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ANALYZING THE EFFECT OF NUMBER OF CARBON ATOMS ON THE PROPERTIES OF THE SYNTHESIZED SURFACTANTS

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An investigation into the synthesis and characterization of surfactants produced by polyethylenepolyamine with dodecanoic, tetradecanoic and hexadecanoic and has been carried out scientifically. IR and UV spectroscopies was utilized in this study to clarify the structure and makeup of the resultant Gemini surfactants. A Tensiometer was used to measure surface tension at the air-water interface while examining several aqueous solutions that contained these Gemini surfactants. Using the same methodology, the electrical conductivity of these solutions was also determined. A number of essential surface activity characteristics were computed and compared in order to determine the relationship between surface tension and concentration as well as electrical conductivity and concentration. These characteristics include the minimum surface area for a single surfactant molecule, the maximal adsorption (Γmax), the surface pressure (πcmc), and the Critical Micelle Concentration (CMC). Besides that, from the dependence of electrical conductivity through concentration change in Gibbs free energy of micellization and adsorbtion has been determined. Finally, petrocollecting and petrodispersing properties of the surfactants have been measured.

KEY WORDS: *dodecanoic acid, tetradodecanoic acid, hexadecanoic acid, polyethylene-polyamine*

КӨМІРТЕК АТОМДАРЫНЫҢ СИНТЕЗДЕНГЕН БАЗ-БАЗЫҚ ЗАТТАРДЫҢ ҚАСИЕТТЕРІНЕ ӘСЕРІН ТАЛДАУ

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МҰНАЙ-ХИМИЯЛЫҚ ПРОЦЕСТЕРІ ИНСТИТУТЫ

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Полиэтиленполиаминнің додекандық, тетрадекандық және гексадекандықпен өндірілетін беттік белсенді заттардың синтезі мен сипаттамасын зерттеу және ғылыми түрде жүргізілді. Бұл зерттеуде алынған Gemini беттік белсенді заттардың құрылымы мен құрамын нақтылау үшін ИҚ және УК спектроскопиялары пайдаланылды. Осы Gemini беттік белсенді заттары бар бірнеше сулы ерітінділерді зерттеу кезінде ауа-су интерфейсіндегі беттік керілуді өлшеу үшін тенсиометр пайдаланылды. Сол әдістемені қолдана отырып, бұл ерітінділердің электр өткізгіштігі де анықталды. Беттік керілу мен концентрация, сондай-ақ электр өткізгіштік пен концентрация арасындағы байланысты анықтау үшін бірқатар маңызды беттік белсенділік сипаттамалары есептелді және салыстырылды. Бұл сипаттамаларға бір беттік белсенді зат молекуласының минималды бетінің ауданы, максималды адсорбция (Гmax), беттік қысым (πcmc) және критикалық мицелла концентрациясы (СМС) жатады. Сонымен қатар, Гиббстің бос энергиясының концентрациясының өзгеруіне байланысты электр өткізгіштігінің мицеллизация мен адсорбцияға тәуелділігі анықталды. Соңында беттік белсенді заттардың мұнай жинау және мұнай тарату қасиеттері өлшенді.

ТҮЙІНДІ СӨЗДЕР: *додекан қышқылы, тетрадодекан қышқылы, гексадекан қышқылы, полиэтиленполиамин*

АНАЛИЗ ВЛИЯНИЯ ЧИСЛА АТОМОВ УГЛЕРОДА НА СВОЙСТВА СИНТЕЗИРОВАННЫХ ПАВ

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Проведены научные исследования синтеза и характеристики поверхностно-активных веществ, полученных на основеполиэтиленполиамина с додекановой, тетрадекановой и гексадекановой кислотами. В этом исследовании с использованием ИК- и УФ-спектроскопиибыла проведена идентификация для выяснения структуры и состава полученных «гемини» поверхностно-активных веществ. Тензиометр использовался для измерения поверхностного натяжения на границе раздела воздух-вода при исследовании различных водных растворов, содержащих поверхностно-активные вещества «гемини» типа. По этой же методике была определена и электропроводность этих растворов. Ряд важных показателей поверхностной активности был вычислен и сравнен, чтобы определить взаимосвязь между поверхностным натяжением и концентрацией, а также электропроводностью и концентрацией. Эти характеристики включают минимальную площадь поверхности для одной молекулы поверхностно-активного вещества, максимальную адсорбцию (Гmax), поверхностное давление (πккм) и критическую концентрацию мицеллообразование (ККМ). Кроме того, по зависимости электропроводности от изменения концентрации определена свободная энергия Гиббса мицеллообразования и адсорбции. В заключении, были исследованы нефтесобирающие и нефтедиспергирующие свойства поверхностно-активных веществ.

