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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

**SYNTHESIS AND PROPERTIES OF NEW SURFACTANTS
BASED ON AMINO ALCOHOL AND EPICHLORHYDRINE**

Speciality: 2314.01 – Petrochemistry

Field of science: Chemistry

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The work was performed at the laboratory “Surface-active reagents and preparations” of the Institute of Petrochemical Processes named by acad. Y.H. Mammadaliyev of the Ministry of Science and Education of the Republic of Azerbaijan

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GENERAL DESCRIPTION OF THE WORK

Relevance of the topic and degree of elaboration.

One of the main pollutants of the hydrosphere is oil and oil products. Oil products are dumped into the rivers, seas and other water bodies of many countries of the world. The main reasons for spilling oil into water bodies are accidents in oil pipelines, oil wells, offshore fass, oil residues spilled into the sea with ballast water from tankers, oil and oil products spilled on the water surface because of military operations, wastewater from oil fields, and others. According to various estimates, up to 15-30 million tons of oil products are spilled into the world ocean every year because of accidents in sea wells, discharge of oil-contaminated waters from ships. 1% of ocean pollution by petroleum products is caused by natural seepage, 54% by marine transport and up to 23% by industrial waste.

Not only in the hydrosphere, but also in the atmosphere, a large amount of oil products is released every year. One of the specific types of hydrocarbon losses is oil and relatively light fractions of oil products entering the environment due to evaporation in reservoirs. This pollutes the air and causes losses of raw materials and changes in its composition. Gasoline losses in bases where oil products are stored amount to several thousand tons every year.

Pollution of both the hydrosphere and the atmosphere with oil and oil products is one of the global problems for humanity. Preventing this is a very important and urgent topic in front of the scientists of the world. Until now, several works have been done in the field of removing oil layers on the water surface and creating tools that slow down the evaporation of oil products. At the prospekte time, it is necessary to mention the creation of colloid-chemical means. However, since these compounds are organic substances, on the one hand they help to clean the environment, on the other hand, they are toxic to a certain extent and lead to its pollution. However, when using well-known complex salts such as ionic-liquid type compounds, the mentioned shortcoming disappears.

It is known that substances of ionic-liquid nature^{1,2} have recently been the center of attention of chemists all over the world. This is due

to a number of valuable properties inherent in them, which are important for the creation of "green chemistry" technologies that ensure environmental protection.

In the presented work, the synthesis of surface-active ionic liquids of aliphatic alcohols, higher monocarbonic acids, as well as alternative raw materials, chloroxypropyl ethers of vegetable acid fractions, which are ecologically safe for cleaning oil and oil products from the surface of water bodies.

The creation of means to slow down the evaporation of petroleum products is of interest both ecologically and economically. For this purpose, the development of ionic liquids of oligomeric nature, which can form a microlayer on the surface of oil products, based on chloroxypropyl ethers of polyatomic alcohols and ethanolamines, is of particular importance at the present time.

The object and subject of the research. Epichlorohydrin, higher alcohols, higher monocarboxylic acids, polyhydric alcohols, fatty acids, and amino alcohols, which are the basis of the synthesis of ionic-liquid surface-active substances (sufactans) as the object of research in the dissertation, and the subject of the research is the structure by investigating the application possibilities of cation-active surfactants - property dependencies are defined.

Research goals and objectives. It is the synthesis of ethanolamine salts of aliphatic mono- and polyhydric alcohols, higher aliphatic monocarbonic acids, as well as chloroxypropyl ethers of acid fractions of vegetable oils, determination of physico-chemical indicators of the obtained compounds and investigation of their application properties by studying structure-property dependencies.

Research methods. IR-, UV- and NMR-spectroscopy, conductometry, tensiometry and other physico-chemical analysis methods were used during the determination of the composition, structure, and properties of the purchased products.

¹Azizov A.G., Asadov Z.G., Akhmedova G.A. Ionic liquids and their applications Baku.: Elm. 2010. 589 p.

²Buettner C.S., Cognigni A., Schröder C., Bica-Schröder K. Surface-active ionic liquids: A review //Journal of Molecular Liquids 347 (2022) 118160.

The main provisions submitted to the defence:

- C₈-C₁₂, C₁₄ higher alcohols were chloroxypropylated with epichlorohydrin (ECH) in the presence of an alkaline catalyst in different mole ratios, and the obtained chloroxypropyl ethers were modified with alkanolamines. The colloidal-chemical parameters of aqueous solutions of triethanolamine (TEA) salts of esters with one degree of chloroxypropylation were determined at different temperatures.

- ethylene glycol, 1,2-propylene glycol, 1,3-butylene glycol and glycerol in different molar ratios (from 1:1 to 1:50) with ECH and obtained ethers were converted into ion-liquid salts with ethanolamines;

- the kinetic regularities of the chloroxypropylation reaction of glycerin in the presence of a pyridine catalyst were studied.

- higher monocarboxylic acids were oligomerized with ECH in the presence of an alkaline catalyst and cation-active ion-liquid salts were obtained by modifying the obtained esters with ethanolamines in the quaternization reaction.

- vegetable oil acid fractions are chloroxypropylated with ECH in the presence of NaOH (catalyst). The received non-ionogenic surfactants were modified by the -CH₂Cl group and turned into ionogenic surfactants;

- the oil collecting, oil dispersing, gasoline evaporation retarding properties of the synthesized ionic-liquid surfactants were studied.

Scientific novelty of the investigation. For the first time:

- ionic-liquid salts of aliphatic diatomic alcohols (C₈-C₁₄) chloroxypropyl ethers with ethanolamines (monoethanolamine (MEA), diethanolamine (DEA), TEA, methylaminoethanol (MAE) and isopropanolamine (IPA)) were synthesized, their physical-chemical, surface-active properties were studied. The oil accumulation and oil dispersing ability of the synthesized ion-liquid surface-active compounds was revealed.

- MEA, DEA, and TEA salts of chloroxypropyl ethers of polyatomic aliphatic alcohols (C₂-C₄) were synthesized and the obtained compounds were studied in detail. The ability of these compounds,

which belong to ionic-liquid type surface-active substances, to slow down the evaporation of oil products has been revealed.

- the kinetic laws of the reaction of chloroxypropylation of glycerol with ECH were studied, the corresponding kinetic equation was derived, the activation energy of the reaction, the value of the multiplication before the exponent was calculated.

