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Anionic cocogem surfactants containing propyl-2-ol groups: Synthesis, surface properties and antibacterial activity against SRB bacteria



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ABSTRACT

Cocogem surfactants are synthesized based on the neutralization reaction of higher monocarboxylic acids (octanoic acid, nonanoic acid, decanoic acid, undecanoic acid, dodecanoic acid, tetradecanoic acid, hexadecanoic acid, heptadecanoic acid, octadecanoic acid) and 2:1 mol of N,N'-bis(propyl-2-ol)ethylenedia mine). Krafft temperatures of these surfactants and colloidal chemical properties of their aqueous solutions were determined by using tensiometric and conductometric measurements. Surface activity parameters of cocogem surfactants, critical micelle concentration (CMC), counter-ion binding degree (β), the surface tension reduction effectiveness (π_{CMC}), excess concentration of surface (Γ_{max}), as well as area per molecule at the interface (Λ_{min}), Gibbs free energy (ΔG_{ad} and ΔG_{mic}) values of adsorption and micellar formation processes are calculated. The size of the aggregates formed by anionic cocogem surfactants in an aqueous solution was determined by the DLS method. It was defined that cocogem surfactants have antibacterial properties against SRB, which is considered to be the most dangerous for the oil industry. © 2022 Egyptian Petroleum Research Institute. Production and hosting by Elsevier B.V. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/).

1. Introduction

Gemini surfactants are substances, the head groups are located at a certain distance and consist of two amphiphilic parts connected by a spacer group. Gemini surfactants have two hydrophilic groups and two hydrophobic groups [1]. Hydrophilic or hydrophobic groups are joined by a spacer chain. Gemini surfactants have a myriad of special features. These features make them superior to their single-chain counterparts. For instance, significantly lower CMCs; super efficiency in case of reducing surface tension; unusual aggregation morphologies; and better wetting, solvents, foaming, etc. features make them potentially useful in many applications [2]. In general, gemini surfactants have higher efficiency that's why they can be considered environmentally preferable and more cost-effective compared to typical surfactants [3]. A class of surfactants is also counter-ion coupled gemini (cocogem) surfactants. These types of substances are sometimes called pseudo-vessel sur-

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factants [4]. There is the main source of information about cocogem surfactants in the literature. Cationic cocogem surfactants are substances based on alkyl amines and dicarboxylic acid derivatives. There is mainly a dicarboxylate anion group of dicarboxylic acid acting as a spacer in this type of cocogem surfactant [5]. It has been synthesized cocogem surfactant based on adipoyl chloride and N,N-dimethylhexadecylamine. M.S. Alam et al. noted, that the obtained cocogem surfactant has good antibacterial and very weak toxic properties. Y. Tang et al. [6] had synthesized various surfactants, based on the cetyl-trimethyl-ammonium bromide and dicarboxylic acid sodium salt. It has been found, that increasing the (CH₂) length of the alkyl chain between carboxyl groups decreases the value of CMC in dicarboxylic acids. These types of cocogem surfactants can foam, emulsify, and act as anticorrosive surfactants [7,8]. Cationic cocogem surfactants can be obtained from renewable resources. Cocogem surfactants, that are synthesized based upon sugars can reduce surface tension at the waterair boundary. Anionic cocogem surfactants, on the other hand, are obtained based on higher monocarboxylic acids and diamines [9,10]. Diamines play the role of a spacer in represented cocogem surfactants. In the presence of propyl-2-ol derivatives instead of diamines, the CMC and γ_{CMC} values of the obtained cocogem sur-