КЛЮЧЕВЫЕ СЛОВА: *додекановая кислота, тетрадодекановая кислота, гексадекановая кислота, полиэтиленполиамин.*

Introduction. In modern times, surface-active substances are widely used in all fields of national economy, medicine, science and everyday life. They are diphilic in nature and consist of hydrophilic and hydrophobic parts [1]. For this reason, surfactants are adsorbed at the boundary between two phases and exhibit different properties. In the proposed work, it is intended to obtain new surfactants based on fatty acids, that is, higher aliphatic monobasic carboxylic acids and polyethylenepolyamine. 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 the surfactants to be purchased will be significantly safer for the environment. The use of polymers makes it possible to synthesize surfactants with a traditional structure, i.e. one hydrocarbon chain, and "gemini" surfactants, i.e. surfactants with two or more hydrocarbon chains. "Gemini" type surfactants have recently been the focus of experts' attention [2-5]. To differentiate Gemini surfactants from traditional types, certain parameters must be considered. These include hydrophilic-lipophilic balance, critical micelle concentration (CMC), Kraft temperature, and molecular packing parameter. CMC represents the concentration at which micelle formation occurs [6]. Gemini surfactants typically exhibit CMC values 10-100 times smaller than those of traditional types [7].

The amphiphilic nature of surfactants is pivotal to their wide array of practical applications. These applications encompass detergents [8], personal hygiene products [9], additives in paints and coatings [10], biocides, agrochemicals, the food industry, oil recovery [11] and environmental protection (utilized in oil residue treatment and explosives [12-15].

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, lowering the surface tension at the boundary between phases, and the possibility of obtaining lower costs of surfactants [16-17]. As dimer (Gemini) surfactants show better properties than their traditional ones, application areas of them are more such as enhanced oil recovery, remediation of oil spill, demulsifying agent, detergents and self cleaning agents, corrosion inhibitors and so on [18].

Experimental part

Starting substances and their characteristics

Dodecanoic acid has a molar mass of 200 g/mol, a density of 0.883 g/ml, and a white, crystalline powder with a faint smell similar to bay oil. Its boiling point is 298.9 °C, while its melting point is 44.2 °C.

Tetradodecanoic acid is a fatty acid that is used in the beauty business for a variety of reasons. It may be found in nutmeg, butter fat, coconut oil, palm oil, and spermacetin, which is the oil from the sperm whale. These include serving as an emulsifier, surfactant, opacifying agent, cleaning agent, and fragrance ingredient.

The Moscow-based Komponent-Reactant Joint Stock Company produces hexadecanoic acid, a white, crystalline material having a molar mass of 256.42 g/mol. It has a solidification point of 62.9 °C and a density of 0.853 g/cm³ at 62 °.

A viscous, very alkaline, reddish-brown or tan liquid called polyetilenpolyamine. Its density is 1.025 g/cm³ at 20°C, and its melting point is -26 °C. Its boiling point is greater than 190 °C (1.33 kPa). It is soluble in ethanol and water but insoluble in benzene and ether. Salt is created when it reacts with acid, and it quickly takes up moisture and carbon dioxide from the atmosphere.

Methodology for studying the structure and composition of synthesized complexes

IR spectra were recorded by an ALPHA FTIR spectrometer (Bruker,USA) using KBr tablets.

Surface tension Measurement: An Israeli Du Nouy ring KSV Sigma 702 tensiometer was used to measure surface tension. A Pt wire ring was dragged through the liquid-air contact and inserted into the solution to put the sample in the glass cell. Three measurements, taken every three minutes, were used to determine the average surface tension. The Pt wire ring was flame-lit using a Bunsen burner and rinsed with water in between trials.