- MEA, DEA and TEA salts of chloroxypropyl esters of higher aliphatic acids (C₈, C₉, C₁₁, C₁₂, C₁₅ and C₁₇) were synthesized, their physico-chemical parameters were determined, and these compounds were ionic-liquid type compounds with surface-active properties was found. It has been proven experimentally that the obtained substances can collect and disperse oil.

- ethanolamine (MEA, DEA, and TEA) salts of chloroxypropyl esters of ecologically safe vegetable acid fractions (separated from sunflower, corn, cotton, soybean, olive, flax and castor oils) were synthesized, and their physicochemical properties were studied. The oil collecting and dispersing abilities of these compounds, which belong to ionic-liquid type surface-active substances, were revealed by laboratory tests.

Theoretical and practical value of the work. By changing the structure of obtained surfactants and studying the structure-property dependences, effective oil collector and oil dispersant in water bodies polluted with oil and oil products of ionic-liquid surface-active salts of chloroxypropyl ethers of acid fractions of aliphatic monoatomic alcohols, higher aliphatic monocarboxylic acids, as well as plant-derived natural oils the possibility of its application as reagents has been determined.

Ethanolamine salts of chloroxypropyl ethers of aliphatic polyatomic alcohols are surface-active ionic-liquid type compounds, the possibility of using them as a reagent capable of retarding the evaporation of petroleum products and as a reagent that forms a microlayer on the surface of petroleum products in reservoirs has been revealed.

Personal participation of the author. All the results given in the dissertation were determined by the author himself. Setting the issues, conducting experiments and analyses, and summarizing the results

were performed with the direct participation of the author.

Publications. 30 scientific works were published on the topic of the dissertation, including 13 articles, 17 abstracts in international and republican conferences.

Approbation. The materials of the dissertation work were presented and discussed at the following conferences: XVIII National Chemistry Congress (Kars, 2004), 11th International Conference on Polymers and Organic Chemistry (Prague, 2004), 5th International Scientific Conference "Environment and Protection of Life Activity" (Sumgayit, 2004), European Polymer Congress (Portarosh, 2007), II International Scientific and Practical Conference (Astrakhan, 2007), European Polymer Conference (Garnano, 2008), dedicated to the 100th anniversary of Academician Hasan Aliyev (Baku, 2007), VI "Ecology and safety of life activity" International scientific conference (Sumgayit, 2007), academician A.M. Republican scientific conference dedicated to the 100th anniversary of Guliyev (Baku, 2012), All-Russian conference with international participation, dedicated. 75 th anniversary of the birth of B.B. Kormacheva (Cheboksary, April 19-20, 2012), XII International scientific conference of doctoral students, masters and young researchers "Actual Problems of Chemistry" dedicated to the 95th anniversary of the birth of national leader Heydar Aliyev (Baku State University, Baku, 2018), International scientific-practical conference and school of young students "Chemistry, chemical technology and ecology: science, production, education" (Dagestan State University, Russia, Makhachkala, 2018), Intern.Sci.Conf." Actual Problems of Modern Chemistry" dedicated to the 90th anniversary of acad. Y.H.Mammadaliyev Institute of Petrochemical Processes (Baku, 2019), Dedicated to the 100th anniversary of the national leader of Azerbaijan, Heydar Aliyev V International scientific conference of young researchers (Baku Engineering University, 2023), full member of ANAS, k.e.d., prof. Conference dedicated to the 90th anniversary of S.M. Aliyev on "Synthesis of petroleum chemistry, polyfunctional monomers, oligomers and polymers" (ARETN, IPCP, 2023).

The name of the institution where the dissertation work was performed. The dissertation work was performed in the laboratory "Surface-active reagents and preparations" of Y.H. Mammadaliyev Institute of Petrochemical Processes of NASA on the research work plan (State registration No. 0113Az2035).

The scope of the work. The dissertation consists of an introduction (12079 marks), six chapters: the first chapter (55746), the second chapter (8971), the third chapter (43985), the fourth chapter (23059), the fifth chapter (37016), the sixth chapter (15398), the conclusions (3872 sign), 200126 signs (204 pages) including 53 tables and 45 pictures from 229 literature references.

Introduction explains and justifies actuality of the topic, the goals and objectives, scientific novelty and practical value of the work.

The first chapter is devoted to the literature materials on the study of the methods of obtaining ionic-liquid type substances and the area of application is given in accordance with the topic. The synthesis and study of surfactants based on individual aliphatic diatomic alcohols, chloroxypropyl ethers of higher aliphatic monocarboxylic acids, and ethanolamines are shown. In the literature review, several unique properties and broad application prospects of ionic-liquid type compounds were discussed.

The second chapter describes the primary substances and their purification, the method of synthesis of ECH oligomers and their ethanolamine modifications, the composition and structure of chloroxypropyl ethers and their ethanolamine modifications, the kinetics of the chloroxypropylation reaction of glycerin, the oil-collecting ability of ethanolamine modifications, the methods of studying the gasoline evaporation retardation abilities of ethanolamine salts of chloroxypropyl ethers of polyatomic alcohols information is provided.

The third chapter deals with the preparation and research of surface-active oligomers of ECH based on aliphatic diatomic alcohols (C₈-C₁₄), the synthesis of ionic-liquid type salts as a result of their reaction with ethanolamines (MEA, DEA, TEA, MAE and iPA), obtained ionic-liquid surfactants- is dedicated to the study of oil accumulation and oil dispersion properties in distilled, drinking, sea

waters and Surakhani formation waters.

The fourth chapter synthesis of polyatomic aliphatic alcohols - ethylene glycol, 1,2-propylene glycol, 1,3-butylene glycol and chloroxypropyl ethers of oligomeric nature with ECH of glycerin, the synthesis of ethanolamine modifications of chloroxypropyl ethers of glycerin and glycols, the determination of their physicochemical parameters, the retarding properties of gasoline evaporation and the properties of glycerin. The kinetic regularities of the reaction of obtaining chloroxypropyl ether are discussed.

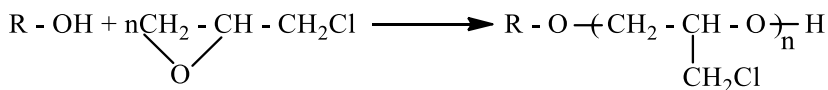
The fifth chapter synthesis of modified ethanolamines based on epichlorohydrin oligomers obtained with the participation of higher aliphatic monocarboxylic acids (pelargonic, capric, lauric, tridecane, palmitic, heptadecane, stearin and olein), their physico-chemical indicators, the results of the study of surface-active, oil-collecting and oil-dispersing properties has been presented.

