

# REPUBLIC OF AZERBAIJAN

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## ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

### **STUDY OF SYNTHESIS OF VARIOUS FATTY OIL ACIDS DERIVATIVES WITH HEXAMETHYLENDIAMINE**

Speciality: 2314.01 – Petrochemical

Field of science: Technical sciences

Applicant: **Rashad Rahim Aghakishiyev**

**Baku – 2024**

The work was performed at the “petrochemical technology and industrial ecology” department of Azerbaijan State Oil and Industry University.


Scientific supervisor: Doctor of Chemical Sciences, acad.  
**Vagif Maharram Abbasov**

Official opponents: Doctor of Technical Sciences, prof.  
**Fariz Ali Amirli**  
Doctor of Technical Sciences, Assoc.prof.  
**Sahib Haji Yunusov**  
Doctor of Technical Sciences, Assoc.prof.  
**Huseyn Jalil Huseynov**

Dissertation council ED 1.17 of Supreme Attestation Commission under the President of the Republic of Azerbaijan operating at the Institute of Petrochemical Processes of the Ministry of Science and Education of the Republic of Azerbaijan

Chairman of the Dissertation council:  Doctor of Chemical Sciences, academician  
**Vagif Majid Farzaliyev**

Scientific secretary of the Dissertation council: Doctor of Technical Sciences, Associate professor  
 **Zaur Zabil Aghamaliyev**

Chairman of the scientific seminar: Doctor of Technical Sciences, Associate professor  
 **Mahaddin Farkhad Abbasov**

## CHARACTERISTICS OF THE WORK

### **Relevance of the topic and the degree of elaboration.**

The reason for the corrosion of metal structures depending on the operating conditions is that metals show thermodynamic discontinuity due to the reaction of metal equipment with aggressive components in the environment. Corrosion of metal equipment in oil and gas extraction, in the processing sector and in various fields of technology, under the aggressive influence of the environment, leads to the emergence of environmental problems and the loss of a large amount of funds.

The rapid development and wide application of technology in the developed countries of the world increases the attention to solving the problem of protecting this technical equipment from atmospheric corrosion during operation and idle stops. It should be noted that since agricultural and military equipment are kept under conservation conditions for most of the year, they should be protected from atmospheric corrosion. External factors affecting atmospheric corrosion - air pollution with various aggressive components, humidity, temperature. It should be noted that since these factors differ sharply in different regions of our republic, the solution to the problem of protection against atmospheric corrosion becomes somewhat complicated.<sup>1</sup>

Pollution of the environment with aggressive components as a result of anthropogenic influence leads to further increase in corrosion of metal constructions and a gradual reduction of the metal stock. US experts have made calculations about this, and based on this calculation, they determined that pollution in the atmosphere will double, and the service life of industrial equipment will decrease by an average of 1.5 times before major repairs. Dispersion of these products into the environment as a result of the breakdown and corrosion of metal equipment during accidents has a very negative effect on ecology.<sup>2</sup> In addition, the subsequent use of the products

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<sup>1</sup> Abbasov, V.M. Korroziya. II nəşr. – Bakı: Elm, – 2023. – 360 s.

<sup>2</sup> Javaherdashti, Reza. How corrosion affects industry and life. Anti-Corrosion Methods and Materials, – 2000. Vol. 47, No. 1, – p. 30-34.

required to produce stainless materials, as well as the creation of low-waste technology for reprocessing spent petroleum products, are considered real scientific problems.

Mineral resources and funds used to fight against corrosion of devices and metal equipment show the exceptional importance of the problem of protection of metals from corrosion and the importance of scientific and technical development in this field.

Corrosion processes, which lead to considerable environmental, economic and technical losses, require the creation of materials that are both economically efficient and ecologically safe, as well as highly effective against corrosion. Based on all this, it can be said that the creation of corrosion inhibitors, conservation fluids and lubricants with various compositions to increase the service life of metal equipment and structures, to increase the quality and efficiency of work in production processes, belongs to promising fields.

Based on the current environmental conditions in our country, it is important to create conservation inhibitors with high protection against corrosion for the protection of metal equipment. By application of additives with different compositions, the scientific research work conducted by us in this direction is dedicated to the achieve of conservation liquids and lubricants composed of various components.

Based on the conducted research work, amidoamines and imidazolines were synthesized on the basis of various vegetable fatty acids and hexamethylenediamine, and compositions with different compositions were prepared on the basis of nitroderivatives. By adding the received compositions to T-30 oil distillate, conservation liquids were created, and the possibility of industrial application was determined based on the high results of tests in the modern "CORROSIONBOX-1000E" experimental chamber.

As we know, bitumens are more widely used in road construction. As is known, road bitumens are obtained due to the oxidation of vacuum residue which is obtained from the distillation of crude oil. The modification of road bitumen is very important for use in construction materials depending on the extraction of tars from the oils contained in it. Due to its effectiveness, the process of wear and tear

of roads built with the use of bitumen without special additives or modifications is ongoing. As one of the causes of road damage is due to poor gravel adhesion capability.

It should be noted that adding additives to bitumen in bitumen production plants is not appropriate. So, at this time, it becomes very difficult to fill and empty bitumen. The listed shows that the creation, production and use of modifiers and additives for road bitumen remains relevant.

This problem is one of the most urgent problems for our republic, and its solution is possible by the construction of roads required by world standards.

**Object and subject of the research.** The object of research provides an opportunity to obtain a nitro compound based on hexamethylenediamine, amidoamines, imidazolines and  $\alpha$ -olefin (teradecene-1), to develop the technology of obtaining multicomponent compositions, including the development of their scientific basis.

Calculation of the electronic structure of multi-component compositions and inter-component interactions, determination of the physical and chemical properties of these compositions, their IR- and  $^1\text{H}$  NMR spectra, and testing of prepared conservation liquids were carried out within the framework of ASTM-85 standards.

**Goals and objectives of the study.**

The aim of the thesis work is to synthesize multifunctional corrosion inhibitors based on raw materials which are sufficiently available in our Republic, to prepare conservation liquids based on them and to improve the quality of bitumen by adding it to road bitumen as an additive.

Preparation of conservative liquids by adding to T-30 oil distillates from compositions based on various fatty acids and derivatives synthesized on the basis of hexamethylenediamine (amidoamines, imidazolines) and nitro compounds is one of our aim. The following studies were conducted in order to carry-out those problems:

- preparation of amidoamines, imidazolines, their compositions with nitro compounds  $\text{C}_{14}\text{H}_{28}$  obtained from the reaction of corn, sunflower,

cotton and soybean fatty acids with hexamethylenediamine;

- conducting tests of prepared conservation liquids in the "Q-4" thermo-humidity chamber, in sea water and H<sub>2</sub>SO<sub>4</sub> environment, and in the new modern "CORROSIONBOX-1000E" test chambers;
- checking the quality indicators of bitumen by adding amidoamines, imidazolines obtained from the reaction of corn, sunflower, cotton and soy fatty acids with hexamethylenediamine (1÷1) to road bitumen;
- checking the quality of bitumen by adding to road bitumen the composition of amidoamines, imidazolines and various fatty acids obtained from the charged reaction of corn, sunflower, cotton and soybean fatty acids with hexamethylenediamine (1÷1);
- study of conservation liquids and physico-chemical properties of road bitumen, determination of qualities by adding corrosion protection and additives to road bitumen by different methods.

