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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

SYNTHESIS AND STUDY OF NEW SURFACTANTS BASED ON FATTY ACIDS AND POLYAMINES

Specialty:	2314.01 - Petrochemistry
Field of science:	Chemistry
Applicant:	**Hajar Tahir Nabiyeva**

Baku - 2025

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

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Dissertation council ED 1.16 of Supreme Attestation Commission under the President of the Republic of Azerbaijan operating at the Institute of Petrochemical Processes named after acad. Y.H. Mammadaliyev of the Ministry of Science and Education Republic of Azerbaijan.

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GENERAL CHARACTERISTICS OF WORK

Relevance of the work and degree of development. The growth of the world's population leads to the increase of anthropogenic effects on the environment and some ecological problems. One of the main problems is the pollution of water bodies. Pollution of the hydrosphere can be different depending on the source of influence. One of them is the extraction of oil from the seas and oceans, the accidents that may occur during the transportation of oil by tankers, and the covering of water bodies with an oil layer[1]. Such layers disrupt the ecological balance at the water-air boundary, because these layers prevent the penetration of sunlight into the deep layers of the water, make it difficult for the normal exchange of gases such as oxygen, carbon dioxide and nitrogen at the indicated boundary, and the living conditions of aquatic organisms deteriorate. The synthesis and research of new, ecologically harmless surfactants that allow to eliminate thin oil layers on the surface of natural water bodies is of great relevance from the point of view of environmental protection[2].

In modern times, surfactants have found wide application in all fields of national economy, medicine, science and everyday life. Surfactants are diphilic in nature and consist of hydrophilic and hydrophobic parts. For this reason, they exhibit different properties by adsorbing at the boundary between the two phases[3].

In the proposed work, it is envisaged to obtain new surfactants based on fatty acids, and polyamines (ethane-1,2-diamine, N^1-(2-aminoethyl)ethane-1,2-diamine and polyethylenepolyamine).

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[1]Silva, I.A. Oil spills: impacts and perspectives of treatment technologies with focus on the use of green surfactants / I.A. Silva, F.C.G. Almeida, T.C Souza [et al.] // Environmental Monitoring and Assessment, - 2022. v. 194. № 3, p. 143.

[2]Asadov, Z.H. Petroleum-collecting and dispersing chemicals for cleaning sea surface from thin petroleum slicks / Z.H. Asadov, I.A. Zarbaliyeva, R.A. Rahimov [et al.] // Bulletin of the Chemical Society of Ethiopia, - 2014. v. 28. № 2, - p. 205-

[3]Damen, M. Structure–delivery relationships of lysine-based Gemini surfactants and their lipoplexes / M. Damen, E. Cristóbal-Lecina, G.C Sanmartí [et al.] // Soft Matter, - 2014. V. 31. № 10, – p. 5702-5714.

Fatty acids, being a component of triglycerides in natural oils, belong to ecologically harmless and renewable types of raw materials. This creates a serious basis that surfactants to be synthesized will be significantly safer for the environment.

The use of higher aliphatic monobasic carboxylic acids and polyamines makes it possible to synthesize surfactants with a traditional structure, i.e. one hydrocarbon chain, and "Gemini" type surfactants, i.e. surfactants with two or more hydrocarbon chains. "Gemini" type surfactants have recently been in the center of attention of experts. Their different structure from traditional surfactants, that is, the presence of several hydrocarbon groups, provides a number of unique properties, for example, the formation of micelles in very small concentrations, lower values of surface tension at the boundary between phases, and thus lower costs of surfactants.

Object and subject of work. The object of the dissertation research is new Gemini-type surfactants synthesized on the basis of various fatty acids and polyamines. The subject of the research includes how the structure and composition of Gemini surfactants obtained on the basis of (C_{12}-C_{18}) fatty acids and ethane-1,2-diamine, N^1-(2-aminoethyl)ethane-1,2-diamine and polyethylenepolyamine affects their many physical-chemical parameters.

The purpose and objectives of the dissertation work. The purpose of the work is the synthesis of new "Gemini" type surface-active substances based on fatty acids and polyamines, the study of their structure and composition by modern spectroscopic and analytical methods, the determination of their main physico-chemical parameters, including surface activity parameters, their petrocollecting and petrodispersing properties which allow to remove ecologically harmful thin oil layer on the water surface, as well as other useful and application-oriented properties. In addition, the petrocollecting and petrodispersing properties which are the ability of substances to rid natural water bodies of ecologically dangerous thin oil layers are investigated in detail, depending on various factors, the petrocollecting and petrodispersing properties, selection of the most effective samples and recommendations for their application.

The tasks of scientific research are the following:

- Synthesis of new Gemini-type surfactants based on (C_{12}-C_{18}) fatty acids and polyamines and study of their structure and properties;
- Studying the effect of the length of the alkyl chain in the synthesized Gemini surfactants on their surface-activity, electrical conductivity and petrocollecting and petrodispersing properties;
- Investigating the influence of the number of nitrogen atoms on their properties in surfactants synthesized on the basis of (C_{12}-C_{18}) fatty acids and polyamines;
- Comparison of the surface-activity, electrical conductivity and petrocollecting and petrodispersing properties of the synthesized Gemini surfactants with their mono-structured counterparts.

Research methods. The synthesis of (C_{12}-C_{18}) fatty acids with polyamines was carried out step by step in laboratory conditions, both equivalently and in twice the molar ratio of the acid. The integrity of the obtained results was identified by IR-, UV-, ^{1}H and ^{13}C NMR-spectroscopy methods. Many physical-chemical properties were studied by analysis methods such as DLS-, TGA-.

The main provisions put forward for defense. (C_{12}-C_{18}) fatty acids and polyamines were synthesized in equimolar and twice the molar ratio of fatty acids to polyamines and their main physical-chemical properties, surface tension, electrical conductivity, petrocollecting and petrodispersing properties were studied.

Scientific novelty of the dissertation. The first time in work:

- Gemini-type surfactants were synthesized from the reaction of (C_{12}-C_{18}) fatty acids with ethane-1,2-diamine, and it was proven that they have petrocollecting and petrodispersing properties;
- New Gemini surfactants based on N^{1}-(2-aminoethyl)ethane-1,2-diamine and (C_{12}-C_{18}) fatty acids were synthesized and confirmed to have petrocollecting and petrodispersing properties;
- New Gemini-type surfactants based on (C_{12}-C_{18}) fatty acids and polyethylenepolyamine were synthesized and proven to have petrocollecting and petrodispersing properties;

- Application-oriented properties of synthesized Gemini surfactant, as well as inhibitory bactericidal properties against sulfate reducing bacteria (SRB) were studied;
- The main physical and chemical parameters of each synthesized surfactant were compared with its mono-structured homologues and the superior parameters were noted.

Theoretical and practical value of the work. Surfactants synthesized on the basis of fatty acids and polyamines, having high surface activity and effective petrocollecting capacity, can be used in the cleaning of surface oil-contaminated water bodies. Surfactants exhibiting high bactericidal properties against SRB can be used as inhibitor-bactericides.

Personal participation of the author. The results reflected in the dissertation were obtained by the author. With the participation of the author, the issues were set, experiments and tests were carried out, the results were systematized and summarized.