The sixth chapter production of ionic liquid type salts with ethanolamine (MEA, DEA and TEA) based on ECH oligomers obtained with the presence of acid fractions of vegetable oils (sunflower, corn, cotton, soy, olive, flax and castor), properties and distillation of obtained Surfactants the study of oil accumulation and oil dispersing properties in drinking water, sea water and Surakhany formation water is explained.

THE MAIN CONTENT OF THE WORK

Synthesis and Properties of oligomeric salts of ionic liquid type based on Epichlorohydrin, Aliphatic Diatomic Alcohols (C₈-C₁₄) and Amino Alcohols

Oligomerization of ECH in the presence of higher aliphatic diatomic alcohols (octanol, nonanol, decanol, undecanol, dodecanol, tridecanol, tetradecanol) was carried out using NaOH (1% by alcohol, mass) as a catalyst. The reaction scheme can be described as follows:



where R-C₈H₁₇; C₉H₁₉; C₁₀H₂₁; C₁₁H₂₃; C₁₂H₂₅; C₁₃H₂₇ and

C₁₄H₂₉ are n-average degree of chloroxypropylation or average degree of oligomerization of ECH.

Physico-chemical parameters of the obtained ammonium salts, including specific electrical conductivity (κ) were determined. It was determined that these salts are ionic-liquid type. This is evidenced by the fact that the value of the specific electrical conductivity of their 0.5% aqueous solutions is significantly higher than that of distilled water (for distilled water, this indicator is equal to 0.001-0.0001 $\text{Om}^{-1}\cdot\text{m}^{-1}$).

When comparing ECH oligomers obtained in the presence of C₈-C₁₂ alcohols and modified ones synthesized based on ethanolamines, it is clear that the value of κ decreases when passing from the salt with DEA to the salt with TEA. This can be explained by the strengthening of the hydration of the cation with the increase in the number of hydroxyl ions.

Chlorinated derivatives with a degree of chloroxypropylation close to 1 based on octanol, nonanol, decanol, and dodecanol and ionic-liquid type surfactants based on TEA were purified by passing through silica gel. The values of the surface tension of the aqueous solutions of the obtained ionic-liquid salts at the air-water boundary were determined at four different temperatures (10, 20, 30 and 40°C) (Fig. 1).

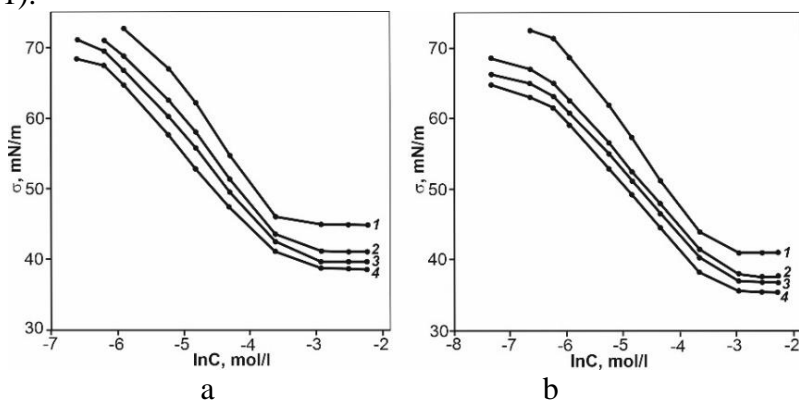


Figure 1. Surface tension isotherms of TEA salts of chloroxypropyl esters of octanol (a) and nonanol (b) at 1-10°, 2-20°, 3-30° and 4-40°C

Figure 1 shows surface tension isotherms obtained at 10, 20, 30

and 40°C in the presence of TEA salts of chlorooxypropyl esters of octanol in (a) and nonanol in (b). As can be seen from the figure, the surface tension isotherm shifts downwards with increasing temperature. It can be seen from the pictures that the value of surface tension stabilizes as the concentration of surfactants solution increases at all four temperatures. When stabilization of the surface tension values is observed, we can talk about the formation of micelles. The first value of the stabilization range corresponds to the critical micelle concentration (CMC)

Surface activity and specific electrical conductivity of TEA salts of chloroxypropyl esters of C₈-C₁₀ and C₁₂ higher aliphatic diatomic alcohols at 10, 20, 30 and 40°C were measured and colloid-chemical parameters were calculated (table 1).

Table 1.
Colloid-chemical parameters of TEA salts of chlorooxypropyl esters of C₈-C₁₀ and C₁₂ higher aliphatic diatomic alcohols

Surfactants	HLB	T= t+273	β	$\Gamma_{\text{maks}} \times 10^{10}$, mol/sm ²	A _{min} , Å	CMC $\times 10^2$, mol/l	π , mN/m	γ_{CMC} , mN/m	pC ₂₀
C ₈	31.3	283	0.78	2.33	71.2	4.24	30.2	44.0	4.30
		293	0.75	2.22	74.8	4.07	31.8	41.0	4.40
		303	0.72	2.09	79.3	3.99	31.5	39.5	4.47
		313	0.69	1.92	86.5	3.84	31.4	38.2	4.51
C ₉	30.9	283	0.83	2.21	75.0	4.11	34.1	40.1	4.60
		293	0.79	1.91	86.9	3.99	35.1	37.7	4.69
		303	0.74	1.74	95.5	3.73	34.2	37.0	4.76
		313	0.70	1.61	103.3	3.49	34.0	35.6	4.81
C ₁₀	30.4	283	0.86	2.14	77.6	3.87	37.7	36.5	4.91
		293	0.81	1.98	84.0	3.68	37.3	35.5	4.97
		303	0.77	1.78	93.5	3.47	37.1	34.1	5.03
		313	0.73	1.46	113.5	3.33	36.2	33.4	5.09
C ₁₂	29.4	283	0.92	2.50	66.4	3.71	39.9	34.3	5.52
		293	0.88	1.99	83.2	3.62	39.6	33.2	5.56
		303	0.85	1.63	101.7	3.53	39.2	32.0	5.58
		313	0.81	1.53	108.8	3.31	38.1	31.5	5.62

Note: β is the degree of deionization of the counterion; CMC - critical micelle concentration; γ_{CMC} surface tension value at CMC; Γ_{maks} - maximum adsorption; A_{min} - minimal surface area on cross-section of polar group; π_{CMC} - surface pressure; pC₂₀ - adsorption efficiency of surfactant.

