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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

STUDY OF SURFACE ACTIVE OLIGOMER NATURE PROPOXY DERIVATIVES OF C_8-C_{18} ALYPHATIC AMINES

Speciality: **2314.01 - Petrochemistry**

Field of science: **Chemistry**

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Baku-2021

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GENERAL CHARACTERISTICS OF WORK

Relevance and development of the topic. The world's population has been growing rapidly over the last century and will increase over the next 50 years. This increase in the world's population and the fact that the population is located mainly in industrialized cities lead to a global problem in the energy industry. Most of the high-capacity energy sources in nature are of hydrocarbon origin. Over the past 40 years, oil and natural gas have dominated more than half of the world's energy industry[1]. Approximately 71% of the crude oil fields and 69% of the natural gas fields are located in Russia and Asia (Central Asia and the Arabian Peninsula) sent to anywhere in the world[2]. Oil is often transported by tankers across three oceans. Thus, accidents during the transportation of oil in the oceans and seas are inevitable[3]. This is the same as the danger that may arise during the extraction of oil.

According to historical facts, thousands of tons of oil and oil products have been wasted during numerous accidents, resulting in the pollution of large areas of water bodies. The largest pollution in the last 60 years occurred in 1964 (about 0.6 billion barrels) and 2005 (about 0.4 billion barrels)[4]. Oil and oil products flowing from tankers spread over water bodies and have an extraordinary impact on nature. It is necessary to clean crude oil and oil products from water basins in a short time. In the first stage of purification, the oil is collected mechanically and removed by pumps. The cleaning of a thin layer of oil on water bodies takes place in the second stage. Thin layers of oil can upset the balance in nature.

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[1] Abas, N. Review of fossil fuels and future energy technologies / N.Abas, A.Kalair, N. Khan // Futures, -2015.Volume 69, -p.31-49.

[2] Rempel, H., Geographical Distribution of Oil and Natural Gas Deposite-Different Means of Transportation to the Consumption Centers // Pipeline Technology, - Berlin, -2006, -p.1-11.

[3] Dittmar, M. Regional Oil Extraction and Consumptions: A simple production model for the next 35 years // BioPhysical Economics and Resource Quality, - 2016. 1:7, -p.1-19.

[4] Etkin, D., Analysis of Oil Spill Trends in the United States and Worldwide // International Oil Spill Conference, -2001, -p.1291-1300.

Thus, the oil layer creates an additional environment between air and water, preventing the dissolution of oxygen in the air, and the sun's rays entering the lower aquifers of the oceans and seas. The vital activity of flora and fauna within water basins, which cannot be enriched with oxygen and deprived of sunlight, is weakening.

One of the most important branches of chemistry is the rapid accumulation of thin layers of oil in order to preserve the balance of nature. To do this, various surfactants are synthesized and investigated. About 10% of the demand for surfactants (more than 12 million tons) falls on nitrogen compounds. Although such substances have been known for about 50 years, they have been used more in the last 20 years due to their high efficacy surfactants. When surfactant collects oil from the water surface, the negative impact on the environment is relatively reduced.

Object and subject of research. The object of the dissertation research is synthesis and generalization of the properties of surface-active oligomeric propoxylic derivatives of various chains of alkylamines. The subject of the study covers how surface activity and petrocollecting and dispersing properties change as a result of changes in the composition and structure of substances obtained by oligomerization of amines with propylene oxide.

Objectives and tasks of the research. The aim of this research is to develop new oligomeric surfactants based on higher aliphatic amines (C_8, C_9, C_{12}, C_{16} and C_{18}) and propylene oxide (PO), their aliphatic monocarboxylic acids, ethylene chloride, salts with chloride, bromide and orthophosphate acids, as well as to carry out their identification by modern research methods, to determine the main physical and chemical parameters, including the main surface activity parameters and to study in detail the properties of petrocollecting property aimed at removing environmentally harmful thin oil layers from the surface of their water basins. The tasks of the research work are as follows:

- obtaining surfactants of oligomeric nature from the interaction between aliphatic amines C_8, C_9, C_{12}, C_{16} and C_{18} and PO and the study of their structure and properties,
- obtaining organic salts from the interaction of propoxylates

obtained on the basis of PO with monocarboxylic acids and a comparative study of their surface activity, electrical conductivity and petrocollecting properties,

- obtaining quaternary ammonium salts from the interaction of synthesized oligomers with ethylene chlorohydrinand studying the effect of alkyl group on the properties of salts,
- synthesis of salts between hydrochloric and bromide acids and oligomers, determination of the effect of acid types and alkyl radicals on the petrocollecting properties,
- to obtain both salts and esters of propoxilates with orthophostate acid, to study their structure and composition and to identify similarities and differences.

Research methods. Synthesis of oligomeric derivatives of alkylamines with propylene oxide, production of various modifications of the obtained compounds with organic acids, ethylene chloride, chloride, bromide, orthophosphate acids were carried out in stages in the laboratory.

Accuracy of results. Elemental analysis, IR-, UV-, ^{1}H and ^{13}C NMR-, DLS-spectroscopy methods were used as research methods, interfacial tension was used to measure surface tension (γ), specific electrical conductivity (κ), irradiance coefficient, dynamic, kinematic viscosity and the density of substances were determined.

The main provisions of the defense. The main provisions of the defense are derived from oligomeric derivatives of alkylamines (C_8-C_{18}) with propylene oxide, various modifications of propoxylates with organic acids, ethylene chloride, chloride, bromide, orthophosphate acids and their main physical and chemical properties, petrocollecting and dispersion properties were studied.

Scientific novelty of the research. The followings were done for the first time:

- oligomeric compounds were synthesized from propoxylation of higher aliphatic amines (C_8, C_9, C_{12}, C_{16} and C_{18}) by PO, their main physicochemical parameters were determined, surface activity parameters were calculated, structure and composition were determined by IR-, UV-, ^{1}H and ^{13}C NMR spectroscopy;
- organic salts were obtained from the interaction of alkylamines

(C_9, C_{12} and C_{16}) with propoxy derivatives with monobasic aliphatic monocarboxylic acids (C_2-C_{18}), their structure and composition were confirmed by IR-, UV-, ^1H and ^{13}C NMR spectroscopy. Organic salts obtained by chemical modification of synthesized propoxyl derivatives of alkylamines (C_9, C_{12} and C_{16}) had a strong petrocollecting effect on thin layers of oil spread on tap, sea and distilled water, petrocollecting coefficient and duration of application were determined;

- surface tension, special electrical conductivity properties of synthesized organic salts were widely studied, appropriate isotherms were plotted, surface activity parameters, including constants of Stauff-Klevens equation and a number of other indicators were calculated;
- ammonium-type salts of propoxy derivatives of alkylamines (C_9, C_{12} and C_{16}) were synthesized with ethylene chloride, their physical and chemical parameters, including surface activity parameters were determined, structure and composition determined by IR-, UV-, ^1H and ^{13}C NMR-spectroscopy methods and element analysis;
- propoxy derivatives of hexadecylamine, sharply reducing surface tension, have been found to be highly effective at very low concentrations;
- hydrochloride salts of propoxy derivatives of alkylamines (C_8 and C_{16}) were synthesized, their structure and composition were determined by IR-, UV-, ^1H and ^{13}C NMR-spectroscopy methods. Solubility, surface tension, specific electrical conductivity and petrocollecting properties of salts were studied. Hydrobromide salt of octadecylamine propoxylate was synthesized, its composition was determined by IR-, UV-spectroscopy methods and physical-chemical properties were studied;
- orthophosphate salts and esters of octylamine and hexadecylamine propoxylates were synthesized, their structure and composition were determined by IR-, UV-, ^1H and ^{13}C NMR-spectroscopy methods. Solubility rates of substances, surface tension and specific electrical conductivity values, petrocollecting coefficients were determined.