Approbation and application of the work. The main provisions and results of the dissertation were reported and discussed at the following republican and international scientific and technical conferences (21 theses):

"I International Scientific Conferences of Students and Young Researchers" dedicated to the 97th anniversary of the birth of national leader Heydar Aliyev (Baku, 2020); II International scientific conference of young scientists and specialists (Baku, 2020); 1st International Scientific and Practical Internet Conferences (Dnipro, 2020); "II International Scientific Conference of Students and Young Researchers" dedicated to the 98th anniversary of the birth of national leader Heydar Aliyev (Baku, 2021); "International Scientific Conference" (Ganja, 2021); XIV International Scientific Conference "Current Problems of Chemistry" of doctoral students, master's students and young researchers dedicated to the 98th anniversary of the birth of national leader Heydar Aliyev (Baku, 2021); “VI International Scientific Conference of Young Researchers" dedicated to the 99th anniversary of the birth of national leader Heydar Aliyev (Baku, 2022); Republican scientific conference dedicated to academician Nadir Mir-Ibrahim Seyidov's 90th anniversary

"Catalysts, olefin-based oils" (Baku, 2022); "Modern problems of theoretical and experimental chemistry" devoted to the 90th anniversary of academician Rafiga Aliyeva (Baku, 2022); National scientific conference dedicated to the 80th anniversary of Nazil Fazil Janibeyov, corresponding member of ANAS, "Actual problems of the chemistry of heteroatomic compounds" (Baku, 2022); Republican conference on "Environmental protection, industrial and household waste recycling" (Ganja, 2022); XXV Republican Scientific Conference of Doctoral Students and Young Researchers (Baku, 2022); "Actual Problems of Modern Natural and Economic Sciences" International Scientific Conference dedicated to the 100th anniversary of the birth of national leader Heydar Aliyev (Ganja, 2023); "Scientific advances and innovative approaches" Proceedings of the VII International Scientific and Practical Conference (Japan, 2023); XXVI Republican Scientific Conference of Doctoral Students and Young Researchers (Baku, 2023); Synthesis and research of metal complex and organometallic catalysis, (so)oligomer, (so)polymers (Baku, 2023); "Women in STEM" International Festival (Tashkent, 2024); Scientific-practical conference on the role of national leader Heydar Aliyev in improving the environment in Azerbaijan (Baku, 2024); LXXXVI-LXXXVII International scientific and practical conference on the topic “Chemistry, physics, biology, mathematics: theoretical and applied research (Moscow, 2024); International Conference on Advances in Biotechnology, Chemistry and Life Sciences (Luxembourg, 2025).

There are 30 scientific works on the subject of the dissertation. 9 of them are articles ("European Chemical Bulletin"-1, "Azerbaijan Chemical Problems"-1, Journal "Oil and Gas"-2 articles, "Processes of Petrochemistry and Oil Refining"-2 articles. "Magyar Tudományos Journal"- 1, "Polish Journal of Science"-1 "Azerbaijan Oil Industry Journal"-1) 9 are conference materials, and the rest are abstracts of reports.

Place of the dissertation work. The work was carried out in the laboratory “Surface active reagents and preparations” of the Institute of Petrochemical Processes named after Academician Y.H Mammadaliyev Ministry of Science and Education of Azerbaijan.

The total volume of the dissertation indicating the volume of structural sections. The thesis is presented on 221 pages (206371 characters) collected on the computer and includes 87 pictures, 56 tables. The text of the dissertation consists of an introduction, 6 chapters, conclusions, and a list of used literature sources. 198 references are given in the list of literature sources. The structure of dissertation - contents 3170, introduction 10843, first chapter 48035, second chapter 10335, third chapter 42193, fourth chapter 43153, fifth chapter 34121, sixth chapter 11935, results 2586 characters.

In the introduction, the relevance, purpose, scientific innovation, theoretical and practical importance of the researches are given and justified.

The first chapter provides a literature review on the structure and characterization, synthesis, properties and application areas of Gemini-type surface-active substances.

In the second chapter, the characteristics of the used substances and devices are given, the methods of the surfactant synthesis, surface activity, specific electrical conductivity, petrocollecting, petrodispersing, inhibitory bactericidal properties againts SRB are described.

In the third chapter, the structure and composition, physicochemical properties (solubility in solvents, surface activity, electrical conductivity) of both mono and Gemini type surfactants obtained from the reaction of fatty acids containing C_{12}-C_{18} carbon atoms with ethane-1,2-diamine, also, the results of the study of petrocollecting and petrodispersing properties are described and analyzed.

The fourth chapter describes the structure and composition, physicochemical properties, as well as the description and analysis of the results of the study of petrocollecting and petrodispersing properties of both mono and Gemini type surfactants obtained from the reaction of fatty acids containing C_{12}-C_{18} carbon atoms with N^1-(2-aminoethyl)ethane-1,2-diamine.

The fifth chapter provides the structure and composition, physical-chemical properties, as well as a description and analysis of the results of the study of petrocollecting and petrodispersing

properties of both mono- and Gemini-type surfactants obtained from the reaction of fatty acids with C_{12}-C_{18} carbon atoms with polyethylenepolyamine.

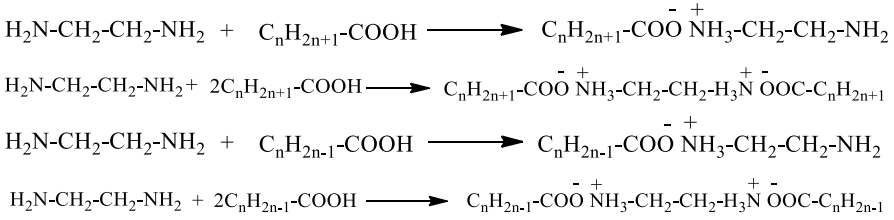
In the sixth chapter, the influence of the number of nitrogen atoms in the final product on many physicochemical properties of the obtained Gemini surfactants was discussed.

At the end of the thesis work, results and a list of literature were presented.

MAIN CONTENT OF THE WORK

Synthesis and properties of new surfactants based on fatty acids and ethane-1,2-diamine

The reaction was carried out with C_{12}-C_{18} carbon series fatty acids and ethane-1,2-diamine in equimolar and twice the molar ratio of the acid to the amine in the temperature range of 50-90°C, under closed conditions, continuously stirring with a magnetic stirrer for 9-10 hours. Based on the reaction, both mono and new Gemini surfactants were synthesized. The reaction scheme is shown below:



Here, n=11-17.

The surfactants obtained from oleic acid are viscous, the others are in solid form. The structure and composition of the obtained Gemini-type surfactants were identified by IR, UV and NMR spectroscopy.

The IR spectrum of Gemini surfactant synthesized on the basis of oleic acid and ethane-1,2-diamine is given in figure 1. When studying the IR spectrum, it is clear that the valence oscillations of the N-H bond in the NH_2 group are observed at the level of 3293.64 cm^{-1}. The absorption bands in the spectrum at 3007.88 cm^{-1} represent the C-

H valence oscillations of the double bond. The valence oscillations of the C-H bond in the CH_3 and CH_2 groups can be determined by absorption bands of 2953.8, 2921.51 and 2852.13 cm^{-1}. The deformation oscillations of valence C=C bond and N^{+}-H bond is observed in absorption bands of 1643.95 and 1542.22 cm^{-1}. Absorption bands corresponding to deformation oscillations of C-H bond in CH_3 and CH_2 groups are reflected at 1462.46 and 1400.23 cm^{-1}. Absorption bands at 1304.68 and 1184.16 cm^{-1} are related to valence vibrations of C-N bond. The deformation oscillations of the C-H bond of the double bond correspond to the absorption bands at 967.20 cm^{-1} and 918.86 cm^{-1} in the spectrum. $(CH_2)X$ dancer vibrations are observed at 721.00 and 721.41 cm^{-1}.

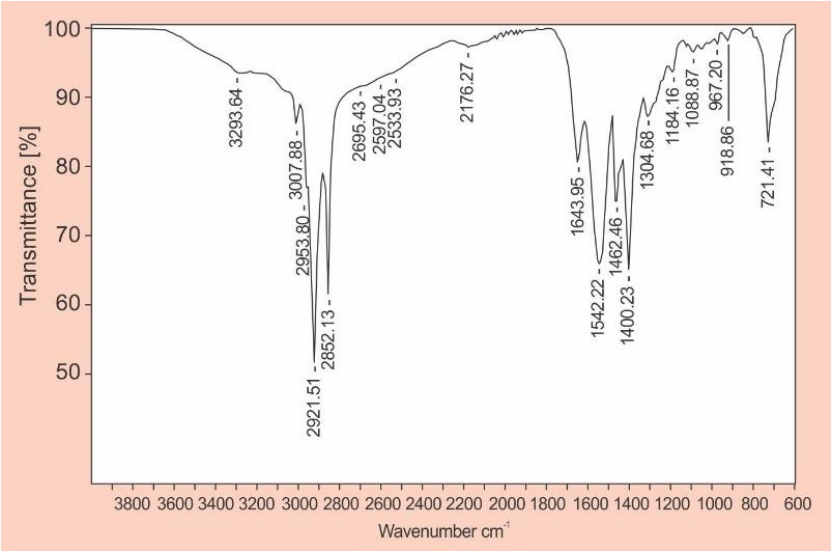
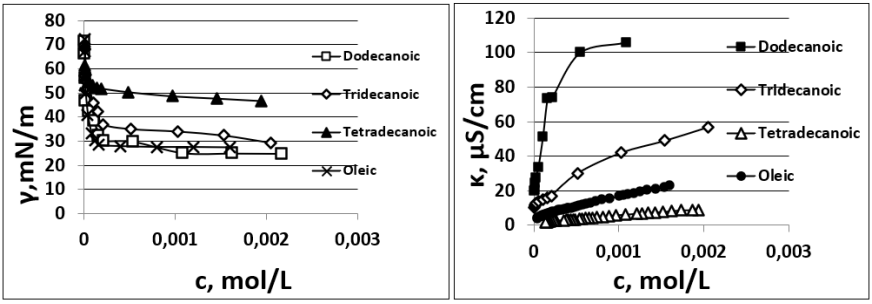


