Для боротьби з корозією використовують корозійно-стійкі сплави, що вимагає великих затрат і не є достатньо надійним засобом, та інгібітори корозії, які потрібно постійно вдосконалювати для підвищення їх ефективності та зменшення собівартості. Оскільки корозія характеризується руйнуванням матеріалу внаслідок взаємодії із навколишнім середовищем, то одним із доцільних методів її мінімізації є використання інгібіторів. Тому актуальним завданням охорони навколишнього середовища та економічного розвитку країни є розробка ефективних засобів захисту металів від корозії. Отриманий інгібітор АС-2 (розчин суміші 2-алкілімідазолінів в метанолі) досить ефективно захищає сталь від корозії у водно-нафтових сумішах при високих концентраціях мінеральних солей у воді. Ефективність інгібітору практично не залежить від співвідношення об'ємів нафти та концентрованих водних розчинів хлориду натрію. При використанні інгібітору при концентрації 50 мг/дм³ ступінь захисту сталі від корозії перевищував 90 %. Пов'язано це з тим, що захист сталі від корозії відбувається за рахунок сорбції імідазолінових складових на поверхні металу та сорбції органічних складових нафти на гідрофобних алкільних групах 2-алкілімідазолінів. При цьому гідрофобізація поверхні металу відбувається в присутності незначних кількостей нафти. Показано, що у суміші, що містила 200 см³ 3 % розчину хлориду натрію та 800 см³ нафти, при концентрації оцтової кислоти відповідно 0,5 та 3,0 г/дм³, при температурі 80 °C при дозі інгібітору 15–50 мг/дм³, ефективність була 72–92 %. Це дозволяє вирішити проблему раціонального природокористування та забезпечує перехід до використання екологічно безпечних енергоефективних технологій

Ключові слова: інгібітор корозії, 2-алкілімідазолін, водно-нафтова суміш, швидкість корозії сталі, ступінь захисту від корозії

DEVISING A CORROSION INHIBITOR FOR STEEL ST37-2 IN A WATER-OIL MIXTURE

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1. Introduction

Despite the significant development of industrial synthetic substances production, metals remain the main structural material that is indispensable in a series of industries. Given the increase in the volume of metal production, there is an enhanced need to protect them against corrosion [1]. It is worth noting that most domestic oil pipelines are operated under conditions of intensive internal corrosion; the operation of wells is associated with the release of mineral particles, in particular sand, which also leads to the wear of the equipment. High corrosion activity at oil extraction is the feature of water from mines as it is characterized by high mineralization [2, 3]. Crude oil causes the corrosion of steel pipelines since it is heated to a temperature of 80 °C and contains up to 30 % of water when it is pumped [4]. One of the common resource-saving methods of ensuring safe operation of pipelines and increasing their durability is the use of anticorrosive protection. The anticorrosive protection of elements, which are exposed to corrosive influence, is an integral part of the operation of oil pipelines that ensure the uninterrupted functioning of the oil industry equipment and an important element of the environmental safety of production. The most effective method of eliminating corrosion influence is the use of corrosion inhibitors [5, 6]. A permanent monitoring of the corrosion condition of pipelines makes it possible to quickly take measures to protect them against corrosion, determine the service life of the equipment, and perform the repair works in time [7].

Therefore, it is a relevant area of research to find effective corrosion inhibitors in order to reduce corrosion aggressiveness of water-oil mixtures depending on their oil-mineral composition and to define the optimal parameters.

2. Literature review and problem statement

It is known that in mineralized waters the best inhibitors are those of the adsorption type, which are capable of adsorbing in the form of a thin film at the metal surface, thereby significantly reducing the rate of metal oxidation when interacting with water [8].

