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Study of The Amides Obtained by The Synthesis of Cottonseed Oil and Diethanolamine as Inhibitor to Prevent Atmospheric Corrosion

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Abstract: Amides of cottonseed oil and diethanolamine were synthesized in 1:1, 1:2 and 1:3 mole ratios and the structures of the substances were confirmed using infrared spectroscopy. Physicochemical properties were also studied. Conservative liquids were developed by adding 3, 5 and 10% of the obtained amides into T-30 oil. Corrosion tests involved immersing metal plates in conservative liquids within a hydrochamber, seawater, and 0.001% H_2SO_4 solution. The studies revealed that synthetic amides can be used to develop conservative liquids of high protective efficiency. The best results were obtained by 10% solutions. It was determined that metal plates immersed in a 10% solution of amide in a mole ratio of 1:2 were protected from corrosion during 205 days in "Г-4" hydrochamber, 165 days in seawater, and 160 days in a 0.001% H_2SO_4 solution. Taking into account that the optimum composition created as a result of research is more favorable from an economic point of view, turbine oil was used as a solvent medium, and the obtained results met the demand.

Keywords: Cottonseed oil, diethanolamine, amide, atmospheric corrosion, turbine oil.

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1. INTRODUCTION

Atmospheric corrosion, being one of the most common types of corrosion, has led to significant attention being focused on the development of protection tools and methods. The occurrence of atmospheric corrosion on steel surfaces causes great losses of material resources (1-3).

Atmospheric corrosion affects all metal structures operating outdoors (about 50% of the total existing metal stock), namely: overhead pipelines, capacity equipment, metal parts of buildings, piers, bridges, transport facilities. During operation, the surfaces of devices are inevitably exposed to moisture and pollution, and it's the main reason for the occurrence and increase of corrosion processes (4, 5).

Atmospheric corrosion can be classified into categories such as dry corrosion, damp corrosion, and wet corrosion according to surface humidity. Humid and wet flow refers to electrochemical mechanisms, but dry flow refers to chemical mechanisms (6, 7).

The main type of corrosion: is atmospheric corrosion (the cause of oxidation is the contact of the metal object with oxygen and water vapor in the air). Rust develops faster when there are pollutants in the air in the form of chemically active substances. Liquid corrosion (occurs on metal equipment in a water environment). If we are talking about seawater, oxidation on it is significantly accelerated due to the high salt content of the liquid. Soil corrosion (metal equipment located on the ground is affected by this type). Chemical reactions begin and continue under the influence of chemical elements that make up the soil, groundwater, various types of seepage. Corrosion in metal products can manifest itself in different ways (8, 9).

- a continuous rusty layer or its separate parts are formed;
- small areas of rust penetrating the part are formed;
- deep cracks are formed;
- one of the alloy components is oxidized;
- deep penetration occurs throughout the volume;
- several symptoms appear at the same time;

The reasons can be of two types (10): Chemically, i.e., the metal dissolves as a result of chemical reactions with active substances. Electrochemical is related to the generation of an electric current when in contact with electroly solutions, under its influence electron exchange occurs in metals. This causes damage to the crystal structure and the formation of rust (11).

There are several main ways to protect metal equipment from corrosion (27):
- metal alloy;
- protective coatings (metal, non-metal);
- electrochemical protection;
- changes in the properties of the corrosive medium;
- rational product structure;

This is one of the really effective ways to increase the rust resistance of metals. Chromium, nickel, molybdenum, etc. is added to the alloy or metal during the alloying process alloying elements such as. This is one of the really effective ways to increase the rust resistance of metals. They lead to passivation of the metal, that is, the corrosion protection of the metal or alloy increases due to the stopping of the anode process (12).