Electroconductivity Measurement: The electroconductivity of surfactant arrangements was measured by electroconductometric estimations using the Russian-manufactured "ANION 4120" conductometer. With an estimation temperature range of -100°C and a relative error of less than 2%, the estimation covered a range of 104 S/m to 10 S/m. After dissolving 0.025 g of each salt in 25 ml of distilled water, the electroconductivity of each salt was measured.

Petrocollecting and petrodispersing properties: The established method was used to evaluate the properties of petrodispersing and petrocollecting. One milliliter of unrefined oil (brand name "Pirrallahi") was spread over 40 milliliters of water in a Petri dish, with a 0.17 mm film thickness. Next, the surfactant was added to the film from the side in a 5% weight solution. The petrocollecting coefficient (K) was computed by dividing the initial oil film's surface area by its surface area before and after surfactant treatment. The petrodispersing percentage (Kd), which represents the level of surface cleaning, was then computed.

Results and discussions

Reactants were added at equimolar ratios, and the process of synthesizing salts with these reactants took nine to ten hours.

Below reaction mechanisms are shown:

$$H_2N\text{-}(CH_2)_2\text{-}NH\text{-}((CH_2)_2\text{-}NH))_n\text{-}(CH_2)_2\text{-}NH_2 \quad + \quad 2C_{11}H_{23}\text{-}COOH \longrightarrow$$

$$\longrightarrow C_{11}H_{23}\text{-}COO\overset{-}{H}\ \overset{+}{N}H_2\text{-}(CH_2)_2\text{-}NH\text{-}((CH_2)_2\text{-}NH))_n\text{-}(CH_2)_2\text{-}H_2\overset{+}{N}\ \overset{-}{H}OOC\text{-}C_{11}H_{23}$$

Figure 1 – **The reaction scheme of dodecanoic acid with polyetylenepolyamine**

$$H_2N\text{-}(CH_2)_2\text{-}NH\text{-}((CH_2)_2\text{-}NH))_n\text{-}(CH_2)_2\text{-}NH_2 \quad + \quad 2C_{13}H_{27}\text{-}COOH \longrightarrow$$

$$\longrightarrow C_{13}H_{27}\text{-}COO\overset{-}{H}\ \overset{+}{N}H_2\text{-}(CH_2)_2\text{-}NH\text{-}((CH_2)_2\text{-}NH))_n\text{-}(CH_2)_2\text{-}H_2\overset{+}{N}\ \overset{-}{H}OOC\text{-}C_{13}H_{27}$$

Figure 2 – **The reaction scheme of tetradodecanoic acid with polyetylenepolyamine**

$$H_2N\text{-}(CH_2)_2\text{-}NH\text{-}((CH_2)_2\text{-}NH))_n\text{-}(CH_2)_2\text{-}NH_2 \quad + \quad 2C_{15}H_{31}\text{-}COOH \longrightarrow$$

$$\longrightarrow C_{15}H_{31}\text{-}COO\overset{-}{H}\ \overset{+}{N}H_2\text{-}(CH_2)_2\text{-}NH\text{-}((CH_2)_2\text{-}NH))_n\text{-}(CH_2)_2\text{-}H_2\overset{+}{N}\ \overset{-}{H}OOC\text{-}C_{15}H_{31}$$

Figure 3 – **The reaction scheme of hexadecanoic acid with polyetylenepolyamine**

The structure of the obtained salts has been confirmed using IR- and UV-spectroscopies and the results are described in *Fig 4-9*.

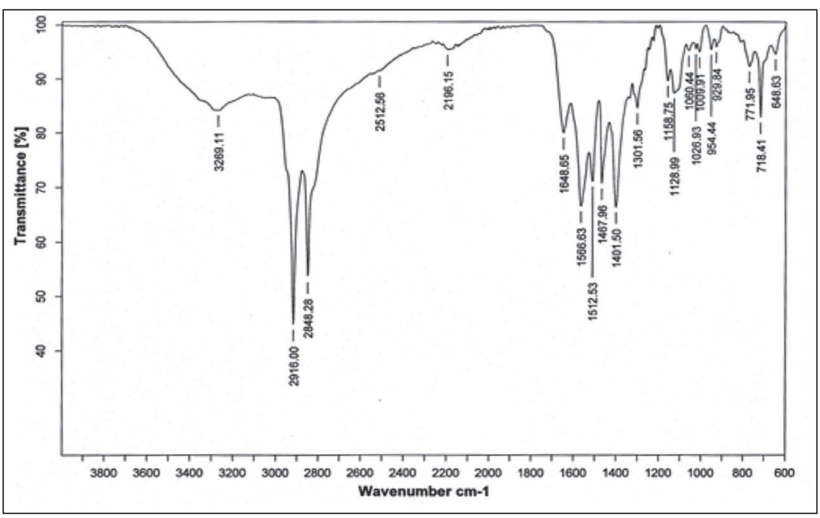

Figure 4 – **IR spectrum of the salt of dodecanoic acid with polyethylenepolyamine**