Fig. 1. IR spectrum of Gemini surfactant synthesized on the basis of oleic acid and ethane-1,2-diamine

Solutions of the synthesized complexes with different concentrations in water were prepared and their surface tension at the air-water boundary was measured with a tensiometer, and electrical conductivity was measured with a conductometer. Based on the obtained results, surface tension and electrical conductivity isotherms

for Gemini surfactants were constructed and shown in figure 2.



a) b)

Fig. 2. a) surface tension and b) electrical conductivity isotherms of Gemini surfactants synthesized on the basis of fatty acids and ethane-1,2-diamine

Thermodynamic parameters of surface activity and electrical conductivity for the synthesized surfactants (CMC - Critical Micelle Concentration, γ_{KMQ} - surface tension corresponding to the CMC point, π_{KMQ} - surface pressure, C_{20} - density with a value 20 units smaller than the surface tension value of water, Γ_{max} - maximum adsorption, A_{min} - minimum surface area per molecule, α - dissociation degree of counterion, β - degree of association of the counterion, ΔG_{mis} Gibbs free energy of micellization, ΔG_{ad} - Gibbs free energy of adsorption, I_{act} – interphase activity) were calculated and listed in table 1 and 2, respectively.

Table 1

Surface activity parameters of surfactants based on ethane-1,2-diamine and fatty acids (C_{12}-C_{18}).

Surfactant	CMC*10^4 (mol/L)	γ_{KMQ} (mN/m)	π_{CMC} (mN/m)	C_{20}*10^4 (mol/L)	Γ_{max}*10^{10} (mol/cm^2)	A_{min}*10^2 (nm^2)
Dodecanoic acid						
Mono 24.8°C	9.62	27.93	43.71	1.92	1.92	86.46
Dimer 22°C	2.17	30	41.37	0.19	1.51	109.78
Tridecanoic acid						
Mono 20°C	2.73	35.97	35.03	0.3	1.9	85.8
Dimer 25°C	2.05	37	34.64	0.2	0.99	167.55
Tetradecanoic acid						
Mono 20°C	3.47	47	24	1.74	0.95	175.32

Continuation of Table 1

Dimer 20.5℃	1.45	52	19	3.44	0.73	227.48
Oleic acid						
Mono 21℃	2.19	25.84	46.54	0.11	2.1	79.1
Dimer 21℃	1.6	28.37	44.01	0.19	1.65	100.7

In the table above, the parameters of surface activity of substances with mono and dimer structures formed by ethane-1,2-diamine of different carbonic acids, which differ from each other by the number of carbon atoms and the presence of double bonds, are mentioned. It was found that surfactants with gemini structure have lower CMC and larger A_{min} value than their mono homologues in all mentioned reaction products. As it is known from the literature, Gemini surfactants are superior to mono-type ones in many respects. Comparing Gemini surfactants among themselves, we can note that the lowest surface tension (28.37 mN/m) belongs to Gemini product obtained from synthesis with oleic acid. Looking at the CMC, it is clear that the smallest concentration ($1.45*10^{-4}$ mol/L) is observed in the complex with tetradecanoic acid. The corresponding CMC points for the complexes obtained with oleic acid and tetradecanoic acid are very close to each other, and oleic acid, as we mentioned, has much less value of the surface tension at the air-water interface than the others. Looking at the surface tension values for the compounds given in the table, it is clear that an increase in surface tension indicators is observed as the number of carbon atoms increases in fatty acids that do not contain double bonds. In addition, based on the obtained results, we can note that in the compounds obtained from the synthesis of these fatty acids with ethane-1,2-diamine, with an increase in the number of carbon atoms in the alkyl chain in the last molecule, a decrease in the value of CMC is observed.

Considering table 2, we can note that the most negative (-32.25 kJ/mol and -33.85 kJ/mol) of both micelle formation and adsorption free Gibbs energy are compounds which belongs to the synthesis of ethane-1,2-diamine and oleic acid. The least negative value of both mentioned parameters is observed in the reaction product of ethane-1,2-diamine with dodecanoic acid. While a decrease in interfacial activity is observed from dodecanoic acid towards the synthesis with

tetradecanoic acid, this parameter has the highest value due to the presence of a double bond in the synthesis with oleic acid.

Table 2

Electrical conductivity and thermodynamic parameters of Gemini surfactants synthesized based on fatty acids and ethane-1,2-diamine

Fatty acid	α	β	ΔG_{mic}, (kJ/mol)	ΔG_{ad}, (kJ/mol)	$I_{act,}$, (kJ/mol*Å)
Dodecanoic acid	0.9	0.1	-22.7	-25.48	23.2
Tridecanoic acid	0.48	0.52	-28.55	-32.23	19.24
Tetradecanoic acid	0.89	0.11	-23.99	-26.59	11.69
Oleic acid	0.51	0.49	-32.25	-33.85	33.6

The petrocollecting and petrodispersing properties of tetradecanoic acid and ethane-1,2-diamine based substances of both structures were comparatively analyzed in three different water sus with different degrees of mineralization, and the results are given in table 3.

Analyzing the monotype compound according to table 3, it can be seen that the highest value of petrocollecting coefficient (K) is 29.59, which is manifested when it is applied with a solution in 5% alcohol in distilled water. An approximate 30-fold reduction of the previous area of the oil spot remains for 144 hours, that is, until the distilled water evaporates, while the reagent retains its potency in its pure form. In the Gemini surfactant, the highest K value (34) can be seen in all three water samples.

Table 3

Petrocollecting and petrodispersing parameters of mono and Gemini surfactants based on tetradecanoic acid and ethane-1,2-diamine

Surfactant	State of the salt	τ_{hour}	K/Kd		
			Sea water	Tap water	Distilled water
Mono	5 wt.% aqueous solution	0-1	6.4	8.54	21.37
		1-2	8	10.12	21.37
		2-44	9	10.12	21.37
		44-144	9, drying	10.12	21.37
		144	9, drying	10.12, drying	10.12, drying

Continuation of Table 3

	5 wt.% ethanolic acid	0-1	25.64	25.6	29.59
		1-2	25.64	25.6	29.59
		2-44	25.64	25.6	29.59
		44-144	25.64	25.6	29.59
		144	25.64, drying	25.6, drying	29.59, drying
	Solid	0-1	10.99	19.2	10.1
		1-2	10.99	19.2	10.1
		2-44	10.99	19.2	10.1
		44-144	10.99	19.2	10.1
		144	10.99, drying	19.2, drying	10.1, drying
Gemini	5 wt.% aqueous solution	0-1	1	1	1
		1-4	1.36	1.36	1.96
		4-28	2.77	3	3
		28-76	34	34	7.24
		76-100	10.99	19	19
		110	10.99, drying	7.63, drying	19, drying
	5 wt.% ethanolic solution	0-1	90%	21.37	25.64
		1-4	10.99	21	25.64
		4-28	13.77	21	25.64
		28-76	13	34	34
		76-100	10.99	19	15
		110	10.99, drying	85%, drying	15, drying
	Solid	0-1	1	1	1
		1-4	3	1.36	1.36
		4-28	12.25	3.78	2.77
		28-100	9.6	12.25	8.5
		110	9.6, drying	5.54, drying	15, drying