As can be seen, as the temperature increases, the CMC

decreases. In the obtained surfactants, with the increase of alkyl radical length from C₈ to C₁₂ and temperature from 10° to 40°C, CMC, Γ_{\max} and γ_{CMC} decrease, while pC₂₀ and A_{min} - increase. With the increase in the length of the alkyl radical from C₈ to C₁₂ in ionic liquid salts, the CMC value decreases from $4.08 \cdot 10^{-2}$ to $3.62 \cdot 10^{-2}$ mol/l at 20°C. This is explained by the increase in the size of that radical and the strengthening of the hydrophobic-hydrophobic interaction.

Figure 2 shows the dependences of the correspondingly synthesized salts on the number of carbon atoms in the alkyl radical of CMC.

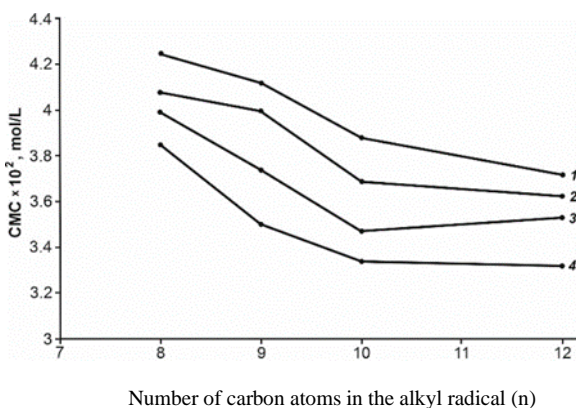


Figure 2. Graph of dependence of alkyl radical length of TEA salts of chloroxypropyl ethers of higher aliphatic diatomic alcohols C₈-C₁₀ and C₁₂ on CMC: 1-10°, 2-20°, 3-30° and 4-40°C

As can be seen from the figure, as the number of carbon atoms in the alkyl group increases, the CMC decreases. The prospective regularity is observed at the four temperatures studied. Table 1 shows the maximum adsorption values of the synthesized ion-liquid surfactant salts at different temperatures. As can be seen, as the temperature increases, Γ_{\max} decreases, and as the number of -CH₂-group in the chain increases, Γ_{\max} increases. When the temperature increases from 10 to 40°C, the value of the pC₂₀ indicator increases. However, it is known that pC₂₀ increases with increasing temperature in non-ionogenic surfactants and decreases in ionogenic and zwitterionogenic surfactants. Such a discrepancy can be explained by the fact

that even though the ionic-liquid surfactants we received are cation-active, their it contains a heterochain with $-\text{OH}$, $\sim\text{O}-\overset{\text{I}}{\text{C}}\text{H}-$ groups belonging to non-ionogenic surfactants. Figure 3 shows the dependence of pC_{20} on the number of carbon atoms in the alkyl radical of the ionic liquid salt. As can be seen from the figure, as the number of carbon atoms in the alkyl chain in the hydrophobic part of surfactants increases, the value of pC_{20} increases linearly (at 10, 20, 30 and 40°C).

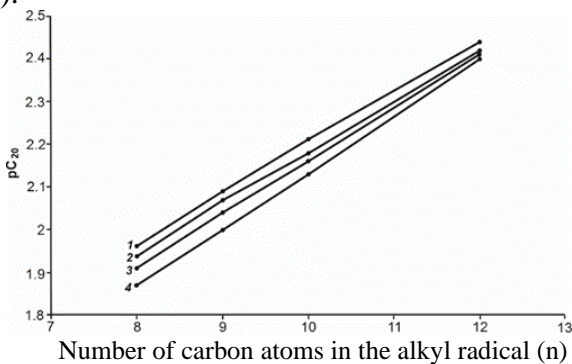


Figure 3. Graph of dependence of alkyl radical length on pC_{20} of TEA salts of chloroxypropyl ethers of higher aliphatic diatomic alcohols $\text{C}_8\text{-C}_{10}$ and C_{12} : 1-10°, 2-20°, 3-30° and 4-40°C

Deionization rates of the counterion of the synthesized ionic-liquid salts were determined by the conductometric method. In this way, the specific electrical conductivity of aqueous solutions of surfactants with different concentrations was determined. Based on the obtained results, the dependences between the concentration of ionic-liquid surface-active salts and the specific electrical conductivity were established (Figure 4). As it can be seen, as the thickness of surfactants increases, the κ line increases, and then this dependence continues along a relatively sloping line. The point of intersection of the lines is equal to the CMC of that surfactants. The degree of deionization of the counterion is equal to the ratio of the angular coefficients of these straight lines. The degree of ionization of surfactants is equal to $\alpha=1-\beta$.

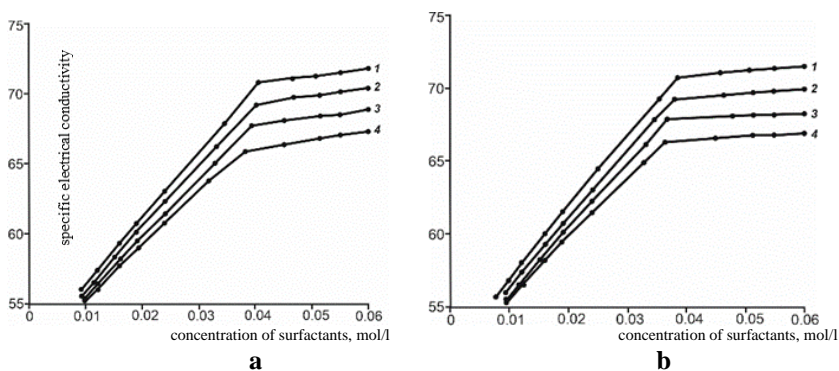


Figure 4. Density dependence of specific electrical conductivity of aqueous solutions of TEA salt of chloroxypropyl esters of octanol (a) and nonanol (b) at different temperatures: 1-10°, 2-20°, 3-30° and 4-40°C

Table 1 lists the values of surface pressures of ionic-liquid surfactant salts. As the temperature and the number of (CH₂) in the hydrophilic group increase, the value of π increases. In the TEA salt of chloroxypropyl ether of octanol, $\pi=31.8$ mN/m at 20°C, while in the TEA salt of chloroxypropyl ether of dodecanol, $\pi=39.6$ mN/m at 20°C.