The sample's infrared (IR) spectrum, shown in *Fig. 4*, shows several unique absorption bands, such as: bends in the C-H bond at 718.41 and 1401.50 cm-^{1} in the CH3 and CH2 groups. stretching vibrations of the C-H bond at 2848.20 and 2916.00 cm-^{3} in the CH3 and CH2 groups. COO group stretching vibrations at 1401.50 and 1512.53 cm-^{1}. H–N bond stretching vibrations at 3269.11 cm-^{1}. At 2196.1 and 2512.56 cm-^{3}, there is an ammonium band.

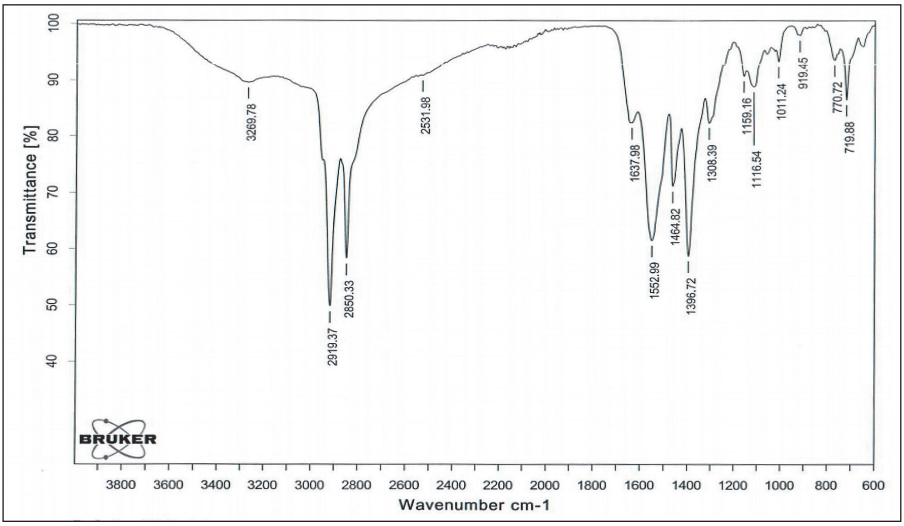

Figure 5 – **IR spectrum of the salt of tetradodecanoic acid with polyethylenepolyamine**

The sample's IR spectra, shown in *Fig. 5*, shows the following absorption bands. Bending vibrations of the C-H bond at 1464.82 cm-^{1} in the CH3 and CH2 groups. Stretching vibrations of the C-H bond at 2850.33 and 2919.37 cm-^{1} in the CH3 and CH2 groups. COO group stretching vibrations at 1396.72 and 1552.99 cm-^{3}. H-N bond stretching vibrations at 3269.78 cm-^{1}. A band of ammonium at 2531.98 cm-^{3}.

