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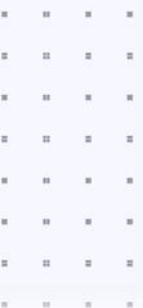
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SYNERGISTICALLY ACTIVE BASE SYNTHESIS FOR CORROSION INHIBITORS

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Abstract. The temperature dependence and catalytic method of synthesis of imidazoline derivatives have been compared. It has been found that fatty acids and ethylenediamine, which are part of cottonseed oil, are achieved by the formation of a 1:1 ratio and monoamides and imidazoline derivatives acting at temperatures 190 and 240°Ct. Compositions have been prepared by adding the vacuum distillation cubic residue of methanol, formaldehyde, and mono-ethanolamine into the composition of the products obtained, and it has been determined that with an increase in the amount of active base, the degree of corrosion protection also increases. The degree of corrosion protection is high when the amount of synergistically effective additives methanol and formaldehyde was up to 10 g, while the amount of MECRVD was 5 g. As keeping the samples under conditions with a dose of hydrochloric acid of 15 and 23%, the inhibitor concentration was 99.6 and 99.01%, respectively, when the concentration of the inhibitor was 10 g/l.

Keywords: heterocycles, imidazoline derivatives, carboxylic acid ammonium salts, fatty acids, ethylenediamine, synergistically active base, corrosion inhibition

One of the important problems of today's oil-gas and chemistry industry is related to the creation of new corrosion inhibitors for construction materials and increasing the efficiency of their use. The weight of metal losses due to corrosion is increasing year by year, and the amount of expenses due to accidents in pipelines, industrial equipment, and devices is increasing. In an aggressive environment, it is important to obtain polyfunctional heterocyclic compounds that have the activity of preventing the corrosion of device materials.

In the period of rapid development of chemistry and oil-gas processing industries in the world, scientific research is being conducted on the methods and structure of organic inhibitors, the nature of corrosion processes, and the protection of structural materials in hydrocarbon production and processing processes. In this regard, certain results have been achieved in the direction of the production of new-generation organic corrosion inhibitors, including the provision of chemical products that substitute imports.

Measures are being implemented in the field of creation and production of highly effective corrosion inhibitors based on local raw materials and secondary material resources in our republic, and certain results are being achieved. It is important to carry out these tasks, including the development of resource-efficient technologies based on domestic raw materials and secondary products, and the creation of an effective corrosion inhibitors composition as an import substitute.

A great deal of experience has been accumulated in the preparation of polyfunctional heterocyclic compounds from multi-ton olefins, glycols, amines, etc., capable of preventing the decomposition of metallic materials in an aggressive environment [1-5].

Methods for obtaining 1-aryl imidazoles (imidazoles and benzimidazole) as synergistically active bases for multicomponent corrosion inhibitors are described in Grimmett's monography and "Complex heterocyclic chemistry" journals [6-9], producing 4-methylphenyl-sulfonyl-methylisocyanide based on aldimines, imidoyl chlorides, nitriles, isocyanates, or imino ethers [10,11], in 1,5-exchanged 4-chloro-and 4 - there are reports of the use of bromimidazoles [12,13], 5-amino-4-tsian-1-arylimidazole [14], 1-aryl-5-aminoimidazole-4-carboxamide [15, 16], and amino alcohols [17].

In order to obtain synergistically active bases of imidazoline derivatives, the reactions of ethylenediamine and oleic acid have been studied under different conditions: in a reactor equipped with a mechanical stirrer, a dropping funnel, a thermometer, and Dina-Stark nozzles, a solvent (as a solvent, pure benzene and an aromatic hydrocarbon mixture (benzene, toluene) and xylols) are used), and fatty acids are added with intensive mixing. As the reactor temperature increases by 60°C, a kind of mass is produced. Heating lasts for ~1 hour. Ethylenediamine is then rapidly transferred to the reactor through the dropping funnel. In this case, the temperature rises to 100°C,

and the color of the mixture changes from pale yellow to dark red or black due to an exothermic reaction.