The authors of work [9] investigated the influence of the *d*-metals ions in the composition with phosphonic acids on the processes of corrosion in a neutral aqueous medium. It was shown that a series of ions, such as zinc, chromium, lead, under high aeration conditions and a temperature of 20 °C at concentrations $2-5$ mg/dm^3, are effective steel corrosion inhibitors. The degree of corrosion protection is maintained at $80-90$ %. It was shown that phosphonic acids, such as oxyethylidene diphosphonic acid and nitrilotrimethylphosphonic acid, provide a degree of corrosion protection at the level of $95-98$ % at acid doses of up to 10 mg/dm^3. At higher temperatures, there is a significant reduction of protective properties due to the destruction of a passivation film at the metal surface when the concentration of oxygen in water is reduced. Consequently, the main drawback of these inhibitors is the reduction of the protective effect at a temperature over 50 °C.

An effective anti-corrosion agent for steel that is used in high-chloride water environments is the «green» organic compounds [10, 11]. The maximum efficacy of inhibition of 94.6 % was achieved using 500 ppm of the inhibitor. However, the cited works [10, 11] considered the selective action only for a neutral water corrosive environment and did not study the efficacy of inhibitors for bi-phase systems that are characteristic of steel corrosion in the petrochemical and oil-refining industries.

It is known that the main components of corrosion inhibitors for the oil-industrial equipment are imidazolines. Imidazolines belong to the corrosion inhibitors of mixed type as they reduce the speed of the anode dissolving of iron and the cathodic reduction of hydrogen. As surfactants, imidazolines have the cation-active properties, are good emulsifiers, and form water-soluble salts. Film-forming and anticorrosion properties contribute to their use in industry as corrosion inhibitors. This is predetermined by that imidazolines adsorb at the metal-solution interface and contribute to the formation of a monomolecular hydrophobic layer that repels water, thereby slowing the rate of steel corrosion. The P110 inhibitor demonstrated an inhibition corrosion efficiency of 97.6 % at a concentration of about 200 mg/dm^3 [12].

At present, there are many known N-, O-, S-containing organic compounds that have proven to be effective corrosion inhibitors for various aggressive environments. More effective for the water-saturated oil deposits saturated with CO_2 is to use the derivatives of heterocyclic compounds, namely, azoles, imidazolines. Two new thiadiazole derivatives were synthesized and investigated as soft-alloy corrosion inhibitors in a water CO_2 saturated oil deposit in paper [13]. It is shown that the corrosion protection efficiency is 98.7 % even at a rather low concentration of 0.0025 mM/dm^3, and reaches 99.37 % at a concentration of 0.025 mM/dm^3. The synthesized inhibitors reported in [14] demonstrate good steel corrosion indicators in the water of reservoir deposits saturated with CO_2. It was determined that the degree of protection increased by increasing the polymer concentration to the maximum values of 92.12 and 96.52 % for 150 ppm at 50 °C in the presence of SR-16 and SR-17, respectively. The findings in [15] showed that the N-, S-containing organic compounds could effectively protect carbon steel against corrosion, while the efficiency of inhibition increases as the concentration of the inhibitor grows, reaching 93 % at 50 mg/l. Even at a low concentration of 10 mg/l, the density of corrosion current decreases from $1.447 \cdot 10^{-4}$ A·cm^{-2} for the model sample to $2.99 \cdot 10^{-5}$ A cm^{-2} after 12 hours of immersion. Paper [16] tackles the effectiveness of imidazoline derivatives as a corrosion inhibitor of steel J55 in the water of a reservoir oil field saturated with CO_2. The effectiveness of inhibition is up to 90 % at 400 mg/l. The addition of an inhibitor reduces the density of anode current, as well as the anode current, and significantly enhances the parameters of electrochemical impedance spectroscopy. The efficiency of a soft steel corrosion inhibitor in a 1.0 M HCl solution with a temperature from 25 °C to 90° was 99.2 % [17]. A major disadvantage of these inhibitors is the high prices for relatively significant expenditures in the corrosive environment [12–17]. Therefore, the unresolved issue is to devise effective affordable corrosion inhibitors for metals in water-organic environments.

Study [18] synthesized alkylimidazolines with the use of sunflower oil and diethylene triamine. However, if we consider that diethylene triamine price reaches USD 56/kg and the price of pure ethylenediamine is USD 48/kg while the consumption of ethylenediamine to produce 1 kg of imidazoline is 30 % less, it would be advisable to perform the synthesis using ethylenediamine.