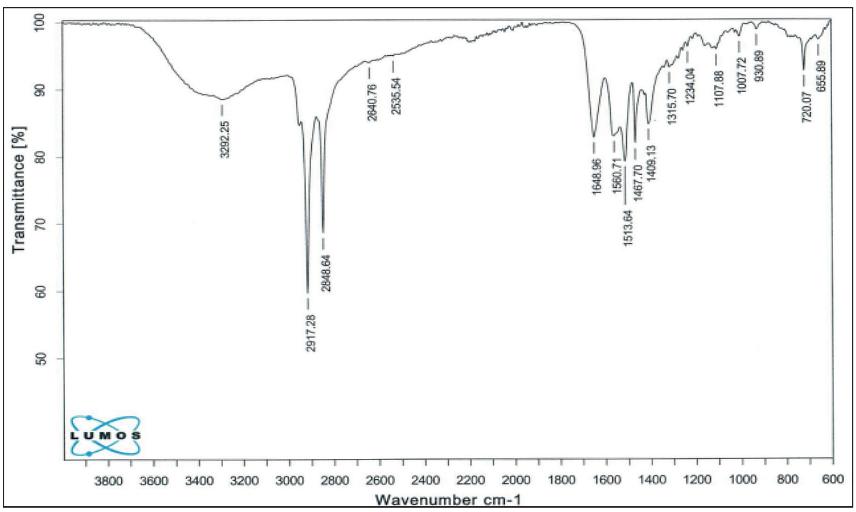

Figure 6 – **IR- spectrum of the salt of hexadecanoic acid with polyethylenepolyamine**

The sample's IR spectra, shown in *Fig. 6*, shows the following clearly visible absorption bands: Bending vibrations of the C-H bond at 1467.70 cm^{-1} in the CH3 and CH2 groups. stretching vibrations of the C-H bond at 2848.64 and 2917.28 cm^{-1} in the CH3 and CH2 groups. COO group stretching vibrations at 1315.10 and 1513.64 cm^{-3}. H-N bond stretching vibrations at 3292.25 cm^{-3}. A band of ammonium at 2535.54 cm^{-3}.

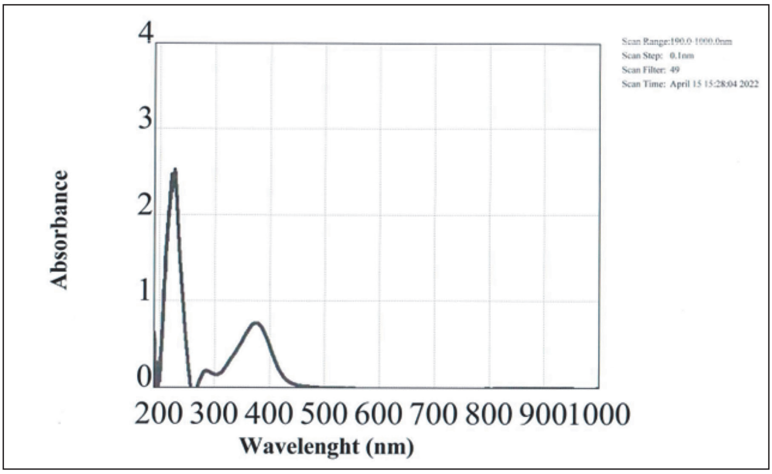

Figure 7 – **UV spectrum of the salt created by combining polyethylenepolyamine and dodecanoic acid**

As shown in *Figure 7*. At 220 nm in the UV spectrum, there is an absorption band that is thought to be related to the complex amino group.

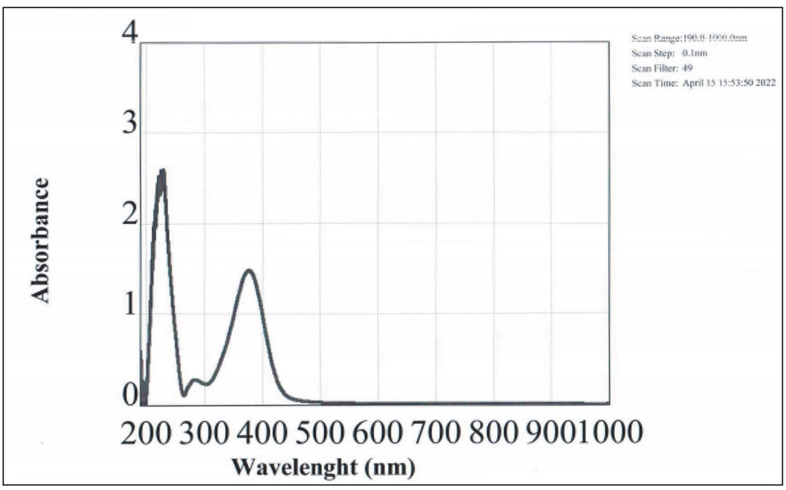

Figure 8 – **UV spectrum of the salt created by combining polyethylenepolyamine and tetradodecanoic**

The ultraviolet spectrum of the salt created by combining polyethylenepolyamine and tetradodecanoic acid is shown in *Figure 8*. *Figure 4* shows that the complex amino group is represented by an absorption band in the UV spectrum, which is located at 220 nm.

