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COMPARATIVE ANALYSIS OF NEW MONOMER AND GEMINI SURFACTANTS SYNTHESIZED ON THE BASIS OF DODECANOIC ACID AND ETHANE-1,2-DIAMINE DEPENDING ON THEIR MOLAR RATIO

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Abstract: *This article includes thorough information on the synthesis process and physicochemical properties of the products generated from the reaction of dodecanoic acid and ethane-1,2-diamine IR spectroscopy was used to confirm the structure and composition of the reaction products. Several parameters of the obtained surfactants were distinguished, including surface tension, electrical conductivity, and petro-dispersing parameters. Comparative analysis has been given based on the properties of the mono and gemini surfactant. At last, final suggestions were made, taking into account the salts' applicable qualities*

Keywords: *dodecanoic acid, ethane-1,2-diamine, surfactant, surface-activity*
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Introduction

Gemini-type surfactants are made up of two hydrophilic and two hydrophobic groups that are linked together by a cavity. A symmetrical structure is formed when both hydrophilic and hydrophobic groups are the same [1]. Bunton and his colleagues began researching bis-quaternary ammonium salts-gemini surfactants in 1974 [2]. They discussed the synthetic strategy as well as the kinetics of nucleophilic events. Years later, Devinsky synthesized and examined the surfactant and micelle-forming properties of a wide range of bis-quaternary ammonium salts [3]. Surfactants' ability to adsorb at the contact is one of their most important features. Surface tension is reduced by the chemical composition of these molecules [4-5]. Pure water has a surface tension of 72 mN.m [6]. Surface tension is reduced by a surfactant by replacing molecules in the solution with surfactant molecules. Surfactants' amphilic characteristic is the primary reason for their extensive variety of practical applications. This essential property of amphiphiles serves as the foundation for their extensive range of practical applications. Surfactants are used in detergents [7], personal hygiene products [8], as paint and coating additives [9], biocides [10-13], organic synthesis [14-15], pharmaceuticals [16-17], textiles and leather production [7, 18, 19], agrochemicals [20], fibers [21-23], plastics [24], food industry [25-26], increasing oil recovery [27-29], environmental protection (oil residue cleanup [30-31], and explosives [32]. Surfactants are also employed to substitute existing solvents in order to lessen hazards and environmental effect [33]. High-performance surfactants are in high demand due to their diverse variety of application fields. As a result, novel surfactants are effectively designed and created [34-37]. Gemini surfactants have lately piqued the curiosity of researchers due to their higher surface active components than traditional surfactants. Surfactants with a gemini (dimer) structure have more than one hydrophobic tail and one hydrophilic head group. They have a lower CMC (critical micelle concentration) and better water solubility than typical surfactants, as well as a lower surface tension [37-41]. The goal of this project is to synthesize both monosurfactant and gemini surfactants and investigate their characteristics.

The aim of the work is the synthesis of new, mono structured and dimer "gemini" type surfactants based on fatty acids and polyamines, determination of their main physical and chemical parameters, including surface activity parameters, their oil collection, which allows removing ecologically harmful thin oil layers on the water surface, as well as the study of the petro-dispersing ability, as well as other useful and application-oriented properties, and the offer of appropriate recommendations.

Experimental part

Ethane-1,2-diamine is a product of Aldrich. It has a molar mass of 60.1 gmol-1 and is a transparent liquid. Ammonia has a smell. Its refractive index is 1.4565, its boiling point is 117-119 °C, and its density is 0.89 $gml^{-1.}$

At a molar mass of 200 g/mol, dodecanoic acid is formed as a white, crystalline powder and has a faint bay oil flavor. It has a 0.883 g/ml density The melting point is 44.2 °C and the boiling point is 298.9 °C. Dodecanoic acid and ethane-1,2-diamine were utilized as reactants in this study. These reactants were used to create two salts. Initially, an equimolar ratio of dodecanoic acid and ethane-1,2-diamine was used in the process. Another product was created by combining the same acid and amine in a 2:1 ratio. The reactions took about 9-10 hours to complete at 70-80°C.

2.1 Tension on the surface measurements: Surface tension was measured using a Du Nouy ring KSV Sigma 702 tensiometer (Israel). The sample was placed in the glass cell, a Pt wire ring was inserted into the sample solution and gradually drawn through the liquid-air interface. The average value of surface tension was calculated using three readings at three-minute intervals. In-between experiments, the Pt wire ring was rinsed with water and flamed with a Bunsen burner.

