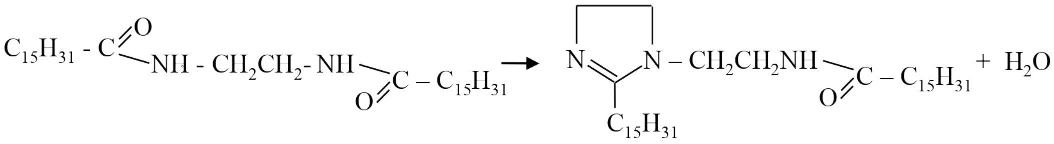
$$C_{15}H_{31}-\overset{O}{\overset{\|}{C}}-NH-CH_2CH_2-NH_2\cdot HO-\underset{O}{\underset{\|}{C}}-C_{15}H_{31}\rightarrow$$

$$\rightarrow C_{15}H_{31}-\overset{O}{\overset{\|}{C}}-NH-CH_2CH_2-NH-\underset{O}{\underset{\|}{C}}-C_{15}H_{31}+H_2O$$

(9)

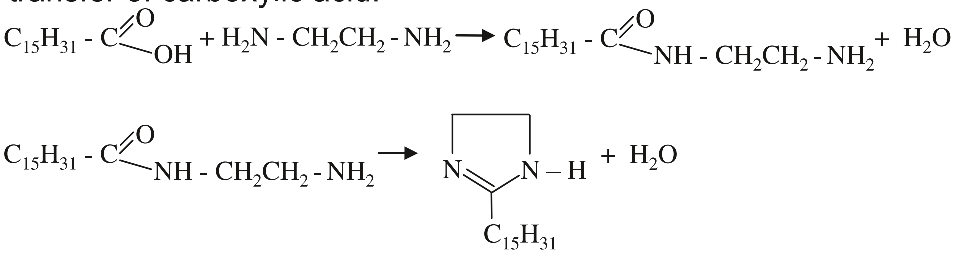
In this case, considering the ease of formation of the acid-amino-acid three-molecular complex compared to the reactions of the initial carboxylic acid and ethylenediamine, and the ease of implementation of the internal complex reaction compared to intermolecular interaction reactions, it is clear that diamide is formed in any excess amount of ethylenediamine (even with a 20-fold increase). Along with the heterocyclization of acid amidoamine, the heterocyclization of diamides is also observed, and the formation of 2-hexadecyl-1- (2-hexadecyl aminoethyl)-2-imidazoline also influences the selectivity of the reaction. In the composition of the products obtained under the conditions of the conducted experiments, peaks close to the molecular mass of 2-hexadecyl-1- (2-hexadecyl aminoethyl)-

2-imidozoline diamides are observed:



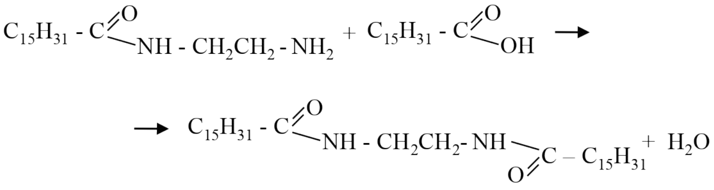
(10)

Thus, to reduce the amount of the secondary product diamide in the synthesis of alkylimidazolines, it is necessary to prevent the formation of the initial acid dimer and thus the formation of the acid-amino-acid three-molecular complex. To achieve the goal set during the research, reactions were carried out in the "ethylenediamine carboxylic acid" system, and the sequence of adding the initial reaction raw materials was studied. At the first stage of heat treatment, ethylene diamine was initially introduced into the reactor, and then the reaction was carried out by bimolecular acylation without the initial stage - the formation of ammonium salts - with the transfer of carboxylic acid:

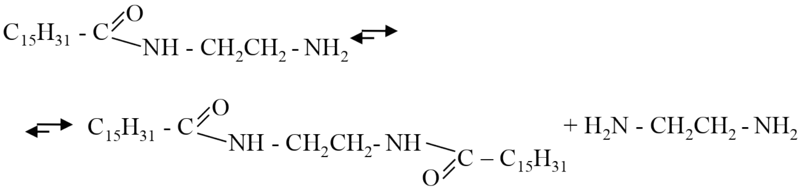


$$C_{15}H_{31}-C(=O)OH + H_2N-CH_2CH_2-NH_2 \rightarrow C_{15}H_{31}-C(=O)NH-CH_2CH_2-NH_2 + H_2O$$