Then the reaction mixture is heated in the temperature range of 130-150 °C for 3 hours: it is observed that the mixture of water and solvent (aromatic hydrocarbons) is collected from the reactor in the Dina-Stark collector. The solvent collected in the collector is returned to the reactor. After that, the process is continued by heating the reaction mixture at a temperature range of 160-165 °C for three hours. In this case, the accumulation of water in the Dina-Stark collector cottage 2. The separation of water from ammonium salts of carbonic acid and the formation of amides continues.

The resulting mixture is heated at a temperature of 225°C for one hour. Here, complete separation of water is carried out. After that, the reaction mixture is cooled and the degree of protection against metals is determined.
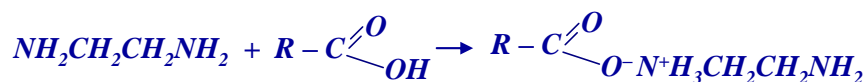
The synthesis of imidazoline derivatives is carried out in different proportions of fatty acids and ethylenediamine.

$$NH_2CH_2CH_2NH_2 \; + \; R-C\underset{OH}{\overset{O}{\diagup}} \longrightarrow \; N\diagdown\diagup N-H \; + \; H_2O$$
$$R$$
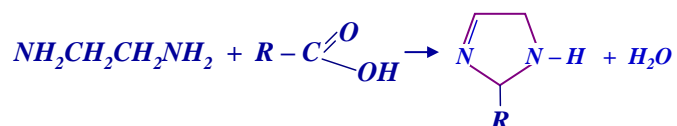
It has been proven that the synthesis of imidazoline goes through the step of formation of linear amides. Depending on the reaction conditions, in the first stage, monoamides and diamides of fatty acids are produced, which ensure the formation of the imidazoline ring.

As mentioned above, the synthesis of imidazoline derivatives is multi-step and involves the formation of many intermediate products. The sequence of the process can be shown by the following chemical reaction schemes:

Stage 1. Formation of ammonium salts of fatty acids with ethylenediamine:

$$NH_2CH_2CH_2NH_2 \; + \; R-C\underset{OH}{\overset{O}{\diagup}} \longrightarrow \; R-C\underset{O^-N^+H_3CH_2CH_2NH_2}{\overset{O}{\diagup}}$$

Stage 2. Separation of water from ammonium salts of carbonic acid and formation of amides:

$$R-C\underset{O^-N^+H_3CH_2CH_2NH_2}{\overset{O}{\diagup}} \longrightarrow \; R-C\underset{NHCH_2CH_2NH_2}{\overset{O}{\diagup}} \; + \; H_2O$$
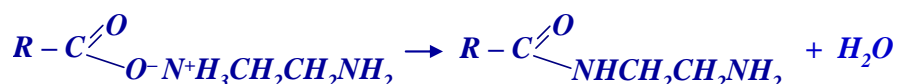
Stage 3. Cyclization of carboxylic acid amides and formation of imidazoline derivatives:

$$R-C\underset{NHCH_2CH_2NH_2}{\overset{O}{\diagup}} \; \Longleftrightarrow \; N\diagdown\diagup N-H \; + \; H_2O$$
$$R$$
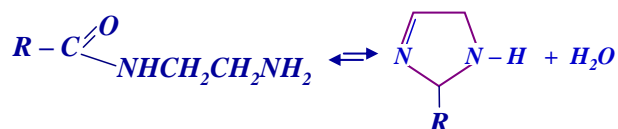
The amount of the separated reaction water has been determined and compared with the calculated amounts (the calculated amount was obtained with respect to the complete conversion of imidazoline).

Table 1

Amount of water released in imidazoline synthesis

Reaction conditions	Separated reactive water	Water whose separation is calculated
after the first stage of two-stage thermal processing	4.3	7.2
after the second stage of two-stage thermal processing	5.3	7.2
in the presence of an acidic catalyst	6.5	7.2

The comparative ratios of the reaction fraction and calculated water content (Table 1) confirm that the starting reagents are carried out with high conversion to the target imidazoline. Although the ratios are large when heat treatment is carried out at a temperature of 190°C, at a temperature of 230°C, a sharp increase in conversion leads to an increase in the yield of imidazoline formation.