Theoretical and practical significance of the research. Synthesized propoxy derivatives of alkylamines, as well as their organic salts of monocarboxylic acids and ethylene chlorohydrinwith high surface activity and effective petrocollecting property can be used to clean surface of petrocollecting water bodies.

Personal presence of the author. The results reflected in the dissertation were obtained by the author. Problem statement, experiments and tests, analysis, systematization and generalization of results were carried out with the participation of the author.

Approbation and application. There are 24 published scientific works on the topic of the dissertation. 9 of them are articles ("Processes of Petrochemistry and Oil Refining" (2 articles), "Chemical Problems", Ganja branch of ANAS "Newsletter", "Scientific works" of AzTU, "Journal of Surfactants and Detergents", "Norwegian Journal of Development of the International Science", "Polish Journal of Science", "Austria-Science"), 5 conference proceedings and abstracts of scientific reports from 10 conferences.

The results of the dissertation were reported and discussed at the following conferences: Scientific respublic conference dedicated to the 50th anniversary of the Institute of Chemistry of Additives (Baku, 2015); International scientific-practical conference on "Emergencies and safe life" (Baku, 2015); IX Baku International Mammadaliyev Conference on Petrochemistry (Baku, 2016); Conference dedicated to the 80th Anniversary of the Institute of Catalysis and Inorganic Chemistry (Baku, 2016); "Actual Problems of Modern Natural Sciences" International Scientific Conference (Ganja, 2017); Conference dedicated to the 100th anniversary of acad. B.K. Zeynalov (Baku, 2017); "Actual Problems of Modern Natural Sciences" International Scientific Conference (Ganja, 2018); BSU, XII International Scientific Conference on "Actual Problems of Chemistry"; BEU, I International Scientific-Practical Conference on "Science and Technology" (Baku, 2018); International scientific-practical conference of young scientists (Almetyevsk, 2018); International Scientific Conference on "Actual Problems of Modern Chemistry" dedicated to the 90th anniversary of the Institute of Petrochemical Processes (Baku, 2019); ANAS, Second International

Scientific Conference of Young Scientists and Specialists on "Multidisciplinary approach to solving modern problems of fundamental and applied sciences" (Baku, 2020); I International Scientific Conference of Students and Young Researchers on "Sustainable Development in Chemistry and Chemical Engineering" (Baku, 2020).

Name of the organization where the dissertation work is performed. Institute of Petrochemical Processes named after academician Y.H. Mammadaliyev of the Azerbaijan National Academy of Sciences.

The total volume of the dissertation. The dissertation consists of 219132 characters (229 pages) - introduction, 5 chapters, results, 154 references, 63 pictures and 68 tables.

The first chapter (42240 characters) consists of a review of the literature on the production of surfactants based on alkylamines and epoxides, the synthesis and study of various salts from them.

The second chapter (12119 characters) gives a description of the substances and devices used. Methods for the study of the synthesis of surfactants (oligomers based on alkylamines and propylene oxide and their various modifications), surface activity, specific electrical conductivity, petrocollecting and dispersion properties, biodegradation.

In the third chapter (51756 points) the structure and composition of surfactants synthesized on the basis of alkylamines and propylene oxide, physical and chemical properties (solubility in solvents, dynamic and kinematic viscosities, density, surface activity, electrical conductivity, etc.), as well as the study of petrocollecting properties described.

In the fourth chapter (54534 points) the synthesis and identification of propoxy derivatives of alkylamines with salts of monocarboxylic acids and ethylene chloride, determination of the main physicochemical parameters, especially surface activity indicators (colloid-chemical and thermodynamic properties), petrocollecting results were described.

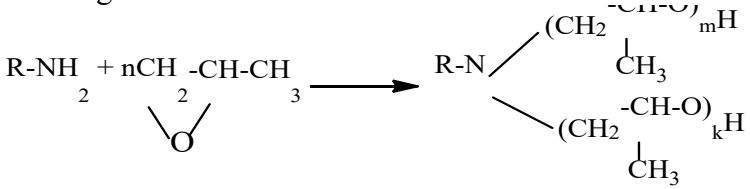
The fifth chapter (336719 points) deals with the synthesis of chloride, bromide, orthophosphate salts and esters of propoxy

derivatives of alkylamines, identification, determination of basic physicochemical parameters, , colloid-chemical and thermodynamic parameters, study of petrocollecting properties.

At the end of the dissertation the results, a list of references and a list of abbreviations and symbols are presented.

Study of oxypropylation of alkylamine with PO

Oligomeric reagents were synthesized from the interaction of PO with alkylamines (octylamine, nonylamine, dodecylamine, hexadecylamine and octadecylamine). The reactions took place at 140-150^{0}C for 14-15 hours, using a 40% aqueous solution of KOH as a catalyst. The general scheme of reactions is as follows:



where R is the alkyl radical (octyl, nonyl, dodecl, hexadecyl and octadecyl), n is the degree of propoxylation (n = m + k).

As a result of calculations, it was found that the propoxylation degrees of octylamine propoxylates were 1.52 and 2.74, the propoxylation degrees of nonylamine propoxylates were 2.07, 2.80, 2.86 and 4.38 [1, 7, 8, 18], the propoxylation degrees of dodecylamine propoxylates were 2.97, 3.33, 3.53, 3.71, 4.04 and 5.80 [2, 9, 14, 16], the propoxylation degrees of hexadecylamine propoxylates were 1.07, 2.28, 2.32, 2.96, 3.22, 3.79 and 10.02 and propoxylation degrees of octadecylamine propoxylates are 0.5 and 6.2 [5].

The composition and structure of synthesized propoxylates were analyzed by IR-, UV-, NMR-spectroscopy methods. Figure 1 shows the ^{1}H and ^{13}C NMR spectra of nonylamine propoxylate with a propoxylation degree of 4.38.