**Research methods.** The testing process of achieved conservation liquids was carried out on steel plates in the "CORROSIONBOX-1000E" experimental chamber, "T-4" thermohumidity chamber, sea water and 0.001% H<sub>2</sub>SO<sub>4</sub> environment within the existing standards. Systematic studies were conducted using <sup>1</sup>H NMR, IR-Fourier spectroscopy method and other modern physico-chemical methods in the analysis of the structural composition of the primary components used in the preparation of conservation liquids.

With the addition of additives to road bitumen for the testing of quality of road bitumen the several devices are used. For breaking point temperature BPA 5 device belongs to Anton Paar company has been used. The conductivity was carried out in the DDA3 device, and for the penetration PNR 12 device has been used.

**The main provisions of the defense:**

- Obtaining suitable acids from vegetable corn, sunflower, cotton and soybean oils by hydrolysis method;
- Synthesis of amidoamine and imidazoline derivatives of hexamethylenediamine and nitro compound based on hexamethylenediamine;
- Investigation of various compositions adding in T-30 oil distillate as a preservative liquid based on amidoamine and imidazoline derivatives hexamethylenediamine;

- Investigation of various compositions adding in T-30 oil distillate as a preservative liquid based on amidoamine and imidazoline derivatives hexamethylenediamine plus nitrocompounds;
- Checking the physico-chemical quality of bitumen by adding the additive prepared in the optimal ratio to road bitumen based on amidoamine and imidazoline derivatives of hexamethylenediamine;
- Checking the physico-chemical quality of bitumen by adding the composition amidoamine and imidazoline derivatives of hexamethylenediamine with various fatty acids to road bitumen as an additive;
- Studying the physico-chemical parameters of multi-functional conservation liquids and lubricants based on binary and ternary components with new composition, determining the corrosion protection of metals by various methods and giving recommendations on practical application.

**Scientific novelty of the research.** For the first time, tests were conducted in the modern "CORROSIONBOX-1000E" experimental chamber as preservative liquids based on various fatty acids (corn, sunflower, cotton and soybean) and hexamethylenediamine, and high results were obtained. In addition, by adding synthesized amidoamine derivatives to road bitumen as an additive, the quality and adhesion of bitumen were further improved.

10% solutions of the compositions obtained from the interaction of various fatty acids with hexamethylenediamine in the optimal ratio (1:1:1) based on amidoamines, fatty acids and nitro compound ( $C_{14}H_{28}$  nitro compound) in mineral oils were studied as a preservation liquid and were tested on demand (90 days, GOST 9054 -75) several times higher indicators were obtained. The experiments were carried out in the "Q-4" thermo-humidity chamber, sea water, condensation and atmospheric phase:

Amidoamine (Cotton fatty acid: hexamethylenediamine 1:1) + corn fatty acid + Nitrocompound ( $C_{14}H_{28}$ ) - protects for 420 days.

Amidoamine (Corn fatty acid: hexamethylenediamine 1:1) + cotton fatty acid + Nitrocompound ( $C_{14}H_{28}$ ) - protects for 450 days.

For the first time, it was determined that the compositions of synthesized amidoamines with various fatty acids have a high

adhesion ability as an additive to road bitumen.

**Theoretical and practical significance of the research.** High-quality conservation liquids, including additives for road bitumen, have been created based on the acids obtained by the hydrolysis method from vegetable corn, sunflower, cotton and soybean oils, which have a large reserve.

Based on the experimental tests carried out in the thermo-humidity chamber (protection of metal plates for 420 and 450 days), it was determined that the prepared conservation liquids are highly effective protective coatings that prevent corrosion of metals in various aggressive environments and can be successfully applied by creating a high protective effect on the surface of the metal.

Oil distillate (T-30) used as a medium in the preparation of conservation liquids, as well as additives containing inhibitors, are produced on the basis of raw materials with a sufficient reserve.

**Approbation and application.**

11 scientific works, including 8 articles, 3 thesis have been published. The printed works fully reflect the content of the dissertation.

The main results of the research were discussed at the following scientific conferences:

Republican conference on "Environmental protection, industrial and household waste recycling" (Ganja, 2022), International scientific conference on "Education and research activities in the new era: realities and challenges" (Mingachevir, 2022); XII International Scientific and Practical Conference "Modern science: actual problems", (UK, Manchester, 2024).

**The organization where the dissertation work was performed.**

The research work was carried out at the "Petrochemical technology and industrial ecology" department of Azerbaijan State Oil and Industry University.

**Author's personal participation.**

The author personally participated in conducting research, summarizing and interpreting experimental results, conducting tests and writing articles.



**The total volume of the dissertation with a sign indicating the volume of the structural sections of the dissertation separately:**

The thesis consists of 160 pages of printed material, including an introduction, 6 chapters, conclusions, a bibliography with 144 sources and 27 pictures, 2 schemes, 19 tables. The volume of the dissertation is consists of 171865 marks, excluding the bibliography, tables and pictures (introduction 14790 marks, chapter I 47280, chapter II 11685, chapter III 24950, chapter IV 33810, chapter V 25560, chapter VI 9990 and conclusion 3800 marks).

**Introduction:** In this part, the relevance of the topic, the object and subject of the research, the goal set in the dissertation, the scientific novelty of the work, and the theoretical and practical significance of the research were given.

**The first chapter:** Consists of a literature review of corrosion inhibitors and atmospheric corrosion inhibitor protection, corrosion protection methods, and preservative liquids containing anti-corrosion compositions, including road bitumen production technology and quality requirements, additives.

The compositions of conservation liquids and inhibitors obtained recently were comparatively analyzed, their advantages and disadvantages were indicated, and scientific literature on the mechanisms of action was examined.

**In the second chapter,** the study of the physico-chemical indicators of various additives necessary for conducting research and the confirmation of their structures by spectral analysis methods, research methods, methods of testing conservation fluids in various aggressive environments, the working principle of the apparatus used for determining the effect of protection against corrosion, determining the quality indicators of bitumen and information about the methods of analysis has been published.

**The third chapter is** devoted to the physico-chemical properties of plant-based fatty acids and hexamethylenediamine, amidoamines and imidazolines synthesized on the basis of hexamethylenediamine, and other used components, as well as the application of preservation liquids.