Stepwise transfer of diamine to carboxylic acid limits the formation of the resulting acylamidoethylenamine by the following reactions:



$$C_{15}H_{31}-C(=O)NH-CH_2CH_2-NH_2 + C_{15}H_{31}-C(=O)OH \rightarrow C_{15}H_{31}-C(=O)NH-CH_2CH_2-NH-C(=O)-C_{15}H_{31} + H_2O$$



$$2\,C_{15}H_{31}-C(=O)NH-CH_2CH_2-NH_2 \rightleftarrows C_{15}H_{31}-C(=O)NH-CH_2CH_2-NH-C(=O)-C_{15}H_{31} + H_2N-CH_2CH_2-NH_2$$

After transferring the entire amount of carboxylic acid to the reaction medium, the control of the reaction was continued in the same manner as in Experiment 1. The synthesis was completed, and the resulting product was analyzed by IR spectroscopy and gas-liquid chromatography methods.

RESULTS

In the IR spectrum of the product obtained as a result of the reaction, bands of C=N valence vibrations characteristic of the imidazoline heterocycle (1608 cm-1), intense bands characteristic of C=O (1668 cm-1), and absorption characteristic of N-H deformation vibrations (1556 cm-1) were observed.

Chromatographic analysis showed that the total surface area of alkylimidazoline peaks is 1:28 relative to diamide peaks, and the quantitative ratio is 0.48: 99.52% by mass. In this case, a sharp decrease in the content of 2-hexadecyl-1- (2-palmityl-aminoethyl) -2-imidazoline was also observed and amounted to 1.52% compared to 2-hexadecyl-2-imidazoline.

Thus, it was possible to minimize the amount of diamide formation by changing the

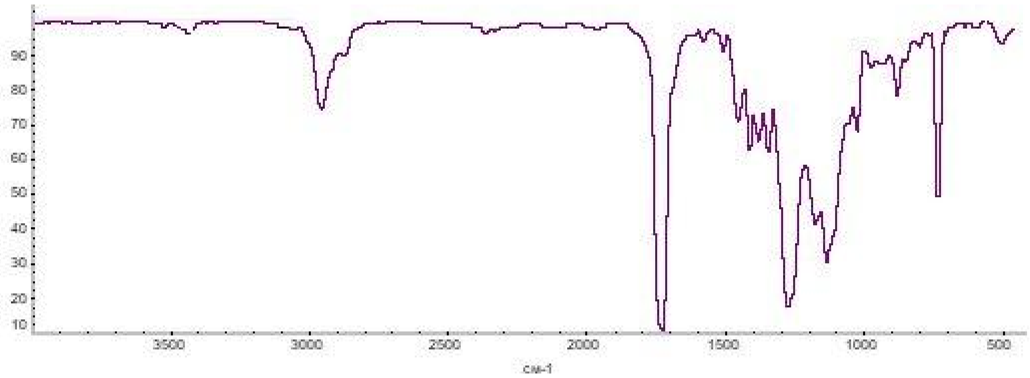


Figure 1. Product obtained in the "Ethylenediamine→ carboxylic acid" system IR spectrum

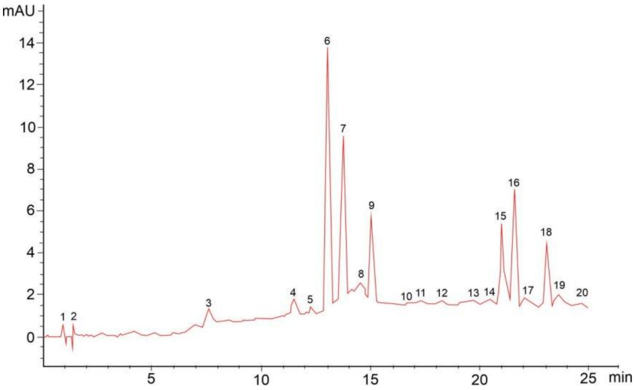


Figure 2. Chromatogram of the product obtained at a ratio of EF: EDA 1:4: 6-,7- and 9-alkylimidazolines; products of acylation of ethylendiamine with palmitic, oleic, and linoleic acids, respectively