Additional layers are artificially formed on the surface of the metal product to protect it from corrosion. Such coatings can be metallic or non-metallic. However, regardless of their composition, they are subject to the same requirements: good adhesion properties, durability, the ability to maintain their properties in an aggressive environment (13). Protection of metal equipment with metal coatings differs from other methods in its unequivocal effect. Non-metallic protective coatings are divided into inorganic and organic. Inorganic coatings, metal oxides, chromium, phosphorus compounds, etc. are used to protect metal equipment from corrosion with non-metallic coatings (28). Organic coatings include paint coatings, resins, plastics, polymer coatings and rubbers (14). Atmospheric corrosion has taken a large place in the destruction of metal equipment, and much attention is paid to the development of protective means and methods. The essence of the protection of metals from atmospheric corrosion inhibitors consists of chemical and physical interaction with moisture, oxygen and other corrosive substances, as a result of which substances neutral in terms of corrosion are formed and passivation of metal surfaces, hydrophobization, or both occur together (15). At the beginning of the 20th century, it was determined that some gas-containing chemical compounds have the ability to precipitate on the surface of metals and protect them from the harmful effects of the environment. These compounds are called volatile corrosion inhibitors or VCI - volatile corrosion inhibitors (16). Among the foreign samples, VIBATAN Metal Antiox 01792 can be distinguished. It is a universal concentrate of volatile corrosion inhibitors for ferrous and non-ferrous metals. Another volatile corrosion inhibitor with a high concentration of VIBATAN Metal Antiox 01801 can also be attributed here (17, 18). Its field of application is the creation of a protective coating against corrosion in the military industry and large machinery. The most domestically produced ЛИАК include derivatives and salts of aliphatic, aromatic, cyclic and heterocyclic amines (19, 20). These are mainly nitrites, nitrates, borates, phosphates, benzoate, nitrobenzoates. These are mainly nitrites, nitrates, borates, phosphates, benzoates, nitrobenzoates (21). In this regard, the issue of protection against atmospheric corrosion and reduction of losses during transportation and storage is very relevant. The application of inhibitors is one of the known methods of corrosion protection, as well as one of the most effective methods among the various methods of preventing the disintegration of a metal surface. The inhibitors synthesized by us are considered to be low-component, ecologically harmless and biodegradable, and especially "Green" inhibitors.

2. MATERIAL AND METHOD

2.1. Materials

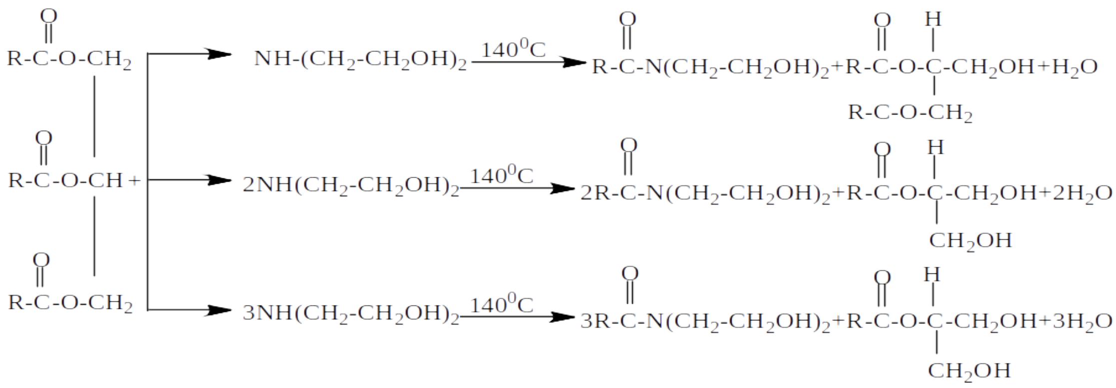
Cottonseed oil is obtained by cold pressing cotton seeds, after removing the fiber from them. This method makes it possible to preserve microelements and nutrients characteristic of natural cotton in the oil. Cotton seeds collected in the Republic of Azerbaijan. DEA (purity > 98.5% Merck®, Germany). Indicators of cotton seed:
Approximate seed weight: about 1 gram,
Approximate seed size: 1 cm,
Seed germination percentage: 90%,
Beginning of fruiting/flowering: in the year of planting,
Estimated yield: usually 10-20 flowers per plant

2.2. Synthesis

Amides of cottonseed oil and diethanolamine (DEA) in different mole ratios were synthesized. The reactions were carried out in a three-necked flask equipped with a stirrer, a heater and a thermometer. The reaction begins by weighing the required amount of cottonseed oil and filling into the flask, and is followed by turning on the heater and stirrer. As the temperature reaches 60-70°C, the required amount of DEA is weighed and added to the flask. It is followed by raising the temperature to 140°C and stirring at this temperature for 4 hours and cooling after the end of the reaction.

3. RESULTS AND DISCUSSION

3.1. The course of The Reaction



3.2. Instrumental Characterization

The structure was Studied by infrared spectroscopy method (IR spectroscopy) ALPHA IR-Fourier spectrophotometer of German BRUKER company.