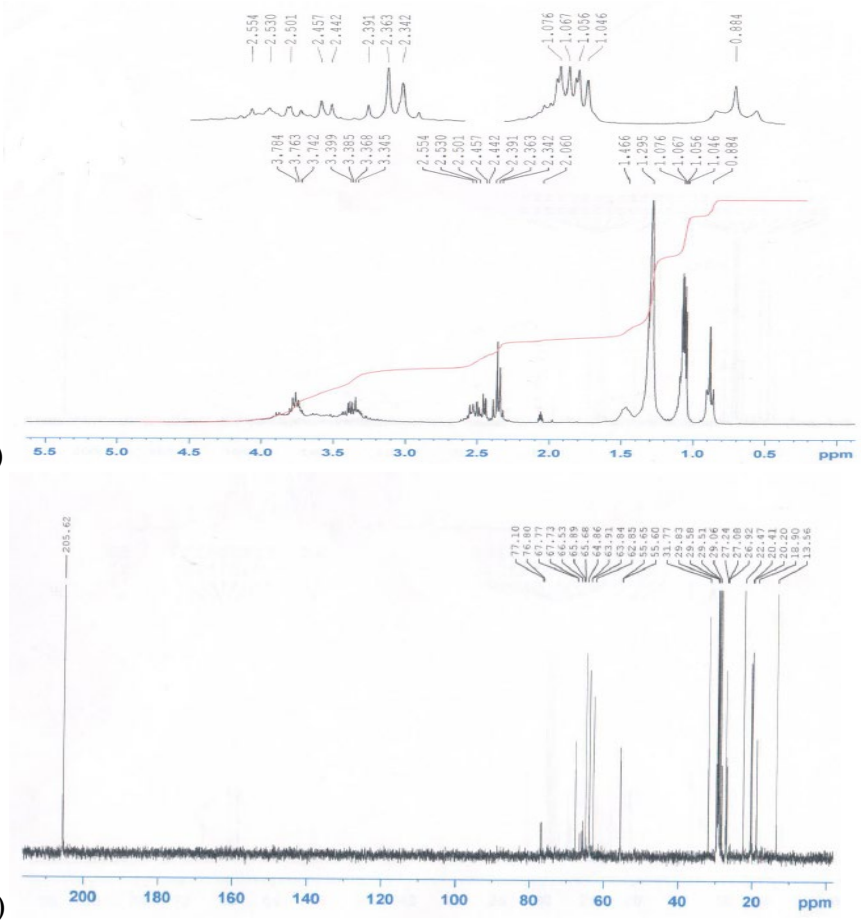


Figure 1. NMR of nonylamine propoxylate with propoxylation degree 4.38: a) ^{1}H NMR, b) ^{13}C NMR spectra

In Figure 1 (a), the signal at 0.884 ppm in the ^{1}H NMR spectrum of nonylamine propoxylate with n = 4.38 is in the CH_3 group of the alkyl chain, the signal is in the CH_3-CH_2 group in the alkyl chain at 1.046-1.076 ppm, and the signal is at 1.295 ppm N-(CH_2 -CH) refers to the CH_2 hydrogen atoms in the groups. The resonance signal at 1.466 ppm corresponds to the N-CH_2-CH_2, 2.342-2.554 ppm multiplet propoxy chain CH_2-CH(CH_3)O, and the multiplet N-CH_2-CH(CH_3)O at 3.346-3.399 ppm. The signal at 3.742-3.784 ppm refers to hydrogen in the OH group.

The resonance signal of the CH_3-CH_2 group at 13.56 ppm in the ^{13}C NMR spectrum of nonylamine propoxylate with n = 4.38, the signal of the CH-CH_3 group in the non-nitrogen-binding PO manganese at 18.90 ppm, and the N-nitrogen-bonded PO manganese in the CH-CH_3 group at 20.20 and 20.41 ppm, N-CH_2-CH(CH_3) group signal, resonance signal of CH_2 manga in alkyl group at 22.47-31.77 ppm, resonance signals of N-CH2-CH group at 55.60-67.77 ppm, CH-OH group signal at 205.62 ppm.

The solubility of alkylamine propoxylates in different solvents has been determined. Solubility rates of octylamine propoxylates (n = 1.52 and 2.74) in organic (isopropanol, isooctane, acetone, ethanol) and inorganic (distilled water) solvents were determined and the results are given in Table 1. The solubility results of surfactants were calculated as the ratio of the mass of solute to the volume of solvent (g/ml).

Table 1

Solubility of octylamine propoxylates in solvents (g/ml)

n	isopropanol	isooctane	acetone	ethanol	distilled water
1.52	0.05	0.17	0.15	0.11	0.04
2.74	0.04	0.13	0.14	0.07	0.03

According to Table 1, the solubility of propoxylate in solvents decreases with increasing molecular size. Oligomers are well soluble in isooctane and acetone. Thus, for every ml of isooctane, 0.17 grams of propoxylate with n = 1.52 and 0.13 grams of propoxylate with n = 2.74 are dissolved. These values are 0.15 and 0.14 grams in acetone, respectively. The least solubility of the obtained substances was observed in distilled water. It was found that only 0.04 grams of propoxylate with 1.52 and 0.03 grams of propoxylate with a degree of propoxylation of 2.74 can be dissolved in distilled water.

Surface tensile properties of alkylamine propoxylates at the air-water boundary were studied. Aqueous solutions of propoxylates in different concentrations were prepared, the surface tension coefficients of the solutions were determined by means of a tensiometer and the specific electrical conductivity was determined by means of a conductometer. Isotherms were plotted using the values of surface tension and specific electrical conductivity of alkylamine

propoxylates and are shown in Figure 2.

The surface tension values of surfactants decrease sharply to CMC, and after these values they almost stabilize. It can be seen from Figure 2 that the minimum value of surface tension between octylamine propoxylates is observed in surfactant with propoxylation degree as n = 2.74.

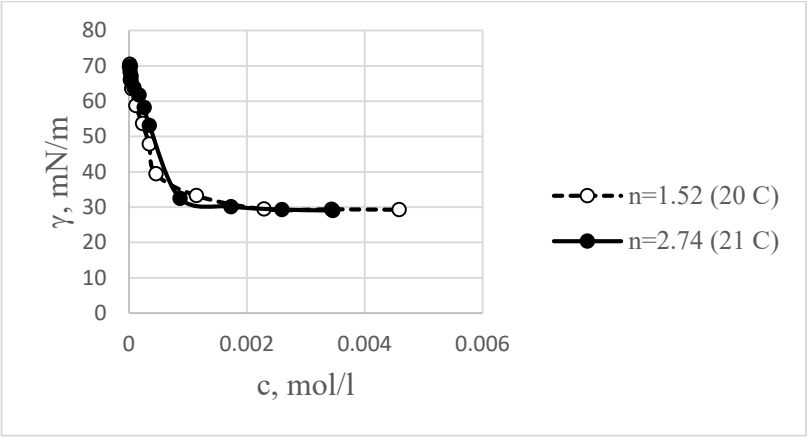


Figure 2. Surface tension isotherms of propyxy derivatives of octylamine

The isotherms of the specific electrical conductivity of propoxyl derivatives of octylamine are given in Figure 3.

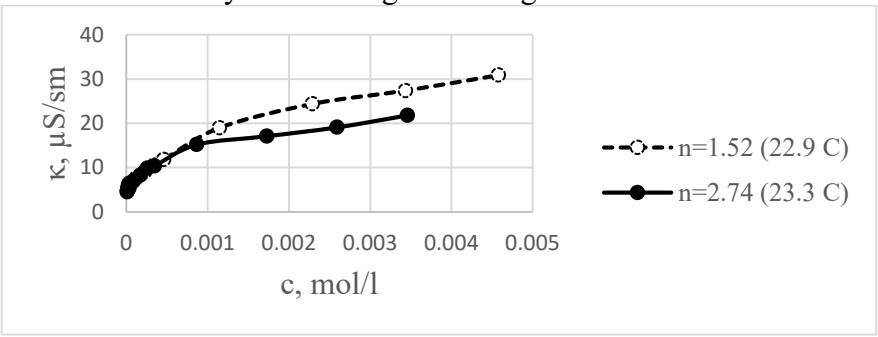


Figure 3. Isotherms of specific electrical conductivity of o propoxy derivatives of octylamine

Depending on the viscosity of alkylamine o propoxylates, it is possible to calculate the surface activity and thermodynamic

parameters by means of applied surface tension and isotherms of specific electrical conductivity. The surface-activity parameters of dodecylamine propoxylates are given in Table 2.