**The fourth chapter** is about the analysis of the results of the

corrosion protection effect of preservation liquids on metal plates based on synthesized amidoamines, imidazolines and nitrocompound. The results of the research were analyzed in a comparative form and it was determined that the composition of synthesized amidoamines with nitro compound and the three-component composition of these compounds with vegetable fatty acids have a higher preservation ability of the amidoamine derivatives separately as a preservative liquid in different oil distillates

**The fifth chapter** is dedicated to the application additive to road bitumen of synthesized amidoamine, imidazoline derivatives with fatty acids of plant origin (corn, sunflower, cotton and soy). In this chapter, the composition of amidoamine and imidazole derivatives with individual and vegetable fatty acids was prepared and the quality indicators of bitumen and their results were shown by adding 0.4 and 0.6% to road bitumen.

**In the sixth chapter**, a generalized scheme of the principle of production of conservation liquids based on multicomponent inhibitors, road bitumen and the change in the properties of bitumen after adding additives to bitumen is given.

At the end of the dissertation work, the results containing the researches conducted, the list of cited literature and appendices are given.

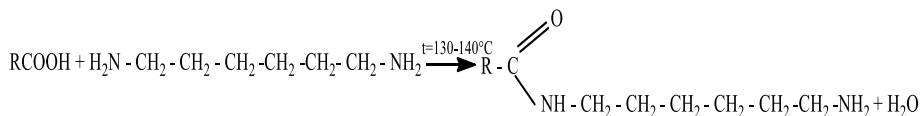
## THE MAIN CONTENT OF THE WORK

### Selection of raw materials and synthesis of inhibitors based on vegetable oils

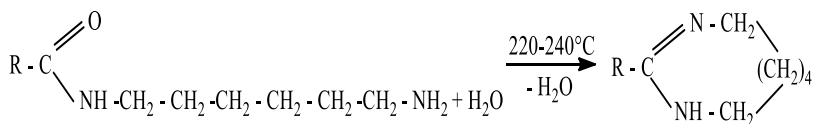
The substances used as primary components in the preparation of inhibitors are the following: 1. Sunflower, cotton, soybean and corn oils; 2. Hexamethylenediamine; 3. T-30 turbine oil.

Vegetable oils taken as components were first heated to 50 °C, and then 10% NaOH solution was gradually added drop by drop to the reaction medium. In order to carry out the hydrolysis of the oil in an alkaline environment, it was intensively stirred at 60-80°C for 2 hours and then the sodium salt of the fatty acids was obtained. At the next stage, the sodium salt of the obtained fatty acids was treated with 20% HCl, and the fatty acids were separated, the acid was washed and the alkali was removed. After that, the water remaining in the acid was evaporated and the fatty acids were separated.

In the second stage, synthesis of amidoamine derivatives of hexamethylenediamine and fatty acids obtained from sunflower, corn, cotton and soybean oils was carried out. Amidoamine derivatives were obtained with approximately 90% yield.



In the third stage, imidazolines were synthesized from amidoamines based on hexamethylenediamine with fatty acids obtained from sunflower, corn, cotton and soybean oils. Imidazoline derivatives were obtained with approximately 90% yield.



By adding different percentages of these synthesized inhibitors to T-30 turbine oil, conservative liquids were prepared.

As a continuation of the research, IR spectra of fatty acids (sunflower, corn, cotton and soybean fatty acids) obtained on the basis of vegetable oils were drawn and their structures were confirmed (pictures 1-4).

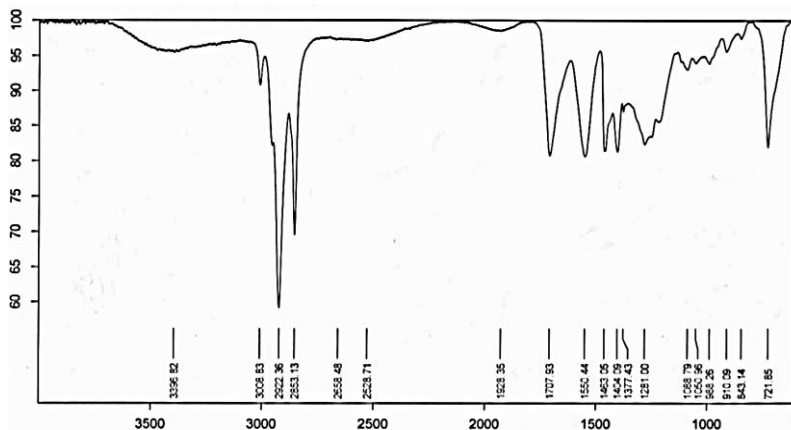


Figure 1. IR spectrum of sunflower oil acid

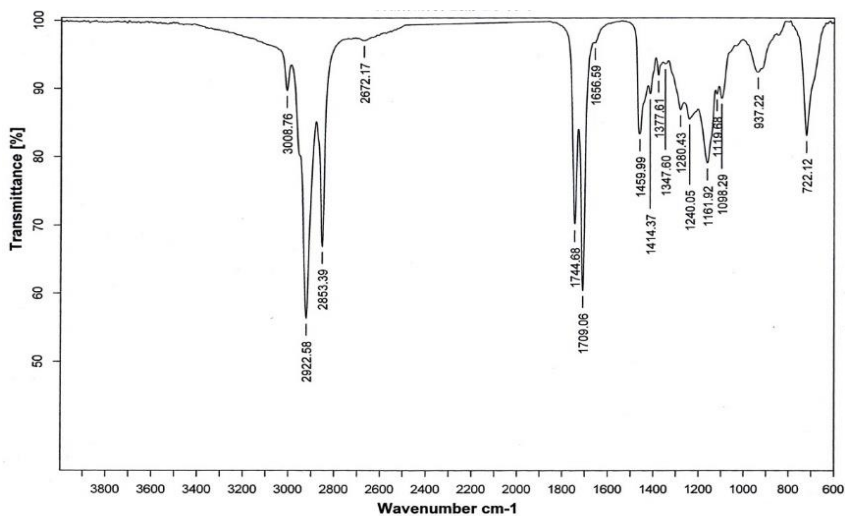
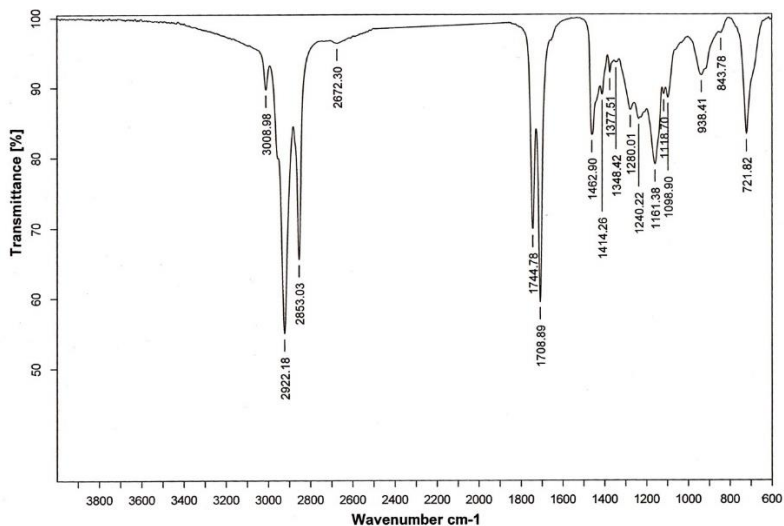
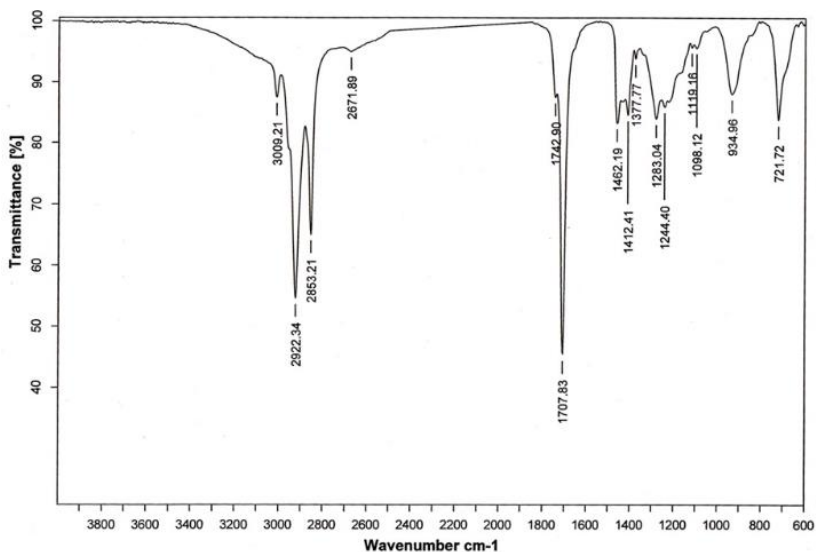