2.2 Electro-onductometric estimations:

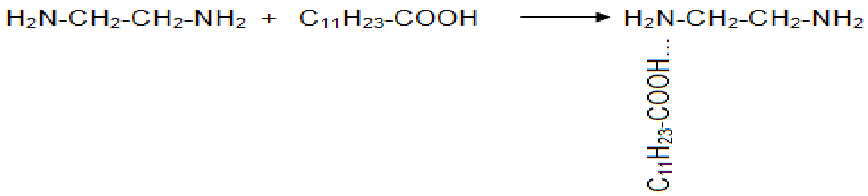
The «ANION 4120» conductometer was utilized to degree the particular electroconductivity of the surfactant arrangements (Russia). The estimation run is 104 S/m-10 S/m, the estimation temperature run is -100 C and the relative blunder is less than 2%. Each salt's electroconductivity was measured and after 0.025 g was broken down in 25 ml of distillated water.

2.3 Ponder of petroleum-collecting and petroleum-dispersing properties:

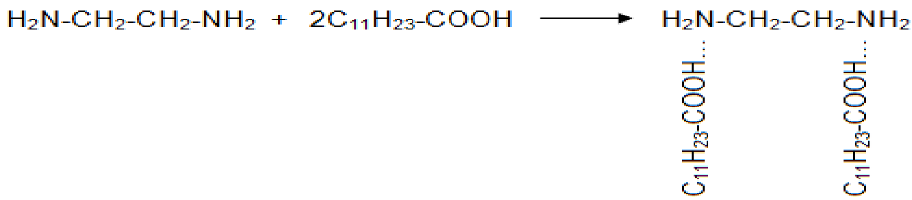
The surfactants' petro-dispersing capacity was decided utilizing the well-known method depicted in [4]. In a Petri dish, 40 mL of water is set. In this work, 1 ml of unrefined oil (trademark "Pirrallahi") is spread over the water (thickness of the film is 0.17 mm). The surfactant (or its 5% wt. arrangement) is at that point included to the film from the side. At standard interims, the surface zone of the introductory oil film and the current zones of the shaped oil slicks are measured. The coefficient $K_{d,}$ which indicates the degree of surface cleaning (in%), is calculated.

Results and discussion

Schemes of the reactions are described below:



Salt of dodecanoic acid with 1,2 ethanediamine (1:1)



Salt of dodecanoic acid with 1,2 ethanediamine (2:1)

The first reaction scheme is the reaction product between dodecanoic acid and 1,2 ethanediamine in an equimolar ratio, and the second reaction is a surfactant obtained from two moles of acid per one mole amine.