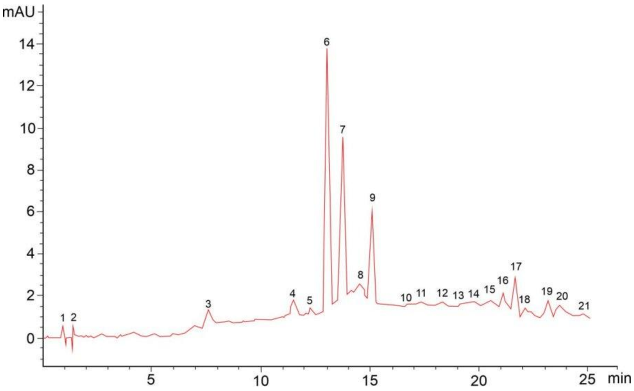


Figure 3. Chromatogram of the product obtained in the "Ethylenediamine →Carboxylic Acid" system: 6- 7-, and 9-alkyl-imidazolines; products of acylation of ethylene diamine with palmitic, oleic, and linoleic acids, respectively

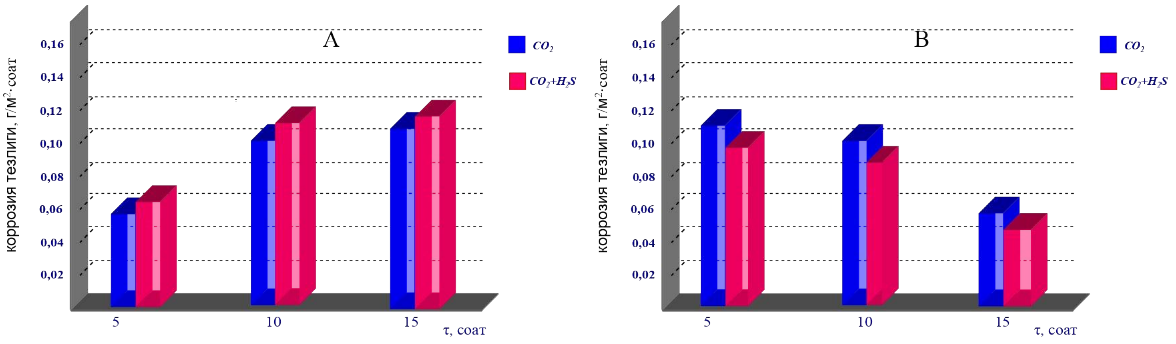
sequence of feedstock transfer in the carboxylic acid system to ethylenediamine.

DISCUSSION

At the initial stage of the research to determine the process of inhibitor adsorption, the corrosion rates of steel samples in various aggressive environments were studied. The use of mineralized water saturated with hydrochloric and sulfamic acids, as well as dissolved gases such as carbon dioxide and a mixture of carbon dioxide and hydrogen sulfide, made it possible to assess the influence of such factors as the composition of the medium and the pH value on the activity and adsorption of the developed inhibitory composition. The change in the movement of the medium from static to 1 m/s helped to assess the influence of the flow rate of the aggressive medium on the adsorption properties of the corrosion inhibitor. The time interval from 3 to 24 hours was used to determine the time required to achieve maximum adsorption of the inhibiting composition on the metal surface.

Fig. 4 (A and B) shows the dependence of the corrosion rates of St.3 steel samples on the duration of the experiment in a water-saturated layer of mineralized model saturated with CO_2 and H_2S.