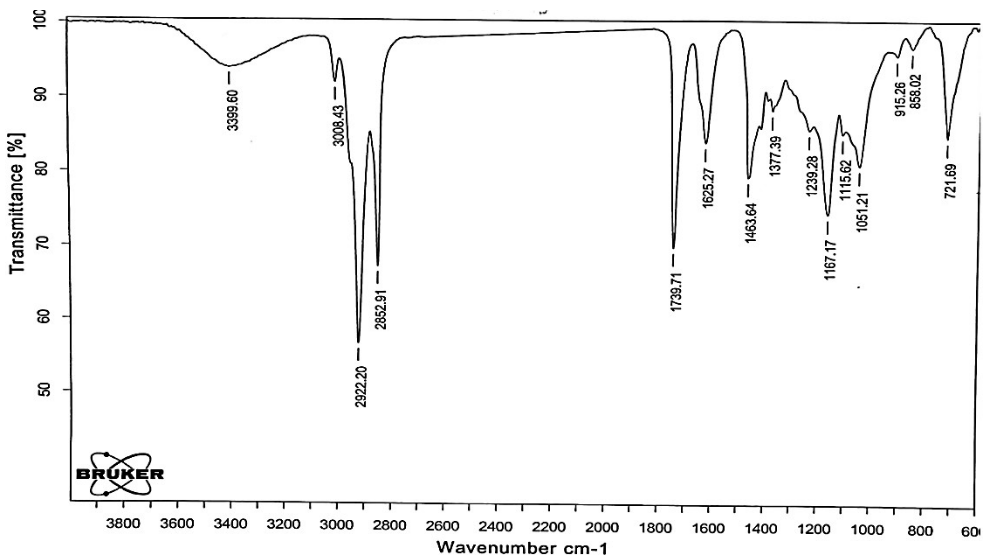


Figure 1: IR spectrum of the amide obtained in 1:1 mole ratio.

So, 250 g of cottonseed oil were taken for the purpose of obtaining amide in a 1:1 mole ratio. After the temperature reached 60-70°C, 30 g of DEA were added and the temperature was raised to 140°C. After stirring for 4 hours, the IR spectrum of the substance was plotted and determined (Figure 1).

The following absorption bands were observed in the spectrum:

721 cm^{-1} – mathematical vibration of C-H bond of CH_2 group;
1625 cm^{-1} – valence vibration of C-N bond of –CO-N- group (tertiary amide);
1739 cm^{-1} – C=O bond of the ester;
1051, 1239 cm^{-1} – C-N bond C-NH group;
1115, 1167 cm^{-1} – C-O-C bond of the ester;
1377, 1463, 2852, 2922 cm^{-1} – valence vibration of C-H bond of CH_3 and CH_2 groups;
3008 cm^{-1} – C-H bond of cottonseed oil C=C group.
3399 cm^{-1} – N-H bond of DEA and O-H bond of C-OH group coincide.

IR spectrum of the sample reveals that the reaction didn't run to completion. Absorption bands are observed at 1739, 1115, 1167 cm^{-1} characteristic for cottonseed oil and the ester. Observation of the absorption band of tertiary amide at 1625 cm^{-1} confirms obtaining the tertiary amide. In the region of 3365 cm^{-1}, an absorption band characteristic for O-H bond of C-OH group also appears and coincides with the absorption band corresponding to N-H bond.

For the purpose of obtaining the amide of cottonseed oil with DEA in a 1:2 mole ratio, 250 g of cottonseed oil were taken and filled into a flask, heated to 60-70°C, and at this temperature, 60g of DEA were weighed and added to it and stirred at this temperature for 4 hours. After the completion of the reaction, IR spectrum was plotted (Figure 2) and determined.