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factants are lower [11]. In certain circumstances, cocogem surfactants have been synthesized, using alkyl benzenesulfonates (or SDS) instead of higher monocarboxylic acids, and jeffamine instead of diamines (or ether diamines) [12–14]. Protic ionic liquid surfactants are obtained by the interaction of higher monocarboxylic acids with amines at room temperature [15,16]. If diamines are utilized contrary to amines, cocogem surfactants are obtained. Therefore, this type of cocogem surfactants can also be called protic ion liquid surfactants. All types of microorganisms (bacteria) lead to corrosion. The most harmful is the sulfate-reducing bacteria (SRB). These bacteria release H₂S into the environment as a final product by using sulfate ions. The large amounts of H₂S under the influence of these bacteria causes great environmental and economic losses in large-scale industrial plants [15]. One of the most affected areas by SRB bacteria is the oil industry. Thus, SRB bacteria is one of the reasons, that cause corrosion of the equipments in the oil industry [16,17]. In addition to their ability to inhibit the growth of SRB bacteria [18] surfactants have many advantages such as high efficiency as corrosion inhibitors, low cost, easy production and low toxicity.

The presented research work is devoted to the synthesis of new anionic cocogem surfactants by the interaction of C_8 - C_{18} monocarboxylic acids with N,N-bis(propyl-2-ol) ethylenediamine, the study of their surface activity, and exploring antibacterial properties against SRB bacteria.

2. Experimental section

2.1. Reagents and instruments

All monocarboxylic acids $C_nH_{2n+1}COOH$ (purity ≥ 97 %, Sigma-Aldrich), epoxypropane (purity 99 %, Alfa Aesar), ethylenediamine $H_2N(CH_2)_2NH_2$ (purity 99 %, Alfa Aesar). The surface tension of the aqueous solutions of the synthesized cocogem surfactants was determined with a KSV Sigma 702 (Finland) tensiometer [19], the specific electrical conductivity was determined with an ANION-410 (Russian production) conductometer, and the size dimensions of the aggregates were determined with a Particle Size Analyzer (HORIBA LB-550, Japan) device.

2.2. Synthesis of N,N'-bis(propyl-2-ol)ethylenediamine

It has been obtained N,N'-bis(propyl-2-ol)ethylenediamine from the reaction of ethylenediamine and epoxypropane in a 1:2 mol ratio. 0.2 mol ethylenediamine and 0.4 mol epoxypropane were added to a round-bottom flask to start the reaction. Epoxypropane was added in slight excess to ensure about the conversion of ethylenediamine was complete [19].

2.3. Synthesis of cocogem surfactants

There were executed reactions of N,N'-bis(propyl-2-ol) ethylenediamine with carboxylic acids in the ratio of 1:2 for obtain cocogem surfactants. The process was carried out at the temperature of 55–58 °C by using alcohol as a solvent. During the reaction, N,N'-bis(propyl-2-ol) ethylenediamine is converted to the ammonium group of ethylenediamine and the carboxyl group to the carboxylate anion. The common scheme of the reaction is described below:

there is $R = C_7H_{15}$ (C_8 -E- C_8); C_8H_{17} (C_9 -E- C_9); C_9H_{19} (C_{10} -E- C_{10}); $C_{10}H_{21}$ (C_{11} -E- C_{11}); $C_{11}H_{23}$ (C_{12} -E- C_{12}); $C_{13}H_{27}$ (C_{14} -E- C_{14}); $C_{15}H_{31}$ (C_{16} -E- C_{16}); $C_{16}H_{33}$ (C_{17} -E- C_{17}); $C_{17}H_{35}$ (C_{18} -E- C_{18}).

2.4. Biocidal properties of surfactants against the Desulfovibrio desulfuricans

The sulfate-reducing bacterium used in this study is *Desulfovibrio desulfuricans* (ATCC 27774). Bacterial cells were pre-cultured for three days at 37 °C within a medium that consisted of 3.00 g trisodium citrate, 2.50 g sodium lactate, 1.0 g yeast extract, 0.20 g MgSO₄, 0.30 g (NH₄)₂Fe(SO₄)₂·6H₂O, 0.50 g K₂HPO₄, 0.05 g NH₄Cl, 0.01 g CaCl₂·2H₂O in 1000 ml of distilled water. The bacterial culture was performed under the anaerobic condition at pH 7.2–7.5 [20]. The effect of the synthesized surfactants on SRBs was tested at three concentrations (50, 100, and 150 mg/l). There was realised the growth of bacteria is about 10^8 cells/ml, in the tested aqueous medium.