Table 2

Surface activity parameters of propoxy derivatives of dodecylamine

$CMC \times 10^4$ (mol/L)	γ_{CMC} (mN/m)	π_{CMC} (mN/m)	$C_{20} \times 10^5$ (mol/L)	$\Gamma_{max} \times 10^{10}$ (mol/cm^2)	$A_{min} \times 10^2$ (nm^2)
n=2.97					
6.99	44.33	27.67	39.87	0.95	175.51
n=3.33					
6.60	39.20	32.80	31.17	1.05	158.91
n=3.53					
6.40	38.60	33.40	27.55	1.31	127.23
n=3.71					
6.24	33.15	38.85	10.95	1.13	146.49
n=4.04					
5.95	36.91	35.09	22.26	1.07	154.68
n=5.80					
4.79	39.95	32.05	28.71	1.05	158.45

As the degree of propoxylation of oligomers increases, it is generally observed that the CMC decreases. Thus, according to Table 2, the CMC value of surfactant with propoxylation degree 2.97 is 6.97×10^{-4}, while propoxylation degree increases to 5.80 the CMC value decreases to 4.79×10^{-4}. However, surfactant with a total $n \approx 1.0$ has the smallest CMC.

The thermodynamic parameters of propoxy derivatives of dodecylamine were calculated and given in Table 3. Based on the calculated values of the Gibbs free energy of micelle formation and adsorption of the corresponding surfactants, the absolute value of the Gibbs free energy of each surfactants is 2-3 kJ / mol higher than the adsorption Gibbs micelle formation. For example, the Gibbs free energy of micelle formation of an oligomer with an propoxylation rate of 4.04 is -33.51 kJ / mol, and the Gibbs free energy of adsorption is -36.78 kJ / mol. This indicates that the corresponding surfactants

molecules are more prone to adsorption than to form micelles.

Table 3

Electrical conductivity and thermodynamic parameters of propoxy derivatives of dodecylamine

n	α	β	ΔG_{mic} (kJ/mol)	ΔG_{ad} (kJ/mol)
2.97	0.25	0.75	-31.52	-34.44
3.33	0.14	0.86	-33.73	-36.87
3.53	0.18	0.82	-33.15	-35.71
3.71	0.09	0.91	-34.93	-38.36
4.04	0.18	0.82	-33.51	-36.78
5.80	0.23	0.77	-33.51	-36.57

Density, dynamic and kinematic viscosity of alkylamine propoxylates (dodecylamine and hexadecylamine) at different temperatures (25, 30, 35, 40 və 45ºC) were determined. The results of hexadecylamine propoxylate with a propoxylation degree of 3.22 are given in Table 4.

Table 4

Density, dynamic and kinematic viscosity of hexadecylamine propoxylates

n	Values	Temperature				
		25 °C	30 °C	35°C	40°C	45°C
3.22	Dinamic viscosity, mPa*s	112.160	81.992	61.450	42.975	36.580
	Kinematic viscosity, mm^2/s	123.220	90.358	67.990	47.650	40.801
	Density, g/cm^3	0.9102	0.9074	0.9038	0.9019	0.8965

According to Table 4, the kinematic viscosity of surfactant is higher than the dynamic viscosity for each temperature. As the temperature increases (from 25ºC to 45ºC), the values of both dynamic (112.160 mPa×s to 36.580 mPa×s) and kinematic (123.220 mPa×s to 40.801 mPa×s) viscosity decreases. In a given temperature range, the highest density of surfactant (0.9102 g / cm3) is observed at 25ºC and the lowest density (0.8965 g / cm3) is observed at 45ºC. Table 4 shows that the dynamic viscosity values are equal to the product of the

kinematic viscosity and the density. The difference between the prices obtained from the device and the calculated prices varies between 0.001 mPa×s and 0.005 mPa×s.To study the petrocollecting and dispersing properties of octadecylamine propoxylates, the substances were applied to distilled, tap and seawater in pure form, in the form of 5% aqueous solution and 5% ethyl alcohol solution by mass. The petrocollecting and dispersing (on Ramana oil) properties of surfactants in distilled, tap and sea (Caspian Sea) water over time are given in Table 5.

Table 5

Results of petrocollecting properties of octadecylamine propoxy derivatives

n	Type of treatment	τ (hour)	K		
			Distilled water	Tap water	Sea water
0.5	Unthinned reagent	0.17-2	33.97	39.06	33.28
		24-30	44.16	46.49	39.06
		48-54	39.06	39.06	39.06
		92	39.06, drying	39.06, drying	39.06, drying
	5% wt. aqueous solution	0.17-2	22.08	39.06	28.70
		24-30	33.97	46.49	40.14
		48-54	33.97	33.97	33.97
		92	33.97, drying	33.97, drying	33.97, drying
	5% wt. ethanolic solution	0.17-2	33.28	33.28	33.28
		24-30	46.49	46.49	46.49
		48-54	46.49	46.49	46.49
		92	46.49, drying	46.49, drying	46.49, drying
6.2	Unthinned reagent	0.17-2	33.28	33.28	28.70
		24-30	39.06	39.06	29.44
		48-54	39.06	39.06	29.44
		92	39.06, drying	39.06, drying	29.44, drying
	5% wt. aqueous solution	0.17-2	29.44	29.44	29.44
		24-54	39.06	39.06	39.06
		92	39.06, drying	39.06, drying	39.06, drying
	5% wt.	0.17-2	33.28	36.80	36.80

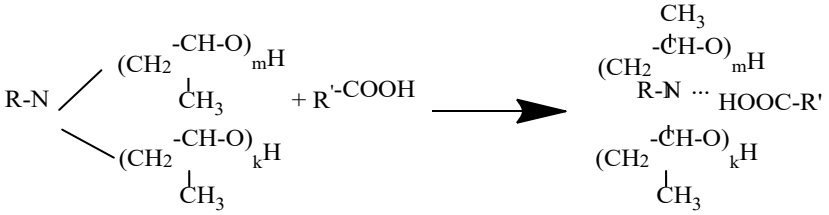
	ethanolic solution	24-54	36.80	36.80	36.80
		92	36.80, drying	36.80, drying	36.80, drying

As can be seen from Table 5, both propoxylates have a strong effect on the thin oil layer spread over the water basins. Comparing the two surfactants, it can be said that they have a high petrocollecting properties factor, regardless of the degree of propoxylation. At the same time, as can be seen from the table, the coefficient of petrocollecting properties during exposure in the range of 0.17-2 hours is less than the coefficient obtained after 24 hours.

The reason for this can be explained by the aggregate cases of synthesized surfactants. Thus, both substances are solid and both are poorly soluble in water. That is why the full effect of surfactants on the oil layer occurs after a few hours. In both cases, the petrocollecting examination of oil resulted in the drying of the water.

