

Розробка ефективних засобів захисту металів від корозії в агресивних середовищах, що містять воду, нафтопродукти, карбонові кислоти та мінеральні солі, є актуальним і практично важливим завданням охорони навколишнього середовища. Для боротьби з корозією використовують інгібітори корозії, які потрібно постійно вдосконалювати та коригувати їх склад. Головним недоліком високоефективних інгібіторів на основі алкілімідазолінів, суміші алкілімідазолінів з алкілірідінієвими та/або четвертинними амонійними сполуками розчинними в середовищі метанолу є високі ціни при відносно значних їх витратах в корозійне середовище. В роботі синтезовано інгібітори корозії сталі у нафтовмісних водних середовищах, що відповідають підвищеним еколого-економічним вимогам. Показано, що при підвищенні рівня мінералізації води відбувається зростання корозійної активності водних середовищ по відношенню до нелегованої сталі. Наявність вуглекислого газу, сірководню чи карбонових кислот призводять до підкислення водно-нафтових сумішей, внаслідок чого спостерігається підвищення швидкості корозії сталі. Досліджено ефективність синтезованих інгібіторів на основі олій та поліетиленполіамінів, які містять імідазоліни. При температурі 80 °С у суміші, що містила 200 см³ 3% розчину хлориду натрію, 800 см³ нафти, концентрації оцтової кислоти 0,5 та 3,0 г/дм³ при дозі інгібітору 50 мг/дм³ досягнуто ступеню захисту сталі від корозії на рівні 90–92%. На основі повного факторного експерименту розраховано рівняння регресії, які дозволяють досить легко розрахувати оптимальну дозу інгібітору корозії сталі у водно-нафтових сумішах. Показано, що синтезований інгібітор перспективний при захисті металів від корозії як в мінералізованих водах, що містять нафту, так і в присутності нафтопродуктів, що містять воду

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1. Introduction

The issue related to the chemical resistance of materials and the protection of metals has remained relevant given the permanent corrosion losses by Ukraine's metal fund, which causes significant material losses in various sectors of industry. Corrosion accounts for 40–70% in the premature destruction of steel structures, which leads to anthropogenic accidents and catastrophes, as well as to the loss of billions of tons of articles. The corrosion-related issues are acute in the chemical, petrochemical, and oil-producing industries [1].

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SYNTHESIS OF HIGH-EFFECTIVE STEEL CORROSION INHIBITORS IN WATER-OIL MIXTURES

N. Gomelya

Doctor of Technical Sciences, Professor,
Head of Department*
E-mail: m.gomelya@kpi.ua

I. Trus

PhD, Senior Lecturer*
E-mail: inna.trus.m@gmail.com

O. Stepova

PhD, Associate Professor
Department of Applied Ecology
and Environmental Sciences
National University «Yuri Kondratyuk

Pershotravnevyyi ave., 24, Poltava, Ukraine, 36011

E-mail: alenastepovaja@gmail.com

O. Kyryliuk

PhD, Senior Lecturer
Department of National Security
National Academy of the Security Service of Ukraine
M. Maksymovycha str., 22, Kyiv, Ukraine, 03022

E-mail: 20kiril20@gmail.com

O. Hlushko

PhD, Associate Professor*
E-mail: alyona_glushko@ukr.net

*Department of Ecology and Technology
of Plant Polymers
National Technical University of Ukraine
«Igor Sikorsky Kyiv Polytechnic Institute»
Peremohy ave., 37, Kyiv, Ukraine, 03056

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The duration of equipment operation depends on the aggressiveness of the media. The corrosion rate is much higher at the elevated concentration of carbon dioxide and hydrogen sulfide. Water, together with the corrosion-active gases dissolved in it, brings the intensive local corrosion damage to steels 20 and St37-2, which are the most common structural materials in the petrochemical and oil and gas extracting industries [2].