Synthesis and properties of new surfactants based on fatty acids and N^1-(2-aminoethyl)ethane-1,2-diamine

The reaction was carried out with C_{12}-C_{18} carbon series fatty acids and N^1-(2-aminoethyl)ethane-1,2-diamine in equimolar and twice the molar ratio of the acid to the amine in the temperature range of 50-90℃, under closed conditions, continuously stirring with a magnetic stirrer for 9-10 hours. Based on the reaction, both mono and

new Gemini surfactants were synthesized. The reaction scheme is shown below:

$$H_2N\text{-}(CH_2)_2\text{-}NH\text{-}(CH_2)_2\text{-}NH_2 + C_nH_{2n+1}\text{-}COOH \longrightarrow$$

$$\longrightarrow C_nH_{2n+1}\text{-}COO^- \; NH_3^+\text{-}(CH_2)_2\text{-}NH\text{-}(CH_2)_2\text{-}NH_2$$

$$H_2N\text{-}(CH_2)_2\text{-}NH\text{-}(CH_2)_2\text{-}NH_2 + 2C_nH_{2n+1}\text{-}COOH \longrightarrow$$

$$\longrightarrow C_nH_{2n+1}\text{-}COO^- \; NH_3^+\text{-}(CH_2)_2\text{-}NH\text{-}(CH_2)_2\text{-}H_3N^+ \; OOC\text{-}C_nH_{2n+1}$$

$$H_2N\text{-}(CH_2)_2\text{-}NH\text{-}(CH_2)_2\text{-}NH_2 + C_nH_{2n-1}\text{-}COOH \longrightarrow$$

$$\longrightarrow C_nH_{2n-1}\text{-}COO^- \; NH_3^+\text{-}(CH_2)_2\text{-}NH\text{-}(CH_2)_2\text{-}NH_2$$

$$H_2N\text{-}(CH_2)_2\text{-}NH\text{-}(CH_2)_2\text{-}NH_2 + 2C_nH_{2n-1}\text{-}COOH \longrightarrow$$

$$\longrightarrow C_nH_{2n-1}\text{-}COO^- \; NH_3^+\text{-}(CH_2)_2\text{-}NH\text{-}(CH_2)_2\text{-}H_3N^+ \; OOC\text{-}C_nH_{2n-1}$$

Here, n=11-17.

The structure and composition of the synthesized surfactants were identified by IR, UV and NMR spectroscopy.

The UV spectrum of tetradecanoic acid is depicted in figure 3. It is clear from the graph that the maximum absorption occurs at a wavelength of 225 nm. This is explained by the presence of amino groups.

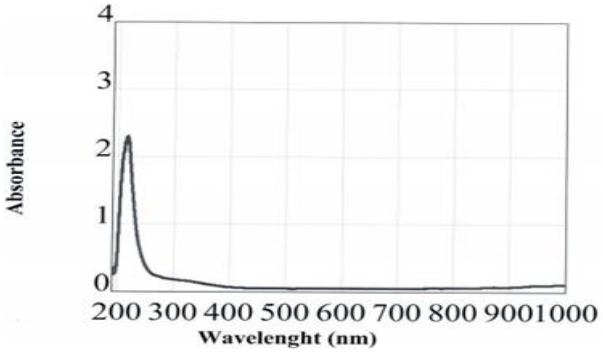


Fig. 3. UV spectrum of Gemini surfactant based on tetradecanoic acid and N[1]-(2-aminoethyl)ethane-1,2-diamine

The structure and composition of Gemini-type product synthesized on the basis of oleic acid and N^1-(2-aminoethyl)ethane-1,2-diamine was confirmed by hydrogen and carbon NMR-spectroscopy. The spectra are given in figures 4 and 5, respectively.

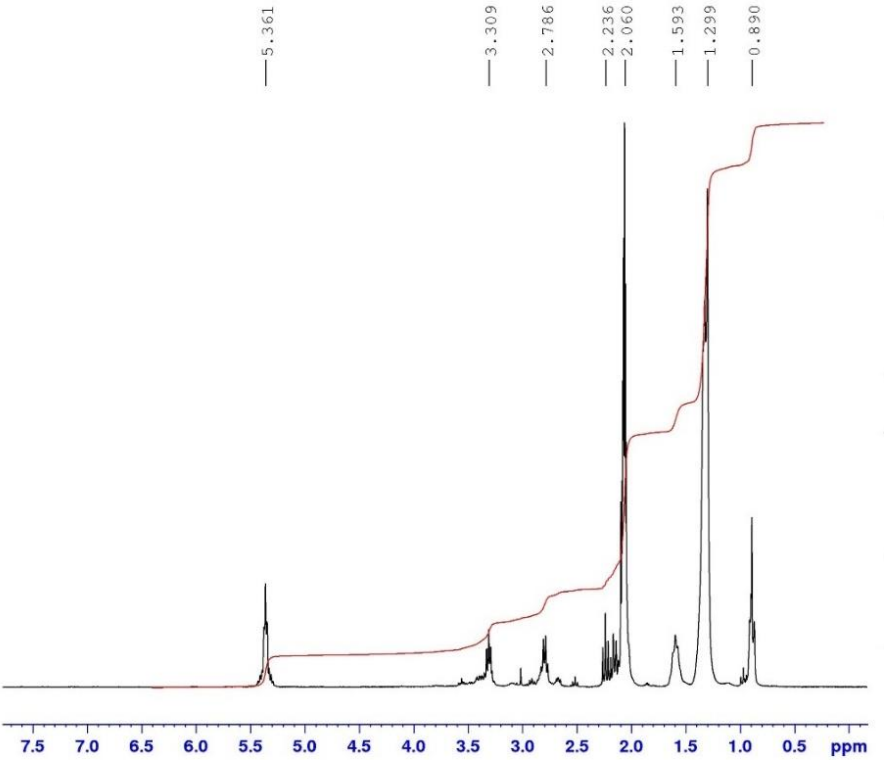


Fig. 4. ^{1}H NMR spectrum of Gemini surfactant based on oleic acid and N^1-(2-aminoethyl)ethane-1,2-diamine

In the spectrum given in figure 4, ^{1}H NMR (BRUKER-Fourier 300.18 MHs, Aseton-D6, δ, ppm.): 0.89 (t., 6H, CH_3), 1.22-1.41 (m., 40H, CH_2), 1.59 (m., 4H, CH_2), 2,23 (t., 4H, CH_2COO), 2.45-3.53 (m., 16H, CH_2-NH, CH_2-CH=), 5.36 (m., 4H, CH=CH).

In the spectrum given in figure 5, ^{13}C NMR ppm.: 13.49 (CH_3), 22.45, 25.10, 25.38, 26.92, 31.75, 34.49, 35.84 (CH_2), 127.89, 129.67 (CH=CH).

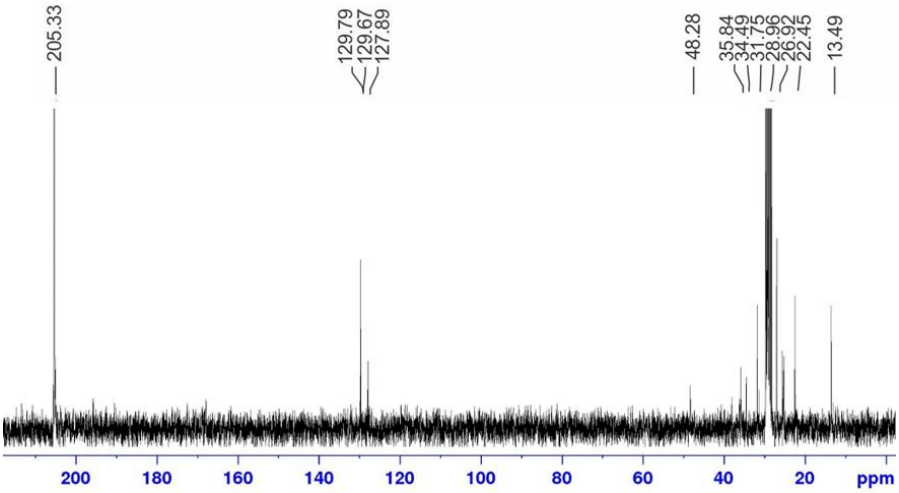


Fig. 5. ^{13}C NMR spectrum of Gemini surfactant based on oleic acid and N^1-(2-aminoethyl)ethane-1,2-diamine

Solutions of the obtained complexes with different concentrations in water were prepared and their surface tensions at the air-water boundary were measured with a tensiometer, surface tension isotherms were constructed based on the obtained results and are depicted in figure 6.