The values of Gibbs free energy (ΔG_{mis}^0), enthalpy (ΔH_{mis}^0) and entropy (ΔS_{mis}^0) of micellar formation process at the water-air boundary of TEA salts of C₈-C₁₀ and C₁₂ chloroxypropyl ethers were calculated (table 2). As can be seen, the values of ΔG_{mis} are negative ($\Delta G_{mis}<0$). So, the process of mycelial formation is a self-paced process. As the temperature and the number of -CH₂- groups in the alkyl radical increase, the value of ΔG_{mis} decreases. As the number of CH₂ groups in the alkyl radical increases, ΔH_{mis}^0 decreases and ΔS_{mis}^0 increases.

The oil collection and dispersing properties of the ammonium salts of ECH oligomers obtained with the presence of aliphatic alcohols, undiluted and in the form of a 5% aqueous dispersion of Ramana oil on the surface of distilled, drinking, sea (Caspian Sea) waters and Surakhany formation waters (density at 20°C is 860 kg/ m³, kinematic viscosity 16.8 sSt) was studied on the example of a thin layer (≤ 0.17 mm).

Table 2.

Thermodynamic parameters of micellization and adsorption processes of TEA salts of chloroxypropyl ethers of C₈-C₁₀ and C₁₂ higher aliphatic diatomic alcohols

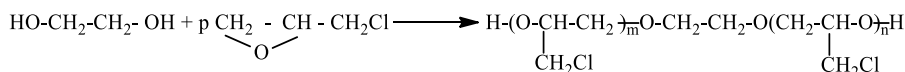
Alkyl radical	T=t+273, K	ΔG_{mis} , kC/mol	ΔG_{ad} , kC/mol	ΔS_{mis} , kC/mol K	ΔH_{mis} , kC/mol	ΔS_{ad} , kC/mol K	ΔH_{ad} , kC/mol
C ₈	283	-13.2	-14.5	0.0360	-3.05	0.0470	-1.25
	293	-13.6	-15.1				
	303	-13.9	-15.5				
	313	-14.3	-15.9				
C ₉	283	-13.7	-15.3	0.0366	-3.34	0.0552	0.32
	293	-14.0	-15.9				
	303	-14.4	-16.4				
	313	-14.8	-16.9				
C ₁₀	283	-14.2	-16.0	0.0368	-3.79	0.0602	1.12
	293	-14.6	-16.4				
	303	-14.9	-17.1				
	313	-15.3	-17.8				
C ₁₂	283	-14.9	-16.5	0.0388	-3.86	0.0700	3.32
	293	-15.2	-17.2				
	303	-15.6	-18.0				
	313	-16.0	-18.5				

The oil collection efficiency of surfactants was evaluated by the oil collection coefficient (K) and retention time (τ), which shows how many times the surface area of the initial oil layer is reduced. It was determined that the 5% aqueous solution of the TEA salt of chloroxypropyl ether of decanol with n=2.5 has a higher oil collection effect ($K_{\text{max}}=30.4$) than other surfactants. In general, as the length of the alkyl chain increases from C₈ to C₁₂ in the synthesized surfactantas, while the number of hydroxyethyl groups increases from MEA to TEA, the oil collection capacity increases. Due to the long alkyl chain in tetradecanol-based surfactants, the low diffusion rate of surfactants (especially in waters with a high degree of mineralization) had a negative effect on the oil accumulation effect.

Chloroxypropylation of C₂-C₄ polyatomic aliphatic alcohols, their modification and study of the physico-chemical and applied properties of the obtained products

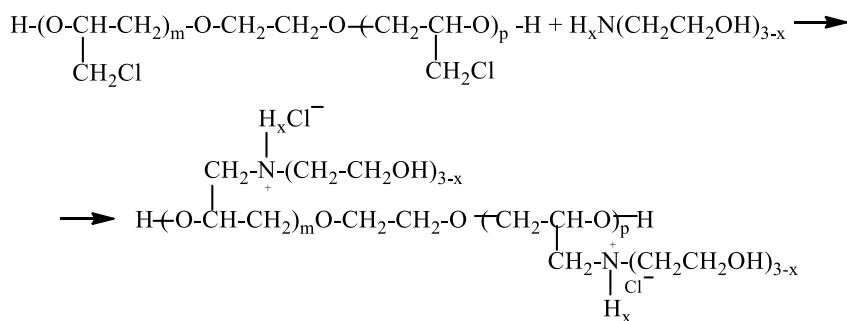
Polyatomic aliphatic alcohols - ethylene glycol, 1,2-propylene

glycol, 1,3-butylene glycol and glycerol were chlorooxypropylated with ECH at 140-160°C in a ratio of five moles. NaOH (3.0% mol of polyol) was used as a catalyst. The reaction time was 32-36 hours. In the case of ethylene glycol, the reaction scheme can be shown as follows:



where $p=m+n$

Ethylene glycol chlorooxypropyl ethers (EGCOE) modified with MEA, DEA and TEA in an equimolar ratio of chlorooxypropylene manganese to ethanolamine at 55-60°C for 18-20 hours follow the Menshutkin reaction scheme shown below:

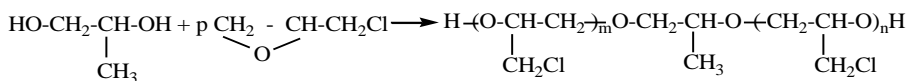


where $x=0, 1, 2$.

Conversion of ethanolamines was 85-92%. The surface activities of the synthesized salts were studied at the kerosene-water phase boundary by stalagmometric method. It was determined that when ethers are modified with ethanolamines, their surface activity increases, while esters modified with DEA and TEA show higher surface activity. Based on the values of specific electrical conductivity, it can be said that the ammonium type salts of EGCOE (except for the MEA salt of EGCOE with $n=47.71$) are ionic liquid type salts. The ability of a number of synthesized modifications of EGCOE to retard the evaporation of A-92 gasoline was revealed by tests. The highest retardation effect (16.9% in 3 days) in case of rinsing is shown by ether modified with MEA with $n=9.17$. The MEA-modified ether with $n=4.53$ in the form of a 5% aqueous solution is the

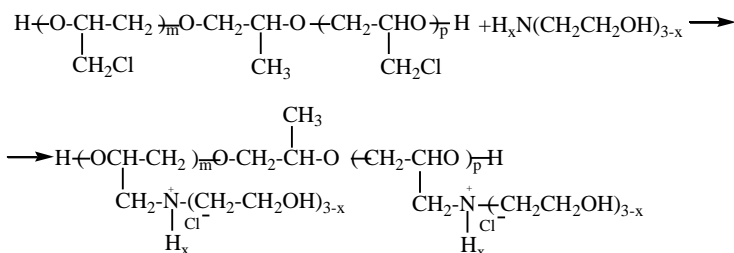
most effective (14.4% retardation effect in 2 days).