Mine waters in oil production processes are characterized by high concentrations of mineral salts [3, 4]. This results in their high corrosion activity and propensity for

sedimentation. The pre-stabilization treatment of solutions [5] is used to prevent sedimentation. Quite significant is the corrosion activity of a medium in crude oil, in which water content reaches 20–30 %, and in water with oil content up to 10 %. This is due to the fact that the pumping of oil implies heating it to a temperature of 70–80 °C, which in the presence of water causes significant corrosion of steel pipelines when using unalloyed steel pipes [6, 7].

Corrosion inhibitors are used to effectively protect pipelines and equipment against corrosion [8, 9]. One of the key trends in the development or application of inhibitors is the concept of environmental friendliness and minimum impact on the environment. Therefore, the priority in the world is the use of «green» anti-corrosion protection means. There are data on the corrosion inhibitors based on plant-derived organic compounds for various corrosive media [10, 11]. These inhibitors are quite effective in neutral and acidic environments. However, under additional exposure to mechanical stresses or aggressive environment, their use is not sufficiently effective. At present, the protection of metals in water-oil mixtures most often involves inhibitors based on alkyl imidazoline, a mixture of alkyl imidazolines with alkyl pyridinium and/or quaternary ammonium compounds that dissolve in a methanol medium [1].

Thus, it is a relevant task to search for such corrosion inhibitors that could demonstrate the high protective effect and would comply with the environmental and economic factors, which would prolong the service life and improve the resistance of metallic materials to the processes of corrosion destruction.

2. Literature review and problem statement

Authors of [12] applied an extractive part of grape processing products and its main components, aromatic and aliphatic aldehydes, as steel corrosion inhibitors in the steam phase. They established the inhibitory efficacy (the degree of protection is 99 %) and the mechanism that forms a protective film. However, the efficiency in aqueous environments was not investigated.

The «green» corrosion inhibitors were investigated as an effective anti-corrosion agent for steel in aqueous environments containing chlorides. The environmentally safe «green» organic compounds of various chemical classes (polyphenol compounds, flavonoids, aldehydes) ensure efficiency at the level of 98 % [13]. The paper considered selective action only for a neutral water corrosion environment; the two-phase systems were not studied that are typical for steel corrosion in the oil and gas extraction industries.

Gel capsules with calcium alginate, loaded with an inhibitor and BaSO₄ were used in paper [14] as the corrosion inhibitors at oil-containing environments. The heavy additive introduced contributes to capsule settling. When the temperature rises from 15 to 70 °C, it increases the release of the inhibitor. However, the paper disregarded many factors that affect the efficiency of using capsules, for example, the time it takes for a capsule to be in a well or the oil pipeline, the optimum release rate of the inhibitor, the optimum immersion capsule speed, etc.

Authors of work [15] synthesized an inhibitor based on polycarbonates with carboxylic and/or phosphonate groups. The derived PCA-COOH inhibitor had a fairly low efficiency; over a week, at 130 °C, one observed a decrease in the PCA-PO₃H₂-COOH inhibitory efficacy; to improve the effec-

tiveness of the process, it must include new functional groups. Rather promising are the inhibitors based on nitrogen-containing heterocyclic compounds. Paper [16] showed that the maximum rate of titanium corrosion (0.260 mg/(cm²·h)) was demonstrated in 1.0 M sulfuric acid at 298 K. Aromatic nitro compounds act as effective titanium corrosion inhibitors. Aromatic nitro compounds demonstrate the maximum inhibitory efficacy of 73.0 % (VI), 82.6 % (VII), and 94.6 % (VIII). The effectiveness of inhibition improves with an increase in the concentration of inhibitors and decreases with an increase in temperature. Work [17] shows that when the temperature rises from 20 °C to 50 °C there is a decrease in inhibitory properties, by 40 %. This is due to both the destabilization of the oxygen passivation film and the increase in the rate of steel oxidation with an increase in temperature. However, studies [16, 17] did not consider corrosion processes for temperatures above 60 °C.