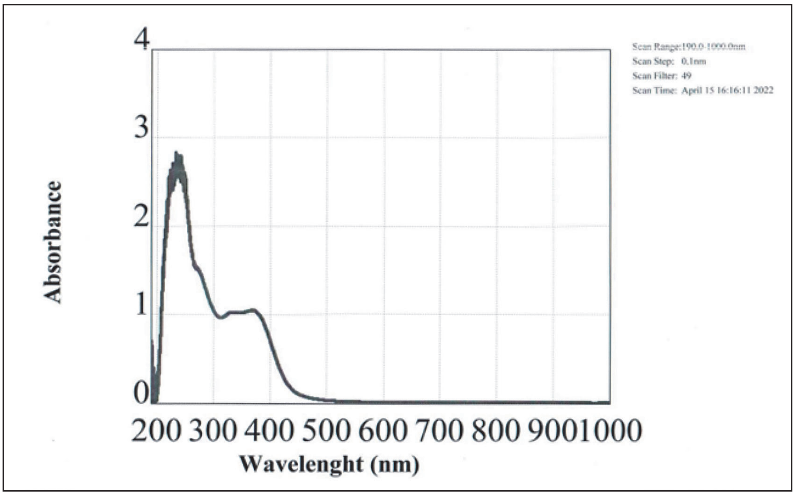

Figure 9 – **UV spectrum of the salt that is produced when polyethylenepolyamine and hexadecanoic acid**

The UV spectrum of the salt that is produced when polyethylenepolyamine and hexadecanoic acid are combined is shown in *Figure 9*. As can be seen in *Figure 4*, the complex amino group is responsible for an absorption band that is prominently visible at 220 nm in the UV spectrum.

Using the tensiometer, the surface tension of the aqueous solution of the surfactants in different concentrations was measured and using these data a graph of the dependence of the surface tension values on the thickness was constructed and the CMC point was determined for each substance from the graph. A number of surface activity parameters of substances were determined using the formulas (3.1) and (3.2).

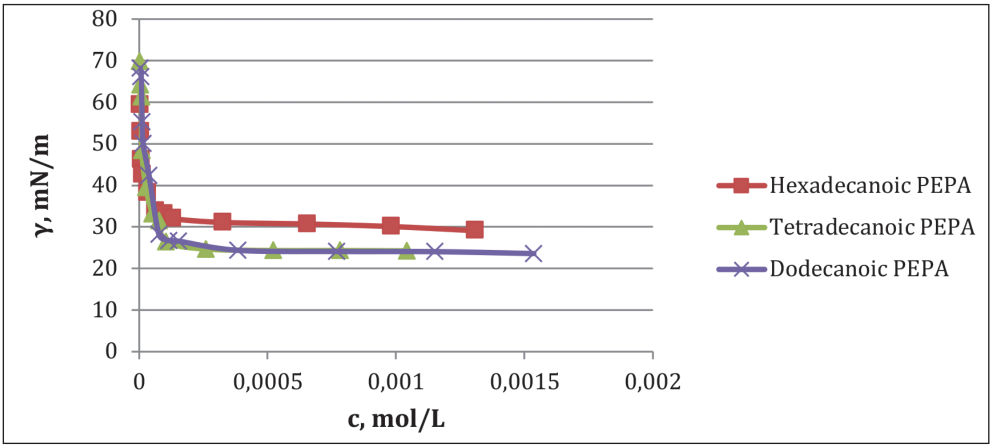

Figure 10 – **Surface tension versus concentration for the obtained salts**

$$\Gamma_{max} = \frac{1}{n*R*T} * \lim_{c \to CMC} \frac{d\gamma}{dlnc} \qquad (3.1)$$

$$A_{min} = \frac{1}{N_A * \Gamma_{max}} \qquad (3.2)$$

Here n represents of dissociated ions which is 3 for the Gemini surfactants, R is universal gas constant (8.314 J/mol*K) and T is absolute temperature. The calculated surface activity parameters using the equations above are shown in *Table 1*.