3. Results and discussion

3.1. Characterization and synthesis of the anionic cocogem surfactants

As can be seen from the IR spectrum of anionic cocogem surfactants, the absorption bands of the COOH group at 1700 cm⁻¹ disappeared, and the absorption bands of symmetric and asymmetric vibrations of the COO group formed at 1560-1580 and 1370-1420 cm⁻¹, respectively. The absence of any peak in the region after 10 ppm in the ¹H NMR spectrum, and the shift of the peak belonging to the C=O group in the ¹³C NMR spectrum, confirm the conversion of the carboxyl group to the carboxylate group. Referring to this, we can conclude that the neutralization reaction is almost complete (see Figs S1-S3 in Supporting Information). The Krafft temperature of surfactant cocogem based on monocarboxylic acids C₈-C₁₀ is less than 0 °C, surfactant cocogem based on monocarboxylic acids $C_{11}\text{-}C_{14}$ is less than 25 °C ($C_{11}\text{-}E\text{-}C_{11}$ for 5 °C, C_{12} -E- C_{12} for 14 °C and C_{14} -E- C_{14} for 20 °C) and C_{16} are higher than 25 °C (C_{16} -E- C_{16} for 27 °C, C_{17} -E- C_{17} for 26 °C and C_{18} -E- C_{18} for 32 °C). In cocogem surfactants based on adipoyl chloride and tertiary amines, alkyl chain elongation also increases in the Krafft temperature [7]. There is a deterioration in the water solubility of other cocogem surfactants based on dicarboxylic acids [13,21-

3.2. Measurement of surface tension

Surface tension values at the boundary with air (at 25 °C) of aqueous solutions of anionic cocogem surfactants with Krafft temperature below 25 °C were determined. Based on the gotten outcomes , the concentration dependence of the surface tension was plotted. (Fig. 1). As evident the surface tension isotherms, the surface tension values of the cocogem surfactants are stabilized due to the complete formation of micelles after a certain concentration. The concentration at which the surface tension value stabilizes is equal to the CMC value. Table 1 lists the CMC values of anionic cocogem surfactants. As can be seen from the table, the CMC values of the synthesized anionic cocogem surfactants vary within 0.16–10.80 mM, and it decreases by increasing the alkyl chain. The CMC values are approximately-two times less than those of coco-

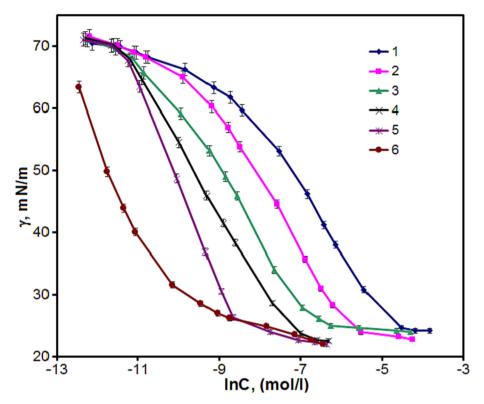


Fig. 1. The plot of the surface tension versus the natural logarithm of the concentration of cocogem surfactants based on caprylic acid (1), pelargonic acid (2), capric acid (3), undecylic acid (4), lauric acid (5) and myristic acid (6).

Table 1 Surface activity parameters of the synthesized cocogem surfactants (25 $^{\circ}$ C).