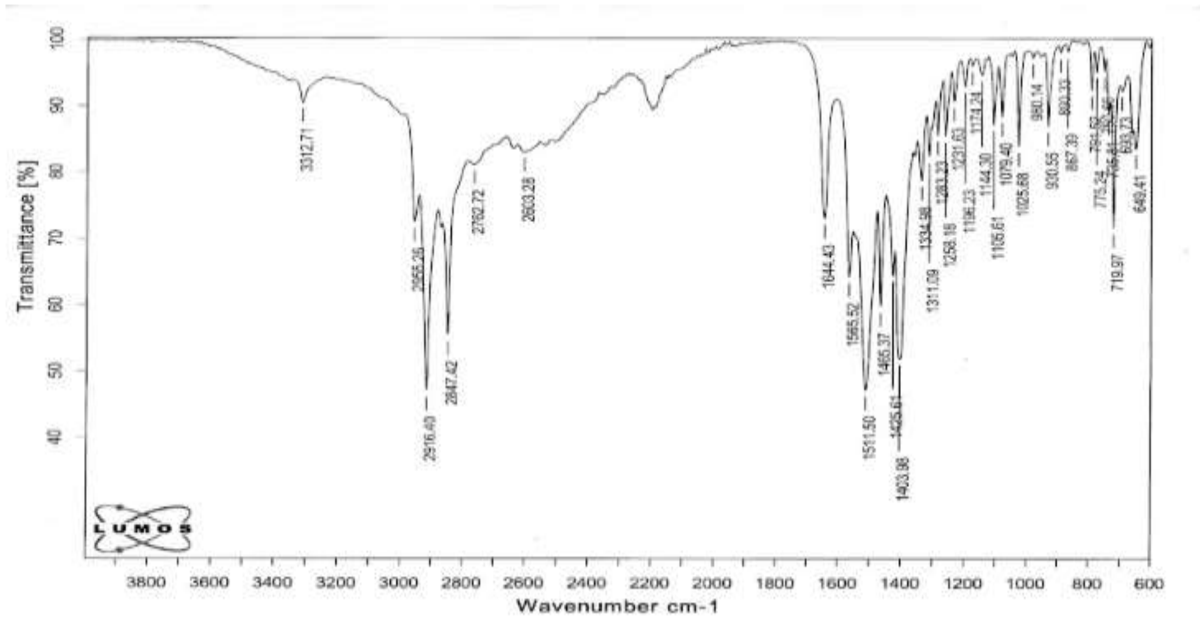


Fig. 1. IR- spectroscopy of salt of dodecanoic acid with 1,2 ethanediamine (1:1)

The IR- spectrum of Fig. 1 of the sample has the following absorption bands: bending (1425, 1465 cm^{-1}) and stretching (2847, 2916, 2955 cm^{-1}) vibrations of the C–H bond of CH_3 and CH_2 groups; stretching (1025, 1079, 1105 cm^{-1}) vibrations of the C–O bond of alcohol (weak bands); stretching (1403, 1511, 1565 cm^{-1}) vibrations of the COO group; stretching (3312 cm^{-1}) vibrations of the H–N bond; ammonium band (2603, 2762 cm^{-1}).

The IR- spectrum of Fig. 2 of the sample has the following absorption bands: bending (1327.24,1305.48 cm^{-1}) and valence (2915.12, 2871.49, 2848.75 cm^{-1}) vibrations of the C–H bond of CH_3 and CH_2 groups; stretching (1180.77, 1105.10, 1079.70 cm^{-1}) vibrations of the C–O bond of alcohol (weak bands); stretching (1553.76, 1517.31, 1467.21, 1404.51 cm^{-1}) vibrations of the COO group; stretching (3388.88 cm^{-1}) vibrations of the H–N bond;

ammonium band (2639.64 , 2537.88 cm^{-1}). The surface-activity parameters of the synthesized components are measured in water-air border using the different concentrated aqueous solutions at room temperature (t=23^{o}C). Surface tension at this border is 72 mN/m without reagent. Also, surface tension of the synthesized mono surfactant is 50.32, 46.13, 43.42, 26.87 mN/m in 0.005%, 0.0075%, 0.01% and 0.075% concentrated solution respectively.