Figure 2. IR spectrum of corn oil acid



**Figure 3. IR spectrum of cotton oil acid**



**Figure 4. IR spectrum of soybean oil acid**

As can be seen from the IR spectrum, absorption bands characteristic of both ether and acid were obtained in all of them.

## **Research and results of conservative liquids based on synthesized amidoamines**

First, conservative liquids were prepared on the basis of compositions of amidoamines synthesized on the basis of plant fatty acids and hexamethylenediamine, separately and with different plant fatty acids (sunflower, corn, cotton and soy), and the effect of corrosion protection in different environments was checked.

In order to simplify the analysis of the results of the synthesized inhibitors and conservation fluids prepared based on their composition, these compositions are named by codes and shown below.

1. Amidoamine synthesized on the basis of "A-1" - corn fatty acid and hexamethylenediamine in a 1:1 mol ratio
2. Amidoamine synthesized on the basis of "A-2" - sunflower fatty acid and hexamethylenediamine in a 1:1 mol ratio
3. Amidoamine synthesized on the basis of "A-3" - cotton fatty acid and hexamethylenediamine in a 1:1 mol ratio
4. "A-4"-synthesized amidoamine based on soy fatty acid and hexamethylenediamine in a 1:1 mol ratio.

Synthesized amidoamines were added to T-30 oil distillate in amounts of 5, 7, 10, and 20% to prepare preservation liquids and were tested in "Г-4" thermohumidity chamber, sea water, and 0.001% H<sub>2</sub>SO<sub>4</sub> environment, and are listed in Table 1 below.

As can be seen from Table 1, the best result was obtained by taking 10 and 20% of the amidoamine synthesized on the basis of cotton fatty acid and hexamethylenediamine as an inhibitor, and 80 and 90% of T-30 oil distillate. The corrosion protection effect of these synthesized amidoamine based preservation fluids on metal plates in all 3 environments is higher than the corrosion protection effect of other synthesized amidoamines on metal plates. Thus, those samples (sample No. 4) with 10 and 20% were 210 and 225 days in the "Г-4" thermohumidity chamber, 117 and 125 days in sea water, and 116 and 120 days in 0.001% H<sub>2</sub>SO<sub>4</sub> environment.

**Table 1.**

**Test results of prepared preservative liquids based on synthesized amidoamine with hexamethylenediamine distillate and vegetable fatty acids by adding to T-30 oil**

№	Solution of compositions in T-30 oil distillate	The total amount of inhibitor, in %	Corrosion protection time, in days		
	Composition of samples		«Г-4» hydrocamera	Seawater	0,001% H <sub>2</sub> SO <sub>4</sub>
1	T-30 oil distillate	100	34	15	9
2	T-30 oil distillate+ “A-1”	5	101	37	35
		7	145	46	45
		10	170	57	55
		20	187	86	85
3	T-30 oil distillate +“A-2”	5	145	50	47
		7	166	62	60
		10	197	95	92
		20	217	120	117
4	T-30 oil distillate + “A-3”	5	185	54	52
		7	200	98	97
		10	210	117	116
		<b>20</b>	<b>225</b>	<b>125</b>	<b>120</b>
5	T-30 oil distillate + “A-4”	5	137	40	38
		7	155	53	52
		10	185	77	75
		20	201	197	195

Also 5, 7, 10 and 20% composition of amidoamine synthesized on the basis of hexamethylenediamine with corn, sunflower, cotton and soybean fatty acids and by adding to T-30 oil distillate, preservation liquids were prepared and tests were conducted (Tables 2-4).

**Table 2.**  
**Conservation liquids prepared on the basis of the composition of amidoamine synthesized on the basis of corn fatty acid and hexamethylenediamine with various fatty acids**

№	Composition of samples	Total amount of inhibitor, in %	Corrosion protection time, in days		
			«Г-4» hydrocamera	Sea-water	0,001% H <sub>2</sub> SO <sub>4</sub>
1	T-30 oil distillate 100 %	0	34	15	9
2	T-30 oil distillate (95%, 93%,90%,80%) + "A-1" + sunflower fatty acid in a 1:1 mole ratio	5	134	42	41
		7	167	52	50
		10	185	65	62
		20	200	95	91
3	T-30 yağ distillatı (95%, 93%,90%,80%) + "A-1" + corn fatty acid in a 1:1 mol ratio	5	165	57	55
		7	180	72	70
		10	220	105	101
		20	232	135	131
4	T-30 yağ distillatı (95%, 93%,90%,80%) + "A-1" + cotton fatty acid in a 1:1 mol ratio	5	200	65	62
		7	217	109	105
		10	226	132	130
		20	240	165	162
5	T-30 yağ distillatı (95%, 93%,90%,80%) + "A-1" + soybean fatty acid in a 1:1 mol ratio	5	153	45	41
		7	175	62	60
		10	210	90	85
		20	228	105	100