The issue of eliminating corrosion of steel, given a significant quantitative and qualitative range of solutions in the water-oil environment, is quite complicated and relevant. In addition, the ever-increasing requirements for environmental safety and economic feasibility of oil and gas equipment predetermine the search for more effective, less toxic, and cheap corrosion inhibitors. Therefore, studies are aimed at developing corrosion inhibitors that have a high protective effect. These inhibitors meet environmental and economic requirements and are widely available for industrial use.

3. The aim and objectives of the study

The aim of this study is to synthesize a new inhibitor that would effectively protect steel against corrosion in water-oil mixtures at high concentrations of mineral salts in water.

To accomplish the aim, the following tasks have been set:
– to determine the effectiveness of the synthesized imidazolines using EDA, depending on the ratio of the volumes of oil and the concentrated aqueous solutions of sodium chloride;
– to determine the rate of corrosion of St37-2 steel and to establish the optimum dose of the synthesized inhibitor AC-2 in a water-oil mixture in the presence of acetic acid at a temperature of 80 °C.

4. Synthesis and evaluation methods of the reagent AC-2 as a steel corrosion inhibitor in water-oil mixtures

For the synthesis, a mixture of 50 % of ethylenediamine (EDA) water solution with oil in Octanol 1 was used.

We added to 950 g of oil 240 g of a 50 % EDA solution and 500 cm³ of Octanol 1. The mixture, at stirring, was gradually heated to 190 °C in a reactor, distilling water and an excess of amine with octanol impurities. After 6-hour heating, when the water ceased to distill, we distilled octanol in a vacuum. The remaining light-brown liquid, which, after the dissolution in methanol, was used as an inhibitor (AC-2). The water-amine-octanol mixture was distilled by sampling a fraction with a boiling point of 116 °C. The resulting aqueous solution of ethylenediamine, as well as the distilled Octanol 1, is suitable for reuse. The resulting inhibitor is well-soluble in methanol, acetone, benzene, chloroform, and other solvents. Given that it was mainly used in the emulsions of water and oil, we selected methanol as a solvent. Excess ethylenediamine is required to prevent the formation of diamines of carboxylic acids, whose conversion into imidazolines requires maintaining high temperatures (>300 °C), which sharply reduces the yield of the main products.

The process of alkylimidazoline synthesis is implemented in two stages (Fig. 1).

$$CH_2-O(O)\,CR_1$$
$$|$$
$$CH-O(O)CR_2 + nNH_2CH_2CH_2NH_2 \rightarrow R_1(R_2,\,R_3)C \qquad\qquad \begin{matrix} O \\ // \\ \\ \backslash \\ NHCH_2CH_2NH_2 \end{matrix} +$$
$$|$$
$$CH_2-O(O)CR_3$$

$$+ CH_2OHCHOHCH_2OH + (n-1)\,NH_2CH_2CH_2NH_2 \qquad (1)$$

$$R_1(R_2,\,R_3)C \begin{matrix} O \\ // \\ \\ \backslash \\ NHCH_2CH_2NH_2 \end{matrix} \xrightarrow{T,^0C} R_1(R_2,\,R_3)C \begin{matrix} N-CH_2 \\ // \\ | \\ \backslash \\ N-CH_2 \\ | \\ H \end{matrix} + H_2O \qquad (2)$$
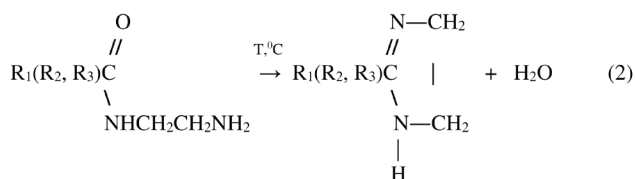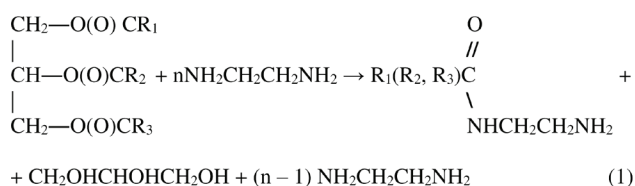
Fig. 1. The reaction of alkylimidazoline synthesis