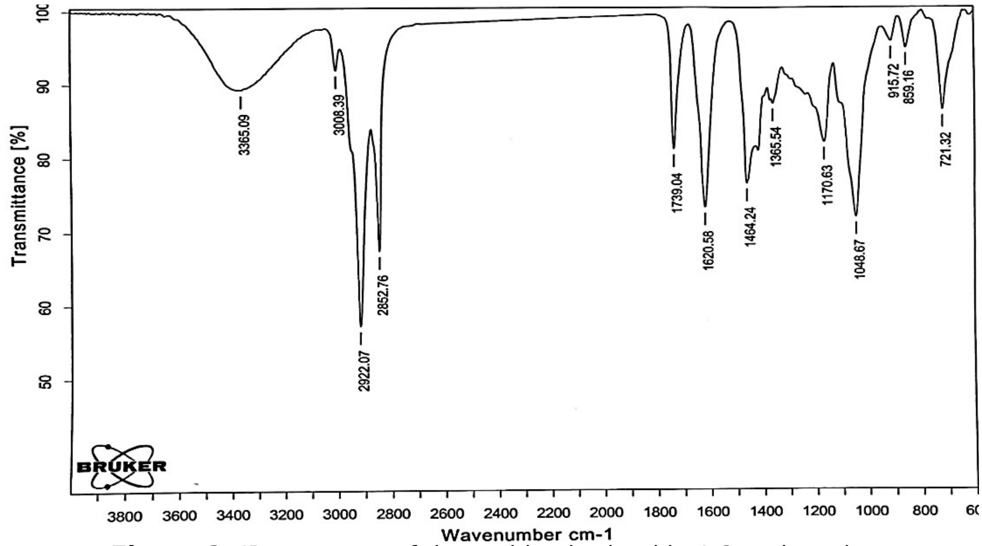


Figure 2: IR spectrum of the amide obtained in 1:2 mole ratio.

The following absorption bands were observed in the spectrum:

721 cm^{-1} – mathematical vibration of C-H bond of CH_2 group;
859, 915 cm^{-1} – deformation vibration of C-H bond of HC=C-group;
1048 cm^{-1} – valence vibration of C-N bond;
1170 cm^{-1} – C-O-C bond of the ester;
1739 cm^{-1} – C=O bond of the ester;
1620 cm^{-1} – valence vibration of C-Nbond of -CO-N-group (tertiary amide);
1365, 1464, 2852, 2922 cm^{-1} – deformation and valence vibrations of C-H bond of CH_3 and CH_2 groups;
3008 cm^{-1} – valence vibration of C-H bond of HC=CH-group;
3356 cm^{-1} – valence vibration of O-H bond of COH group.

The reaction didn’t run to complete even at 1:2 mole ratio. Absorption bands characteristic for the tertiary amide obtained at 1621 cm^{-1} proves the intensity of the absorption bands in comparison to 1:1 mole ratio. Absorption band intensity of C=O group of the ester characteristic for cottonseed oil at 1739 cm^{-1} was also decreased.

In order to obtain the amide of cottonseed oil and DEA in 1:3 mole ratio, 250 g of cottonseed oil were taken and filled into a flask, heated to 60-70°C, then 90 g of DEA were added to it, mixed at 140°C for 4 hours. IR of the obtained substance was determined by plotting the spectrum (Figure 3).

The following absorption bands were observed in the spectrum:

720 cm^{-1} – mathematical vibration of C–H bond of CH_2 group;
858, 915 cm^{-1} – deformation vibration of C-H bond of CH=C-group;
1047 cm^{-1} – valence vibration of C-N bond;
1185, 1209, 1257 cm^{-1} – C-O-C bond (a small amount) of the ester;
1364, 1421, 1464, 2852, 2922 cm^{-1} – deformation and valence vibrations of C-H bond of CH_3 and CH_2 groups;
1617 cm^{-1} – valence vibration of C-N bond of -CO-N-group (tertiary amide);
1739 cm^{-1} – C=O bond of the ester (a small amount)
3008 cm^{-1} – valence vibration of C-H bond of HC=C group;
3358 cm^{-1} – valence vibration of O-H bond of COH group.

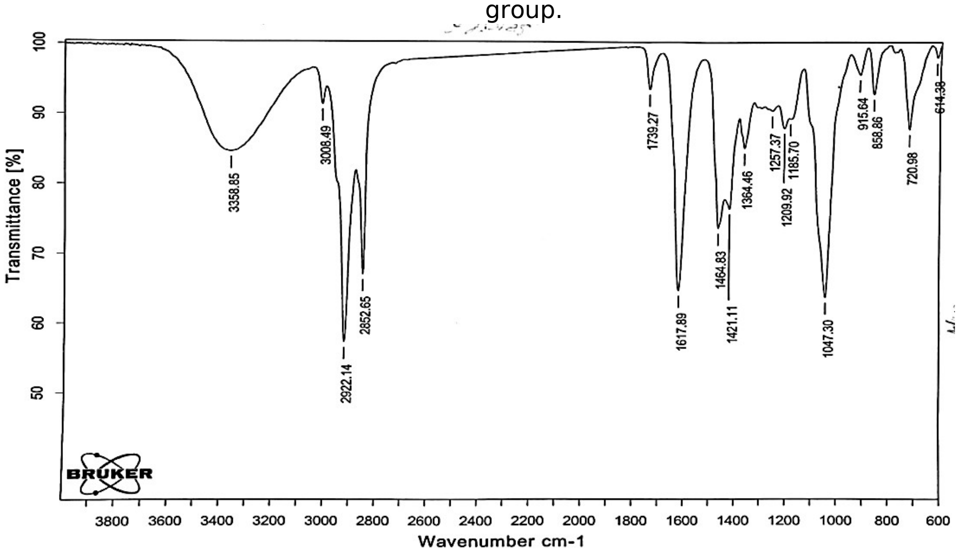


Figure 3: IR spectrum of the amide obtained in 1:3 mole ratio.