**Table 3.**  
**Preservation liquids prepared on the basis of the synthesized amidoamine on the basis of sunflower fatty acid and hexamethylenediamine with various fatty acids**

№	Solution of compositions in T-30 oil distillate	Total amount of inhibitor, in %	Corrosion protection time, in days		
	Composition of samples		«Г-4» hydrocamera	Seawater	0,001 % H <sub>2</sub> SO <sub>4</sub>
1	2	3	4	5	6
1	T-30 oil distillate 100 %	0	34	15	9
2	T-30 oil distillate (95%, 93%,90%,80%) + "A-1" + sunflower fatty acid in a 1:1 mol ratio	5	160	62	60
		7	209	102	100
		10	265	196	192
		20	279	200	200



followed

1	2	3	4	5	6
3	T-30 oil distillate (95%, 93%,90%,80%) + "A-1" + corn fatty acid in a 1:1 mol ratio	5	157	60	57
		7	195	92	88
		10	211	98	95
		20	217	103	98
4	T-30 oil distillate (95%, 93%,90%,80%) + "A-1" + cotton fatty acid in a 1:1 mol ratio	5	210	115	112
		7	253	124	122
		10	292	197	194
		<b>20</b>	<b>300</b>	<b>203</b>	<b>200</b>
5	T-30 oil distillate (95%, 93%,90%,80%) + "A-1" + soy fatty acid in a 1:1 mol ratio	5	161	92	90
		7	225	97	93
		10	286	145	137
		20	290	147	145

Table 4.

**Preservative liquids prepared on the basis of the composition of amidoamine synthesized on the basis of cotton fatty acid and hexamethylenediamine with various fatty acids**

№	Solution of compositions in T-30 oil distillate	Total amount of inhibitor, in %	Corrosion protection time, in days		
	Composition of samples		«Г-4» hydro-camera	Seawater	0,001 % H <sub>2</sub> SO <sub>4</sub>
1	T-30 oil distillate 100 %	0	34	15	9
2	T-30 oil distillate (95%, 93%,90%,80%) + "A-1" + sunflower fatty acid in a 1:1 mol ratio	5	120	50	48
		7	165	57	54
		10	182	90	88
		20	195	95	91
3	T-30 oil distillate (95%, 93%,90%,80%) + "A-1" + corn fatty acid in a 1:1 mol ratio	5	157	65	62
		7	175	70	68
		10	207	102	100
		20	225	121	118
4	T-30 oil distillate (95%, 93%,90%,80%) + "A-1" + cotton fatty acid in a 1:1 mol ratio	5	197	72	70
		7	210	105	104
		10	222	130	125
		<b>20</b>	<b>237</b>	<b>135</b>	<b>132</b>
5	T-30 oil distillate (95%, 93%,90%,80%) + "A-1" + soybean fatty acid in a 1:1 mol ratio	5	140	45	41
		7	165	62	61
		10	192	80	77
		20	210	95	91

Table 5.

**Preservative liquids prepared on the basis of the composition of amidoamine synthesized on the basis of soy fatty acid and hexamethylenediamine with various fatty acids**

№	Composition of samples	Total amount of inhibitor, in %	Corrosion protection time, in days		
			«Г-4» hydrocamera	Seawater	0,001% H <sub>2</sub> SO <sub>4</sub>
1	T-30 oil distillate	100	30	12	8
2	T-30 oil distillate 95%, 93%, 90%, 80% + "A-4" + corn fatty acid in a 1:1 mol ratio	5	175	139	135
		7	184	145	142
		10	197	155	152
		20	297	161	158
3	T-30 oil distillate 95%, 93%, 90%, 80% + "A-4" + sunflower fatty acid in a 1:1 mol ratio	5	212	107	102
		7	285	110	105
		10	291	112	107
		20	305	115	110
4	T-30 oil distillate 95%, 93%, 90%, 80% + "A-4" + cotton fatty acid in a 1:1 mol ratio	5	225	112	110
		7	291	126	124
		10	310	142	139
		20	326	145	142
5	T-30 oil distillate 95%, 93%, 90%, 80% + "A-4" + soy fatty acid in a 1:1 mol ratio	5	95	67	65
		7	108	90	87
		10	175	103	98
		20	190	105	100

From the test results (tables 1-4), it can be concluded that the corrosion protection effect of metal plates of preservation fluids prepared on the basis of the composition of amidoamines synthesized on the basis of cotton fatty acid and hexamethylenediamine with various fatty acids showed a higher result than the protection effect of the prepared preservation liquids by adding to the T-30 oil distillate individually. Thus, the corrosion protection effect of metal plates of conservation fluids prepared by adding 10 and 20% of amidoamine, synthesized on the basis of cotton fatty acid and

hexamethylenediamine, to T-30 oil distillate as an inhibitor, 210 and 225 days in thermohumidity chamber "Г-4", sea 117 and 125 days in water, and 116 in 0.001% H<sub>2</sub>SO<sub>4</sub> and 120 days (table 1, sample No. 4), the preservation effect of the preservative liquid prepared based on the composition of synthesized amidoamine with cotton fatty acid was 222, 237 days in the "Г-4" thermohumidity chamber, and 130 and 135 days in sea water and 125 and 132 days in 0.001% H<sub>2</sub>SO<sub>4</sub> environment (table 4, sample number 4).

If we look at Table 5, we can see that the corrosion protection effect of metal plates of conservation liquids prepared by adding these inhibitors in the amount of 10 and 20% to T-30 oil distillate is 310 and 326 days in the "Г-4" thermohumidity chamber, 142 and 145 in sea water and 139 and 142 days in 0.001% H<sub>2</sub>SO<sub>4</sub> medium (table 5, sample no. 4).

### **Research and results of preservation liquids based on synthesized imidazolines**

Amidoamines were synthesized on the basis of various vegetable fatty acids and hexamethylenediamine and were added to T-30 oil distillate in various percentages as inhibitors by preparing preservation liquids and research was carried out. As a result of these studies, it was determined that the conservation liquids prepared on the basis of the combined composition of the synthesized amidoamines have a higher corrosion protection effect on "Polad-3" brand metal plates.

Compositions based on synthesized imidazolines are named by codes and shown as follows:

1. "I-1" - imidazoline synthesized in a 1:1 mol ratio based on corn fatty acid and hexamethylenediamine;
2. "I-2" - imidazoline synthesized in a 1:1 mol ratio based on sunflower fatty acid and hexamethylenediamine;
3. "I-3" - imidazoline synthesized in a 1:1 mol ratio based on cotton fatty acid and hexamethylenediamine;
4. "I-4" - imidazoline synthesized on the basis of soy fatty acid and hexamethylenediamine in a 1:1 mol ratio.

These imidazolines were added to T-30 oil distillate in amounts of 5, 7, 10, and 20% to prepare preservation liquids and were tested in

"Г-4" thermohumidity chamber, sea water, and 0.001% H<sub>2</sub>SO<sub>4</sub> environment.