SYNTHESIS AND STUDY OF ALKYLAMINE PROPOXILATES WITH ORGANIC REAGENS

The interaction of propoxy derivatives of alkylamines (nonylamine, dodecylamine and hexadecylamine) with organic acids was studied. Reactions are carried out indoors at a temperature of 50-60^{0}C for 5-6 hours. Organic salts were synthesized from nonylamine propoxylate with a degree of propoxylation of 2.54 with pentane, hexane, octane, nonane, decane, undecane and dodecanoic acids, dodecylamine propoxylate with a degree of propoxylation of 3.53 with ethane, pentane, octane, nonane, octane, undecan, dodecane, hexadecylamine propoxylate with a degree of propoxylation 2.96 with ethane, pentane, octane, nonane, undecane, dodecane, tridecane, tetradecane and octadecanoic acids. Their properties were studied. The general scheme of the reaction is shown below:



where m + k is the total with a degree of propoxylation rate. The alkyl group (nonyl, dodecyl and hexadecyl) in the radical amine, and R` indicates the radical of the organic acid [11-13, 15, 17, 20].

The composition and structure of the organic salts of the obtained with a degree of propoxylates were confirmed by IR-, UV- and NMR-spectroscopy. The solubility of organic salts in both organic and inorganic solvents has been determined. Depending on the density of the synthesized surfactants, the surface tension and electrical conductivity isotherms were constructed and the relevant parameters were calculated.

In order to calculate the Stauff-Klevens constants of organic salts of alkylamines (nonylamine, dodecylamine and hexadecylamine) synthesized with propoxy derivatives with saturated monocarboxylic acids, surface tension isotherms of these salts were plotted and parameters were calculated. According to the Stauff-Clevens equation, there is a linear relationship between the number of carbon atoms (N) in the alkyl radical of surfactants with the same functional groups and the log (CMC):

$$\lg(KMQ) = B * N + A$$

To determine this dependence and to find the Stauff-Klevens constants, the salts synthesized by pentanoic acid of the propoxylates of alkyl amines (nonylamine, dodecylamine and hexadecylamine) were studied and the results are given in Table 6. The dependence of the lg (CMC) values given in Table 6 on the number of carbon atoms in the alkyl group of the amine is shown in Figure 4.

According to Figure 4, the graph of the dependence of the number of carbon atoms on the values of lg (CMC) is almost linear ($R^2 = 0.98$; 98% of these values show a linear dependence).

Table 6

CMC and lg (CMC) values of propoxylates of alkylamines (nonylamine, dodecylamine and hexadecylamine) synthesized with pentanoic acid

Acid	Propoxylates of alkylamines	CMC, mol/l	lg(CMC)
Pentanoic acid	Nonylamine	0.00135	-2.86
	Dodecylamine	0.00102	-2.99
	Hexadecylamine	0.00021	-3.68

The constant B in the Stauff-Clevens equation is equal to the angle coefficient of the straight line, B = -0.1431. The constant A of the equation indicates the intersection with the y-axis, A = -1.355. As a result, the Stauff-Klevens equation of salts formed by the interaction of alkylamines with pentanoic acid can be expressed as follows:

$$\lg(KMQ) = -0.1431 * N - 1.355$$

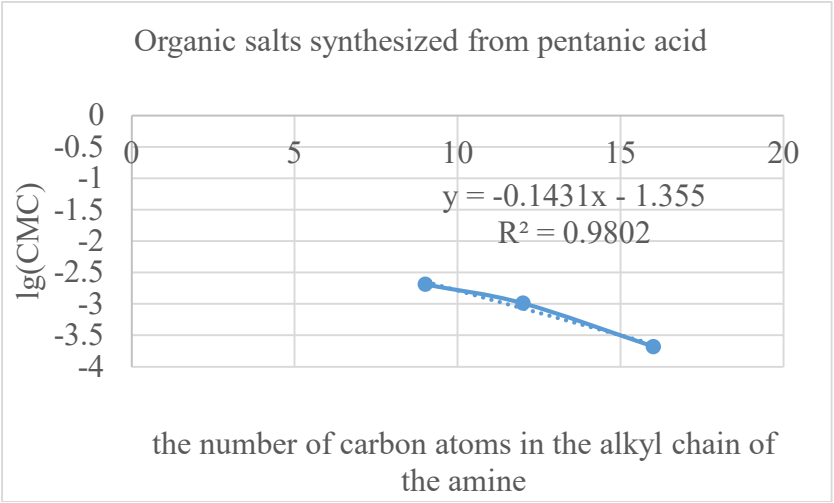
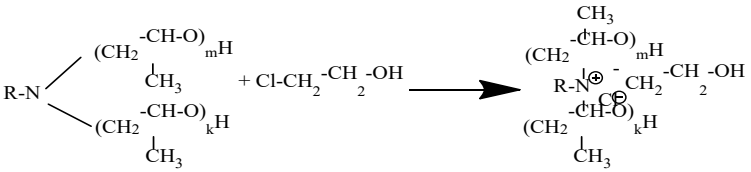


Figure 4. Graph of lg(CMC) values of salts of alkylamines (nonylamine, dodecylamine and hexadecylamine) synthesized with pentanoic acid of propoxylates depending on the number of carbon atoms

Preparation and study of alkylamine propoxylates with ethylene chlorohydrin and ammonium salt type derivatives

The interaction with ethylene chlorohydrin and derivatives of alkylamine propoxylates has been studied in order to obtain new, quaternary ammonium salt. The reaction of propoxylates of all three amines (nonylamine, dodecylamine, hexadecylamine) with ethylene chlorohydrin was carried out at a temperature of 70-80ºC for 15-16 hours using a magnetic stirrer. The components of the reaction were taken in a 1:1 mole ratio. Color change was observed during the reaction [23]. Thus, the yellow-light-brown color of propoxylates changed to dark brown during the reaction. The general scheme of

reactions can be shown as follows:



where R amine radical (C_9H_{19}, $C_{12}H_{25}$, $C_{16}H_{33}$), (m + k) indicates the total propoxylation rate.

Conversion of ethylene chlorohydrin with nonylamine propoxylate (n = 4.38) was determined to be 88%, conversion of ethylene chlorohydrin to dodecylamine propoxylate (n = 5.80) to 83%, and conversion of ethylene chlorohydrin to hexadecylamine propoxylate (n = 3.79). It has been observed that the conversion of ethylene chlorohydrin decreases with the elongation of the alkyl chain in the amine.

The composition and structure of salts of alkylamines synthesized with ethylene chlorohydrin were determined by IR-, UV- and NMR-spectroscopy. The UV spectrum of the salt obtained from the action of nonylamine propoxylate (n = 4.38) with ethylene chlorohydrin is given in Figure 5. In the UV spectrum, the absorption peak of the σ → σ * transition (at ~ 205.0 nm) is observed due to the nitrogen atom in the amine fragment of the quaternary ammonium salt.

