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SYNTHESIS OF SURFACTANTS BASED ON OCTYLAMINE, PROPYLENE OXIDE, AND DICARBOXYLIC ACIDS AND INVESTIGATION OF THEIR ANTIBACTERIAL PROPERTIES

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ABSTRACT

Using a twofold molar excess of propylene oxide at 25 °C, octylbis(2-hydroxypropyl) amine was synthesized. A series of gemini surfactants was obtained by reacting this product with dicarboxylic acids (oxalic, succinic, adipic, and sebacic) in a 2:1 molar ratio at 53–55 °C. The structures of the synthesized products were confirmed using IR spectroscopy. Tensiometric measurements of surface tension over a wide concentration range at the air–water interface revealed that the obtained compounds exhibit high surface activity. For example, the product based on oxalic acid reduces the surface tension from 70.33 mN/m at 0.0001% to 29.51 mN/m at 0.1%. It was also shown that their specific electrical conductivity significantly exceeds that of deionized water, confirming the ionic nature of the synthesized compounds. Furthermore, disk diffusion tests demonstrated that the antibacterial activity of these products depends not only on the length of the spacer group but also on the type of bacterial strain.

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5. INTRODUCTION

The current stage of scientific development is marked by growing interest in surfactants, which is driven by their wide application in various fields, including industry, agriculture, medicine, and everyday life. In recent years, researchers have increasingly focused on the synthesis of surfactants with novel structural architectures [1-3]. Among these, gemini surfactants deserve special attention. The distinctive feature of their structure is the presence of two hydrophobic moieties and two headgroups (typically charged), linked by spacer fragments of varying length and composition [4-9]. Compared to their monomeric analogues, gemini surfactants exhibit a combination of high positive surface charge, conformational flexibility, and the ability to adapt spatially during association. They are also characterized by low critical micelle concentration values, allowing their use in significantly lower dosages [10]. These attributes impart enhanced performance properties [11–13]. Due to their high efficiency, gemini surfactants represent a more

environmentally friendly and economically advantageous alternative to conventional surfactants.

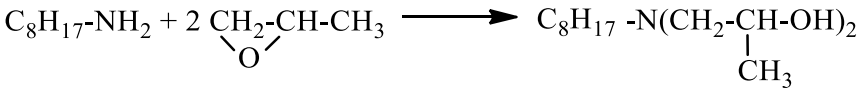
The present study focuses on the synthesis of new gemini surfactants based on octylamine, propylene oxide, and dicarboxylic acids, as well as on investigating how the structure of the spacer group influences their surface-active and antibacterial properties.

6. MATERIALS AND EXPERIMENTAL METHODS

Octylamine (99% purity; Alfa Aesar, Shore Road, Heysham), propylene oxide (99.9%; Alfa Aesar, Great Britain), oxalic acid dihydrate (99%), succinic acid (99%), adipic acid (99%), and sebacic acid (98%) were all supplied by Alfa Aesar. The chemical structures of the prepared compounds were confirmed by ALPHA FT-IR analysis. The surface tension values of the aqueous solutions of the synthesized gemini surfactants were measured at the air–water interface using a du Nouy tensiometer (KSV Sigma 702, Finland) with the ring method. The specific electrical conductivity was determined using an ANION-410 ionometer (Russia). The antibacterial activity of the synthesized substances was assessed by the disk diffusion method in accordance with standard microbiological procedures [14]. The antibacterial activity was evaluated against the following microorganisms: *Staphylococcus aureus* (MRSA and MSSA) and *Escherichia coli*.