Since the determination of the acid number of the produced substances is one of the important parameters indicating the degree of conversion of fatty acids, we considered it the goal to determine the same indicators during our research. Since temperature is the main influencing factor of synthesis, the determination of the acid number can be carried out at an optional step of the synthesis.

Table 2

Amount of water released in imidazoline synthesis
The calculated amount of separated water is 7.2 CON/g

Reaction conditions	Acid number, Con/g
after the first stage of two-stage thermal processing	4.3
after the second stage of two-stage thermal processing	4.5
in the presence of an acidic catalyst	8.1

The analysis results showed that the acid number is 4-5 mg CON/g at 190 and 230°C, and it is KC = 8.1 KOH/g for the product obtained with the presence of a catalyst, and we have considered this to be related to the acidic nature of the catalysis process.

Another method of determining the composition of reaction products is the use of IR-spectroscopy. Figures 1 - 3 show the IR-spectra of the mixture of fatty acids included in cottonseed oil and the products obtained from the reaction of ethylenediamine ("Agilent Technology FTIR-640" device: recording range 4000-400 cm^{-1}, the number of cups - 12).

There have been observed that the absorption lines at 1605 cm^{-1} C = N-bond, and absorption lines at 1650 and 1550 cm^{-1} C = O-bond corresponding to linear amides in all conditions of the synthesis.
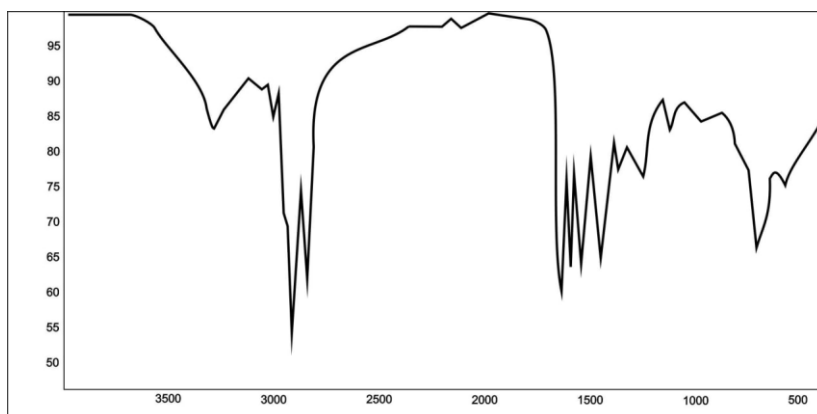

Figure 1. IR-spectrum of the sample obtained at the temperature of 190°C
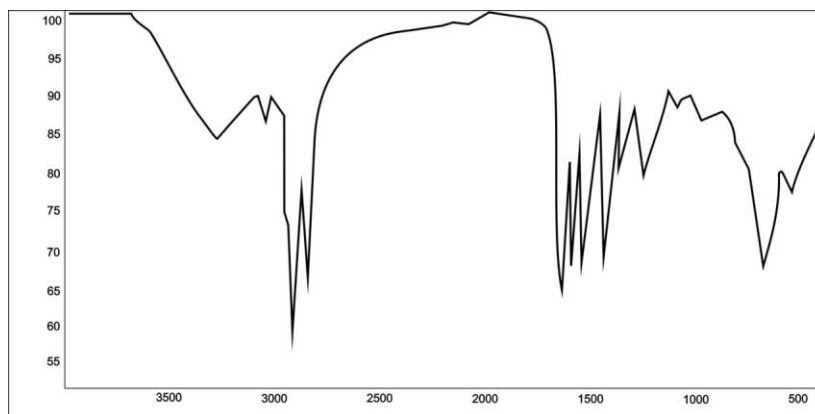
Figure 2. IR-spectrum of the sample obtained at the temperature of 240°C

In the IR-spectrum of the sample taken at a temperature of 190°C (Fig. 1), the characteristic absorption lines for the C=N-bond are less observed in the IR-spectrum of samples taken at a temperature of 230 °C (Fig. 2). C=N-bond intensive absorption characteristic of the imidazoline group is greater in the sample obtained by acid catalysis. Here, we can see that the intensity of absorption for C = N - and C = O-bonds is interrelated, as the intensity of one bond increases, the intensity of the other decreases. It is possible to prove the existence of imidazoline derivatives by comparing the intensities of the C=N- bond in the IR-spectra of the studied samples under different conditions.