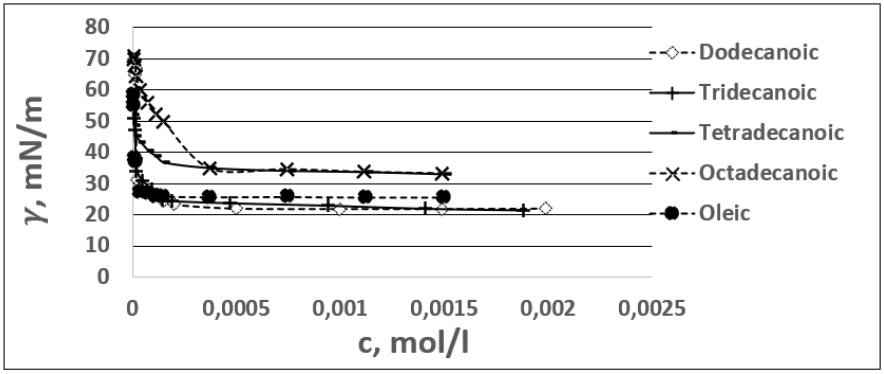


Fig. 6. Surface tension isotherms of Gemini surfactants based on fatty acids and N^1-(2-aminoethyl)ethane-1,2-diamine

Based on the obtained results, thermodynamic parameters of surface activity and electrical conductivity for surfactants are listed in tables 4 and 5, respectively.

Table 4

Surface activity parameters of surfactants based on N^1-(2-aminoethyl)ethane-1,2-diamine and fatty acids

Surfactant	CMC*10^4 (mol/L)	γ_{CMC} (mN/m)	π_{CMC} (mN/m)	C_{20}*10^4 (mol/L)	Γ_{max}*10^{10} (mol/cm^2)	A_{min}*10^2 (nm^2)
Dodecanoic acid						
Mono 25°C	3.29	23.49	48.09	0.12	1.69	97.86
Dimer 22°C	1.99	23.24	49.14	0.16	1.72	96.30
Tridecanoic acid						
Mono 21°C	2.36	25	45.35	0.12	2.2	75.54
Dimer 22°C	1.41	24.65	46.9	0.05	1.06	157
Tetradecanoic acid						
Mono 20°C	0.75	27.1	44.21	0.04	0.74	226
Dimer 22°C	1.51	36.58	34.98	0.08	0.67	248
Octadecanoic acid						
Mono 23°C	2.58	40	31.7	0.92	1.59	104
Dimer 23°C	3.72	34.8	36.90	3.07	1.07	248.23
Oleic acid						
Mono 26°C	1.94	28.17	43.87	0.16	2.03	82
Dimer 26°C	1.12	25.93	45.09	0.08	1.29	129.21

Looking at Table 4, it is clear that the surfactants with the Gemini structure mainly have lower CMC and greater A_{min} values than their mono counterparts in the reaction products. When comparing the surfactants with a gemini structure, it is clear that, except for oleic acid, in the complexes obtained with the presence of other acids, an increase in the value of the surface tension is almost observed with the increase in the number of carbon atoms. The fact that the surface tension in the compound with octadecanoic acid (34.8 mN/m) is smaller than that in the compound with tetradecanoic acid (36.58 mN/m), which slightly

disturbs this increase. However, looking at the CMC values, it is possible to see that the regularity is uniform. So, with the exception of oleic acid, the CMC value increases mainly with the extension of the carbon chain, as well as the surface tension value. Comparing the values of CMC and surface tension of surfactants based on dodecanoic acid and tridecanoic acid, it is possible to see that both compounds have almost the same surface activity. Thus, despite the fact that the first compound exhibits a lower surface tension, it shows it at a higher concentration than the second complex. As in the synthesis of oleic acid with ethane-1,2-diamine, despite the maximum carbon number, the minimum CMC point ($1.12*10^{-4}$ mol/L) and very effective surface tension value (25.93 mN/m) shows its advantage.

Table 5

Electrical conductivity and thermodynamic parameters and of surfactants based on N^1-(2-aminoethyl)ethane-1,2-diamine and fatty acids (C_{12}-C_{18})

Surfactant	α	β	ΔG_{mic}, kJ/mol	ΔG_{ad}, kJ/mol	I_{act}, kJ/mol*Å
Tridecanoic acid					
Mono	0.82	0.18	-24.17	-26.22	34.71
Gemini	0.5	0.5	-32.57	-36.96	23.55
Tetradecanoic acid					
Mono	0.9	0.1	-23.89	-29.12	12.9
Gemini	0.42	0.58	-34.27	-39.48	15.9
Octadecanoic acid					
Mono	0.88	0.12	-22.87	-24.85	23.78
Gemini	0.22	0.78	-34.80	-38.27	24.54
Oleic acid					
Mono	0.9	0.1	-23.89	-29.12	35.61
Gemini	0.32	0.68	-37.81	-41.32	32

Looking at the table above, it is clear that while the Gibbs free energy of micelle formation remains largely constant with alkyl chain elongation in the mono-structured surfactants, the opposite is true for the Gemini compound. In addition, for the Gemini structure, an increase in both the Gibbs free energy of micelle formation and the Gibbs free energy of adsorption is observed with the lengthening of the carbon chain.

DTA analyzes were performed to study the thermal properties of

Gemini surfactant obtained from the synthesis of hexadecanoic acid with N^1-(2-aminoethyl)ethane-1,2-diamine and the result is shown in figure 7.

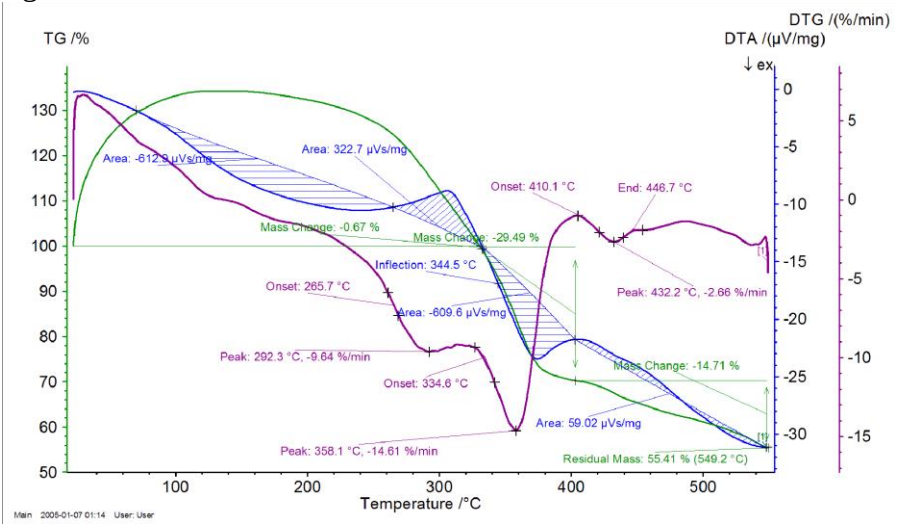


Fig. 7. DTA plot of Gemini surfactant based on hexadecanoic acid and N^1-(2-aminoethyl)ethane-1,2-diamine

Based on the TG/DTG curve of the conducted analysis, it can be said that as the temperature rises, a number of physical processes occur in the surfactants, such as a change in the aggregate state. At this time, depending on its composition and structure, the molecules that are weakly connected to the surface and turned into a light gas move from one surface to another surface, settle in the spaces between the layers, and in connection with this, it is possible to observe a relative increase in volume. The surfactant was stable up to 265.7°C, with a mass loss of 0.67%. In these temperature intervals, as the temperature rises in the DTA curve, it is possible to observe the change of enthalpy in the processes of thermal destruction and generation of phase transitions of chemical reactions. Thus, endothermic heat effects are observed in the range of 90-265°C and the consumed heat area is -612.9 μVs/mg. In the DTA curve in the range of 265.7°C-320°C, the exo effect, which is accompanied by the internal oxidation process due to the decomposition, is observed, the thermal area is 322.7 μVs/mg. At 334-410°C, the thermal destruction process with a maximum of 358.1°C

occurs in the surfactant and the mass loss is 29.9%. The heat area used for this process is -609.6 μVs/mg. In the next temperature range of 400-550°C, the mass loss is 14.71%, creating endo and exothermic heat effects. The total thermal area is 59.02 μVs/mg.