Three new corrosion inhibitors based on dynamic imidazole (IL1, IL2, and IL3) were synthesized in paper [18], with their effectiveness reaching 91.5 %, 98.4 %, and 83.3 %, respectively. However, the unresolved issue is the high price of the inhibitor at its relatively significant consumption in the corrosive environment.

Thus, our analysis of works [12–18] allows us to conclude that up to now the issues related to the development of highly effective, and affordable, metal corrosion inhibitors in water-organic environments have remained insufficiently studied.

So, the best-known inhibitors for water-oil systems are the reagents based on imidazolines. However, these inhibitors are quite costly. They are obtained through the condensation of polyamines by carbonic acids. Carbonic acids with the alkyl radical C₁₆–C₁₈ are currently priced at the level exceeding USD 40/kg. In addition, they are absent in the Ukrainian market. Sunflower oil, even of high quality, is priced at about USD 1.2/kg. If you take a technical product for wholesale use, its cost does not exceed USD 0.8/kg. Given that the mass of carbonic acid in the synthesis of imidazolines accounts for 80–85 % of the total mass of the reagents, it is clear that it is advisable to replace carbonic acids in the production of imidazolines with sunflower oil. In this case, the sunflower oil, which is an ether of fatty acids, is much easier to react with polyalkylenepolyamines in comparison with acids. In addition, the fatty acids of plant-based oils contain a significant amount of unsaturated bonds, which is why their products are melted at lower temperatures compared to the saturated carbonic acids. That is, in contrast to solid imidazolines obtained from the saturated carboxylic acids, using the sunflower oil would yield products with a low melting point; at room temperature, they will be liquid. The resulting products should be well dissolved both in hydrophobic organic solvents and in polar organic solvents. This allows us to argue about the prospects of research aimed at the synthesis of new corrosion inhibitors in order to create the environmentally-safe corrosion inhibitors.

3. The aim and objectives of the study

The aim of this study is to develop new steel corrosion inhibitors in water-oil mixtures, to assess their effectiveness depending on the composition of an aqueous environment and temperature.

To accomplish the aim, the following tasks have been set:

- to devise a procedure for synthesizing imidazolines in the interaction between sunflower oil and polyethylene polyamines;

– to study the corrosion processes of steel St37-2 in a water-oil mixture in the presence of acetic acid and to determine the rate of corrosion in salt water solutions, in a mixture of mineralized solution and oil at temperatures of 30–80 °C, to assess the effectiveness of inhibitors under these conditions;

– to simulate corrosion processes in order to optimize the conditions for protecting metal against corrosion and to determine the optimal dose of the imidazoline inhibitor.

4. Materials and methods to assess corrosion inhibitors in water-petroleum environments

4.1. Examined materials used in the experiment

Samples of steel St37-2 were used in this study. Corrosion was examined by the massometric method. The corrosive environment used was a water-oil mixture (200 cm³ of a 3 % sodium chloride solution, 800 cm³ of oil, 0.5 g and 3 g of CH₃C(O)OH). The temperature was 30, 60, and 80 °C. Corrosion time – 8–10 hours.

The following reagents were used to synthesize the inhibitor: sunflower oil (DSTU 4492:2017), diethylene triamine (TU 2413-357-00203447-99) and Octanol-1 (TU 6-09-3506-78).

4.2. Procedure for determining the efficiency of metal corrosion inhibitors in water-oil environments

The rate of corrosion and the degree of protection against corrosion were calculated from formulae:

$$V = \frac{V_p \cdot (m_1 - m_2)}{S \cdot t}, \text{ g}/(\text{m}^2 \cdot \text{h}),$$

$$Z = \left(1 - \frac{V_i}{V_0}\right) \cdot 100, \%$$

where m_1 is the initial mass, g; m_2 is the mass of samples after the experiment, g; S is the surface area of steel samples, m²; t is the experiment duration, h; V_i , V_0 is the steel corrosion rate with and without an inhibitor, g/(m²·h).