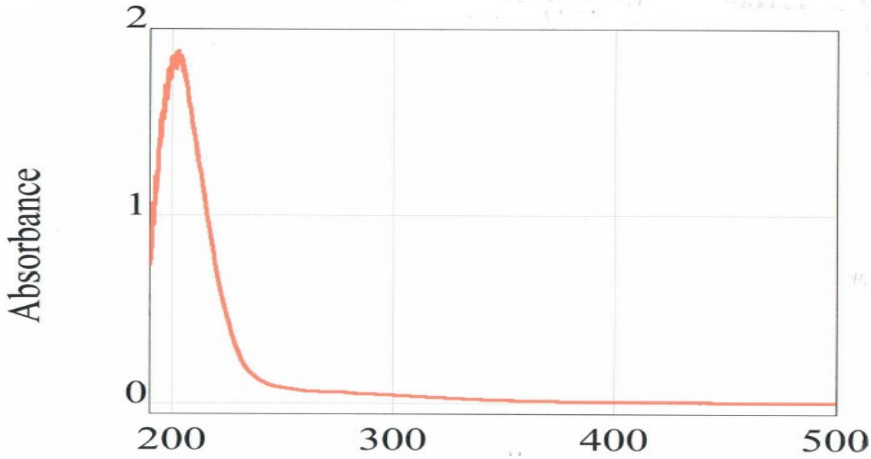


Figure 5. UV spectrum of salt obtained by the action of nonylamine

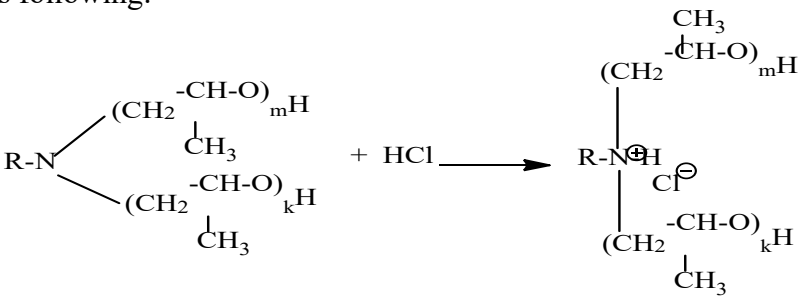
propoxylate (n = 4.38) with ethylene chlorhydrin

The amount of chlorine in the synthesized salts was determined by the Argentometric method. According to the analysis, 0.079 part of the salt derived from nonylamine is a chlorine atom. According to mathematical calculations, this is equal to 0.074. In the case of hexadecylamine-derived salt, these results are 0.057 according to the analysis and 0.065 according to the calculations. As can be seen from both results, the amount of chlorine in the salts is consistent with the Argentometric method and mathematical calculations. Chlorine in the salt of dodecylamine o propoxylate was determined by the same method.

Surface tension, special electrical conductivity and collection of thin oil layers over water surface of salts of alkylamines (nonyl, dodecyl, hexadecyl) synthesized with ethylene chlorohydrin were studied.

Synthesis and study of ammonium salt-type derivatives of alkylamine propoxylates with hydrochloric acid

Salts were synthesized from alkylamines by the interaction of propoxy (n = 10.02) derivatives of octylamine (n = 2.74) and hexadecylamine with hydrochloric acid. The reactions were carried out at room temperature for 5-6 hours. The general scheme of reactions is as following:



where R is the octyl and hexadecyl radicals, and n (n = m + k) is the degree of propoxylation.

The composition and structure of hydrochloride salts of propoxylates of synthesized alkylamines were determined by IR-, UV- and NMR-spectroscopy.

Solubility coefficients of octylamine and hexadecylamine propoxylates in organic and inorganic solvents of hydrochloride salts were determined. Surface tension of aqueous solutions of hydrochloride salts of alkylamine propoxylates, special electrical conductivity and collection of thin oil layers over water bodies were studied.

The DLS results of 0.1, 0.05, 0.005, 0.0025 and 0.0010% aqueous solutions of hexadesylamine propoxylate hydrochloride salt by mass are given in Table 7.

The results show that as the density of the aqueous solution decreases (from 0.1% to 0.0025%), the value of the media increases (from 7.9 nm to 382.3 nm), but decreases slightly when the density is 0.0010% (341.1 nm). The value of moda is increasing (from 8.1 nm to 317.0 nm). The nature of the change in the geometric mean depending on the hardness is similar to that of the median.

Table 7

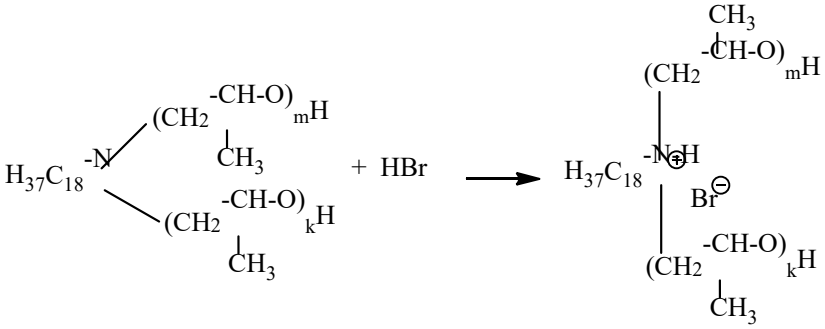
DLS results of 0.1, b) 0.05, c) 0.005, d) 0.0025 and e) 0.0010% aqueous solutions of hexadecylamine propoxylate (n = 10.02) hydrochloride salt by mass

Aqueous solution, %	0.1	0.05	0.005	0.0025	0.0010
Diameter interval, nm	5-13.1	6.6-17.1	58.1-1298.5	76.2-2598.5	87.3-1980.8
Median, nm	7.9	26	231.5	382.3	341.1
Moda, nm	8.1	9.5	183.6	316.6	317.0
Geometric mean, nm	7.8	9.6	290.3	415.8	353.7
Dispersion, nm^2	1.1054	1.4281	1.4272	1.3058	1.1636
Standard deviation, nm	1.2061	2.4720	2.4720	2.1899	1.8053
Refractive index	1.361	1.361	1.361	1.361	1.361
Dissociation coefficient*10^{12} m^2/s	49.435	40.692	1.6899	1.0258	1.1508

As the density decreases in the 0.1-0.0025% interval, the geometric mean increases (from 7.8 nm to 415.8 nm), and decreases slightly at the 0.0010% density (353.7 nm). The diffusion coefficient decreases in the range of 0.1-0.0025% and increases slightly in 0.0010%.

Synthesis and study of octadecylamine propoxylate with ammonium salt type derivative with bromic acid

The reaction of octadecylamine propoxylate (n = 6.2) with hydrobromide salt was carried out at room temperature for 5-6 hours. The reaction scheme is as following:



where n (n = m + k) is the degree of propoxylation.

The UV-spectrum of the hydrobromide salt of octadecylamine propoxylate (n = 6.2) was drawn, the effect of surface tension, specific electrical conductivity and petrocollecting coefficients on thin oil layers were studied.

The IR spectrum of the hydrobromide salt of octadecylamine propoxylate (n = 6.2) is shown in Figure 6. The bands 3373.10 cm^{-1} in the IR spectrum belong to the valence oscillations of the O-H bond in the OH group. The bands 2957.47-2849.91 cm^{-1} in the spectrum correspond to the valence oscillations of the C-H bond in the CH_3, CH_2 and CH groups in the oligomer. The bands 1470.02-1373.93 cm^{-1} correspond to the asymmetric and symmetric deformation oscillations of the C-H bond. The valence oscillations of the C-N bond are reflected at 1278.28 cm^{-1}. The bands at 1134.80 and 1060.72 cm^{-1} are the result of the valence oscillations of the C-O-C simple ester group and the C-O bond in the C-OH group, respectively. The $(CH_2)_x$ stripes of the chain are expressed in the stripe at 716.79 cm^{-1}.