Chloroxypropylation of 1,2-propylene glycol in the above-mentioned way follows the following scheme:



where $p=m+n$

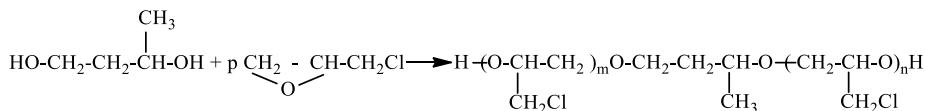
The reaction scheme of chlorooxypropyl ether of propylene glycol (PGCOE) with ethanolamines (MEA, DEA, TEA) is described as follows:



where $x=0, 1, 2$.

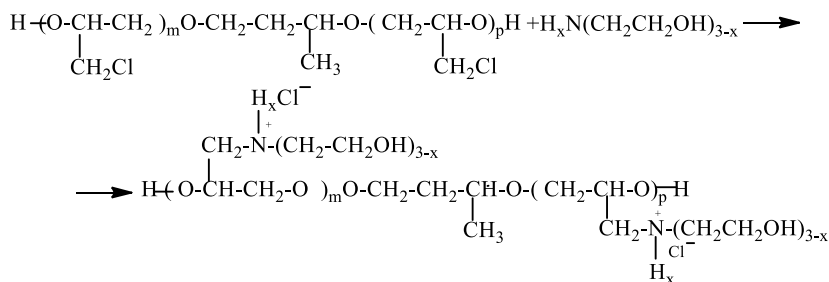
Based on the κ values of the aqueous solutions of the synthesized salts, it was determined that these salts have an ionic-liquid nature. When PGCXOE is treated with ethanolamines, their surface-active properties increase in most cases. The highest surface-active property is the MEA salt of PGCXOE with $n=14.01$ ($\gamma=5.3-3.6$ mN/m in the concentration range of 0.5-5%) and the TEA salt of the ether with $n=47.95$ (in the concentration range of 0.5-5% $\gamma=5.3$ mN/m). The ability of synthesized PGCXOE salts with MEA, as well as modified with TEA with $n=47.95$, to retard gasoline evaporation was studied. It was determined that the MEA modification of ether with $n=4.67$ shows higher results. The undiluted reagent showed a retardation effect of 33.2% and the 5% aqueous solution showed a retardation effect of 23.5% within 1 day. The MEA-modified ether with $n=47.95$ showed 16.0% inhibition in 1 day as a 5% aqueous solution. Then this ability decreases to 7.83% within 14 days.

Chloroxypropylation of 1,3-butylene glycol proceeds according to the following scheme:



where $p=m+n$

MEA, DEA and TEA modifications of chlorooxypropyl ethers (BGXOE) with different degree of chlorooxypropylation of 1,3-butylene glycol ($n=2.65, 9.16, 28.24$ and 46.52) were synthesized. The reaction scheme of BGC OE with ethanolamines is as follows:



where $p=m+n$

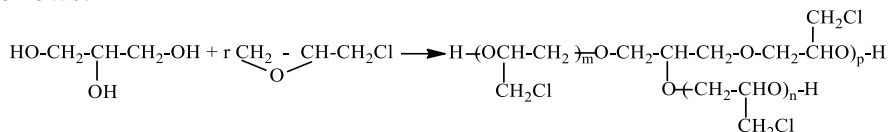
The values of surface tension of 5% aqueous solutions of MEA, DEA and TEA salts of BQXOE with $n=2.65$ are equal to 14.2, 9.4 and 8.9 mN/m, respectively. For comparison, let's note that the surface tension value of 0.5% aqueous solution of BGC OE with $n=2.65$ is 18.3 mN/m. Therefore, the surface-active properties of the synthesized salts are higher than the original ether.

The specific electrical conductivity of BGC OE ($n=2.65, 9.16, 28.24$ and 46.52) salts with MEA is lower than the electrical conductivity of salts with DEA and TEA. The specific electrical conductivity of these salts is many times higher than water, so they can be considered substances of ionic-liquid nature.

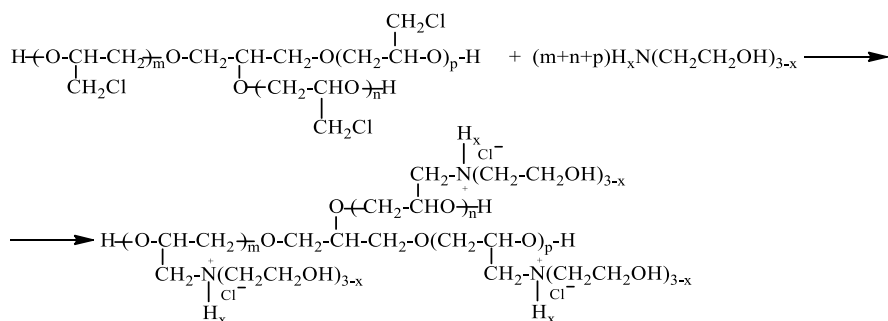
The ability of MEA ($n=9.16$ and 28.24) and TEA ($n=46.52$) salts of BGC OE with a high degree of chlorooxypropylation to retard gasoline evaporation was studied. TEA-modified chlorooxypropyl ether with $n=46.52$ is more effective. The undiluted reagent has a retardation effect of 40.86% in 1.5 hours, 29.08% in 2.5 hours, and 12.82% in 1 day. After 3 days, this amount becomes 10.51%. The results with 5% aqueous solution are slightly lower (22.73, 25.82,

12.39 and 8.29, respectively). After 10 days, the results for both application cases are close to each other (6.46-6.85%). The MEA salt of chlorooxypropyl ether with n=9.16 exhibits 12.8% retardation in solution form in 2 days.

The reaction scheme of glycerol with ECH can be described as follows:



The modification of chlorooxypropyl ethers of glycerin (n=2.27, 4.26, 9.29, 26.17 and 47.5) (QLCOE) with ethanolamines also follows the Menshutkin reaction scheme mentioned earlier:



The values of specific electrical conductivity suggest that all the synthesized ethanolamine salts of QLCOE are ionic-liquid type compounds.