Preservative liquids based on imidazolines synthesized on the basis of various fatty acids and hexamethylenediamine were prepared, tested and the results were as follows.

1. When "I-3" + sunflower fatty acid is taken in a 1:1 mol ratio in the amount of 5, 7, 10 and 20%, and T-30 oil distillate in the amount of 95, 93, 90 and 80%, "Г-4" thermohumidity in the cell 135, 155, 167, 188 days, in sea water 55, 82, 87, 100 days and 52, 80, 85, 97 days in 0.001% H<sub>2</sub>SO<sub>4</sub> medium.

2. "I-3" + corn fatty acid in a 1:1 mol ratio in the amount of 5, 7, 10 and 20%, and T-30 oil distillate in the amount of 95, 93, 90 and 80%, "Г-4" 145, 165, 180, 190 days in a thermo-humidity chamber, 58, 85 in sea water, 92, 102 days and 55, 83, 90, 101 days in 0.001% H<sub>2</sub>SO<sub>4</sub> medium.

3. "I-3" + cotton fatty acid in a 1:1 mole ratio in the amount of 5, 7, 10 and 20%, and T-30 oil distillate in the amount of 95, 93, 90 and 80%, "Г-4" 175, 210, 220, 230 days in a thermo-humidity chamber, 87, 104, 107, 112 days and 85, 102, 106, 110 days in 0.001% H<sub>2</sub>SO<sub>4</sub> medium.

4. "I-3" + soy fatty acid in a 1:1 mole ratio in the amount of 5, 7, 10 and 20%, and T-30 oil distillate in the amount of 95, 93, 90 and 80%, "Г-4" 95, 107, 130, 142 days in a thermo-humidity chamber, 42, 62, 70 in sea water, 82 days and 40, 61, 67, 80 days in 0.001% H<sub>2</sub>SO<sub>4</sub> medium.

From the conducted tests, it was determined that the corrosion protection effect of the preservation liquids made on the basis of the composition of imidazolines synthesized on the basis of cotton fatty acid and hexamethylenediamine with various fatty acids is higher than the protection effect of the preservation liquids made on the basis of other imidazolines.

The composition of imidazoline synthesized on the basis of soy fatty acid and hexamethylenediamine with various fatty acids was added to T-30 oil distillate in the amount of 5, 7, 10 and 20%, conservation liquids were prepared and tested, and the results were as follows:

1. When "I-4" + sunflower fatty acid is taken in a 1:1 mole ratio in the amount of 5, 7, 10 and 20%, and T-30 oil distillate in the amount of 95, 93, 90 and 80%, "Г-4" thermohumidity in the cell 81, 121, 139, 165 days, in sea water 29, 37, 46, 57 days and 27, 36, 43, 56 days in 0.001% H<sub>2</sub>SO<sub>4</sub> medium.

2. When "I-4" + corn fatty acid is taken in a 1:1 mol ratio in the amount of 5, 7, 10 and 20%, and T-30 oil distillate in the amount of 95, 93, 90 and 80%, "Г-4" thermohumidity in the cell 105, 140, 157, 172 days, in sea water 34, 44, 57, 62 days and 32, 41, 55, 61 days in 0.001% H<sub>2</sub>SO<sub>4</sub> medium.

3. When "I-4" + cotton fatty acid is taken in a 1:1 mol ratio in the amount of 5, 7, 10 and 20%, and T-30 oil distillate in the amount of 95, 93, 90 and 80%, "Г-4" thermohumidity 111, 167, 172, 185 days in the cell, 37, 45, 70, 85 in sea water days and 35, 42, 67, 84 days in 0.001% H<sub>2</sub>SO<sub>4</sub> environment.

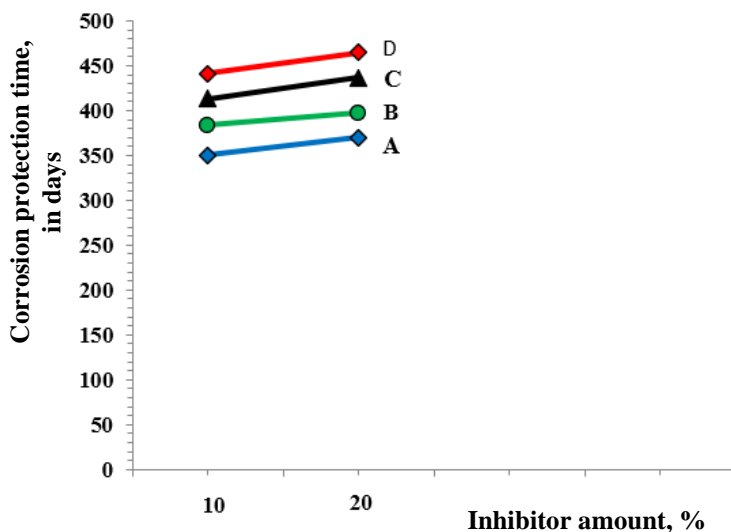
4. When "I-4" + soybean fatty acid is taken in the amount of 5, 7, 10 and 20% in a 1:1 mole ratio, and T-30 oil distillate is taken in the amount of 95, 93, 90 and 80%, "Г-4" thermohumidity 68, 115, 121, 137 days in the cell, 25, 28, 34, 51 days in sea water and 24, 26, 31, 50 days in 0.001% H<sub>2</sub>SO<sub>4</sub> medium.

The tests of conservation fluids made from synthesized imidazolines and the composition of these imidazolines with vegetable fatty acids revealed that imidazolines synthesized on the basis of cotton fatty acid and hexamethylenediamine and their composition have a higher protective capacity as inhibitors. Based on this, it is considered more convenient and appropriate to prepare preservation liquids based on these inhibitors.

### **Research and results of preservation liquids based on the composition of synthesized amidoamines, vegetable fatty acids and nitrocompound.**

In this section we decided to prepare preservation liquids based on triple-component inhibitors and conduct tests in different environments rather than addition of the synthesized amidoamines separately and the composition with vegetable fatty acids to mineral oils. As we know, the presence of a certain amount of water of hydration in the inhibitor makes the solubility of these inhibitors worse

in oil distillates and, therefore, causes a relatively low protective effect as a corrosion inhibitor. On the other hand, the water ligand enters the coordination sphere of the metal ion and weakens its connection with the metal surface. Therefore, inhibitors were prepared on the basis of synthesized amidoamines with vegetable fatty acids and end component with nitro compound, and by adding different percentages of 5, 7, 10 and 20% to T-30 oil distillate, conservation liquids were prepared and researches were conducted.



**Figure 5. Indications of the test of the composition of amidoamine with various fatty acids and nitro compounds in the "CORROSSIONBOX-1000E" experimental chamber, in the condensation phase, as a preservative liquid:**

A. Amidoamine (corn fatty acid:hexamethylenediamine 1:1 mol ratio) + nitrocoumpoundt and soybean fatty acid 1:1 mol ratio.