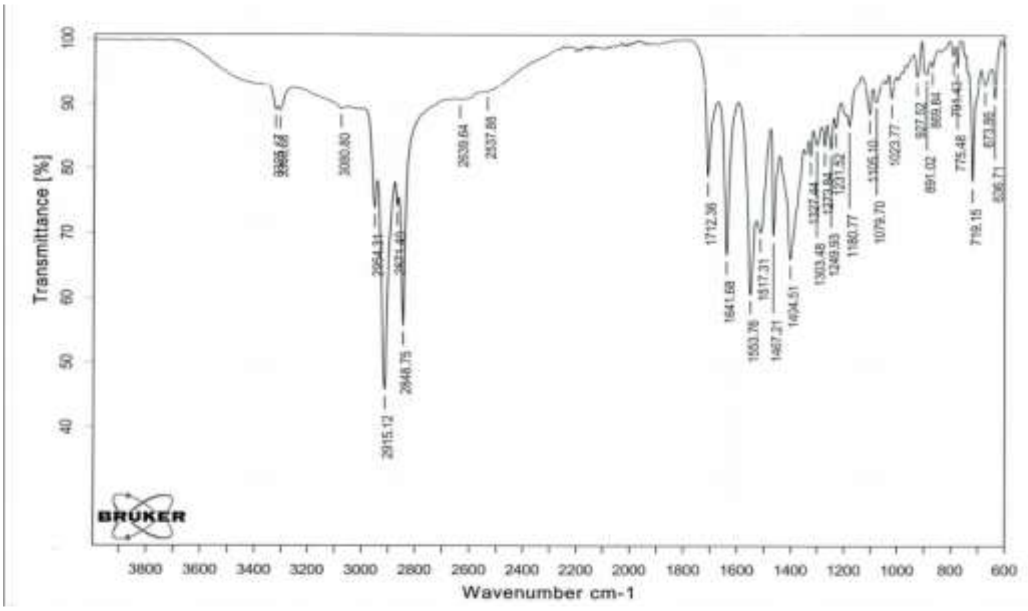


Fig. 2. IR- spectroscopy of salt of dodecanoic acid with 1,2 ethanediamine (2:1)

However, the surface tension of the gemini surfactant is 38.15, 32, 30 and 24.8 mN/m in 0.005%, 0.0075%, 0.01% and 0.075% concentrated solution respectively. When comparing the both surfactants, it can be noted that gemini surfactant shows better surface activity than its monomer one in the same concentration. Using the measured values, isotherms of the surfactants were sketched in Fig.3. The tangent line was sketched through the point after the surface tension starts being stabilized which is the critical micelle concentration and the specific parameters owned to surfactants were calculated (Table 1).

Surface tension of the surfactants as a function of concentration was determined and shown in Fig.3.

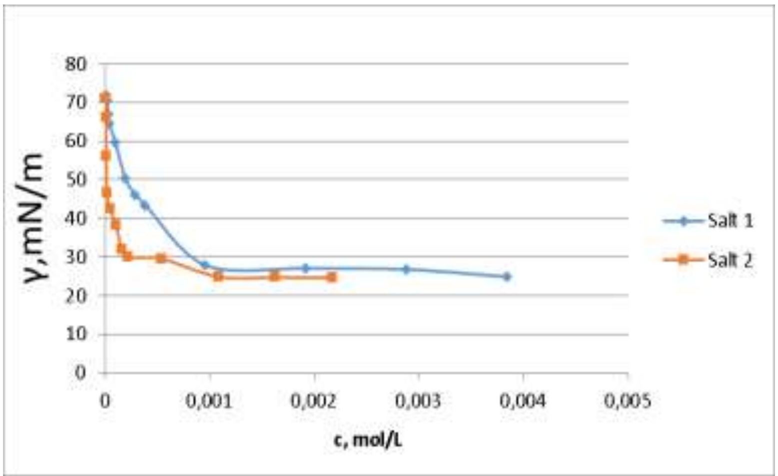


Fig. 3. Surface tension versus concentration

Surface activity parameters are calculated and shown in the Table 1. CMC, γ_{CMC}, surface pressure (π_{CMC}), C_{20} (concentration for decrement of γ by 20 mN/m), adsorption efficiency ($pC_{20} = -logC_{20}$), as well as CMC/C_{20} (interfacial activity) parameters of obtained surfactants were determined. Maximum surface excess concentration (Γ_{max}) and minimum area of one surfactant molecule at water-air border (A_{min}) were determined using the equation below.

$$\Gamma_{max} = \frac{1}{n * R * T} * \lim_{c \to CMC} \frac{d\gamma}{dlnc}$$

$$A_{min} = \frac{1}{N_A * \Gamma_{max}}$$

Table 1. Surface activity parameters of surfactants

Surfactant	CMC*10^4 (mol/L)	γ_{CMC} (mN,m)	π_{CMC} (mN,m)	C_{20}*10^4 (mol/L)	pC_{20}	CMC/ C_{20}	Γ_{max}*10^{10} (mol/cm^2)	A_{min}*10^2 (nm^2)
Salt of dodecanoic acid with 1,2 ethanediamine 1:1	9.6	27.93	43.71	1.92	3.72	5	1.92	86.4
Salt of dodecanoic acid with 1,2 ethanediamine 2:1	2.2	28.34	43.03	0.14	4.83	14.5	1.58	104.9

From the above Table 1 it can be seen that the value of the minimum concentration of CMC for the salt of dodecanoic acid with 1.2 ethanediamine (1:1) is 9.6 * 104 mol/l, and for the salt of dodecanoic acid with 1.2 ethanediamine (2:1)–2.2*104 mol/l, therefore, micellization occurs at low concentrations. Besides, it can be added that the minimum surface area for one molecule of gemini surfactant (Amin = 104.9 mm^2) is greater than its value for mono surfactant (Amin = 86.4 mm^2). Since Table 1 shows that the salt of dodecanoic acid with 1,2 ethanediamine (2:1), which is a gemini type surfactant, has a higher surface activity.