Gemini surfactants	$CMC^{a} \times 10^{3}$, $mol \cdot dm^{-3}$	$CMC^b \times 10^3$, $mol \cdot dm^{-3}$	β	$\begin{array}{c} \Gamma_{max} \times 10^{10}\text{,} \\ \text{mol} \cdot \text{cm}^{-2} \end{array}$		$A_{min} \times 10^2$, nm^2		pC ₂₀	γ _{CMC} , mN·m ⁻¹	π_{CMC} , mN·m $^{-1}$	ΔG _{mic} , kJ·mol ⁻¹	Δ G_{ad} , kJ · mol^{-1}
				n = 2	n = 3	n = 2	n = 3					
C ₈ -E-C ₈	10.80	10.4	0.42	1.86	1.24	89.4	134.1	3.22	24.7	47.3	-19.48	-24.15
C ₉ -E-C ₉	4.06	6.40	0.45	1.99	1.33	83.5	125.2	3.61	24.0	48.0	-22.41	-26.89
C ₁₀ -E-C ₁₀	1.40	1.33	0.52	2.02	1.45	82.2	123.4	3.98	25.0	47.0	-26.76	-31.11
C ₁₁ -E-C ₁₁	0.91	1.15	0.61	2.15	1.44	77.1	115.7	4.25	23.8	48.2	-30.30	-34.52
C ₁₂ -E-C ₁₂	0.43	0.78	0.63	3.26	2.17	50.9	76.4	4.46	24.0	48.0	-32.95	-36.01
C ₁₄ -E-C ₁₄	0.16	0.175	0.66	3.42	2.28	48.6	72.9	5.14	26.2	45.8	-36.66	-39.53

^a The CMC is determined by the surface tension method.

gem surfactants obtained based on N,N'-bis(propyl-2-ol) hexamethylenediamine with a similar structure [11].

The maximum adsorption of cocogem surfactants is calculated at the water–air interface - Γ_{max} (in mol m⁻²) and the minimum surface area occupied by the hydrophilic group (A_{min}) applying the following formulas [24].

$$\Gamma_{\text{max}} = -\frac{1}{nRT} \lim_{c \to c_{\text{CMC}}} \frac{d\gamma}{dlnC} \tag{1} \label{eq:gamma_max}$$

$$A_{\min} = 10^{16} / N_A \Gamma_{\max} \tag{2}$$

 N_A is the Avogadro constant. "n" depends on the sum of adsorbed particles at the interface. It is arduous to determine the "n" value directly for cocogem surfactants. However, even if the value applied for "n" affects the calculated values of Γ_{max} and A_{min} , it does not affect the change in the values of Γ_{max} and Λ_{min} and the number of carbon atoms in the (CH₂)_x chain. Thereby, in our study, the value of "n" was taken as 2 and 3 [25]. The synthesized coco-

gem surfactant is somewhat higher, so we can assume that n=3 is more accurate. Table 1 shows the values of Γ_{max} and A_{min} . As can be seen, with an elongation in the (CH₂) length of the alkyl chain, Γ_{max} increases, and A_{min} decreases. Thus, the elongation of the alkyl chain leads to rising in the packing density of the cocogem surfactant. This is because the process of adsorption at the air–water interface proceeds better as a result of increasing in the hydrophobicity of the surfactant molecule with elongation of the alkyl chain. The same situation is also observed in structurally similar cocogem surfactants [11]. The negative logarithmic value of the anionic cocogem surfactant concentration required to reduce the γ by 20 mN/m (pC₂₀) and it is called the adsorption efficiency. The Table 1 provide information about the adsorption efficiency of the synthesized anioinic cocogem surfactants.

As can be seen from the table, the pC_{20} values in the cocogem surfactant change in the range of between 3.22 and 5.14. The pC_{20} value made up with the length of the alkyl chain in cocogem surfactants. The pC_{20} values of the synthesized cocogem surfac-

^b The CMC is determined by the electroconductivity method.

tants are higher compared to the cocogem surfactant with the same length of spacer chain n=6 as the $(CH_2)_x$ one [11]. The values of surface pressure of cocogem surfactant are calculated according to the following formula:

$$\pi_{CMC} = \gamma_0 - \gamma_{CMC}$$

where γ_0 is the surface tension of the aqueous solution without surfactant at the border with the air, and γ_{CMC} is the value of the surface tension of the surfactant at the CMC. Table 1 are given the values of π_{CMC} synthesized cocogem surfactants. As can be seen from the table, the values of π_{CMC} obtained anionic cocogem surfactants are high enough.