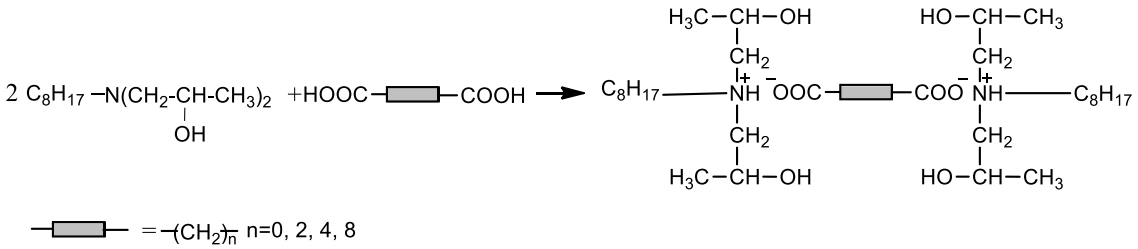
7. RESULTS AND DISCUSSION

In the first stage, octylamine and propylene oxide were synthesized at a molar ratio of 1:2 at room temperature (20–25 °C) for 15–35 hours. The obtained compound, octylbis(2-hydroxypropyl) amine, is a transparent, viscous substance. It is partially soluble in water and highly soluble in ethanol, acetone, hexane, kerosene, CCl_4, and isopropanol. The reaction scheme can be described as follows:



In the IR spectrum of octylbis(2-hydroxypropyl)amine (cm^{-1}), the following absorption bands were identified: 3391 ν (OH), 2959, 2824 and 2854 ν (CH), 1458, 1373 and 1332 δ (CH), 1279 ν (C–N), 1132 ν (C–O), and 722 δ $(CH_2)_x$.

In the second stage, gemini surfactants were obtained from the interaction of octylbis(2-hydroxypropyl)amine with dicarboxylic acids (oxalic, succinic, adipic, and sebacic). The reactions were carried out in a flask equipped with a magnetic stirrer at 70–80 °C at a molar ratio of 2:1. The main components obtained are illustrated in the reaction scheme below:



The gemini surfactants obtained as a result of the synthesis can be designated as follows: $C_8C_0C_8$ (based on oxalic acid) – a yellow liquid; $C_8C_2C_8$ (based on succinic acid) and $C_8C_4C_8$ (based on adipic acid) – viscous, light brown substances; $C_8C_8C_8$ (based on sebacic acid) – a viscous brown liquid.

The structures of the synthesized products were confirmed using IR spectroscopy. The IR spectrum of the compound obtained from octylbis(2-hydroxypropyl)amine and sebacic acid is shown in Fig. 1. In the IR spectrum,cm^{-1}: 3311 ν (OH and NH), 2953, 2924 and 2854 ν (CH), 2540 and 2387 (NH^+), 1561 ν_{as} (COO^-), 1376 ν_s (COO^-), 1457 δ (CH), 1135 ν (C-N), 1079 (C-O), 722 δ $(CH_2)_x$.

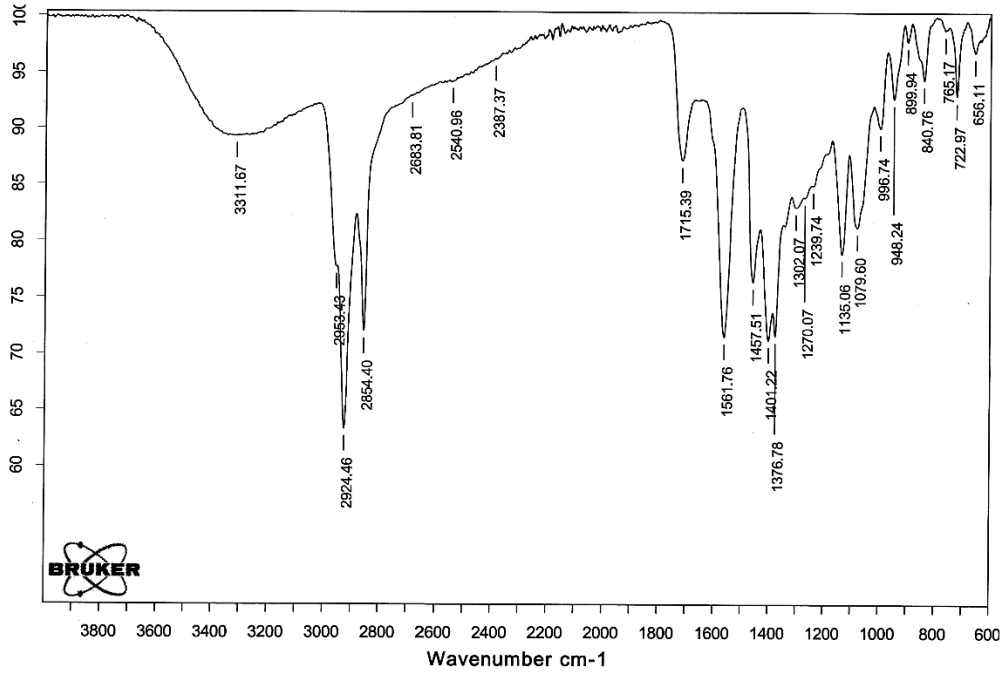


Fig. 1. IR spectrum of the gemini surfactant obtained from **octylbis(2-hydroxypropyl)amine** and sebacic acid