The samples of steel St37-2 were used for the current work. Corrosion was studied by a gravimetric method. The corrosive environment used was a water-oil mixture (the ratio of oil to a 3 % sodium chloride solution is 80:20, 20:80, 5:95) and a mixture that contained 200 cm³ of a 3 % sodium chloride solution, 800 cm³ oil at the concentration of acetic acid of 0.5 and 3 g/dm³. The temperature was 80 °C. We changed the inhibitor concentration from 5 to 50 mg/dm³. Corrosion time is 8–10 hours.

To confirm the reaction mechanism, we have synthesized 2-alkylimidazoline on the basis of ethylenediamine and enanthic acid in the Octanol 1 solution, under conditions identical to those when obtaining the inhibitor AC-2. The yield of imidazoline after the recrystallization from hexane is 69 %, so in a given case we received an individual product, rather than a mixture, in contrast to AC-2. The PMR spectrum was acquired and analyzed using the «TeslaBS-487s» spectrophotometer in a DCl_3 solution. The internal standard is GMDS.

The characteristics of the obtained 2-hexylimidazoline: The melting point T_m=58 °C, PMR, σ, ppm: 0.80 t (3H CH₃), J_{CCH}=7.15 Hz; 0.98–1.56 m (8H CH₂); 2.01 t (2H CH₂); J_{CCH}=7.97 Hz; 3.38 c(4H CH₂); 5.80 s (1H NH).

The steel samples of 2×3 cm with a thickness of 1 mm were mechanically cleaned from corrosion products by grinding paper and sanded to metallic luster. Next, the metallic samples were degreased in ethanol, dried in a drying chamber

at 90–100 °C, and weighed on analytical scales with an accuracy of the fourth character.

Following the corrosion test, the St-37-2 samples were cleaned from petroleum products in a solution of ethanol, aged for 30 minutes in 0.5 % sulfuric acid in the presence of urotropine (C=10 g/dm³). Next, the corrosion products were completely removed by rubber, the samples were washed in ethanol and dried in a drying chamber. After that, we re-weighed them with the accuracy of the fourth character.

The corrosion rate and corrosion protection level were calculated from formulae [18].

$$V = \frac{m_1 - m_2}{S \cdot t},\ \ g/(m^2 \cdot h), \qquad (1)$$

$$Z = \left(1 - \frac{V_i}{V_0}\right) \cdot 100\,\%, \qquad (2)$$

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

5. Results of studying the inhibitor effectiveness

5. 1. Determining the effectiveness of the synthesized imidazolines using EDA (AC-2)

Our study determined the efficacy of the inhibitor synthesized in the condensation of oil with ethylenediamine (AC-2). Fig. 2 shows that a given inhibitor was quite effective in a water-oil mixture with a volumetric share of oil of 80 % and a 3 % solution of NaCl 20 %. In the absence of acid in the mixture, even at 80 °C, the rate of corrosion of steel St37-2 was relatively small and reached 0.3191 mm/year. The inhibitor was used in concentrations from 5 to 50 mg/dm³. When using the AC-2 inhibitor, the protection level reached 88 % at a concentration of 5 mg/dm³. At 25 mg/dm³, the degree of protection exceeded 90 %.

High was the efficacy of the inhibitor in a water-oil mixture medium containing in 1 dm³ 800 cm³ 3 % of the solution NaCl and 200 cm³ of crude oil (the ratio of oil to water in the mixture was 2:8). At a temperature of 80 °C, the degree of protection against corrosion of steel St37-2 at a concentration of the inhibitor of 5 mg/dm³ reached 66–68 % (Fig. 3), and at 50 mg/dm³ the degree of protection for the inhibitor exceeded 90 %. A somewhat lower was the efficacy of the inhibitor in an environment that consisted of 95 % of the solution of 3 % sodium chloride and 5 % of oil (Fig. 4).
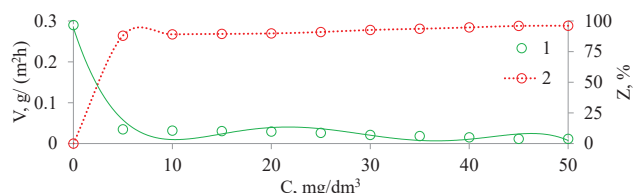