According to the DLS analysis results of dodecanoic acid and N^1-(2-aminoethyl)ethane-1,2-diamine based dimer structured compound in water with different concentrations given in table 6, it is clear that as while the concentration of particles in the solution increases up to a solution with a concentration of 0.00099 mol/L, an increase was observed in the median, mode, and geometric mean, however, after the concentration of 0.0019 mol/L, the decrease in the median, mode, and geometric mean was observed. It is clear that the diffusion coefficient is inversely proportional to the median, mode and geometric mean.

Table 6

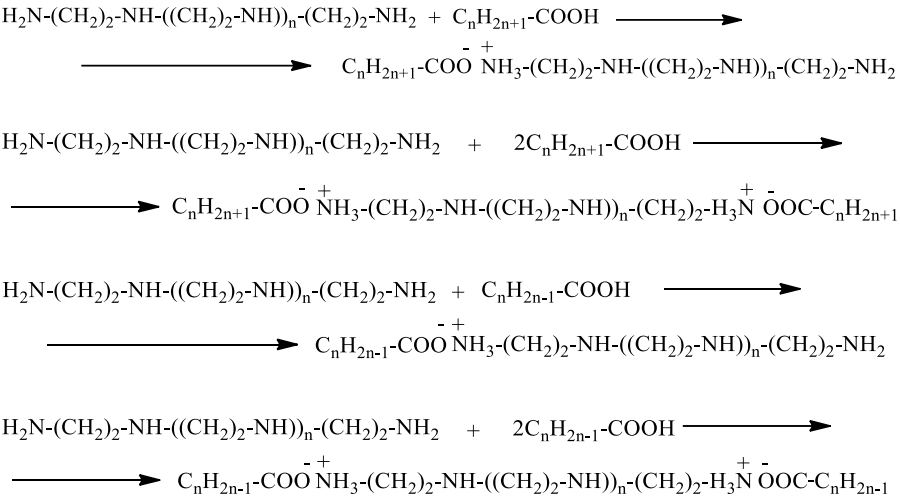
DLS analysis results of surfactant with dimer structure based on dodecanoic acid and N^1-(2-aminoethyl)ethane-1,2-diamine

Aqueous solution, mol/L	$1.98*10^{-5}$	$9.9*10^{-5}$	0.00019	0.00099	0.0019
Diameter interval, nm	2.3-9	45-1500	670-4500	1500-5000	1500-4000
Median, nm	4.8	125.2	1894.1	2805.8	2448.2
Moda, nm	4.8	122.4	1862.5	2803.5	2448.7
Geometric mean, nm	4.7	132.7	1875.4	2785.8	2432.1
Refractive coefficient	1.67	1.67	1.67	1.67	1.67
Diffusion coefficient $m^2/s*10^{13}$	540	20.6	1.36	0.92	1.05

Synthesis and study of Gemini surfactants based on fatty acids and polyethylenepolyamine

Fatty acids of C_{12}-C_{18} carbon range and polyethylenepolyamine were reacted in the temperature interval of 50-90°C, with continuous stirring with a magnetic stirrer, for 9-10 hours. Based on the reaction,

both mono and new Gemini surfactants were synthesized. The reaction scheme is shown below:



Here, n=11-17.

The structure and composition of the synthesized surfactants were confirmed by IR, UV and NMR spectroscopy.

In order to determine the thermodynamic parameters of the surface activity and electrical conductivity of the synthesized Gemini compounds, the corresponding isotherms were constructed and are given in figure 8:

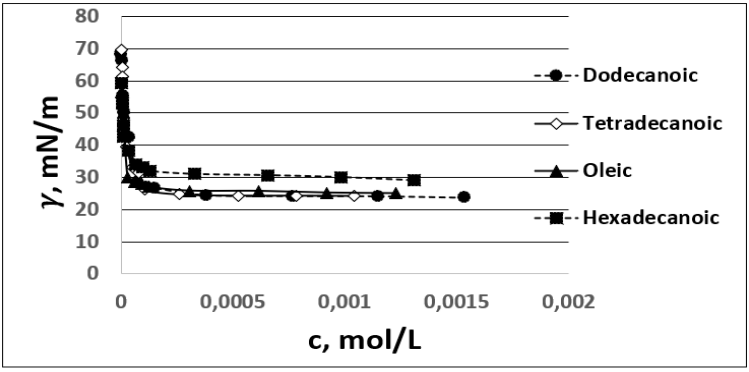


Fig. 8. Surface tension isotherms of Gemini surfactants based on fatty acids and polyethylenepolyamine

The calculated surface activity parameters of the surfactants based on fatty acids and polyethylenepolyamine are listed in Table 7.

Table 7

Surface activity parameters of the surfactants based on fatty acids and polyethylenepolyamine

Surfactant	CMC*10^4 (mol/L)	γ_{CMC} (mN/m)	π_{CMC} (mN/m)	C_{20}*10^4 (mol/L)	Γ_{max}*10^{10} (mol/cm^2)	A_{min}*10^2 (nm^2)
Dodecanoic acid						
Mono 25℃	5.5	26.95	44.77	0,19	1.6	107.12
Gemini 22℃	1.2	26.73	45.61	0,15	1.7	100.12
Tetradecanoic acid						
Mono 21℃	1.56	25.98	46.66	0.104	1.77	93.72
Gemini 21℃	1.04	26.38	46.26	0.009	1.61	103.38
Hexadecanoic acid						
Mono 21℃	1.5	35.3	36.91	0.092	1.5	253
Gemini 21℃	1.3	32	39.71	0.065	0.99	166
Oleic acid						
Mono 24.8℃	0.18	28.32	44.35	0.937	4.18584	40
Gemini 22℃	1.23	27	44.66	0.91911	1.08537	153

Based on the parameters given for the compounds in Table 7, it can be noted that there is no noticeable difference between the surface tension values of mono and Gemini type surfactant based on polyethylenepolyamine and fatty acids. When comparing the Gemini surfactants, it is clear that the compound obtained from tetradecanoic acid is able to reduce the surface tension at the air-water interface the most (26.38 mN/m). In addition, the smallest CMC ($1.04*10^{-4}$ mol/L) belongs to the complex synthesized from tetradecanoic acid.

As with other amines, in the compound obtained from the reaction of oleic acid with polyethylenepolyamine, despite the maximum number of carbon atoms (18), a sufficient improvement in surface tension is observed with the presence of a double bond.

Evaluation of the effect of the number of nitrogen atoms on the properties of the synthesized Gemini-type surfactants

In order to determine the effect of the number of nitrogen atoms in the Gemini-type compounds obtained from the synthesis with ethane-1,2-diamine, N^1-(2-aminoethyl)ethane-1,2-diamine and polyethylenepolyamine in the presence of tetradecanoic acid, a series of graphs were constructed and relevant parameters were calculated. The graph of dependence of surface tension on concentration is shown in figure 9. The surface activity parameters were determined using the graphs and are listed in Table 8.