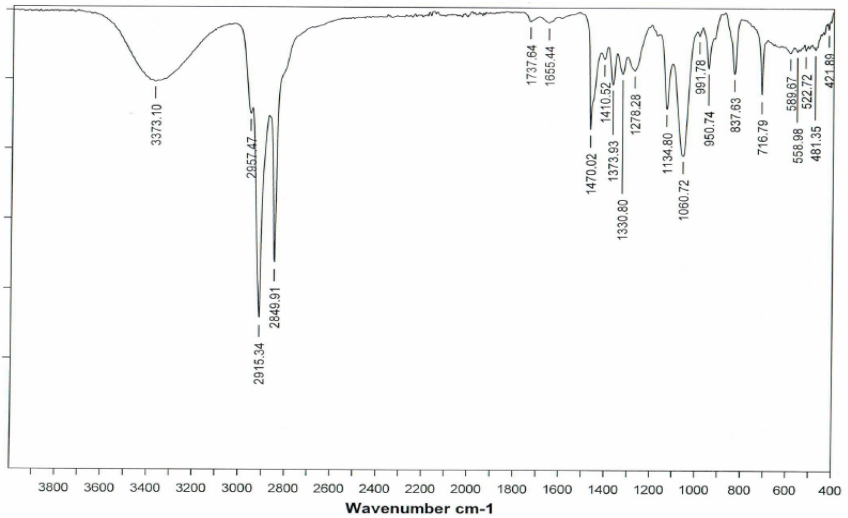
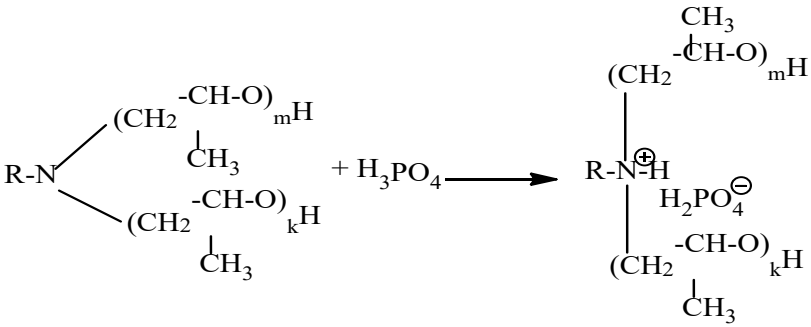


Figure 6. IR spectrum of hydrobromide salt of octadecylamine propoxylate (n = 6.2)

Synthesis ans study of alkylamine propoxylates with orthophosphate acid and ammonium salt type derivatives

Salts were obtained from alkylamine propoxylates by the interaction of octylamine propoxylate (n = 2.74) and hexadecylamine propoxylate (n = 10.02) with orthophosphate acid. The reactions were carried out at room temperature for 6-7 hours. The general scheme of reactions is as follows:



where R is the octyl and hexadecyl radicals, and n (n = m + k) is the degree of propoxylation.

The composition and structure of dihydroorthophosphate salts of

propoxylates of synthesized alkylamines were determined by IR-, UV- and NMR-spectroscopy methods. The diameter distribution of the aggregates of the hexadecylamine propoxylate (n = 10.02) salt with orthophosphate acid was studied by DLS histograms of 0.1, 0.02, 0.01, 0.006 and 0.0006% solutions by weight.

Solubility, electrical conductivity and petrocollecting properties of octylamine propoxylate (n = 2.74) and hexadecylamine propoxylate (n = 10.02) dihydroorthophosphate salts in organic and inorganic solvents were studied.

The surface tension values of the dihydroorthophosphate salt of octylamine propoxylate are given by the viscosity isotherm in Figure 7. The parameters calculated based on this isotherm are shown in Table 8.

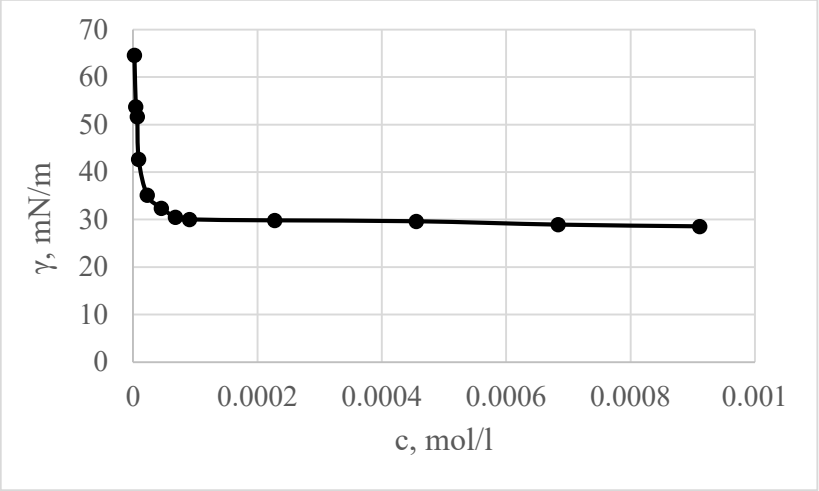


Figure 7. Surface tension isotherms of dihydroorthophosphate salts of hexadecylamine propoxylate (n = 10.02)

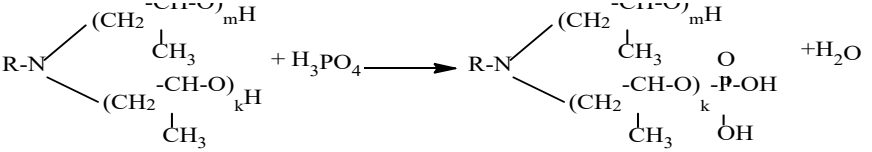
Table 8

Surface active parameters of dihydroorthophosphate salt of hexadecylamine propoxylate (n = 10.02)

$CMC \times 10^4$ (mol/L)	γ_{CMC} (mN/m)	π_{CMC} (mN/m)	$C_{20} \times 10^5$ (mol/L)	$\Gamma_{max} \times 10^{10}$ (mol/cm^2)	$A_{min} \times 10^2$ (nm^2)
0.68	30.52	41.48	0.65	2.01	82.85

Systhesis and study of ester derivative of alkylamine propoxylate with orthophosphate

Esters were obtained from the interaction of octylamine propoxylate (n = 2.74) and hexadecylamine propoxylate (n = 10.02) with orthophosphate acid at a temperature of 50-60ºC. Reactions were carried out within 6-7 hours. The general scheme of reactions is as follows:



where R is the octyl and hexadecyl radicals, and n (n = m + k) is the degree of propoxylation.

The composition and structure of orthophosphate esters of propoxylates of synthesized alkylamines were studied by IR- and UV-spectroscopy methods.

The isotherm of the surface tension values of the orthophosphate ester of hexadecylamine propoxylate (n = 10.02) is plotted, and the parameters calculated based on this isotherm are given in Table 9.

Compairing the orthophosphate ester of hexadesylamine propoxylate (n = 10.02) with the dihydroortaphosphate salt of that propoxylate, special sequences can be observed. While the CMC value in orthophosphate salt is 0.68×10^{-4} mol/l, the CMC value of orthophosphate ester of propoxylate is 0.28×10^{-4} mol l.