The structure of imidazoline derivatives obtained from a mixture of fatty acids has also been studied by ^{1}H NMR-spectroscopy (Fig. 3).

In the ^{1}H NMR-spectrum in Fig.3, the chemical shift of the proton of the N-H - group attached to the carboxyl group is 1.45-1.92 ppm. are observed as singlet signals. The doublet signals of the protons of the imidazoline ring are 7.09-7.22 ppm. seen in the fields. The proton signals corresponding to imidazoline methylene are 4.42-4.47 ppm. in the field. The proton signals of the methylene group belonging to the carboxyl group are in the branches 2.2 - 2.4, and the chemical shift of the protons of the methylene group belonging to carbonic acid is 5.34 ppm. seen in the fields. Many signals not found in the analysis have been observed and related to substances in the solvent mixture.
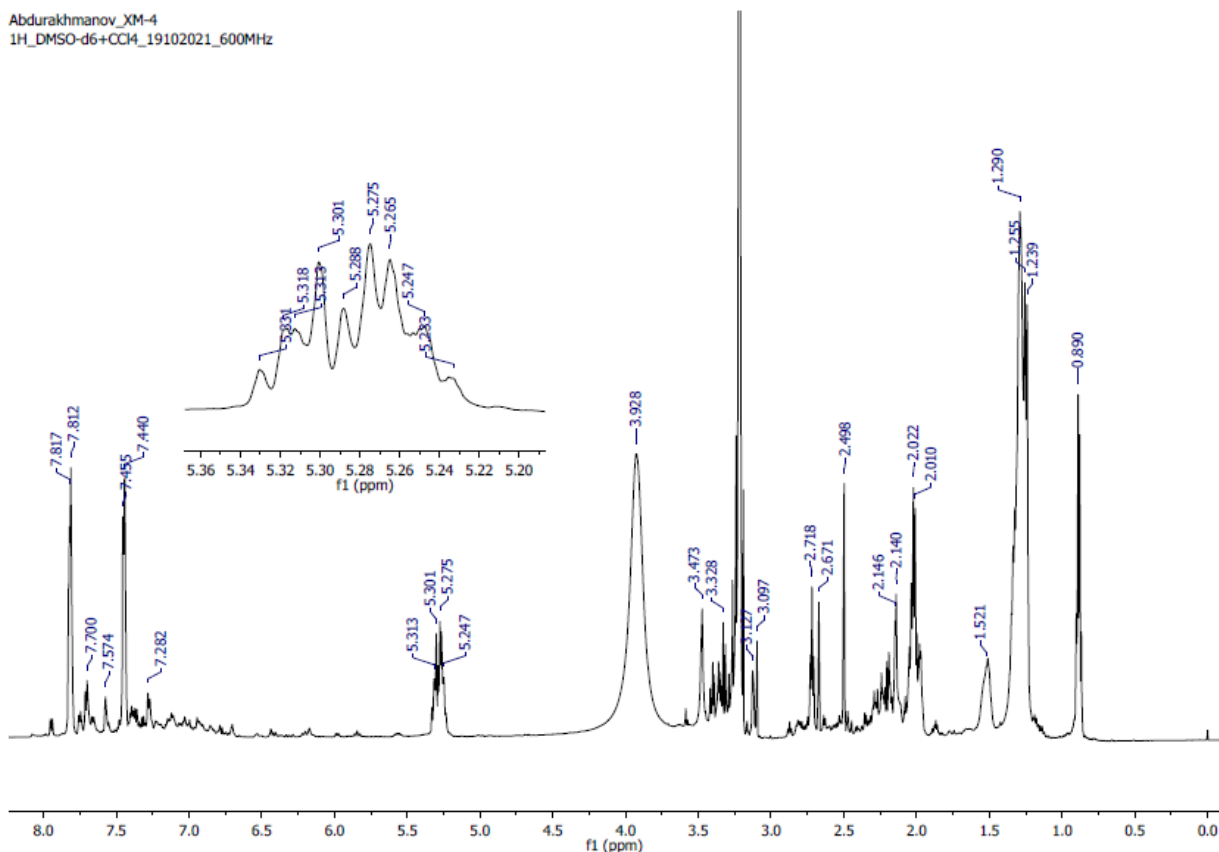
Figure 3. ^{1}H NMR-spectrum of the sample obtained by acid catalysis