3.3. Specific electrical conductivity of anionic cocogem surfactants

The values of the specific electrical water solution's conductance of the obtained anionic cocogem surfactant were determined. Dependence graphs of the specific electrical conductance of water solutions in various concentrations are given in Fig. 2. As can be seen from the figure, the curve of conductivity depending on concentration consists of a straight line with two different slopes. The intersection point of these lines is equal to the CMC value. The CMC values determined by the conductometric method are shown in Table 1. As can be seen from the table, the CMC values determined by the conductometric method differ somewhat from the values determined by the tensiometric method. This is because cocogem surfactants are organic molecules with a large molecular weight of the opposite ion [5,26]. The ratio of the slope (S2) of the dependence line at high concentrations from CMC to the slope (S1) of the dependence line at low concentrations from CMC is equal to the dissociation degree of the counter-ion:

$$\alpha = S_2/S_1 \tag{3}$$

The counter-ion binding degree (β) is computed using the given formula:

$$\beta = 1 - \alpha \tag{4}$$

The values of counter-ion binding of the obtained cocogem surfactants are given in Table 1. As can be seen from the table, the degree of counter-ion binding of the surfactants increases with the increasing $(CH_2)_x$ length of their alkyl chain. Therefore, increasing the length of the alkyl chain complicates the dissociation abil-

ity of counter-ions in the cocogem surfactants. Surfactants with a similar structure based on N,N'-bis(propyl-2-ol) hexamethylenediamine also distribute an increase in β the (CH₂)_x length of the alkyl chain elongation from C_{10} to C_{14} .

3.4. Aggregates values of surfactants

The diameters of the aggregates formed by the obtained anionic cocogem surfactants in water solution were studied by dynamic light scattering (DLS). For this, there were prepared solutions in two different concentrations above the CMC values and the dimension of anionic cocogem surfactant aggregates formed in them, were has been identified (Figs. 3-5). As shown in Fig. 3 (a), as the concentration of C₈-E-C₈ had a growth from 0.05 % to 0.1 %, the diameter of the aggregate changes a little, increasing from 9 nm to 10 nm. The bimodal peak is observed both in 0.05 % and 0.1 % solutions (Fig. 3 b) in C_{10} -E-C10. There are formed aggregates with a diameter of 260 nm and 3000 nm in a 0.05 % solution. When the concentration is doubled, the diameter of both aggregates increases to 300 nm and 3400 nm. With an increase in concentration, the proportion of large-diameter aggregates also increases several times. The average diameter of the aggregates of C₁₂-E-C₁₂ is 3400 nm in a 0.05 % aqueous solution. As the concentration of the solution increases, the monomodal peak is replaced by a bimodal one. There are formed polydisperse aggregates of a smaller diameter (150 nm), although the diameter of the aggregates does not change. There are formed aggregates with a diameter of 4500 nm in a 0.05 % solution of C_{14} -E- C_{14} and aggregates with a diameter of 5100 nm in a 0.1 % solution. With increasing concentration aggregates increase in size and monodispersity increases. As can be seen from Figs. 3-5, the average diameter of the units is increased by the (CH₂) length of the alkyl chain of cocogem surfactants.