In the case of taking the initial substances in a 1:3 mole ratio, a small amount of cottonseed oil remained in the product obtained by the reaction. Intensity of the absorption band characteristic of cottonseed oil (1739 cm^{-1}) was very low. This ratio provides high intensity of the absorption band (1617 cm^{-1}) of C-N bond of the tertiary amide. The analysis of the spectra proved that the amide yield was higher by taking of the initial substances in this ratio. A small amount of unreacted cottonseed oil remained in the system. Physicochemical parameters of the synthesized amides were determined. The results are given in Table 1.

3, 5, 7, and 10% of synthesized amides were added to T-30 oil and physicochemical properties of the solutions were determined. The results are set into Tab. 2. Protective ability of the developed conservative liquids was determined by the direct tests in different structural chambers. The tests were carried out as in the first section. Purified and dried metal plates were tested in a thermohumidity chamber in a 0.001% H_2SO_4 solution in seawater. The obtained results are given in Table 2. As is evident from the table, the highest results were shown by the composition obtained by adding 1:2 mole ratio of 10% amide of cottonseed oil and DEA to T-30 oil. A metal plate immersed into a conservative liquid developed by this way was protected from corrosion for 205 days in “Г-4” hydrochamber, 165 days in seawater, and 160 days in a 0.001% sulfuric acid solution.

Also, the hydrochamber "Г-4" test results of the 10% solutions obtained are given in the form of a diagram (Fig. 4).

Test results of 10% solutions in the hydrochamber "Г-4" are given in the following diagram (Fig. 4).

Table 1: Physicochemical properties of the resulting amides.

Properties	Device	Method	Samples		
			Cottonseed oil+DEA (1:1 mole ratio)	Cottonseed oil+DEA (1:2 mole ratio)	Cottonseed oil+DEA (1:3 mole ratio)
Kinematic viscosity, mm^2/sec, 20°C	Stabinger SVM	ASTM D445	Impossibility of measuring	636.32	1350.4
Density, g/cm^3 20°C	DMA 4500M	ASTM D5002	Impossibility of measuring	0.9660	0.9792
Refractive index, 20°C	Abbemat 500	Device method	1.4778	1.4810	1.4821
Iodine number, gY/100 g	Methodics	GOST 2070-82	51.11	47.84	54.75
Acid number, mgKOH/g	Methodics	GOST 11362	1.13	1.86	3.50
Freezing point, °C	Stanhope seta	ASTMD 97	+3	0	+2

Table 2: Test results of the conservative liquids developed on the basis of the synthesized amides.

	Composition of conservative liquids		“Г-4” hydrochamber	Seawater	0.001% H_2SO_4
	T-30 oil, %	Amides, %			
1	97	Cott.oil+DEA 1:1,3	126	120	125
2	95	Cott.oil +DEA 1:1,5	152	140	147
3	93	Cott.oil +DEA 1:1,7	164	158	150
4	90	Cott.oil +DEA 1:1,10	185	160	157
5	97	Cott.oil +DEA 1:2,3	162	124	126
6	95	Cott.oil +DEA 1:2,5	136	132	130
7	97	Cott.oil +DEA 1:2,7	162	146	154
8	90	Cott.oil +DEA 1:2,10	205	165	160
9	97	Cott.oil +DEA 1:3,3	164	136	142
10	95	Cott.oil +DEA 1:3,5	165	148	146
11	97	Cott.oil +DEA 1:3,7	175	156	149
12	90	Cott.oil +DEA 1:3,10	184	165	154