5. Results of studying the inhibitor efficacy

5.1. Synthesis of the reagent AC-1 as a corrosion inhibitor for water-oil mixtures

In the course of this study, we synthesized alkyl imidazolines using sunflower oil and diethylene triamine. The reaction was performed in Octanol-1. To this end, we added, to 0.1 mol of oil (based on the calculation of an average molecular mass of oil of 932 a. u. at the chosen mean molecular mass of a fatty acid of 280 a. u.) 0.3 mol of diethylene triamine and 250 cm³ of Octanol-1. In this case, the principal process was the progress of the reaction shown in Fig. 1.

The mixture was heated at stirring to 190 °C in a reactor. The mixture was aged at a given temperature for 6 hours while continuously extracting the reaction water. After that, the mixture was cooled; the solvent was released in a vacuum. The residue was a viscous light-brown liquid. After the dissolution of methanol, it was used as a corrosion inhibitor, encoded AS-1. The progress of the reaction yielding imidazoline was registered based on the signals in the PMR spectrum in the region of 3.2–3.7 ppm.

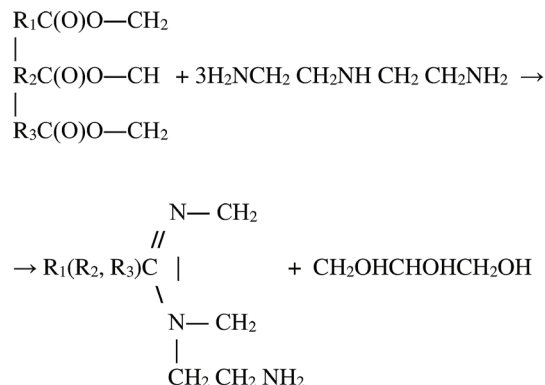


Fig. 1. The reaction of alkyl imidazoline synthesis using sunflower oil and diethylene triamine: R₁, R₂, R₃ are the radicals of carbonic acids in oil

5.2. Determining the efficacy of the AC-1 reagent as a corrosion inhibitor for water-oil mixtures

The water-oil mixtures obtained during extraction of oil and during its transportation are corrosion-active for metals due to the presence of impurities of mineralized water and carbonic acids. In this case, acidic impurities increase the rate of steel corrosion by tens of times. However, in the presence of oil and corrosion inhibitors, this effect is slightly reduced.

The effectiveness of the imidazoline-based corrosion inhibitor depends to a larger extent on temperature (Fig. 2, 3). The corrosion rate of steel St20 increases dramatically in a water-oil mixture with an increase in temperature from 30 to 80 °C. However, the increase in temperature also improves the efficiency of the corrosion inhibitor. At 30 °C, at the inhibitor concentration of 25 mg/dm³, the degree of protection reaches only 44 %, at 60 °C – 60 %, and at 80 °C – 76 %. At 80 °C, and at the inhibitor concentration of 50 mg/dm³ in a water-oil mixture, the protection degree exceeds 90 %.

The imidazoline inhibitor ensures a high degree of corrosion protection for steel St20 even at the ratio of a 3 % solution of NaCl to oil as 80:20 and in the presence of acetic acid in the mixture. With the increase in the acetic acid concentration in a water-oil mixture to 3 g/dm³, the effectiveness of the AC-1 inhibitor is sharply reduced at low temperatures. Even at the inhibitor dose of 25 mg/dm³, the degree of protection against corrosion at 30 °C is reduced to 17 %, at 40 °C – to 43 %. At 80 °C, at the inhibitor concentration of 5 mg/dm³, the degree of protection reaches 68 %, and at a concentration of 25 mg/dm³, it exceeds 90 %. The corrosion rate is reduced in this case from 33.83 mm/year to 0.382 mm/year.

Our study has determined the efficacy of the AS-1 reagent in an environment at the ratio of oil volumes to a 10 % solution of NaCl of 90:10, containing acetic acid. Thus, in the mixture that contained 50 cm³ of a 10 % sodium chloride solution and 950 cm³ of oil at the concentration of acetic acid respectively, 0.5 and 3 g/dm³, at a temperature of 80 °C, the inhibitor efficacy was quite high (Fig. 4).