Thus, the temperature dependence and catalytic method of synthesis of imidazoline derivatives have been compared. It has been determined that the synthesis of monoamides and imidazoline derivatives goes under the conditions of the 1:1 ratio of fatty acids and ethylenediamine included in cottonseed oil. The ratio of monoamides and imidazoline derivatives depends more on the temperature: imidazoline derivatives have been proven to be formed with high efficiency under acid catalysis carried out at relatively high temperatures of 230°C or 130 - 140°C under solvent boiling temperatures.

Conditional names of inhibitors based on imidazoline derivatives: "IIK-D2" - a reaction mixture with a maximum temperature of synthesis of 190°C; "IIK-D3" is the reaction mixture with the maximum temperature of synthesis of 240°C, and determination of their level of corrosion protection has been carried out by gravimetric and potentiometric methods on St.20 mark steel samples. The inhibitor dose has been set at 20 - 40 mg/l. The experiments have been carried out for 24 hours at room temperature, in two-phase systems of gas condensate-saline artificial solution and oil-saline artificial solution. The analyzed media were enriched with hydrogen sulfide for 10 minutes. The composition of the mineralized salt solution consists of KCl (163 g/l) + $CaCl_2 \cdot 2N_2O$ (34 g/l) $MgSO_4$ (0.14 g/l). Table 3 shows the results of determining the level of corrosion protection of St.20 mark steel by inhibitors based on imidazoline derivatives.

Table 3

The results of determining the degree of corrosion protection of St.20 mark steel of the inhibitor based on imidazoline derivatives*

Experiment environment	Mass loss, g	Corrosion rate g/m²·h	Protection level, %
"IIK-D2"			
Control	0,0255	0,0331	
H$_2$S brine + 70 ml gas condensate	0,0004	0,00519	84,3
Control	0,0012	0,01557	-

H$_2$S brine + 70 ml oil	0,0013	0,01428	91,6
Control	0,0107	0,1388889	-
H$_2$S + brine	0,00115	0,0149273	89
"IIK-D3"			
Control	0,00306	0,3952	-
1:2 = water: brine with H$_2$S	0,0053	0,00365	90,7
Control	0,0051	0,196	-
H$_2$S brine + 70 ml oil	0,00215	0,02791	85,8
Control	0,001	0,01298	-
H$_2$S + brine	0,00015	0,00128	90,1

In the recommended composition, "IIK-D3" has been selected as a synergistically active base, and compositions have been prepared by adding formaldehyde and monoethanolamine cube residue in the vacuum-distillation (MECRVD):

Active base   15,0 – 20,0   Formaldehyde   5,0 – 15,0   Water   40 – 70
Methanol   5,0 – 15,0   MECRVD   5,0 – 10,0

The results of research on determining the degree of corrosion protection of inhibitors of synergistically effective compositions are presented in Tables 3 and 4.

Table 3

The degree of corrosion protection of synergistically effective compositions, the aggressive environment is 15% H$_2$S-, and the temperature is 24 °C

№	Inhibitor composition (g)	Inhibitor dose (g/l)	Corrosion rate, (g/m2)	Protection level, %
1	Active base - 15,0 Methanol - 15,0 Formaldehyde - 15,0 MECRVD - 10,0 Water - the rest	2	0.48	96.7
		4	0.47	96.8
		6	0.38	97.4
		8	0.34	97.7
		10	0.27	98.2
2	Active base - 15,0 Methanol - 5,0 Formaldehyde - 15,0 MECRVD – 10,0 Water – the rest	2	0.45	97.0
		4	0.44	97.01
		6	0.40	97.03
		8	0.36	97.6
		10	0.35	97.62
3	Active base – 17,0 Methanol – 15,0 Formaldehyde - 5,0 MECRVD - 10,0 Water – the rest	2	0.46	96.9
		4	0.44	97.01
		6	0.37	97.5
		8	0.34	97.7
		10	0.21	98.6
4	Active base – 17,0 Methanol – 10,0 Formaldehyde - 10,0 MECRVD – 10,0 Water – the rest	2	0.34	97.7
		4	0.30	97.96
		6	0.28	98.1
		8	0.24	98.4
		10	0.20	98.6
5	Active base – 18,0 Methanol – 5,0 Formaldehyde - 5,0 MECRVD – 5,0 Water – the rest	2	0.28	98.1
		4	0.27	98.2
		6	0.19	98.7
		8	0.14	99.0
		10	0.10	99.3