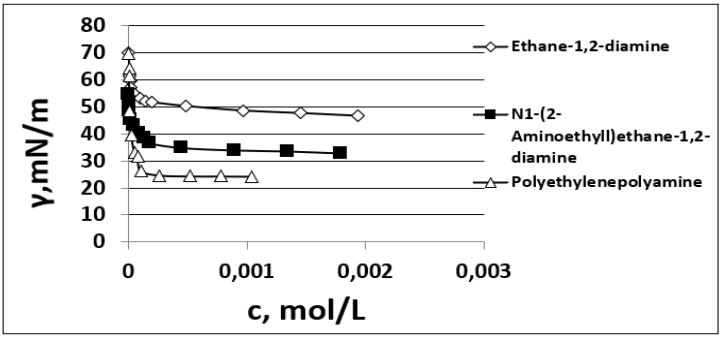


Fig. 9. Surface tension isotherms of tetradecanoic acid and polyamine-based Gemini surfactants

Table 8

Surface activity parameters of tetradecanoic acid and polyamine-based Gemini surfactants

Polyamine	CMC*10^4 (mol/L)	γ_{CMC} (mN/m)	π_{cmc} (mN/m)	C_{20}*10^4 (mol/L)	Γ_{max}*10^{10} (mol/cm^2)	A_{min}*10^2 (nm^2)
Ethane-1,2-diamine	1.45	52	19	3.44	0.73	227.48
N^1-(2-Aminoethyl) ethane-1,2-diamine	1.5	36.58	34.98	0.075	0.67	248.23
Polyethylene-Polyamine	1.04	26.38	46.26	0.12	1.61	103.38

Referring to Table 8, we can say that the polyethylenepolyamine

salt of tetradecanoic acid has the lowest value of both γ_{CMC} and CMC, maximum surface pressure (46.26 mN/m). As can be seen from the table, as the number of nitrogen atoms in the final product increases, the surface tension value decreases. When comparing the CMC point according to the surface tension value, improvement is observed with the increase in the number of nitrogen atoms. The smallest CMC point (1.04 mol/L) coincides with the third product, that is, the synthesis with polyethylenepolyamine. The surface pressure in the final product appears to be proportional to the increase in the number of nitrogen atoms.

The maximum petrocollecting and petrodispersing properties of Gemini surfactants with different amino compounds of tetradecanoic acid in sea, drinking and distilled water are given in table 9.

Table 9

Maximal petrocollecting and petrodispersion parameters of various surfactants synthesized on the basis of tetradecanoic acid and polyamines

Amine	State of the reagent	Sea water		Tap water		Distilled water	
		τ, hours	K/K_d	τ, saat	K/K_d	τ, saat	K/K_d
Ethan-1,2-diamine	5 wt.% aqueous solution	48	34	48	34	24	19
	5 wt.% ethanolic solution	24/4	13/ 90%	48/dry	34/85%	24	34
	Solid	24	12.25	72	12.25	48	12.25
N^1-(2-amino ethyl) ethane-1,2-diamine	5 wt.% aqueous solution	72	10.5	72	12.25	72	12.25
	5 wt.% ethanolic solution	2/ 508	91%/ 19	24/dry	19/84%	26/460	25/ 94%
	Solid	24	78%	48/460	16/93%	508	19

Continuation of Table 9

Polyethylenepolyamine	5 wt.% aqueous solution	19	94%	72	16.7	1	12.28
	5 wt.% ethanolic solution	72/ 144	9/ 89%	1/72	12/89%	1/216	12/ 88%
	Solid	168	87%	1	12.03	1	12.26

As can be seen from table 9, all three complexes obtained from the synthesis of tetradecanoic acid with polyamines have both petrocollecting and petrodispersing properties. The highest petrocollecting coefficient was 34 in the first complex, except for the pure state of the reagent in all three water samples. In the synthesis with ethane-1,2-diamine, the highest oil dispersion is 90% and occurs when applied with a 5% ethanolic solution in sea water.

Below are some graphs and comparative tables with the study of the effect of the number of nitrogen atoms on various parameters in all three compounds obtained from the synthesis of oleic acid and ethane-1,2-diamine, N^1-(2-aminoethyl)ethane-1,2-diamine and polyethylenepolyamine.

First, graphs of dependence of the surface tension of the compounds and electrical conductivity on the density are given in figures 10 and 11, respectively, and the parameters calculated based on the isotherms are described in tables 10 and 11, respectively.

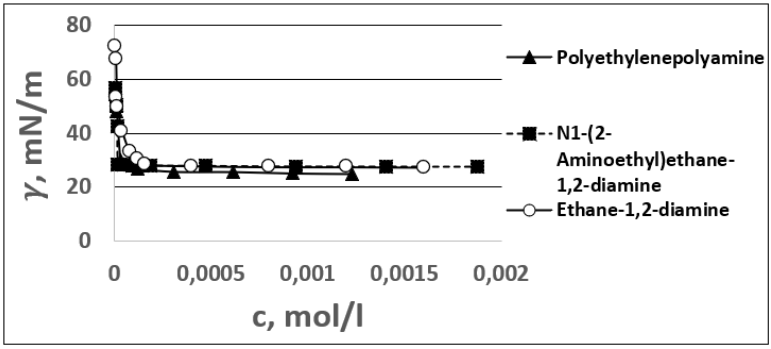
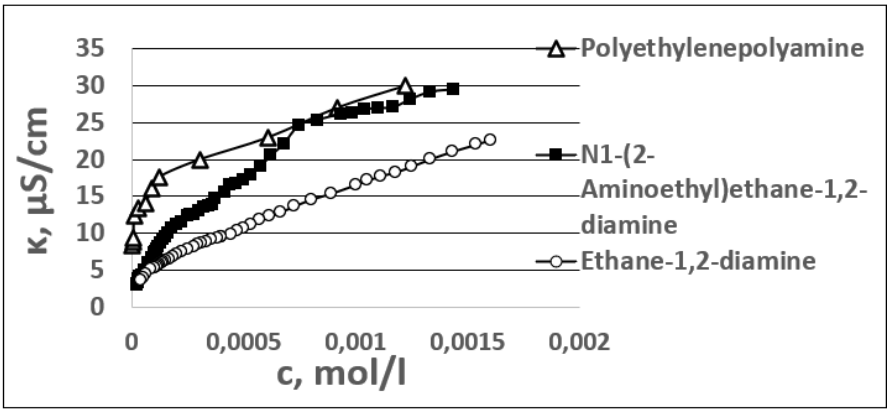


Fig. 10. Surface tension isotherms of surfactants obtained with the reaction of oleic acid with polyamines



Fig, 11. Electrical conductivity isotherms of various surfactants synthesized on the basis of oleic acid and polyamines

Table 10

Surface activity parameters of various surfactants synthesized on the basis of oleic acid and polyamines

Polyamine	CMC*10^4 (mol/L)	γ_{CMC} (mN/m)	π_{CMC} (mN/m)	Γ_{max} *10^{10} (mol/cm^2)	A$_{min}$* 10^2 (nm^2)
Ethane-1,2-diamine	1.6	28.37	44.01	1.65	60.6
N^1-(2-aminoethyl)ethane-1,2-diamine	1.12	25.925	45.085	1.29	129.2
Polyethylene polyamine	1.225	27	44.66	1.08	153.04

According to the table above, it can be noted that the synthesis of oleic acid with ethane-1,2-diamine, N^1-(2-aminoethyl) ethane-1,2-diamine and polyethylenepolyamine lead to the greatest reduction of surface tension at the air-water interface (25,925 mN/m) is the surfactant obtained from the synthesis with diethylenetriamine. For other compounds, this value does not lag behind. Similar to the value of surface tension, the complex with the smallest CMC (1.12 mol/L) is the compound obtained in the presence of N^1-(2-aminoethyl) ethane-1,2-diamine. The surface tension values are not very different from

each other, and the surface pressures are also close to each other. However, the slightly larger surface pressure naturally belongs to N^1-(2-aminoethyl)ethane-1,2-diamine. This, in turn, indicates that the petrocollecting indicators will be better than others, which will be explained in more detail in the following pages. Since the Γ_{max} value is the minimum in the synthesis with polyethylenepolyamine, the surface area per molecule has the maximum value for this synthesis compared to the others.