Fig. 2. Dependence of the characteristics of corrosion of steel St37-2 on the concentration of the AC-2 inhibitor at 80 °C in a water-oil mixture (80 % oil and 20 % of 3 % solution NaCl): 1 — rate of steel corrosion; 2 — degree of protection against corrosion; $y = -3.9 \cdot 10^{-08} x^5 + 5.604 \cdot 10^{-06} x^4 - 2.97258 \cdot 10^{-04} x^3 + 7.095380 \cdot 10^{-03} x^2 - 7.3801132 \cdot 10^{-02} x + 0.283422395$; $R^2 = 0.97549$
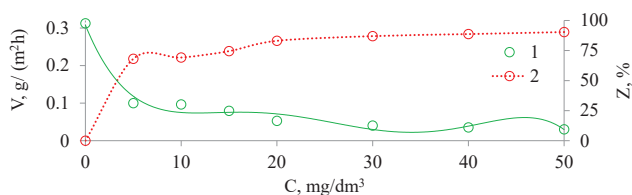
Fig. 3. Dependence of the characteristics of corrosion of steel St37-2 on the concentration of the AC-2 inhibitor at 80 °C in the environment of a water-oil mixture (20 % oil and 80 % of 3 % solution of NaCl): 1 — rate of corrosion of steel St37-2; 2 — degree of protection against corrosion; 1: $y=-4.25\cdot10^{-08}x^5+5.73\cdot10^{-06}x^4-2.818479\cdot10^{-04}x^3+6.2268652\cdot10^{-03}x^2-6.26596094\cdot10^{-02}x+0.3078147804$; $R^2=0.97846$
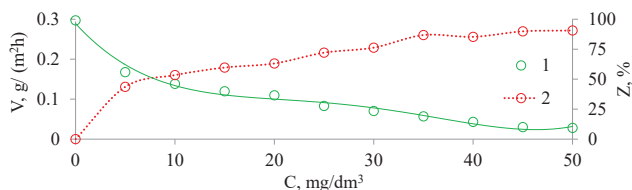

Fig. 4. Dependence of the characteristics of corrosion of steel St37-2 on the concentration of the AC-2 inhibitor at 80 °C in the environment of a water-oil mixture (5 % oil and 95 % of 3 % solution of NaCl): 1 — rate of corrosion of steel St37-2; 2 — degree of protection against corrosion; 1: $y=-1.5\cdot10^{-08}x^5+2.225\cdot10^{-06}x^4-1.20930\cdot10^{-04}x^3+2.989695\cdot10^{-03}x^2-3.616078\cdot10^{-02}x+0.294878$; $R^2=0.98681$

In this case, at a concentration of the inhibitor of 5 mg/dm³, the degree of protection of steel St37-2 against corrosion for the AC-2 inhibitor was 43.6 %. At a concentration of 50 mg/dm³, the degree of protection of steel against corrosion reached 90 %.

5. 2. Determining the efficacy of the synthesized AC-2 inhibitor in a water-oil mixture in the presence of acetic acid

Further study determined the efficacy of the AC-2 reagent in acetic acid-containing environments. Thus, the mixture that contained 200 cm³ of a 3 % sodium chloride solution and 800 cm³ of oil at the concentration of acetic acid, respectively, of 0.5 and 3 g/dm³ at a temperature of 80 °C demonstrated quite high efficacy of the inhibitor. At the concentration of acetic acid of 0.5 g/dm³ for a dose of the inhibitor of 10 mg/dm³, the corrosion rate decreased from 1.6 to 0.7 g/m²·h (Fig. 5).
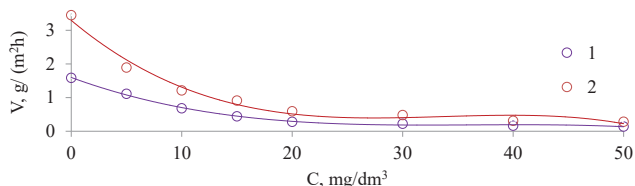