6	Active base – 18,0 Methanol – 10,0 Formaldehyde - 10,0 MECRVD – 5,0 Water – the rest	2	0.27	98.2
		4	0.24	98.4
		6	0.17	98.84
		8	0.13	99.1
		10	0.06	99.5
7	Active base – 20,0 Methanol – 15,0 Formaldehyde - 15,0 MECRVD – 10,0 Water – the rest	2	0.26	98.23
		4	0.24	98.4
		6	0.20	98.6
		8	0.19	98.7
		10	0.15	98.9
8	Active base – 20,0 Methanol – 10,0 Formaldehyde - 10,0 MECRVD – 10,0 Water – the rest	2	0.27	98.2
		4	0.25	98.3
		6	0.23	98.4
		8	0.21	98.6
		10	0.18	98.8

Table 4

The degree of corrosion protection of synergistically effective compositions, the aggressive environment is 23% - H_2S and the temperature is 24 C

№	Inhibitor composition (g)	Inhibitor dose (g/l)	Corrosion rate, (g/m2)	Protection level, %
1	Active base - 15,0 Methanol - 15,0 Formaldehyde - 15,0 MECRVD - 10,0 Water - the rest	2	0.54	96.3
		4	0.50	96.6
		6	0.48	96.7
		8	0.44	97.01
		10	0.40	97.03
2	Active base - 15,0 Methanol - 5,0 Formaldehyde - 15,0 MECRVD – 10,0 Water – the rest	2	0.53	96.40
		4	0.50	96.60
		6	0.47	96.80
		8	0.43	97.08
		10	0.39	97.36
3	Active base – 17,0 Methanol – 15,0 Formaldehyde - 5,0 MECRVD - 10,0 Water – the rest	2	0.44	97.01
		4	0.40	97.30
		6	0.38	97.40
		8	0.34	97.70
		10	0.27	98.20
4	Active base – 17,0 Methanol – 10,0 Formaldehyde - 10,0 MECRVD – 10,0 Water – the rest	2	0.44	97.01
		4	0.38	97.4
		6	0.35	97.62
		8	0.27	98.2
		10	0.22	98.5
5	Active base – 18,0 Methanol – 5,0 Formaldehyde - 5,0 MECRVD – 5,0 Water – the rest	2	0.38	97.4
		4	0.29	98.0
		6	0.18	98.8
		8	0.14	99.0
		10	0.10	99.3
6	Active base – 18,0 Methanol – 10,0 Formaldehyde - 10,0 MECRVD – 5,0	2	0.38	97.4
		4	0.28	98.1
		6	0.15	98.9
		8	0.13	99.1

		10	0.07	99.5
7	Active base – 20,0 Methanol – 15,0 Formaldehyde - 15,0 MECRVD – 10,0 Water – the rest	2	0.37	97.5
		4	0.28	98.1
		6	0.22	98.5
		8	0.20	98.6
		10	0.14	99.0
8	Active base – 20,0 Methanol – 10,0 Formaldehyde - 10,0 MECRVD – 10,0 Water – the rest	2	0.36	97.6
		4	0.24	98.4
		6	0.18	98.8
		8	0.14	99.0
		10	0.11	99.2

The data in Tables 3 and 4 show that the level of corrosion protection increases with the increase in the amount of active base, the level of corrosion protection is high when the amount of synergistically effective additives methanol and formaldehyde is up to 10 g, and the amount of MECRVD is 5 g. from storage under the conditions of 15 and 23% acid dose, 99.6 and 99.01%, respectively, when the inhibitor concentration is 10 g/l.