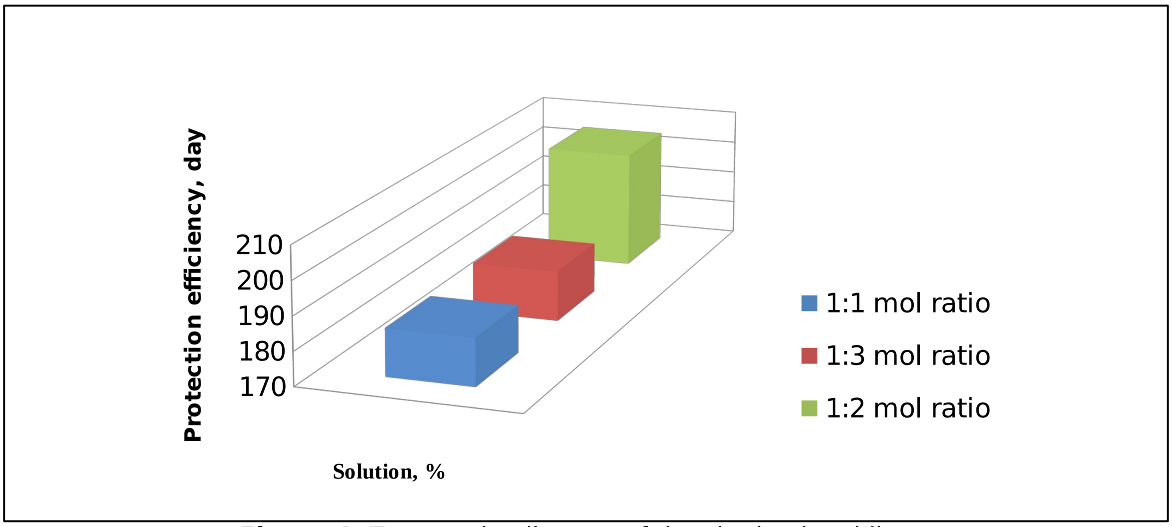


Figure 4: Test results diagram of the obtained amidines.

As is evident from the diagram, the solution of cottonseed oil and DEA shows the best results in 1:2 mole ratio.

If we look at the research works carried out in our country and abroad, tests were carried out in the "Г-4" hydrochamber by adding nitro compounds synthesized in the presence of nitric acid to T-30 turbine oil based on $C_{12}H_{24}$, $C_{14}H_{28}$ and $C_{16}H_{32}$-$C_{18}H_{36}$ α-olefins. A nitro compound synthesized on the basis of $C_{14}H_{28}$ α-olefin was observed for 108 days of corrosion protection (22). For the first time, amidoamines synthesized by reacting diethylenetriamine (DETA), triethylenetetraamine (TETA) and polyethylene polyamines (PEPA) with technical or distilled petroleum acids (PA) in different molar ratios are added to T-30 turbine oils at different concentrations (3, 5, 7 and 10%) preservation fluids were prepared and tested in different harsh environments. Preservation fluid with a 2:1 molar ratio of PA to PEPA showed a preservation effect for 41 days (23). K-17, Mifol KM, НГ-203Р and Mayacor can be preserved in a hydrochamber for 70 days to 100 days, the protective effects of which have been observed (24).

In other works, compositions of obtaining nitrated oil with Mg, Co, and Ni salts of natural petroleum acids were prepared and their properties as conservation liquid were studied. The obtained results show that the corrosion protection of the Steel-3 metal plate in the ″Г-4″ hydrochamber is weak, either alone or in the presence of compositions with Co, Ni and Mg salts of petroleum acid. While nitrous oil protected the steel-3 metal plate from corrosion for 55 days, the protection index with petroleum acid salts was between 45–62 days (25). In other works, compositions of obtaining nitrated oil with Mg, Co, and Ni salts of natural petroleum acids were prepared and their properties as conservation liquid were studied. The obtained results show that the corrosion protection of the Steel-3 metal plate in the ″Г-4″ hydrochamber is weak, either alone or in the presence of compositions with Co, Ni and Mg salts of petroleum acid. While nitrous oil protected the steel-3 metal plate from corrosion for 55 days, the protection index with petroleum acid salts was between 45–62 days (26).

4. CONCLUSIONS

1. The structures of the synthesized substances were confirmed using IR spectroscopy method, and physicochemical properties were determined. 3, 5, 7 and 10% solutions of cottonseed oil were prepared in T-30 oil and studied as conservative liquids. The best result was shown by a 10% solution in 1:2 mole ratio. It was determined that metal plates immersed in a 10% solution exhibited corrosion protection efficiency lasting 205 days in a hydrochamber, 165 days in seawater, and 160 days in a 0.001% H_2SO_4 solution.

2. As a result, it can be noted that, unlike other researche, our test results showed a relatively high protection effect and, besides being and economically efficient, ecologically safe, it has a simple structure in the manufacturing technology.

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