At the concentration of acetic acid of 0.5 g/dm³ at the inhibitor dose of 10 mg/dm³, the degree of protection of steel against corrosion at the level of 56–57 % was reached. When increasing the concentration of the inhibitor to 50 mg/dm³, the degree of protection reached 90–91 %. Thus, we managed to reduce the corrosion rate from 1.5872 to 0.1583 g/(m²·h). Good results were obtained when using the inhibitor in

a water and oil mixture containing 3 g/dm³ of acetic acid. At the inhibitor dose of 10–50 mg/dm³, the degree of protection against corrosion of steel St20 reached 64–92 %. In this case, the rate of corrosion decreased from 3.4508 g/(m²·h) without the inhibitor to 0.3015 g/(m²·h) with the inhibitor in an amount of 50 mg/dm³.

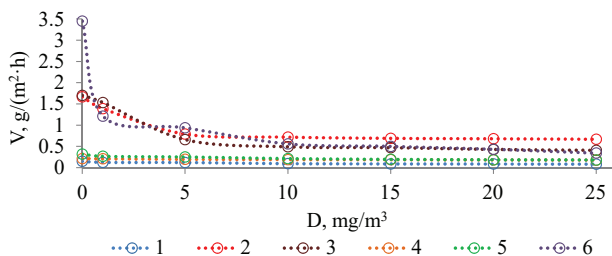


Fig. 2. Dependence of corrosion rate of steel St20 on concentration of the AC-1 inhibitor in a water-oil mixture: 1, 4 – *t*=30 °C; 2, 5 – *t*=60 °C; 3, 6 – *t*=80 °C; 1, 2, 3 – 0.5 g CH₃C(O)OH); 4, 5, 6 – 3.0 g CH₃C(O)OH

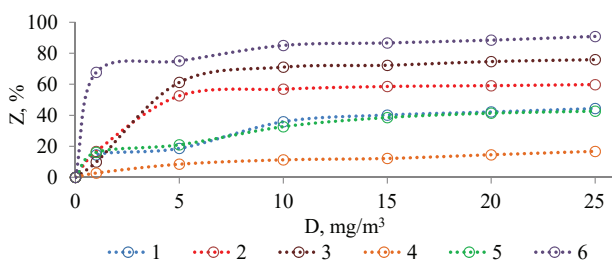


Fig. 3. Dependence of corrosion protection degree of steel St20 on concentration of the AC-1 inhibitor in a water-oil mixture: 1, 4 – *t*=30 °C; 2, 5 – *t*=60 °C; 3, 6 – *t*=80 °C; 1, 2, 3 – 0.5 g CH₃C(O)OH); 4, 5, 6 – 3.0 g CH₃C(O)OH

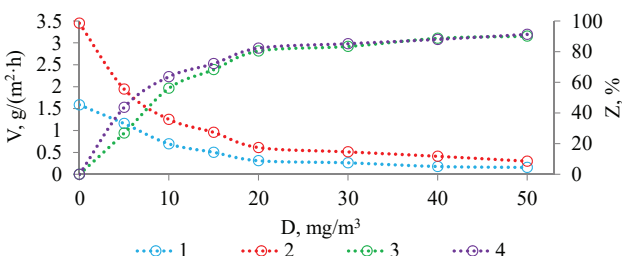


Fig. 4. Dependence of corrosion characteristics of steel St20 on the concentration of the AC-1 inhibitor at a temperature of 80 °C in a water-oil mixture: 1 – 0.5 g CH₃C(O)OH); 2 – 3.0 g CH₃C(O)OH); 1, 2 – corrosion rate, 3, 4 – degree of protection

Thus, one can argue that the synthesized inhibitors based on sunflower oil and polyethylene polyamines containing imidazolines are not inferior in terms of quality to the best known inhibitors of steel corrosion in water-oil mixtures.