The electrical conductivity of the surfactants depending on concentration values is described in Fig. 4.

The ratio of the slopes of best fitted linear line before (Salt 1) and after (Salt 2) CMC values were found and thermodynamic parameters as Gibbs free energy of micellization (ΔGmic) and Gibbs free energy of adsorption (ΔG_{ad}) were identified using equations given below:

$$\Delta G_{mic} = (2 - \alpha) * R * T * \ln(CMC)$$

$$\Delta G_{ad} = \Delta G_{mic} - 0.6023 * \pi_{CMC} * A_{CMC}$$

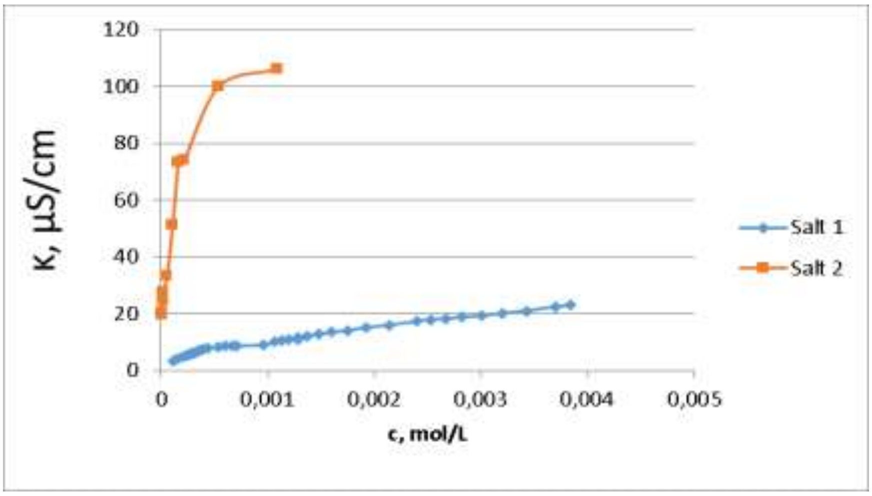


Fig. 4. Electrical conductivity versus concentration

Table 2. Electro thermoconductivity parameters

Surfactant	α	β	ΔG_{mic}, kJ/mol	ΔG_{ad}, kJ/mol
Salt of dodecanoic acid with 1,2 ethanediamine (1:1)	0.52	0.48	-25.72	-27.48
Salt of dodecanoic acid with 1,2 ethanediamine (2:1)	0.9	0.1	-22.77	-25.48

It can be seem from Table 2, Gibbs' free energy of micellization (ΔG_{mic}) and Gibbs' free energy of adsorption (ΔG_{ad}) for both salts are negative. It means that these two processes are spontaneous. However, there is a greater increase in the negative value of ΔG^{o}_{ads} as compared to those of micellization. This suggests the tendency of the molecules to be adsorbed at the interface. Using the method described in [11], petrocollecting and petrodispersing parameters of the salts were measured and shown in Table 3.

Table 3. Petrocollecting and petrodispering parameters of the synthesized salts

Ratio	State of surfactant	Sea water		Tap water		Distilled water	
		K, K_d	Duration-τ, hours	K, K_d	Duration-τ, hours	K, K_d	Duration-τ, hours
1:1	*5 wt. % aqueous solution*	1.92	0-1	3.5	0-1	7.22	0-1
		1.92	1-25	17.4	1-25	3	1-25
		1.92	25-49	17.4	25-49	79.3%	25-49
		18%	49-169	1.96	49-169	89.3%	49-169
	5 wt. % ethanolic solution	97%	0-1	94%	0-1	12	0-1
		50%	1-25	94%	1-25	3	1-25
		50%	25-49	1.95	25-49	1.95	25-49
		16%	49-169	1.9	49-169	1.96	49-169
	Solid	96%	0-1	98%	0-1	83.6%	0-1
		95%	1-25	95%	1-25	89.3%	1-25
		95%	25-49	97.3%	25-49	97.3%	25-49
		95%	49-169	97.3%	49-169	97.3%	49-169
	5 wt. %	2.12	0-1	1.8	0-1	10	0-1