Table 11

Electrical conductivity and thermodynamic parameters of various surfactants synthesized on the basis of oleic acid and polyamines

Polyamine	α	β	ΔG_{mic}, kJ/mol	ΔG_{ad}, kJ/mol
Ethane-1,2-diamine	0.52	0.48	-32.25	-33.85
N1-(2-Aminoethyl)ethane-1,2-diamine	0.33	0.67	-37.81	-41.31
Polyethylene polyamine	0.15	0.85	-40.58	-44.7

Looking at the thermodynamic parameters of electrical conductivity given in Table 11, it is clear that with the increase in the number of nitrogen atoms in the final product, both micelle formation and adsorption Gibbs free energy values become more negative. The largest numerical value of both parameters coincides with the synthesis with polyethylenepolyamine. This is a sign that both the micelle formation and the adsorption process occur more naturally and freely. A regularity is also observed in the α value of compounds. So, for the first product, the electrical conductivity before and after CMC occurs with the same intensity depending on the concentration. In the second joint, after the CMC point, the electrical conductivity varies less depending on the concentration, and in the third joint, with the least intensity. In the synthesis with ethane-1,2-diamine, the thermodynamic parameters of electrical conductivity show very close values. This shows that these processes occur at the same time. For the

other two compounds, these parameters are equally different from each other.

The solubility of various polyamine derivatives of oleic acid in different solvents was tested and the results are given in Table 12.

Table 12

Solubility properties of various surfactants synthesized on the basis of oleic acid and polyamines

Solvent	Solubility index (g/ml) g-quantity of substance ml-volume of solvent		
	Ethane-1,2-diamine	N^1-(2-aminoethyl) ethane-1,2-diamine	Polyethylenepolyamine
Water	0.0084	0.067	Partially soluble
N-heptane	0.086	0.035	-
Cyclohexane	0.261	0.034	0.017
Acetone	0.110	0.11	0.059
Ethyl alcohol	0.258	0.024	0.032
Methanol	0.039	0.018	0.062

Looking at Table 12, it is clear that the highest solubility among the surfactants is 0.258 g/ml, which is the compound based on oleic acid and ethane-1,2-diamine. The worst solubility index can be seen again in the aqueous solution of the surfactant compound. As the number of nitrogen atoms in the final product of the reaction increases, the solubility indicators in solvents mainly decrease.

The bactericidal properties of solutions of different concentrations of Gemini-type surfactants obtained from the synthesis of oleic acid with N^1-(2-aminoethyl)ethane-1,2-diamine and polyethylenepolyamine were tested. The obtained results are given in Table 13.

Table 13

Analysis of bactericidal properties of Gemini surfactants based on oleic acid and polyamines

The name of the complexes	Concentration of the substance, C-mg/l	Bacterial count (cell count/ml)	Amount of H_2S, mg/l	Bactericidal effect, Z-%
N-1/ Oleic acid and N^1-(2-aminoethyl) ethane-1,2-diamine	50	-	-	100
	100	-	-	100
	200	-	-	100
N-2/ Oleic acid and polyethylenepolyamine	50	-	-	100
	100	-	-	100
	200	-	-	100
Control-I amount of H_2S in medium without SRB	24 mg/l			
Control-II amount of H_2S in medium with SRB	375 mg/l			
Control-III number of bacteria in nutrient medium	10^8 cell count /ml			

The tested surfactants have a 100% bactericidal effect in all three concentrations, completely stopping the life activity of sulfate-reducing bacteria.

CONCLUSIONS

1. New Gemini-type surfactants based on C_{12}-C_{18} fatty acids ethane-1,2-diamine, N^1-(2-aminoethyl)ethane-1,2-diamine and polyethylenepolyamine were synthesized, their structure and composition were confirmed by various analytical methods, important physico-chemical parameters were calculated, petrocollecting and petrodispersing properties were studied.
2. As the number of nitrogen atoms in the spacer increases in surfactants obtained from the synthesis of dodecanoic acid with

amines, a decrease in the CMC point is observed with increasing hydrophilicity. The highest petrocollecting coefficient (K=25.5) can be seen in the compound obtained from the synthesis of dodecanoic acid with both N^1-(2-aminoethyl)ethane-1,2-diamine and polyethylenepolyamine [10, 17, 21-23, 26, 29].

3. As the number of nitrogen atoms in the final product increases in the surfactants obtained from the synthesis of tetradecanoic acid with amines, a decrease in both CMC and surface tension (from 52 mN/m to 26.38 mN/m) is observed with increasing hydrophilicity. The salt of tetradecanoic acid with ethane-1,2-diamine and N1-(2-aminoethyl)ethane-1,2-diamine has mainly petrocollecting properties, while polyethylenepolyamine-based surfactant has both petrocollecting and petrodispersing properties [2, 4, 6, 7-8, 9, 24].
4. From surfactants obtained from the synthesis of oleic acid with ethylenedamine, N^1-2-aminoethyl) ethane-1,2-diamine and polyethylenepolyamine, both the surface tension (25,925 mN/m) and the CMC value at the air-water interface are the smallest (1.12 mol/L) belongs to the synthesis with N^1-2-aminoethyl) ethane-1,2-diamine. Surfactants obtained from the synthesis of oleic acid with N^1-(2-aminoethyl)ethane-1,2-diamine and polyethylenepolyamine show 100% bactericidal properties against SRB. An increase in the hydrophilicity of the spacer leads to a negative increase in both micelle formation and adsorption Gibbs free energy values [1, 3, 5, 19, 20].
5. Comparing the Gemini-type compounds obtained from the reaction of C_{18} saturated and unsaturated fatty acids with amines, it can be noted that unsaturated fatty acid (oleic) is superiror than saturated (octadecane) fatty acid in all properties [15-16, 20].
6. The smallest surface tension value (γ_{CMC}=30 mN/m) of the surfactants obtained from the reaction of ethane-1,2-diamine with various C_{12}-C_{18} fatty acids is observed in the synthesis of dodecanoic acid. Increasing hydrophobicity with alkyl chain elongation lowers the surface activity of surfactant [12, 15, 18, 30].
7. In the reaction products of N^1-(2-aminoethyl)ethane-1,2-diamine with C_{12}-C_{18} saturated fatty acids, the length of the alkyl chain and

thus the hydrophobicity of the molecule increase mainly in the surface tension values (from 23.24 mN/m to 36.58 mN/m) is observed. In all compounds, the value of ΔGad is more negative than the value of ΔGmic, and the value of both parameters increases towards C_{18} (-37.81 kJ/mol and -41.32 kJ/mol) [6, 7, 14, 27-28].

8. As the number of carbon atoms in the molecule increases in the reaction products of polyethylenepolyamine with C_{12}-C_{18} saturated fatty acids, both surface tension and CMC value increase with the increase of hydrophobicity of the molecule [11, 13, 25].

The following scientific works on the dissertation materials have been published:

1. Asadov, Z.H., Nabiyeva, H.T. Synthesis and study of surface-active salts based on ethylene diamine and oleic acid // I International Scientific Conferences of Students and Young Researchers, -Baku: - 06-16 April, - 2020, - p. 19-20.
2. Nabiyeva, H.T. Synthesis and study of surface-active salt based on ethylenediamine and myristic acid // Second International Scientific Conference of Young Scientists and Specialists Multidisciplinary approaches in solving modern problems of fundamental and applied sciences, - Baku: - 03-06 March, - 2020, - p. 351.
3. Asadov, Z.H, Nabiyeva, H.T. Synthesis and study of new surfactants based on ethylene diamine and oleic acid // Magyar Tudományos Journal, - 2020. V. 39, - p. 14-19.
4. Asadov, Z.H., Nabiyeva, H.T., Zarbaliyeva, I.A., Huseynova, S. Synthesis and study of surface-active salts based on ethylene diamine and myristic acid for removing thin petroleum layers from water surface // 1st International Scientific and Practical Internet Conferences, Ways of science development in modern crisis conditions, - Dnipro: - 28-29 May, - 2020, - p. 29-31.
5. Zarbaliyeva, I., Nabiyeva, H., Isayev, J., Gahramanov, F. Synthesis and study of new surfactants based on oleic acid and diethylene triamine // The 2nd International Scientific

Conferences of students and young researchers dedicated to the 98th anniversary of the National Leader of Azerbaijan Heydar Aliyev, - Baku: - 2021, - p. 204-206.
6. Zarbaliyeva, I.A., Nabiyeva, H.T., Alimova, A.N. Synthesis and study surface tension of new surfactants based on myristic acid and diethylenetriamine // International Scientific Conference, - Ganja: - 06-07 May, - 2021, - p. 97-99.
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