Table 9

Surface activity parameters of orthophosphate ester of hexadecylamine propoxylate (n = 10.02)

$CMC\times10^4$ (mol/L)	γ_{CMC} (mN/m)	π_{CMC} (mN/m)	$C_{20}\times10^5$ (mol/L)	$\Gamma_{max}\times10^{10}$ (mol/cm^2)	$A_{min}\times10^2$ (nm^2)
0.28	31.75	40.25	0.36	2.44	68.18

The value of surface tension at CMC is 30.52 mN/m in hexadesylamine dihydroorthophosphate salt and 32.26 mN/m in orthophosphate ester. The value of orthophosphate ester C_{20} (0.36×10^{-5} mol/l) of hexadecylamine propoxylate is lower than that of the dihydroorthophosphate salt of hexadecylamine propoxylate (0.65×10^{-5} mol/l). The Γ_{max} values of dihydroorthophosphate salt of

hexadecylamine propoxylate and orthophosphate ester are also 2.01×10^{-10} mol/cm^2 and 2.44×10^{-10} mol/cm^2, respectively. The A_{min} values of the same surfactants are decreasing. This value is equal to 82.85×10^{-2} nm^2 in the dihydriorphosphate salt of propoxylate and 68.19×10^{-2} nm^2 in the orthophosphate ester of propoxylate.

The petrocollecting effects of orthophosphate esters of octylamine propoxylate (n = 2.74) and hexadecylamine propoxylate (n = 10.02) were studied and the results are given in Table 10.

Table 10

Results of petrocollecting properties of orthophosphate esters of octylamine propoxylate (n = 2.74) and hexadecylamine propoxylate (n = 10.02)

Esters	Type of application	τ (hour)	K		
			Distilled water	Tap water	Sea water
Orthophosphate esters of octylamine propoxylate (n = 2.74)	Unthinned reagent	0.17-2	39.06	29.44	25.50
		18-96	33.97	29.44	25.50
		118	33.97, drying	29.44, drying	25.50, drying
	5% wt. aqueous solution	0.17-2	28.70	22.08	22.08
		18-96	25.00	22.08	22.08
		118	25.00, drying	22.08, drying	22.08, drying
	5% wt. ethanolic solution	0.17-2	25.00	25.00	22.08
		18-96	33.28	33.28	25.97
		118	33.28, drying	33.28, drying	25.97, drying
Orthophosphate esters of hexadecylamine propoxylate (n = 10.02)	Unthinned reagent	0.17-4	22.08	22.08	19.00
		23-79	19.00	19.00	19.00
		122	19.00, drying	19.00, drying	19.00, drying
	5% wt. aqueous solution	0.17-4	22.08	22.08	22.08
		23-79	19.00	19.00	19.00
		122	19.00, drying	19.00, drying	19.00, drying
	5% wt. ethanolic solution	0.17-4	22.08	22.08	22.08
		23-79	22.08	19.46	19.46
		122	22.08, drying	19.46, drying	19.46, drying

As can be seen from Table 10, both esters have an effect on a

thin layer of oil spread over water surfaces. Although the orthophosphate ester of hexadecylamine propoxylate (n = 10.02) predominates in its surface activity properties, this cannot be said for the accumulation of thin oil layers on water surfaces. Although the results of both esters are close to each other, it should be noted that orthophosphate ester of octylamine propoxylate (n = 2.74) is more effective. The duration of action of surfactants is approximately the same. After 5 days, the study of both esters was completed by drying the water.

RESULTS

1. Studies of propoxylates synthesized on the basis of PO with alkylamines have shown that the alkyl chain has a direct effect on surface-activity properties and petrocollecting coefficients. The surface tension values and isotherms of hexadecylamine propoxylates from alkylamines are very similar, but differ from other amine propoxylates. Thus, the surface tension isothermal curves of all substances are sharply reduced to 30 mN / m. Thus, it is possible to obtain CMC values even at very low concentrations of surfactants. At the same time, it was found that the coefficients of octylamine, nonylamine and dodecylamine propoxylate vary between 10-17, 4-14 and 4-16, respectively. In hexadecylamine and octadecylamine propoxylates, the petrocollecting coefficients (in the range of 14-38 and 22-46) are higher than in other amines.
2. Biodegradation of nonylamine propoxylate (n = 2.54) salt obtained with capric acid was observed. It was found that 100% in a 0.01% solution for 20 days; 100% for 50 days in 0.025% solution; In 0.1% solution, 91.6% decomposition occurred within 50 days. The results show that the structure of these compounds is largely unchanged during the first 10 days (biodegradation is weak during the first 10 days).
3. Isotherms of both surface tension and electrical conductivity of organic salts obtained from dodecylamine propoxylate (n = 3.53) were constructed and the corresponding parameters were calculated. It was found that the elongation of the alkyl radical of the acid leads to a decrease in the values of CMC, γ_{CMC}. In

thermodynamic parameters, Gibbs free energy values of micelle formation and adsorption decrease with elongation of the alkyl radical. It is observed that the petrocollecting properties of salts increases. The initial petrocollecting coefficients of these salts are usually 44.16.

4. Surface tension values of salts obtained from the synthesis of hexadecylamine propoxylate (n = 2.28) with organic acids are reduced to 23.02-29.54 mN/m. The initial petrocollecting coefficients of surfactants with smaller acid radicals (up to the salt obtained with dodecanoic acid) are higher than 30. When the number of carbon in the alkyl radical of the acid is more than 12, the petrocollecting coefficient decreases.
5. The solubility of alkylamine oxypopylates derivatives of ethylene chlorohydrin and ammonium salt in isopropanol, toluene and distilled water decreases, while the solubility in isooctane and ethanol increases. Alkyl chain micelle formation and adsorption have been found to affect Gibbs free energy values. As the nitrogen-bonded alkyl group increases from nonyl to hexadecyl, both the micelle formation and adsorption Gibbs free energy values decrease. The petrocollecting properties of surfactants have been studied by applying them to Pirallahi petroleum spread over water. The petrocollecting coefficients are often close to 30 in most cases.
6. Hydrochloride salts of octylamine propoxylate (n = 2.74) and hexadecylamine propoxylate (n = 10.02) were obtained. Particle diameters were determined in 0.1, 0.05, 0.005, 0.0025 and 0.0010% aqueous solutions of hexadecylamine propoxylate (n = 10.02) hydrochloride salt. Based on the results, the median, mode, dominant fraction, geometric mean, variance, standard deviation, refraction coefficient and diffusion coefficient of each aqueous solution were calculated.
7. The reaction of octadecylamine propoxylate (n = 6.2) with bromic acid was carried out at room temperature and a salt was obtained. The surface tension and special electrical conductivity of the synthesized salt were studied. Distillation of undiluted and 5% aqueous solutions of salt, petrocollecting coefficients on drinking

and sea water has a maximum of 39.06.

8. The interactions of octylamine propoxylate (n = 2.74) and hexadecylamine propoxylate (n = 10.02) with orthophosphate acid were studied both at room temperature and at 50-60ºC. Salt was obtained from the reactions carried out at room temperature, and ester was obtained as a result of the reactions carried out at 50-60ºC. Comparative analysis of salts and esters are given.

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The defense will be held on **24th of September 2021** at 14^{00} at the meeting of the Dissertation council ED 1.16 of Supreme Attestation Commission under the President of the Republic of Azerbaijan operating at Institute of Petrochemical Processes of the National Academy of Sciences of Azerbaijan.

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Dissertation is accessible at the Institute of Petrochemical Processes of the National Academy of Sciences of Azerbaijan.

Electronic versions of dissertation and its abstract are available on the official website of the Institute of Petrochemical Processes of the National Academy of Sciences of Azerbaijan.

Abstract was sent to the required addresses on **16th of July 2021**

Signed for print:05.07.2021
Paper format:A5
Volume:37662
Number of hard copies:20