Surface-active properties of QLCOE and their ethanolamine salts with different degrees of chlorooxypropylation were studied at the water-kerosene interface. It has been established that QLCOE are themselves surfactants. Their surface-active properties are significantly strengthened when their ethanolamine salts are taken. The MEA, DEA and TEA salts of QLCOE with n=9.29 taken in the concentration range of 0.025-5.0% show higher surface-active properties than other ethanolamine-modified ones ($\gamma=8.1-4.0$, $9.4-4.0$ and $10.1-3.4$ mN/m). The highest surface-active property is shown by the TEA salt of the ether with n=26.17. A 5.0% aqueous solution of this salt reduces the surface tension at the interface with kerosene from 46-46.5 mN/m to

2.0 mN/m.

The DEA salt of glycerol ester with $n=4.26$ exhibits a retardation effect of 66.7% within 1 day, 10.1% after 8 days, and 9.4% within 19 days. The salt of ether with TEA, which is $n=47.55$, shows an effect of 19.1% in 1 day, 13.8% in 2 days, 9.0% in 3 days, 6.9% in 4 days, and 5.6% in 19 days. Ethanolamine salts of QLCOE are superior to ethanolamine salts of chloroxypropyl ethers of other synthesized glycols in retarding gasoline evaporation.

Taking this into account, it is appropriate to determine the regularities of the chloroxypropylation reaction of glycerol and a number of kinetic parameters of the reaction. The kinetics of this reaction was studied by the manometric method and the influence of various factors on the overall rate of the reaction was studied. Glycerin chloroxypropylation was carried out without a catalyst and mainly in the presence of KOH, NaOH, pyridine, triethylamine, as well as the acidic catalyst H_2SO_4 . The chloroxypropylation reaction of glycerol proceeds poorly in the absence of a catalyst. In the initial minutes, the reaction is faster in the presence of pyridine than other basic catalysts. But after 5.0% conversion of ECH, the reaction rate is even higher in the presence of triethylamine. In the presence of KOH and NaOH, the reaction is noticeably weaker.

The chloroxypropylation reaction of glycerol was carried out in various solvents in the presence of pyridine. It should be noted that the initial mixture of glycerol+ECH is insoluble in water, acetone, CCL_4 and xylene. However, at $160^\circ C$, the reaction can be carried out in both polar (acetone) and non-polar (isooctane, benzene) solvents.

Chloroxypropylation reactions were carried out under the condition of changing the concentration of glycerol, ECH, pyridine (separately) and the rate constants for glycerol (1.66), ECH (1.29) and pyridine (1.56) were calculated.

We can write the rate equation for the chloroxypropylation reaction of glycerol in the presence of pyridine as:

$$W = kC_1^n \cdot C_2^m \cdot C_3^p$$

where k is the rate constant of the reaction, C_1 , C_2 , C_3 are concentrations of glycerol, ECH, and pyridine, respectively; n , m and p are the reaction formulations according to their solidities,

respectively.

Taking into account the found values of the compositions, the reaction rate equation looks like this:

$$W = kC_1^{1.66} \cdot C_2^{1.29} \cdot C_3^{1.56}$$

In order to determine the activation energy of the reaction, the kinetic curves of the chloroxypropylation process at several temperatures at constant concentrations of the reaction components were constructed. Based on the values of the initial velocities (W_0) calculated from the kinetic curves, the rate constant for each temperature was determined from the kinetic equation:

$$k_{140} = 1,22 \cdot 10^{-3} \cdot 1^{3,51} \text{mol}^{-3,51} \cdot \text{s}^{-1};$$

$$k_{150} = 4,80 \cdot 10^{-3} \cdot 1^{3,51} \text{mol}^{-3,51} \cdot \text{s}^{-1};$$

$$k_{160} = 5,81 \cdot 10^{-3} \cdot 1^{3,51} \text{mol}^{-3,51} \cdot \text{s}^{-1}$$

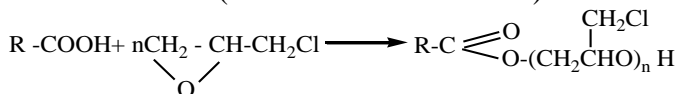
Based on the Arrhenius equation $2.303 \lg k_1/k_2 = E/R(1/T_2 - 1/T_1)$, $\ln k - 1/T$ dependence was established. In the equation, k_1 and k_2 are the rate constants of the reaction at absolute temperatures T_1 and T_2 , respectively, R -universal gas constant (8.314 C/mol×K). The activation energy of the chloroxypropylation reaction of glycerol is equal to (E) 120.15 kC/mol.

Based on the calculated values of the activation energy and the pre-exponential multiplication, the Arrhenius equation takes the following concrete expression:

$$k = A \times e^{-E/RT}; A^{\text{or}} = 2.38 \cdot 10^{12}.$$

Production and study of oligomeric salts of ionic liquid type based on ECH, higher aliphatic monocarboxylic acids and amino alcohols

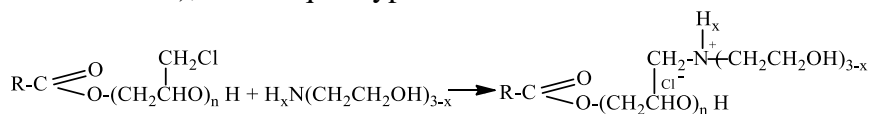
The reaction scheme of chloroxypropylation of pelargonic, capric, lauric, tridecane, palmitic, heptadecane, stearic and oleic acids in the presence of ECH (in different molar ratios) is as follows:



where R is the alkyl radical of the acid, n is the average degree of chloroxypropylation.

Oligomerization was carried out in an autoclave at 140-160oC using basic catalysts (3% mol acid) for 22-26 hours. During synthesis, pelargonic acid with n=3.72, n=4.68, capric acid with n=2.78, n=4.92, lauric acid with n=1.3, n=1.7, n=2.7, tridecanoic acid with n=0.46, n=2.0, n =2.42, n=2.85, n=9.53, palmitic acid n=0.91, n=2.12, n=3.7, heptadecanoic acid n=0.9, n=1.6, n=1.9, stearic acid n=4.87, olein chloroxypropyl esters of acid with n=0.79, n=1.33, n=3.83, n=9.36 were obtained.

As a result of the interaction of the synthesized chloroxypropyl ethers with MEA, DEA and TEA (quaternization reactions in the equimolar ratio of chloroxypropylene manganese to ethanolamine), ionic-liquid type salts were obtained:



where x=0, 1, 2.