2:1	*aqueous solution*	1.67	1-25	1.67	1-25	3	1-25
		87%	25-49	1.56	25-49	1.68	25-49
		12	49-169	1.56	49-169	1.68	73-265
	5 wt. % ethanolic solution	5	0-1	5	0-1	19.15	0-1
		1	1-25	1.95	1-25	3	1-217
		9	25-49	1.56	25-49	1.36	
		9	49-169	1.56	49-169	1.36	
	Solid	50%	0-1	3	0-1	24.4	0-1
		9.07	1-25	4.79	1-25	97%	1-25
		13	25-49	12.8	25-49	97.3%	25-49
		13	49-169	12.8	49-169	97.3%	49-169

According to the Table 3, the salt of dodecanoic acid with 1,2 ethanediamine (1:1) shows both petrocollecting and petrodispersing properties. Specially, in solid phase it totally gives petrodispering result. However, salt of dodecanoic acid with 1,2 ethanediamine (2:1) shows petrocollecting property much more often than salt of dodecanoic acid with 1,2 ethanediamine (1:1). The highest petrodispersing property of salt of dodecanoic acid with 1,2 ethanediamine (1:1) in sea water which is 97%, occurred when treated with 5 wt % ethanolic solution. Similarly, the highest petrocollecting number for salt of dodecanoic acid with 1,2 ethanediamine (2:1) in sea water is seen when treated with solid phase.

As a whole, both surfactants can be recommended using as cleaning agent with due regard for their physical and chemical properties.

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DODEKAN TURŞUSU VƏ 1,2 ETANDİAMİN ƏSASINDA YENİ MONOMER VƏ “GEMİNİ” SƏTHİ-AKTİV MADDƏLƏRİN SİNTEZİ VƏ ONLARIN MOL NİSBƏTİNDƏN ASILI OLARAQ MÜQAYİSƏLİ TƏHLİLİ

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Xülasə: Məqalədə dodekanoik turşunun 1,2-etandiamin ilə reaksiyası nəticəsində yaranan məhsulların sintez prosesi və fiziki-kimyəvi xassələri haqqında məlumat verilmişdir. Reaksiya məhsullarının strukturunu və tərkibini təsdiqləmək üçün İQ- spektroskopiya üsulundan istifadə edilmişdir. Alınan səthi-aktiv maddələrin bir sıra parametrləri, o cümlədən səthi gərilmə, elektrik keçiriciliyi, neft disperslämə və neftyığma parametrləri müəyyən edilmişdir. Mono- və "gemini" səthi-aktiv maddələrin xassələrinin müqayisəli təhlili verilmişdir. Nəticədə duzların tətbiqi xüsusiyyətləri nəzərə alınmaqla yekun təkliflər verilmişdir.
Açar sözlər: dodekan turşusu, 1,2 etandiamin, səthi aktiv maddə, səthi gərilmə, neftyığma

СРАВНИТЕЛЬНЫЙ АНАЛИЗ НОВЫХ МОНОМЕРНЫХ И «ГЕМИНИ» ПОВЕРХНОСТНО-АКТИВНЫХ ВЕЩЕСТВ (ПАВ), СИНТЕЗИРОВАННЫХ НА ОСНОВЕ ДОДЕКАНОВОЙ КИСЛОТЫ И 1,2 ЭТАНДИАМИНА, В ЗАВИСИМОСТИ ОТ ИХ МОЛЯРНОГО СООТНОШЕНИЯ

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Аннотация: Статья содержит информацию о процессе синтеза и физико-химических свойствах продуктов, образующихся в результате реакции додекановой кислоты с 1,2 этандиамином. Структура и состав продуктов реакции исследованы ИК-спектральным методом анализа. Определены некоторые параметры полученных ПАВ, в том числе поверхностное натяжение, электропроводность, а также нефтедиспергирующие и нефтесобирающие свойства. Дан сравнительный анализ свойств моно- и «гемини» ПАВ. В итоге были сделаны окончательные предложения с учетом применимых свойств солей.
Ключевые слова: додекановая кислота, 1,2 этандиамин, поверхностно-активное вещество, поверхностное натяжение