3.5. Thermodynamic parameters of the anionic cocogem surfactants

Gibbs standard free energy of micellization process (ΔG_{mic}) values for synthesized cocogem surfactants can be computed from Eq. (5) [24].

$$\Delta G_{mic} = RT(0.5 + \beta) \ln X_{CMC} - (RT/2) \ln 2 \tag{5}$$

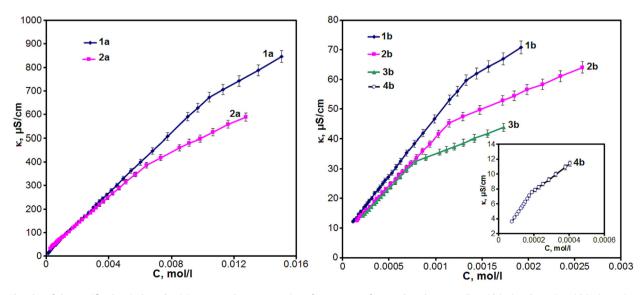


Fig. 2. The plot of the specific electrical conductivity versus the concentration of cocogem surfactants based on caprylic acid (1a), pelargonic acid (2a), capric acid (1b), undecylic acid (2b) lauric acid (3b), and myristic acid (4b).

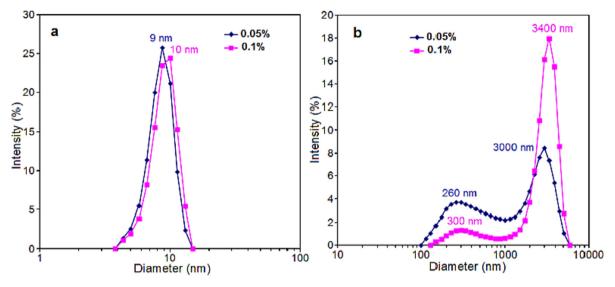
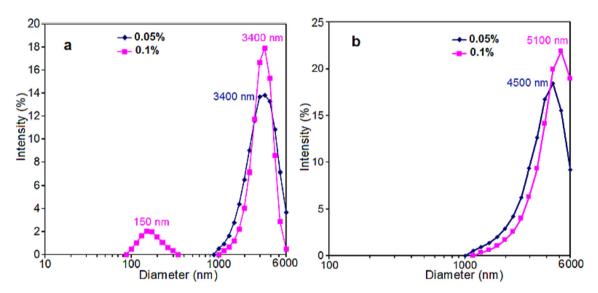


Fig. 3. Size distribution of aggregates in an aqueous solution of C_8 -E- C_8 (a) and C_{10} -E- C_{10} (b) at two different concentrations.



 $\textbf{Fig. 4.} \ \, \textbf{Size distribution of aggregates in an aqueous solution of } C_{12}\text{-}E-C_{12}\left(a\right) \ \, \textbf{and} \ \, C_{14}\text{-}E-C_{14}\left(b\right) \ \, \textbf{at two different concentrations}.$

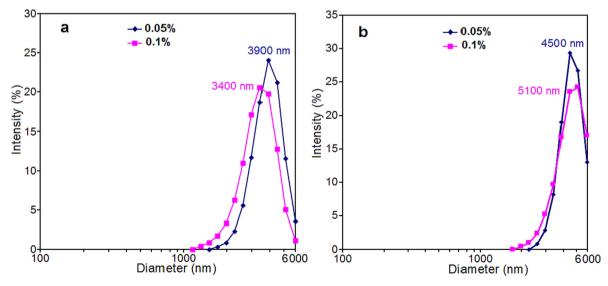


Fig. 5. Size distribution of aggregates in an aqueous solution of C_{16} -E- C_{16} (a) and C_{18} -E- C_{18} (b) at two different concentrations.

where X_{CMC} is the CMC in mole fraction units [i.e., CMC/(CMC + 55.5)].

Standard Gibbs free energy change of adsorption process (ΔG_{ad}) of the obtained anionic cocogem surfactants are using Eq. (6) [24].

$$\Delta G_{ad} = \Delta G_{mic} - 0.6023 \pi_{CMC} A_{CMC} \tag{6}$$

where, A_{CMC} is in Å 2 /molecule, and π_{CMC} is the surface pressure at CMC in the air/water interface (mN m $^{-1}$). The standard values of the change in the Gibbs free energy for the process of micellization and adsorption are showed in Table. 1. It is easy to conclude that the processes of micellization and adsorption are spontaneous since all data are negative. Both the ΔG_{ad} and ΔG_{mic} values diminish with the increasing CH $_2$ length of the hydrophobic chain of the anionic cocogem surfactants, due to rising in the interaction between the alkyl chain processes of adsorption and micellization. The ΔG_{ad} values in cocogem surfactants are lower than ΔG_{mic} , indicating that the micelle formation process predominates over adsorption at the air/water interface.