Table 1 – **Surface activity parameters of the 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}	$A_{min}*10^{10}$ (mol/cm²)	$A_{min}*10^2$ (nm²)
Dodecanoic acid PEPA	1.2	26.73	45.61	0,15	4,8	7.5	1.7	100.12
Tetradodecanoic acid PEPA	1.04	26.38	46.26	0.009	5.04	11.4	1.61	103.38
Hexadecanoic PEPA	1.3	32	39.71	0.065	5.18	20	0.99	166

Looking at the table given above, we can note that the compounds obtained from the synthesis of dodecanoic acid and tetradodecanoic acidwith polyethylene polyamine are capable of reducing the surface tension at the air-water interface at room temperature to a lower value than the other substance. In the complex obtained with hexadecanoic acid, it has a slightly higher value. At the same time, the amount of minimum concentration required to obtain the mentioned surface tension value is also higher than others. This is explained by the fact that the compound obtained with hexadecanoic acid has a larger molecular mass. A similar dependence manifests itself in the value of surface pressure. In addition, there seems to be a dependence between the number of carbon atoms in the molecule and the maximum excess pressure. Thus, as the number of carbon atoms increases, the maximum excess pressure decreases, and naturally, in this case, the minimum surface area per molecule also increases.

Electrical conductivity

In order to determine the thermodynamic parameters of the synthesized compounds, their solutions of different concentrations in water were prepared and the electrical conductivity values were calculated with a conductometer. A graph of electrical conductivity as a function of concentration was constructed and, based on the graph, the value of α, which is the ratio of slopes of the section before and after the CMC point, was calculated. Gibbs free energy value of micellar formation and adsorption was calculated using the formulas (3.3 and 3.4).

$$\Delta G_{mic} = (2-\alpha) * R * T * \ln(CMC) \quad (3.3)$$

$$\Delta G_{ad} = \Delta G_{mic} - 0.6023 * \pi_{CMC} * A_{CMC} \quad (3.4)$$

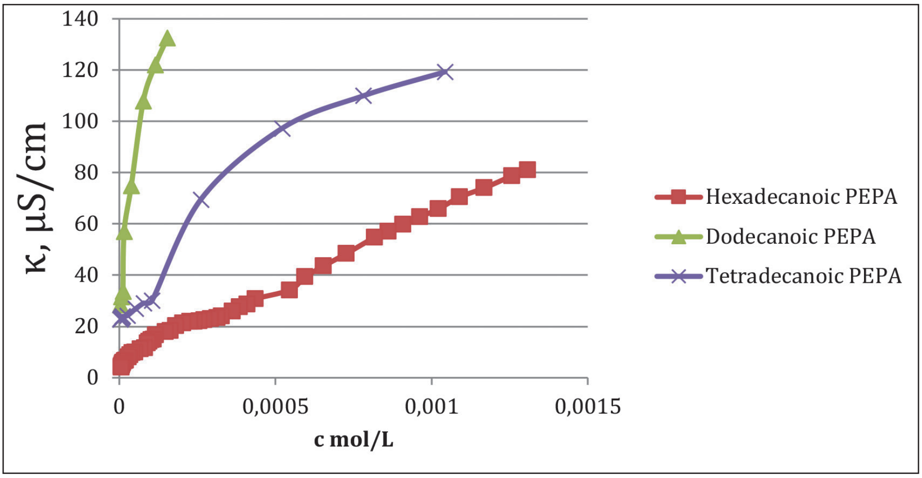

Figure 11 – **Electrical conductivity versus concentration for the synthesized salts**

Table 2 – **Electroconductivity parameters**

Surfactants	α	Δ	ΔG_{mic}, kJ/mol	ΔG_{ad}, kJ/mol
Dodecanoic acid PEPA	0.82	0.18	-26.26	-29
Tetradodecanoic acid PEPA	0,93	0,07	-22,73	-25,60
Hexadecanoic PEPA	0,57	0,43	-31.9	-35.8

Looking at the parameters given in Table 2, it is clear that for all three combinations, both parameters have negative values, which indicates that both processes occur naturally. It can be seen from *Table 2*, for all the salts ΔG_{ad} value is more negative than ΔG_{mic} . It means when surfactant was applied to the air-water border firstly adsorption process happens following withmicellization. For the compound obtained by the synthesis of hexadecanoic acid, these parameters have a more negative value.