According to the obtained data, it is established that the corrosion rate in an



A - 0 m/s; B - 1,0 m/s

Figure 4. Dependence of the corrosion rate of St.3 steel on the experimental time in an MPV medium saturated with a mixture of carbon dioxide and hydrogen sulfide. Experimental conditions: at a temperature of 20±2°C, with the addition of an alkylimidazoline +PAV inhibiting composition in an amount of 0.06% by mass, at the following rotational speeds:

When adding an inhibiting composition, at concentrations selected based on the results of previous studies: 0.06% by mass in acidic media and 60 g/m3 in aqueous media, at room temperature and rotational speeds from 0 m/s to 1.0 m/s.

aggressive medium saturated only with carbon dioxide is higher than in an aqueous medium with the addition of carbon dioxide and hydrogen sulfide. This can be explained by the formation of corrosion products with different properties and different mechanisms of inhibitor adsorption. While iron forms a porous corrosive compound ($FeO{\cdot}FeCO_3$) with carbonic acid that has no protective properties, sulfides are denser compounds compared to carbonates, therefore in some cases they can serve as a protective layer. In addition, the HS^- ions formed in the solution are adsorbed on the metal surface, displacing the adsorbed H_2O and OH^-, which ultimately leads to a decrease in the corrosion rate. Adding a small amount of H_2S to a CO_2 medium can reduce the reduction rate of H_2O and H_2CO_3, limiting the corrosion process. The lowest values of the corrosion rate in both media are reached 18 hours after the start of the experiment.

Determination of the protective effectiveness of the considered inhibiting composition was carried out based on the values of the corrosion rate in pure aggressive media without the addition of an inhibitor. The results obtained in an aqueous medium saturated with dissolved gases are presented in Fig. 5 (A and B).

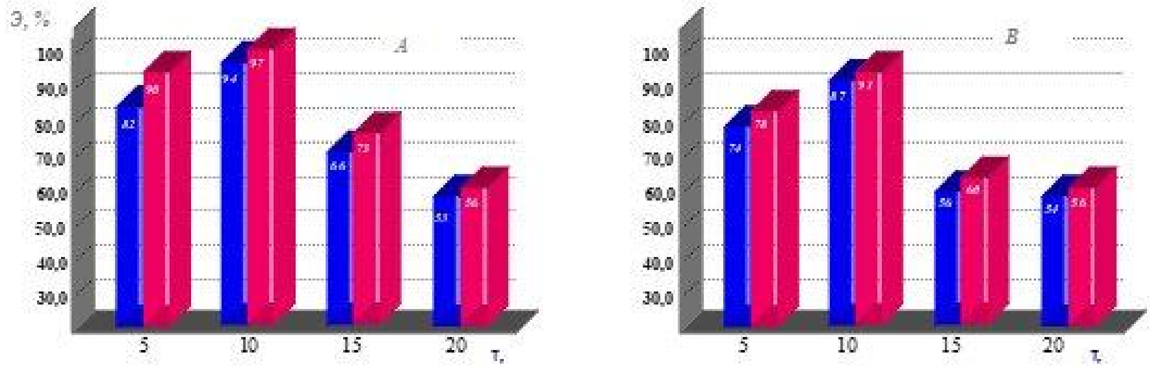


Figure 5. Dependence of the protection effectiveness of inhibitor composition No. 3 (0.06% by mass) on the experimental time in an MPV medium saturated with carbon dioxide and hydrogen sulfide (at a temperature of t=20±2°C). Rotational speeds: A = 0 m/s; V - 1.0 m/s

CONCLUSION

From these graphs, it can be concluded that the protective effect of the inhibiting composition in the presented media changes over time. The indicators of the protective effect are closely related to the values of the corrosion rate and show the highest results after 18 hours. After the experiment, they decrease insignificantly or do not change, which indicates competing processes of inhibitor adsorption and desorption, as well as the formation of reaction products as a result of the corrosion process. The protective effect is higher in a mineralized water environment saturated with both hydrogen sulfide and carbon dioxide, which is explained by the positive effect of HS^- ions on the process of inhibitor adsorption. The adsorption of these anions on the steel surface facilitates the interaction of positively charged molecules of the inhibitor with the metal, which leads to the formation of stronger Fe (H-S-R) compounds, their release from the steel surface is reduced, and as a result, the metal is protected more effectively. The appearance of a dynamic factor initially leads to an increase in the values of the protective effect, which can be explained by an increase in the diffusion of inhibitor molecules to the surface. However, with a further increase in the flow rate of the aggressive medium, the indicators of the protective effect decrease somewhat, which is explained by a shift in the equilibrium towards desorption during the adsorption-desorption processes of the inhibitor on the steel surface. In addition, dynamic conditions contribute to the separation of corrosion products formed during the reaction, which, in turn, can have a positive effect on corrosion protection.

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