3.6. Antibacterial properties of obtained anionic cocogem surfactants

SRB is one of the main bacteria that accelerates the corrosion of metals. These batteries cause increased corrosion of metals, especially in petroleum processing and oil production. One way to protect metal equipment from SRBs is to use bactericides, which can prevent bacterial growth. There were studied (Table 2) the antibacterial properties of the obtained cocogem surfactant against SRB bacteria. As can be seen from Table 2, the obtained anionic cocogem surfactants have antibacterial properties against SRB bacteria. Although this property is weak at low concentrations, the efficiency increases at high concentrations.

The surfactants based on lauric acid have the highest effect. Cocogem surfactant based on caprylic acid also has high antibacterial action. With an increasing value in the (CH₂)_x chain starting from C₈, the antibacterial properties first weaken and then increase. The antibacterial properties of the cocogem surfactant based on myristic acid are relatively weak. The antibacterial properties of surfactants depend on their electrostatic interaction with bacteria and the adsorption ability of the alkyl chain. For high bactericidal ability, the adsorption capacity of the surfactant on the cell membrane must be high [27]. An increase in the $(CH_2)_x$ length of the alkyl chain of surfactants improves their adsorption properties. One of the main reasons for its effective biocidal properties is its high adsorption capacity [27]. In some cases, with an increased CH₂ chain in the alkyl group [28] the electrostatic interaction decreases because of growing dissociation degree in the surfactants. The aggregate dimensions of surfactants are also considered one of the main factors affecting their antibacterial properties [29]. Thus, if the aggregates are large, they cannot be evenly distributed in the bacterial environment. Therefore, it is impossible to completely stop the growth of bacteria due to their weak contact. There were observed that the antibacterial properties are weakened, due

 Table 2

 Antibacterial effect of the obtained anionic cocogem surfactants against SRB.

Gemini surfactants	Concentration of cocogem surfactants, mg/					
	50	100	150			
C ₈ -E-C ₈	10 ³	10 ²	10			
C ₉ -E-C ₉	10 ⁵	10^{4}	10 ³			
C ₁₀ -E-C ₁₀	10 ⁷	10^{6}	10 ⁵			
C ₁₁ -E-C ₁₁	10^{4}	10^{2}	10			
C ₁₂ -E-C ₁₂	10^{3}	10	Nil			
C ₁₄ -E-C ₁₄	10 ⁵	10^{4}	10 ³			

to the large size of the cocogem surfactant aggregates based on surfactants derived from myristic acid, despite the high adsorption capacity.

4. Conclusions

There have been synthesized cocogem surfactants with various alkyl chains based on N,N'-bis(propyl-2-ol)ethylenediamine and (C_8 - C_{18}) monocarboxylic acids. There have been determined the Krafft temperatures of cocogem surfactants and the colloid-chemical parameters of compounds with Krafft temperatures below 25 °C by tensiometric and conductometric methods in their aqueous solutions. It has been established, that by rising in the length of the (CH_2)_x chain of the cocogem surfactants, the values of CMC, A_{min} , ΔG_{ad} , and ΔG_{mic} decrease, in contrast the values of Γ_{max} , β , ρC_{20} increase. It has been determined by DLS that, the size of micellar aggregates, which formed by the anionic cocogem surfactants in water increases sharply with an elongation in the length of the (CH_2)_x chain. It was found that the bactericidal effect of cocogem surfactants based on lauric acid is higher than other anionic cocogem surfactants.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.ejpe.2022.12.001.

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