Table 1 presents the surface tension values of aqueous solutions of the synthesized gemini surfactants with spacer groups of different lengths over a wide concentration range. The results show that all investigated compounds reduce the surface tension depending on their concentration; however, the extent of this reduction varies significantly with the structure of the spacer group.

For $C_8C_0C_8$, the most pronounced and consistent decrease in surface tension is observed: from 70.33 mN/m at 0.0001% to 29.51 mN/m at 0.1%. This behavior indicates high surface activity and effective adsorption of the molecules at the air–water interface even at low concentrations.

Table 1. Interfacial tension at the air–water interface of aqueous solutions of gemini surfactants at various concentrations

Gemini surfactants	Concentration of substances in water (by weight), %												
	0.0001	0.00025	0.0005	0.00075	0.001	0.0025	0.005	0.0075	0.01	0.025	0.05	0.075	0.1
	Surface tension at the water-air interface, mN/m (20 °C)												
$C_8C_0C_8$	70.33	67.43	62.39	60.75	59.43	56.22	52.95	48.27	44.57	40.32	35.52	30.72	29.51
$C_8C_2C_8$	70.97	70.56	70.69	69.08	67.02	67.62	67.42	63.53	62.05	56.09	49.49	46.67	44.47
$C_8C_4C_8$	71.58	67.78	61.53	62.70	66.63	62.29	49.35	56.42	58.32	49.18	48.17	48.28	46.00
$C_8C_8C_8$	70.78	69.15	62.41	62.53	50.52	52.93	52.65	52.53	52.07	54.97	39.12	39.30	38.41

The compound $C_8C_2C_8$ shows a less intensive decrease in surface tension at low concentrations. The values remain relatively high up to 0.005–0.01%, but at higher concentrations the surface tension decreases to 44.47 mN/m. This suggests lower efficiency compared to $C_8C_0C_8$, which may be attributed to reduced packing density at the interface due to the longer spacer.

For $C_8C_4C_8$, an irregular pattern of surface tension changes is observed. Along with notable decreases (e.g., 49.35 mN/m at 0.005%), local increases are also present. Such behavior is likely associated with structural rearrangements and conformational changes of the monomers and aggregates in solution, leading to unstable adsorption at the interface.

The compound $C_8C_8C_8$ exhibits particularly interesting behavior. Despite having the longest spacer group, it shows a noticeable reduction in surface tension at intermediate concentrations (~50–52 mN/m at 0.001–0.01%). As the concentration increases further, very low surface tension values (38.41 mN/m at 0.1%) are achieved, comparable to the most efficient compound, $C_8C_0C_8$. This indicates the possibility of a more compact intermolecular organization at higher concentrations, compensating for the effect of the long spacer.

Overall, the results demonstrate a strong influence of spacer length on the surface-active properties of the gemini surfactants. The most stable and efficient compound in reducing surface tension across the entire concentration range is $C_8C_0C_8$. Increasing the spacer length generally leads to lower efficiency; however, $C_8C_8C_8$ shows a distinct improvement in performance at higher concentrations, which may be attributed to specific features of its aggregation behavior.

Table 2 presents the data on the specific electrical conductivity of the new gemini surfactants synthesized on the basis of octyldiisopropylolamine and various dicarboxylic acids. The measurements were carried out in aqueous solutions at 22 °C within the mass concentration range of 0.00625% to 0.1%.