Surface activity and specific electrical conductivity values of aqueous solutions of synthesized esters and their ethanolamine salts at the kerosene-water phase boundary were determined. Comparisons show that when chloroxypropyl ethers are modified with ethanolamines, their surface activity increases in most cases. The fact that the values of the specific electrical conductivity have increased many times and are several times higher than water indicates that these salts are ionic-liquid in nature. The oil accumulation and oil dispersing capabilities of chloroxypropyl ethers of higher monocarboxylic acids and their ethanolamine modifications were studied on a thin layer of oil spread over mineralized waters of varying degrees. It has been determined that ethanolamine salts act as an accumulator-disperser on the oil layer in distilled and drinking water, and as a dispersant in sea and Surakhani reservoir waters. Among ethanolamine salts, TEA salt has a higher effect. Among the acids, the maximum oil accumulation coefficient of the 5% aqueous dispersion of the TEA salt of chloroxypropyl ether of palmitic acid with n=3.7 was equal to 17.4.

Synthesis of ECH oligomers, their amino alcohol modifications, surface-active, oil-collecting and dispersing properties obtained with the presence of vegetable oil acid fractions

Acidic fractions separated from vegetable oils (sunflower, corn, cotton, soybean, olive, linseed and castor) were oligomerized in the presence of ECH. The reaction was carried out under conditions of monocarboxylic acids. The reactions were carried out in an autoclave made of steel, equipped with a heater and a stirrer for 20-24 hours at a temperature of 140-160°C in the presence of a basic catalyst (sodium hydroxide 3% by mol acid).

As a result of the reaction, sunflower oil acid fraction $n=1.93$, corn oil acid fraction $n=2.16$, cotton oil acid fraction $n=1.54$, soybean oil acid fraction $n=1.71$, olive oil acid fraction $n=1.92$, linseed oil acid chloroxypropyl ethers of fraction $n=1.70$ and castor oil acid fraction $n=4.22$ were obtained. Ion-liquid type salts were obtained by interaction of synthesized ECH oligomers MEA, DEA, TEA. These reactions were carried out at 55-60°C for 7-8 hours in a 1:1 mol ratio of ECH origin mangas to ethanolamines. It was determined by the stalagmometric method that the surface activities of ethanolamine salts of chloroxypropyl ethers of vegetable oil acid fractions are significantly higher than primary ethers. The increase in specific electrical conductivities confirms that these salts are ionic-liquid in nature.

Ethanolamine salts of chloroxypropyl esters of vegetable oil acid fractions have mainly oil-accumulating and oil-dispersing effects in soft waters, and mainly mixed oil-accumulating-oil-dispersing effects in hard waters. The maximum oil accumulation coefficient in sea water was equal to 15.2.

CONCLUSION

1. Chloroxypropyl ethers of octyl, nonyl, decyl, undecyl, dodecyl and tetradecyl alcohols with a degree of chloroxypropylation up to 6 were synthesized with the presence of an alkaline catalyst, and they were converted into cation-active ionic liquid type salts

by quaternization reaction with ethanol- and isopropanolamines, their structures were confirmed, physical - chemical properties, as well as surface activities were studied. It was determined that the TEA salt of decyl alcohol with $n=2.5$ of chloroxypropyl ether decreased the value of surface tension at the kerosene-water interface from 46.5 mN/m to 5.48 mN/m (at $t=17^{\circ}\text{C}$), at the air-water interface. and the TEA salt of chloroxypropyl ether of nonyl alcohol with $n=1$ reduces from 72.5 mN/m to 31.8 mN/m (at $t=20^{\circ}\text{C}$) [4,6,10,11,15,19,23 ,30].

2. The surface tension and specific electrical conductivity of aqueous solutions of TEA salts of chloroxypropyl ethers of octyl, nonyl, decyl and dodecyl alcohols with $n=1$ at different temperatures (10, 20, 30 and 40°C) were determined and, based on them, important colloid-chemical parameters (critical micelle adhesion density, value of surface tension at critical micelle adhesion density, maximum adsorption, minimal surface area on the cross-section of the polar group, surface pressure, adsorption efficiency of surfactant), as well as thermodynamic parameters of micellar adhesion and adsorption processes (Gibbs energy of micellar adhesion and adsorption processes, enthalpy, entropy) was calculated [24].
3. By chloroxypropylating ethylene glycol, 1,2-propylene glycol, 1,3-butylene glycol and glycerol with ECH in different molar ratios (from 1:1 to 1:50), oligoethers with different degrees of chloroxypropylation were obtained, which were treated with ethanolamines (MEA, DEA and TEA) modified and converted into ionic liquid type salts. It was determined that the TEA salt of chloroxypropyl ether of glycerin with $n=26.17$ (5% solution) reduces the value of the surface tension between kerosene-water phases from 46.5 mN/m to 2.0 mN/m [2,5,9 ,26,28].
4. The kinetic regularities of the chloroxypropylation reaction of glycerol were studied, and based on the effect of the concentration of the components on the speed of the reaction, their compositions were determined (1.66 for glycerol, 1.29 for ECH, 1.56 for the catalyst), and the activation energy of the reaction ($E=120.15$ kC/mol) was calculated. [8].

5. Pelargonic, capric, lauric, tridecane, palmitic, heptadecane, stearic and oleic acids were chloroxypropylated with EXH in the presence of the main catalyst in various proportions and cation-active surfactants were synthesized by their quaternization reaction with ethanolamines, their physicochemical parameters were determined and their high surface area have been determined to have activity. Thus, $\sigma=2.2$ mN/m was equal to TEA salt of chloroxypropyl ether of palmitic acid with $n=2.12$ [1,3,12-14,16,17,20-22,25,27,28].
6. The acid fractions of sunflower, corn, cotton, soybean, olive, flax and castor oils were oligomerized with ECH in different mole ratios with the presence of NaOH catalyst. Obtained oligoethers were modified with ethanolamines and turned into cation-active surfactants. These substances were found to have high surface activity at the kerosene-water interphase boundary ($\sigma=3.6$ mN/m in 0.5% aqueous solution) [1,7,13].
7. Laboratory tests were carried out on distilled, drinking, sea and formation waters as a thin layer of Raman oil, it was determined that salts obtained on the basis of higher alcohols and higher acids (as well as acid fractions) show a high oil accumulation and oil dispersing ability ($K_{\max.}=20.3$, $\tau\sim 70$ hours) [1,10,25].
8. The ability of surfactants of the ionic liquid type obtained on the basis of polyatomic alcohols was found to reduce the evaporation losses during the storage of gasoline under static conditions (66.7% retardation in one day) [2,26,28].

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