Fig. 5. Dependence of the rate of corrosion of steel St37-2 on the concentration of the AC-2 inhibitor in a water-oil mixture: 1 — 0.5 g CH₃C(O)OH; 2 — 3.0 g CH₃C(O)OH; 1: $y=-3\cdot10^{-05}x^3+0.00334x^2-0.11979x+1.59891$; $R^2=0.999778$; 2: $y=-8\cdot10^{-05}x^3+0.00846x^2-0.27474x+3.29281$; $R^2=0.98247$

The degree of protection against corrosion under these conditions reached 56–57 % (Fig. 6). At a concentration of the inhibitor at the level of 50 mg/dm³, the degree of protection reached 90–91 %. The high results were also obtained with the use of the AC-2 inhibitor in a mixture of water and oil containing 3 g/dm³ of acetic acid. At the inhibitor doses of 10–50 mg/dm³, the degree of corrosion protection of steel St37-2 reached 64–92 %.
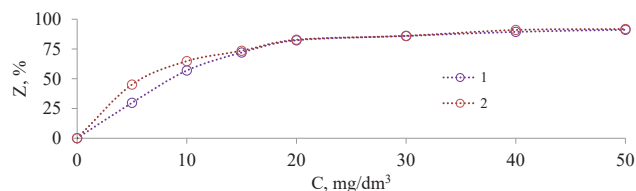

Fig. 6. Dependence of the degree of protection against corrosion of steel St37-2 on the concentration of the AC-2 inhibitor in a water-oil mixture: 1 — 0.5 g CH₃C(O)OH; 2 — 3.0 g CH₃C(O)OH

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

6. Discussing the results of the process of synthesis and estimation of the effectiveness of the AC-2 inhibitor (a mixture of 2-alkylimidazolines)

It is known that the synthesis processes of 2-alkylimidazolines, when using relatively cheap ethylenediamine and carboxylic acids, are implemented rather inefficiently. This is because carboxylic acids, when mixed with EDA, form ammonium salts on both amine groups, regardless of the excess of amine. Another issue is the fact that when an EDA mixture with carboxylic acids is heated at a temperature of >116 °C the EDA azeotrope passes, along with water, into a steam phase. This results in that the excess of amine in the reaction zone is hard to with the basic product of the process being the thermally resistant diamides of EDA and carboxylic acids. When the latter transform to imidazolines, they must be heated to 400 °C. It is obvious that this significantly increases energy costs for the process. In addition, a large part of the products decomposes at high temperatures, which considerably reduces the yield of the main product, which, even at the perfect implementation of the process, cannot theoretically exceed 50 % of the amount of the used carboxylic acid.

The processes occur better when using the esters of carboxylic acids. However, given the high price of the higher carboxylic acids and the costs on its esterification, obtaining imidazolines in this way could prove to be too expensive.

Much cheaper is to use oil, especially technical fractions, which are much cheaper than carboxylic acids. In addition, oils are a complex ether of carboxylic acids and glycerin. Therefore, when mixing it with EDA, ammonium salts do not form. Furthermore, the formation of EDA amides and carboxylic acids when using the esters of the latter proceeds much easier, at lower temperatures, than when applying pure acids. That is, the first stage of the process can be implemented at temperatures below 100 °C with the excess of EDA in the reaction mixture. This contributes to the formation of mostly monoamides that can easily condense with the formation of imidazolines.

The formation of monoamides and imidazolines is also contributed to by the use of a solvent – Octanol 1. This is primarily preventing the formation of diamides. In addition, octanol makes it possible to maintain the temperature in a reactor at the level of 190 °C, regardless of excessive EDA and the amount of water. Water is easily distilled in the form of azeotrope from the reaction zone, which assists in the acceleration of the process of formation of monoamide and the acceleration of its cyclization to imidazoline.

In general, when conducting reactions in solutions, the intramolecular transformations proceed better when the concentration of the solution is lower.