5.3. Optimizing the AC-1 reagent dose to ensure effective anti-corrosion protection

Implementing the method necessitates the knowledge of detailed dependences linking the basic parameters of the process under the optimal conditions for its progress. Therefore, we have additionally derived regression equations for the dependence of corrosion rate on the AC-1 inhibitor dose and the amount of CH₃C(O)OH.

Underlying the calculation is a full factorial plan (FFP), type 2². The FFP plan-matrix 2² and the results of the experiment into corrosion rate on the AC-1 inhibitor dose and the amount of CH₃C(O)OH are given in Table 1.

The result of appropriate calculations, after checking the results of the research, estimating the significance of the obtained coefficients, and verifying the regression equation for adequacy, is the following form of the desired dependence:

$$Y = 0,8910 - 0,6611 \cdot X_1 + 0,2305 \cdot X_2 - 0,1604 \cdot X_1 \cdot X_2$$

Table 1

The FFP 2² plan-matrix and the results of studying the extraction of hardness ions from aqueous solutions

Planning matrix		Natural values of factors		Parameter values
<i>x</i> ₁	<i>x</i> ₂	Dose of AC-1, mg/m ³	<i>m</i> CH ₃ COOH, g	<i>V</i> , g/m ² ·h
-1	-1	5.0	0.5	1.1595
+1	-1	50.0	0.5	0.1583
-1	+1	5.0	3.0	1.9445
+1	+1	50.0	3.0	0.3015

After replacing the encoded values in the derived equation with natural values,

$$X_1 = \frac{D((AC-1) - 27.5)}{22.5}$$

$$X_2 = \frac{m(CH_3COOH) - 1.75}{1.25}$$

the following equation regression in the natural form was obtained:

$$Y = 1,0995 - 0,0194 \cdot D(AC-1) + 0,3425 \cdot D(CH_3COOH) - 0,0057 \cdot D(AC-1) \cdot D(CH_3COOH)$$

The resulting dependence is shown in Fig. 5 in the form of a plane hosting the solution to the reduced equation. It shows the dependence of corrosion rate on the AC-1 inhibitor dose and the amount of CH₃C(O)OH.

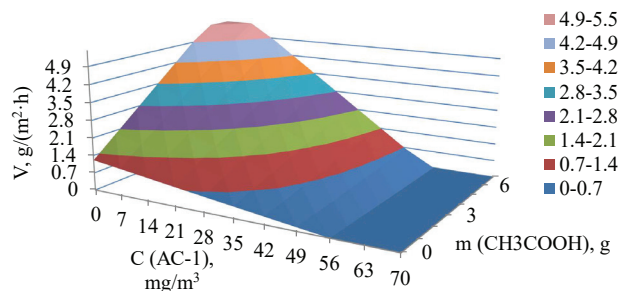


Fig. 5. Graphic representation of results from the type 2² FFP in terms of corrosion rate on the AC-1 inhibitor dose and the amount of CH₃C(O)OH

By using these regression equations, it becomes easy to calculate the optimum dose of a steel corrosion inhibitor in water-oil mixtures depending on the AC-1 inhibitor dose and the amount of CH₃C(O)OH. The obtained results suggest that the corrosion rate decreases with an increased

dose of the inhibitor AC-1, to 70 mg/dm³, while reducing the amount of CH₃C(O)OH.

6. Discussion of results from studying the synthesis of, and estimating, the new corrosion inhibitor in a water-oil mixture

It is known that the corrosion processes of metals in aqueous mineralized media are mainly due to the presence of oxygen. At the complete removal of oxygen from water, unalloyed steels are quite resistant to corrosion in aqueous environments. Such conditions are created in wells and oil pipelines, where in general the anaerobic conditions are maintained. Therefore, in the presence of petroleum products only, with the admixtures of mineralized water in the reducing environment, the corrosion of steel is slow, regardless of the level of water mineralization. Typically, when aerating mineralized waters, their corrosion activity increases by tens of times by stimulating the anode dissolution of the metal and the low stability of the protective layer made from corrosion products, which is mainly formed at a considerable distance from the anode zone. This is contributed to by the solution electrical conductivity.