In the course of the research, the temperature dependence of the level of anticorrosion protection of the obtained compositions was also studied (in the range of 20-60°C).

Table 5

Dependence of the degree of protection of the selected inhibitor No. 6 on temperature, experimental time 24 hours

The solution, % mass.	Corrosion rate $g/m^2.ch$	Protection level, %	Corrosion rate $g/m^2.ch$	Degree of protection, %	Corrosion rate $g/m^2.ch$	Protection level, %
	T=20°C		T=40°C		T=60°C	
HCl ÷ 25.0	0,042	-	0,99	-	25,80	-
HCl+№6 ÷ 25.0+0.5	0,0091	78,30	0,079	92,02	0,40	98,45
H₂SO₄ ÷ 20,0	15,7	-	7,7	-	31,20	-
H₂SO₄+№6 ÷ 20,0+0,5	2,2	85,99	3,0	61,03	16,00	48,72
HCl+H₂S ÷ 25,0 + saturated solution	0,23	-	0,94	-	3,10	-
HCl+H₂S+№6 ÷25,0 + saturated solution + 1,0	0,02	91,30	0,072	92,34	0,099	96,81

Table 5 shows the temperature dependence of the protection level of acid corrosion inhibitor №6 and it can be seen that the protection level increases to 98.4–99.6% when the corrosion rate of St.3 steel without inhibitor is 14.675 g/m^2ch.

Based on the collected data, the composition of active base - 18.0, methanol - 10.0, formaldehyde - 10.0, MECRVD - 5.0, and water 57 g was proved to be effective and recommended for production.

Conclusion. The following conclusions were made based on the results of research on obtaining imidazoline-derived corrosion inhibitors and determining their effectiveness in protecting metals from corrosion:

Optimum conditions for the synthesis of imidazoline derivatives based on a mixture of fatty acids and ethylenediamine were determined, and the effect of temperature was set at 180 - 250°C, the duration of the reaction was 3 hours: it was found that when the reaction was continued at 190°C

for 8 hours a brown liquid adduct was formed, 50% consists of acid amides with a normal structure, and at the temperature of 230°C, a homogeneous liquid of brown color is formed.

The amount of reactive water was determined and compared with the calculated amounts. Since the determination of the acid number of the produced substances is one of the important parameters indicating the degree of conversion of fatty acids, the acid number is 4 - 5 mg CON/g at 190 and 230°C, and for the product obtained with the presence of a catalyst, KS = 8.1 CON/g and this was thought to be due to the acidic nature of the catalysis process.

The composition and structure of the obtained products were confirmed using IR-spectroscopy and ^{1}H NMR-spectroscopy methods.

Temperature dependence and catalytic method of synthesis of imidazoline derivatives have been compared. It has been found that the synthesis of monoamides and imidazoline derivatives takes place under the conditions of a 1:1 ratio of fatty acids and ethylenediamine included in cottonseed oil. The ratio of monoamides and imidazoline derivatives depends more on the temperature: imidazoline derivatives have been proven to be formed with high efficiency under acid catalysis at relatively high temperatures of 240°C or 130 – 140°C at solvent boiling temperatures.

Based on the obtained imidazoline derivatives, a composition of synergistically effective protective inhibitors was created. The recommended composition is the product of catalytic hetero-cyclization of polyethylene polyamine with fatty acids, the active base of which is a composition prepared by adding formaldehyde and monoethanolamine cube residue in the vacuum-distillation (MECRVD):

Important features of the obtained inhibitors are related to the fact that imidazoline derivatives are not isolated in their free state, but used as an organic base in the mixture. In this way, product productivity is achieved and the level of corrosion protection is also high.

With an increase in the amount of the active base, the level of corrosion protection also increases, with the amount of synergistically effective additions of methanol and formaldehyde up to 10 g, and with the amount of MECRVD 5 g, the level of corrosion protection is high, at an inhibitor concentration of 10 g/l, 99.6 and 99.01%, respectively. Based on the data collected, the effectiveness the composition of the active base - 18.0, methanol - 10.0, formaldehyde - 10.0, MECRVD - 5.0, water - 57 g has been proved and recommended for the production.

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