The high product yield is also contributed to by the use of oil in which carboxylic acids have organic radicals C_{16}–C_{20}. This creates steric barriers to attack the free ammonium group of EDTA monoamide and glycerol ester carboxylic acid.

Judging by the ratio of signals in the PMR spectra of the product in the region of 3.4 ppm (the protons of CH_2 groups of the imidazoline ring) with the intensity of signals of alkyl groups, it can be said that in a given case (with a five-fold excess of EDA), there occurs an almost full conversion of oil to imidazoline. The inhibitor itself, given that the oil molecules contain the components of various carboxylic acids, mainly consists of a mixture of 2-alkylimidazolines (Fig. 7).
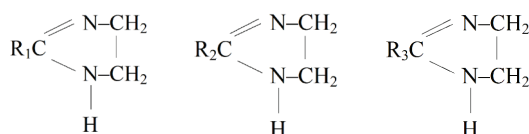

Fig. 7. A mixture of 2-alkylimidazolines included in the composition of AC-2

At a temperature of 80 °C, the degree of protection against the corrosion of steel at a concentration of the inhibitor of 50 mg/dm^3 exceeded 90 % (Fig. 2–4). The efficiency of the AC-2 reagent in acetic acid-containing environments amounted to 92 % (Fig. 5, 6).

Since in the water-oil mixtures the best inhibitors are those based on imidazoline, we chose the AC-1 inhibitor for comparison, which is quite close to the resulting inhibitor AC-2. The synthesis of the AC-1 inhibitor involved diethylenetriamine, which is much more expensive than ethylenediamine, which was used for the synthesis of the AC-2 inhibitor. At the concentration of acetic acid of 0.5 g/dm^3 at a dose of the inhibitor AC-1 of 50 mg/dm^3 the corrosion rate decreased from 1.5872 to 0.1583 g/(m^2·h) [18]; when using the AC-2 inhibitor, this indicator decreased to 0.1412 g/(m^2·h). When using the inhibitor in a water and oil mixture containing 3 g/dm^3 of acetic acid, the corrosion rate decreased from 3.4508 g/(m^2·h) without the inhibitor to 0.3015 g/(m^2·h) when using the AC-1 inhibitor in the amount of 50 mg/dm^3 [18] and to 0.2845 g/(m^2·h) when using the AC-2 inhibitor (Fig. 5). Thus, it can be concluded that the inhibitor synthesized in the current work is more efficient and cheaper.

The advantage of a given inhibitor is the fact that 2-alkylimidazolines at elevated temperatures are easily distilled with petroleum products and water vapors. Therefore, they can be used not only in oil transportation but also at its processing.

This study addressed the effectiveness of the steel corrosion inhibitor AC-2 only, at a temperature of 80 °C. It is important to investigate and to determine the effectiveness of the synthesized inhibitor at higher temperatures. Therefore, the subject of further research is to study the effectiveness of the developed steel corrosion inhibitor AC-2 at elevated temperatures (100–400 °C) in the steam-gas phase with the impurities of water vapor. That would make it possible to use the inhibitor both in oil pipelines and in the technological processes of oil refining.

7. Conclusions

1. It has been shown that the imidazoline-based inhibitors are quite effective in the water-oil mixtures. We have devised a method to synthesize alkylimidazolines on the basis of oil and ethylenediamine in the octanol solution, which makes it possible to obtain highly effective corrosion-resistant steel inhibitors in water-oil mixtures at the high concentrations of mineral salts in the water. The effectiveness of the inhibitor almost does not depend on the ratio of the volumes of petroleum and the concentrated aqueous solutions of sodium chloride. At a concentration of the inhibitor of 50 mg/dm^3, the degree of protection of steel against corrosion reached 90–92 %.

2. It has been established that the efficiency of the AC-2 reagent in acetic acid environments is rather high. At a dose of the inhibitor of 10 mg/dm^3 at a temperature of 80 °C, the degree of corrosion protection reached 57–65 %, while increasing the dosage of the inhibitor to 15–50 mg/dm^3 increases the protection degree to 72–92 %.

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