B. Amidoamine (corn fatty acid: hexamethylenediamine 1:1 mol ratio) + nitro compound and sunflower fatty acid 1:1 mol ratio.

C. Amidoamine (corn fatty acid:hexamethylenediamine 1:1 mol ratio) + nitrocoumpound and corn fatty acid 1:1 mol ratio.

D. Amidoamine (corn fatty acid:hexamethylenediamine 1:1 mol ratio) + nitrocompound and cotton fatty acid 1:1 mol ratio.

If we look at Figure 5, we can see that the preservation liquids prepared on the basis of the composition of corn fatty acid with

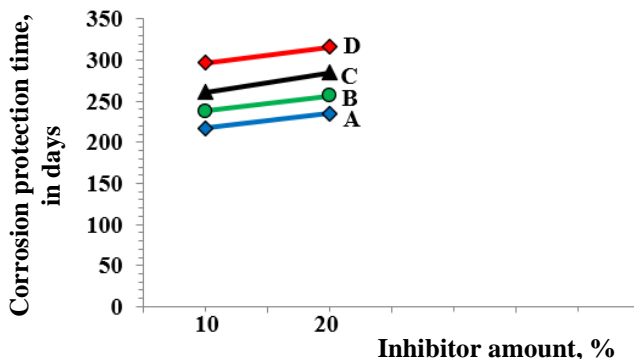
hexamethylenediamine in a 1:1 mol ratio of amidoamine and cotton fatty acid have a higher protective capacity (curve D). Thus, in the experimental chamber "CORROSIONBOX-1000E" of conservation liquids prepared based on the addition of these compositions to T-30 oil distillate in the amount of 10 and 20%, the corrosion protection effect of metal plates showed the highest results of 441 and 465 days in the condensation phase.

The composition of synthesized amidoamines with various fatty acids, as well as the end-component composition of these compositions with nitro compounds, as a result of test studies of the corrosion protection effect of "Polad-3" brand metal plates in various aggressive environments, it was determined that the composition of amidoamines with cotton fatty acids conservation liquids based on this are higher than the conservation effect of conservation liquids based on other fatty acids. By adding 10, 20% of the three-component composition of these compositions with nitro compounds to T-30 oil distillate, the corrosion protection effect of the metal plates of the conservation liquids was more than 2 times higher, which is related to the creation of synergism.

### **Research and results of preservation liquids based on the composition of synthesized imidazolines, vegetable fatty acids and nitro compound.**

As a result of tests of the corrosion protection effect of "Polad-3" brand metal plates of preservation fluids prepared on the basis of the composition of synthesized imidazolines with vegetable fatty acids, it was determined that the composition of imidazolines synthesized on the basis of cotton fatty acid and corn fatty acid with vegetable fatty acids with the addition to T-30 oil distillate prepared preservation liquids have the higher ability to protect metal plates from corrosion.

If we look at Figure 6, we can see that the preservation liquids prepared based on the composition of cotton fatty acid with hexamethylenediamine in the ratio of 1:1 mol, synthesized amidoamine, cotton fatty acid have a higher preservation ability (curve D). Thus, adding these compositions to T-30 oil distillate in the amount of 10 and 20% showed results of 297 and 316 days.



**Figure 6. Test results of the composition of imidazoline with various fatty acids and nitro compounds in the "CORROSSIONBOX-1000E" experimental chamber, in the condensation phase, as a preservative liquid.**

A. Imidazoline (cotton fatty acid: hexamethylenediamine in 1:1 mol ratio) + nitrocompound and soybean fatty acid in 1:1 mol ratio.

B. Imidazoline (cotton fatty acid:hexamethylenediamine 1:1 mol ratio) + nitrocompound and sunflower fatty acid 1:1 mol ratio.

C. Imidazoline (cotton fatty acid:hexamethylenediamine 1:1 mole ratio) + nitrocoupling and corn fatty acid 1:1 mole ratio.

D. Imidazoline (cotton fatty acid:hexamethylenediamine 1:1 mol ratio) + nitrocompound and cotton fatty acid 1:1 mol ratio.

The analysis of the conducted tests shows that the conservation liquids prepared on the basis of the composition of synthesized imidazoline, cotton fatty acid and nitro compound have a higher protection effect.

### **Study of synthesized amidomines and their composition with different fatty acids as additives to road bitumen.**

The quality of bitumen were studied by adding 0.4 and 0.6% of additives prepared on the basis of synthesized amidoamines ("A-1"; "A-2"; "A-3"; "A-4") and their composition with various fatty acids (sunflower, corn) to road bitumen. As a result of these studies, it was determined that "A-1" and "A-2" were prepared on the basis of synthesized amidoamines and their composition with various fatty acids by adding these additives to road bitumen in certain percentages improves bitumen quality and changes bitumen adhesion from 3 points to 1 point. Thus, by adding these additives to road bitumen in the



amount of 0.4 and 0.6%, the brittleness temperature of bitumen increased from  $-18^{\circ}\text{C}$  to  $-29^{\circ}\text{C}$ , ductility increased from 75cm to  $>100\text{cm}$ .

The conducted studies showed that it is considered more appropriate to use these additives to improve the quality of road bitumen.

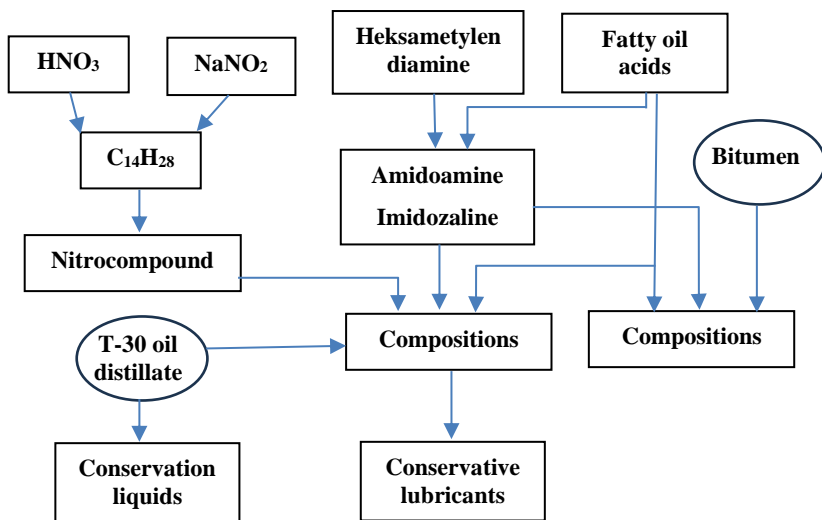
### **Study of synthesized imidazolines and their composition with different fatty acids as additives to road bitumen.**

Research studies were conducted in order to see how bitumen affects quality of bitumen after prepared additives on the basis of the composition of synthesized imidazolines ("I-1", "I-2", "I-3" and "I-4") with different fatty acids added in amounts of 0.4 and 0.6% to road bitumen. It was determined that "I-1" and "I-2" imidazoline compositions made on the basis of sunflower and corn fatty acids had a better effect on the quality of bitumen, but after the addition of these additives, the adhesion of bitumen changed from 3 points to 2 points. As can be seen from the conducted studies, after the addition of additives made on the basis of the composition of synthesized amidoamines and imidazolines with various fatty acids to bitumen, the additives made on the basis of the composition of amidoamines with fatty acids further improve the quality of bitumen and change its adhesion from 3 points to 1 point.