Table 2. Specific electrical conductivity of new gemini surfactants obtained on the basis of octyldiisopropylolamine and dicarboxylic acids

Gemini surfactants	Concentration of gemini surfactants in water (by weight), %				
	0.00625	0.0125	0.025	0.05	0.1
	Specific electrical conductivity, μS/cm (22 °C)				
$C_8C_0C_8$	10.6	22.9	39.0	85.5	226.1
$C_8C_2C_8$	10.8	20.7	37.7	82.5	146.3
$C_8C_4C_8$	12.0	25.0	45.0	96.0	170.1
$C_8C_8C_8$	10.8	19.7	35.5	83.9	150.2

As seen from the table, an increase in the concentration of all studied surfactants leads to a rise in specific electrical conductivity, which is associated with an increased number of ions in the solution. The highest conductivity values at the maximum concentration (0.1%) are observed for the compound $C_8C_0C_8$, while $C_8C_2C_8$, $C_8C_4C_8$, and $C_8C_8C_8$ exhibit slightly lower but comparable values. The differences in conductivity among the series members are attributed to the length of the spacer group and its influence on the degree of ionization and the micellar structure in solution.

The antimicrobial activity of the synthesized gemini surfactants was evaluated using the disk diffusion method, and the results are presented in Table 3. The data show varying degrees of inhibitory activity depending on the structure of the spacer group and the type of microorganism tested.

Table 3. Antimicrobial activity of gemini surfactants

Microbes	Inhibition zone diameter (mm)			
	$C_8C_0C_8$	$C_8C_2C_8$	$C_8C_4C_8$	$C_8C_8C_8$
Gram-positive bacteria				
Escherichia coli	15	8	7	-
Gram-negative bacteria				
Staphylococcus aureus MRSA	8	-	7	8
Staphylococcus aureus MSSA	9	-	13	8

For the gram-positive bacterium *Escherichia coli*, the highest activity is observed for $C_8C_0C_8$, which forms an inhibition zone of 15 mm. The compounds $C_8C_2C_8$ and $C_8C_4C_8$ exhibit significantly weaker activity (8 and 7 mm, respectively), while $C_8C_8C_8$ shows no activity. This indicates high sensitivity of *E. coli* to structures with a short spacer and a gradual decrease in activity as the spacer length increases.

In the case of *Staphylococcus aureus* MRSA, a different trend is observed. The compounds $C_8C_0C_8$ and $C_8C_8C_8$ inhibit bacterial growth with equal efficiency (8 mm), whereas $C_8C_4C_8$ shows moderate activity (7 mm). The compound $C_8C_2C_8$ is ineffective against MRSA. These findings suggest that spacer length influences the interaction of gemini surfactants with resistant *S. aureus* strains in a different manner compared to other microorganisms.

For *Staphylococcus aureus* MSSA, the largest inhibition zone (13 mm) is observed for $C_8C_4C_8$, which differs from its behavior in other tests. The compounds $C_8C_0C_8$ and $C_8C_8C_8$ show moderate activity (9 and 8 mm, respectively), while $C_8C_2C_8$ remains inactive.

Overall, the data demonstrate that the antimicrobial activity of gemini surfactants depends not only on the length of the spacer group but also on the specific bacterial strain. The compound $C_8C_0C_8$ is the most universal, exhibiting consistent activity against all tested microorganisms, whereas $C_8C_2C_8$ is the least effective in most cases. These differences may be related to how the surfactant molecules interact with bacterial cell walls and their aggregation behavior in aqueous media.

8. Conclusion

New gemini surfactants based on octyldiisopropylolamine and a series of dicarboxylic acids (oxalic, succinic, adipic, and sebacic) were synthesized. The structures of the obtained compounds were confirmed by IR spectroscopy. The surface tension values of their aqueous solutions were determined using a tensiometric method. It was established that the length of the spacer group significantly influences the surface-active properties of the gemini surfactants. The most stable and efficient compound in terms of reducing surface tension across the entire investigated concentration range is $C_8C_0C_8$. An increase in spacer length generally leads to a decrease in efficiency; however, for $C_8C_8C_8$, a pronounced improvement in properties is observed at higher concentrations, which may indicate specific features of its aggregation behavior. The antibacterial activity of the synthesized gemini surfactants was evaluated using the disk diffusion method. Analysis of the obtained data shows that the antibacterial effect is determined not only by the length of the spacer group but also by the type of bacterial strain. The compound $C_8C_0C_8$ exhibits the most universal properties, demonstrating consistent activity against all tested microorganisms.

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