In water-oil mixtures, corrosion occurs mainly at the expense of hydrogen depolarization, which is contributed to by certain oxidation of water due to the formation and dissolution of carbon dioxide, hydrogen sulfide, and carbonic acids. In this case, the medium pH is often lower than 7, and in some cases decreases to values of 5.3–5.6. It is obvious that at low temperatures in the presence of oil, covering a significant part of the metal surface, the rate of steel corrosion is relatively low and reaches 0.1–0.3 mm/year. When the temperature rises much of the oil desorbs from the metal surface. In addition, there is an improvement in the diffusion of water and carbonic acids to the metal surface, which contributes to increasing the corrosion rate to 33 mm/year (Fig. 2, 3). In the presence of the imidazoline inhibitor, the protection of steel against aggressive environments significantly improves. The imidazoline cycle, as well as the ethylene-amine group, is well adsorbed at the surface of a metal due to the interaction of electronic pairs of nitrogen atoms with the d-orbitals of iron atoms with the formation of stable complexes. At the same time, the hydrophobic radicals of imidazolines adsorb well the hydrophobic components of oil. This results in the reliable protection of steel against contact with the aggressive aquatic environment.

An important advantage of imidazoline inhibitors is their stability at high temperatures (Fig. 4, 5). Due to this, they protect metal equipment against corrosion not only during oil transportation but also in the processes of its processing, which imply the stages of distillation, rectification, conversion, and pyrolysis. In these processes, the imidazo-

line-based inhibitors are used quite extensively. That is why it is interesting to apply the proposed approach to synthesize imidazolines from the waste of available raw materials – sunflower oil using solvents. The use of a solvent in a given process makes it possible to prevent the formation of di- and polyamides whose conversion to imidazolines occurs at temperatures above 300 °C and requires significant energy costs. It should be noted that the proposed method of synthesis implies the multiple uses of a solvent. Products from oil refining could be used as the solvents. Very promising and interesting is to study the corrosion processes of metals in the presence of imidazolines at high temperatures.

This study has addressed the interaction between sunflower oil and diethylene triamine only. It is advisable to use other polyethylene polyamines, first of all, the most affordable and cheapest of them – ethylene diamine. It is expedient to study the effectiveness of the process of polyamine condensation with oil while optimizing the amount of the solvent used and to assess the efficiency of the process when using cheaper solvents. For example, oil refining products – gasoline, petroleum ether, etc. It is also interesting to determine the effectiveness of the inhibitor in watered oil at temperatures above 100 °C. That would make it possible to use these reagents in order to protect equipment against corrosion not only in the oil pipelines but also in the technological processes of oil refining.

7. Conclusions

1. We have devised a new method to synthesize alkyl imidazolines based on sunflower oil and polyethylene polyamines in the Octanol solution, which makes it possible to obtain relatively inexpensive highly effective steel corrosion inhibitors in water-oil mixtures.

2. It has been shown that most known inhibitors are ineffective in mineralized aqueous environments. In water-oil mixtures, the best inhibitors are those based on imidazoline. It has been determined that the synthesized inhibitors based on oil and polyethylene polyamines, which contain imidazolines, ensure, at a dose of 50 mg/dm³, the protection of steel against corrosion at the level of 90–92 %, that is, in terms of quality, they are not inferior to the best known inhibitors of steel corrosion in water-oil mixtures.

3. Based on a full factorial plan, we have derived a regression equation, linear in character, that makes it possible to optimize the calculation of a steel corrosion inhibitor dose in water-oil mixtures. When using the inhibitor in the amount of 40 mg/dm³ in a solution containing acetic acid in the amount of 1.0 g, the corrosion rate is 0.4379 g/(m²·h), and it decreases to 0.3112 and 0.1870, respectively, at the inhibitor dose of 45 and 50 mg/dm³, respectively.

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