In order to discuss the using of the surfactants as an oil spill cleaning agent, petrocollecting and petrodispersing properties have been measured and shown in *Table 3*.

Table 3 – **Petrocollecting and petrodispersing properties**

Ratio	State of surfactant	Sea water		Tap water		Distilled water	
		Kd	Duration-τ, hours	Kd	Duration-τ, hours	Kd	Duration-τ, hours
Dodecanoic acid PEPA	5 wt. % aqueous solution	90%	0-24	3,83	0-24	25,5%	0-24
		96,4%	24-48	96%	24-48	98,6%	24-48
		78,5%	48-240	90%	48-240	94,5%	48-240
		Spilling		Drying 18%		88 %	
	5 wt. % ethanolic solution	91,5%	0-24	98,2%	0-24	99%	0-24
		96,4%	24-48	96,4%	24-48	90%	24-48
		94,5%	48-240	90%	48-240	90%	48-240
		Spilling		Drying 16%		Spilling	
	Solid	91,20%	0-24	15,32	0-24	12,2	0-24
		98,2%	24-48	98,2%	24-48	97,2%	24-48
		88%	48-240	94,2%	48-240	90%	48-240
		Spilling		Drying 17		Drying 80%	
Tetradecanoic acid PEPA	5 wt. % aqueous solution	94%	0-19	16.7	0-72	12.8	0-1
		88%	19-163	9	72-168	5.57	1-73
		1.5	163-513	Spilling		5	73-265
		Spilling				Spilling	
	5 wt. % ethanolic solution	9.89	0-72	12.03	0-1	12.26	0-1
		84.5%	72-88	89%	1-73	88%	1-217
		89%	88-232	86%	73-169	Spilling	
		2.03	232-328	Spilling			
		Spilling					
	Solid	87%	0-168	12.03	0-1	12.26	0-1
		Spilling		9.625	1-3	10.13	1-3
				11	3-19	9.625	3-19
				12.83	19-312	Spilling	
				Spilling			
Hexadecanoic acid PEPA	5 wt. % aqueous solution	91,5%	0-48	83,5%	0-48	7,25	0-48
		94,3%	48-144	94,3%	48-144	94,3%	48-144
		88,00%	144-174	89%	144-174	90%	144-174
		Spilling		Spilling		Spilling	
	5 wt. % ethanolic solution Solid	91,4%	0-48	91,4%	0-48	20	0-48
		94,3%	48-144	94,3%	48-144	94,3%	48-144
		89%	144-174	92%	144-174	93%	144-174
		Spilling		Spilling		Spilling	
	Solid	2,5	0-48	9,8	0-48	5	0-48
		88,8%	48-144	94,3%	48-144	86,2%	48-144
		83%	144-174	80%	144-174	92%	144-174

When looking at the petrocollecting and petrodispersing properties it can be seen that the compounds obtained with dodecanoic acid, although it has more oil dispersing ability, it also shows petrocollecting ability in the first moments of the application of the reagent. The highest petrocollecting coefficient was 25.5, obtained when applied with a 5% aqueous solution of the compound in distilled water, and maintained its potency for 24 hours. The highest petrodispersing percentage is 99%, and it is obtained when it is applied in distilled water with a solution in 5% alcohol. The highest petrodispersing percentage in seawater is 98.2% and is achieved when the reagent is applied in solid form. In this case, the reagent is active for 24 hours.

Looking at the combination with tetradodecanoic acid, it can be noted that it has more petrocollecting capacity than dodecanoic acid. A greater petrocollecting capacity can be observed in the solid state of the compound. The highest percentage of petrodispersing is 94% and is observed in seawater with a 5% aqueous solution. In this case, the time of the reagent's effectiveness is 19 hours.

Looking at the petrocollecting and petrodispersing results of the synthesis with hexadecanoic acid, it is clear that the compound has more petrodispersing ability. The highest percentage of petrodispersing is 94.3%, observed in all cases of the reagent, and the duration of action lasted 96 hours.

Conclusion:

Regarding the physical properties of the synthesized salts it can be deduced that CMC and corresponding surface tension at this point increases as increasing the length of carbon chain. Coming to the petrocollecting and petrodispersing properties compound obtained with tetradodecanoic acid acid is better at petrocollecting effect than the others, while the salt getting from the dodecanoic acid shows higher petrodispersing property.

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