Since the compositional additives of synthesized amidoamines with corn and sunflower fatty acids have a better effect on the quality of road bitumen, (i.e. bitumen tension after the addition of these additives from 75 cm; to  $>100\text{ cm}$ , brittleness temperature from  $-18^{\circ}\text{C}$ , - up to  $29^{\circ}\text{C}$ , and because the adhesion varies from 3 points to 1 point) the addition of these additives to bitumen is considered more appropriate.

### **A generalized scheme for the process.**

As a final result of the conducted research, a generalized scheme of the technology of obtaining additives with the purpose of increasing the quality of road bitumen and high protection effect conservation liquids with the addition of newly synthesized inhibitors to T-30 oil distillate (Scheme 1) is given.



**Scheme 1. Generalized scheme of the technology of obtaining new conservation liquids for the purpose of improving the quality of road bitumen.**

According to the scheme, it can be said that the process of preparation of preservation liquids consists of 4 stages:

1) Components for compositions were synthesized based on selected reagents in the first stage. Amidoamines and imidazolines were synthesized from nitrogen organic compounds - hexamethylenediamine based on various vegetable fatty acids.

2) In the second stage, compositions of synthesized amidoamines and imidazolines, vegetable fatty acids, and nitro compounds were prepared for conservation ingredients. Hexamethylenediamine and corn, sunflower, cotton, soybean fatty acids were used for the synthesis of imidazolines.

3) Nitration of aliphatic-based tetradecene ( $\text{NaNO}_2$ ) with the participation of an initiator was used in the third stage to obtain nitro compounds.

4) In the fourth stage, various compositions of plant fatty acids, amidoamines, imidazolines, were prepared and added to bitumen in amounts of 0.4 and 0.6%.

## RESULTS

1. Preparation of amidoamines, imidazolines synthesized with sunflower fatty acid, various compositions based on various fatty acids and creating conservation liquids with T-30 oil distillate (5, 7, 10 and 20%) in the "Г-4" hydrochamber environment, tests were carried out in sea water, in a 0.001% solution of  $H_2SO_4$  in water [1, 2, 4].

2. For the first time, the compositions of the amidoamines of hexamethylenediamine synthesized with various fatty acids were prepared with sunflower, corn, cotton and soybean fatty acids and by adding 5, 7, 10, 20% to T-30 oil distillate, preservation liquids were created. These preservation liquids were tested in the hydrochamber environment "Г-4", in sea water, in a 0.001% solution of  $H_2SO_4$  in water. It has been determined that preservation liquids prepared with these compositions of amidoamines synthesized on the basis of cotton and corn fatty acids have a higher preservation capacity [8, 9].

3. Amidoamines of hexamethylenediamine synthesized with various fatty acids and nitro compounds were prepared and added to T-30 oil distillate in amounts of 5, 7, 10 and 20% to create preservation liquids. These conservation liquids were tested in 2 phases (condensation and atmospheric phase) in the experimental chamber "CORROSIONBOX-1000E". It has been determined that the preservation liquids prepared with these compositions of amidoamines synthesized on the basis of cotton and corn fatty acids have a higher preservation capacity [11].

4. The composition of the synthesized amidoamines of hexamethylenediamine with sunflower and corn fatty acids in a 1:1 mol ratio with fatty acids and a nitro compound was tested as a preservative liquid in T-30 oil distillate medium and showed the highest result. Thus, the preservation effect of hexamethylenediamine synthesized with sunflower fatty acid, amidoamine, corn fatty acid and nitrocompound in the amount of 10 and 20% added to T-30 oil distillate, preservation effect in the condensation phase in the experimental chamber "CORROSIONBOX-1000E", 320, 345 days, and this composition of amidoamine synthesized with hexamethylenediamine corn fatty acid consecutive preservation liquid

showed results of 413 and 437 days in that environment [6, 9].

5. Bitumen quality by adding 0.4 and 0.6% additives to road bitumen tested and received high results with compositions of hexamethylenediamine with various fatty acids in 1:1 mol ratios, compositions of corn, sunflower, cotton and soybean fatty acids (in 1:1 mol ratio). In particular, while the adhesion of bitumen itself is 3 points, the adhesion of the additive in the amount of 0.4% and 0.6% has reached 1 point [5, 7, 8].

6. The addition of 0.4 and 0.6% of synthesized amidoamines of hexamethylenediamine with different fatty acids in 1:1 molar ratio, compositions with corn fatty acids to road bitumen improved the quality of bitumen and obtained higher results. So, after the addition of this additive, the softening temperature of bitumen increased from 48.5°C to 47.1°C, ductility from 75 cm to >100 cm, brittleness temperature from -18°C to -29°C, and adhesion from 3 points changed to 1 point [10].

7. By adding 0.4 and 0.6% of hexamethylenediamine to road bitumen in 1:1 mol ratio of synthesized imidazolines, corn and sunflower fatty acids (1:1 mol ratio), bitumen quality were improved. These additives improved the quality of bitumen and had a positive effect on improving its adhesion [1, 3].

8. Based on the obtained results, the mass change of road bitumen and bitumen after adding additives to bitumen was checked. The results obtained by heating bitumen in a thermostat at 163°C after adding 0.6% additive to road bitumen itself showed that the mass of bitumen itself changes after heating as well as after addition of additive. Thus, the mass change of bitumen was 0.06 g (0.12%), but after the addition of some additives, the mass change of bitumen was less than that of bitumen, 0.05 g (0.1%) [10].

## **LIST OF SCIENTIFIC WORKS PUBLISHED ON THE TOPIC OF THE DISSERTATION:**

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The defense will be held on 27 December 2024 at 10<sup>00</sup> at the meeting of the Dissertation council ED 1.17 of Supreme Attestation Commission under the President of the Republic of Azerbaijan operating at Institute of Petrochemical Processes named after academician Y.H.Mammadaliyev of Republic of Azerbaijan Ministry of Science and Education

Adress: Khojali ave., 30, AZ-1025, Baku, Azerbaijan

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Abstract was sent to the required addresses on 26 November 2024.

Signed for print: 15.11.2024

Paper format: A5

Volume: 37020

Number of hard copies: 20