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SYNTHESIS AND CHARACTERIZATION OF LACTIC ACID-BASED AMIDO-AMINE CATIONIC SURFACTANTS FROM FATTY ACIDS AND BENZYL CHLORIDE

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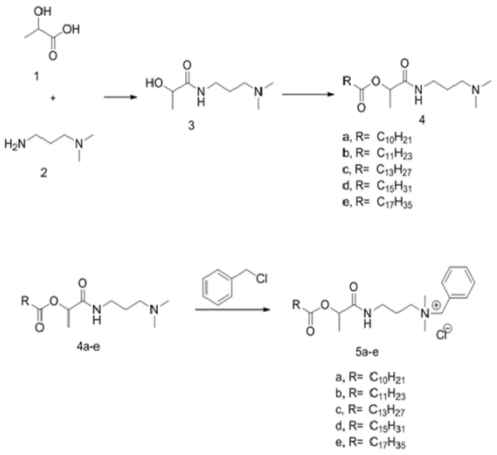
A series of cationic surfactants were synthesized from lactic acid and 3-(*N*,*N*-dimethylamino)-1-propylamine, followed by acetylation with fatty acid esters and quaternization with benzyl chloride. The structures of the synthesized quaternary ammonium compounds were confirmed by FTIR spectroscopy. Their surface-active properties, including critical micelle concentration (CMC), contact angle, foam stability, and wetting time, were systematically evaluated. Among the prepared compounds, those with longer hydrocarbon chains demonstrated superior wetting properties and good foaming behaviour. These products show potential for use in formulations such as shampoos and liquid soaps. Additionally, antibacterial activity was assessed, revealing that hydrocarbon chain length significantly influenced antimicrobial performance.



INTRODUCTION

Quaternary ammonium compounds with ester functionalities are industrially important due to their non-toxicity, biodegradability, and environmental compatibility.[1] Ester-based cationic surfactants degrade readily under aerobic and anaerobic conditions, enabling high removal rates in sewage treatment plants, and pose no harm to aquatic life.[2] Their antibacterial activity depends on structural features and physicochemical properties.[3]

Lactic acid is widely used as an antibacterial agent in home care, institutional, and industrial cleaning products. Quaternary ammonium salts derived from lactic acid are biodegradable, non-toxic, and non-sensitizing to skin, making them valuable in industrial formulations.[4] Functional properties such as foaming stability, detergency, wetting time, and antibacterial activity vary with chemical structure. High foaming ability at low concentrations and increased foam stability with longer hydrocarbon chains make these surfactants suitable for shampoos and liquid soaps.

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In this study, amido-amine-based cationic surfactants were synthesized from lactic acid and various fatty acids with improved yields, using benzyl chloride for quaternization.[5] The work focuses on the synthesis, surface-active properties, and antibacterial activity of compounds with varying alkyl chain lengths. All compounds were evaluated against Gram-positive and Gram-negative bacteria. The general structure of the synthesized surfactants is shown in (Fig. 1).

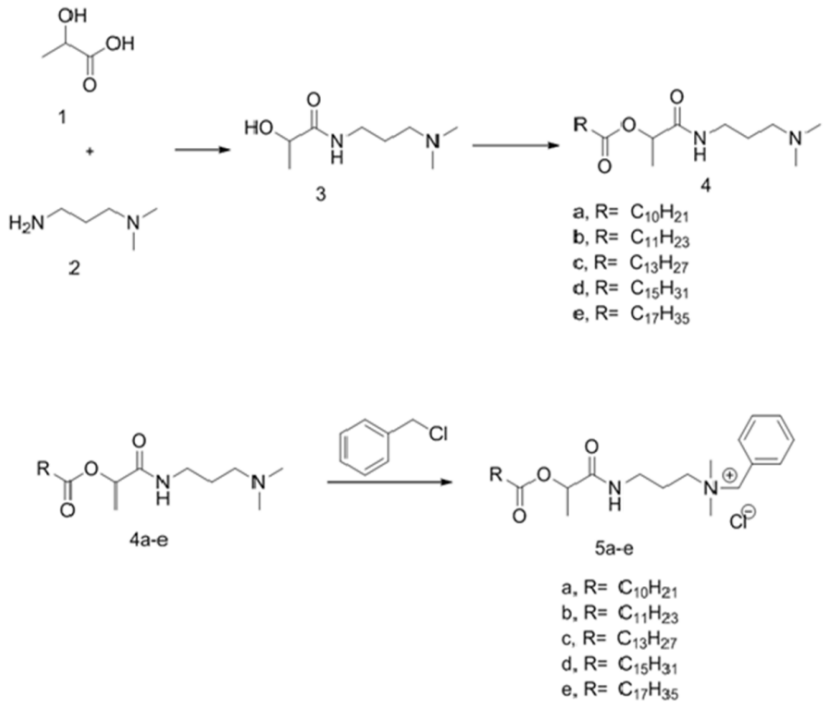

Fig. 1 – Synthetic pathway for the preparation of a series of amido-amine-based cationic surfactants.

EXPERIMENTAL SECTIONS

Materials

Lactic acid, fatty acids (C_8–C_{15} range), and benzyl chloride were purchased from Thomas Baker Pvt. Ltd., Mumbai, India**.** *N,N*-Dimethyl-*N*-propylamine was obtained from Sigma-Aldrich Pvt. Ltd. All chemicals were of analytical grade and used without further purification. Solvents were dried and distilled prior to use when necessary.

Experimental Procedure

Synthetic route for cationic surfactant

Synthesis of N,N-dimethylamino-N-propyl-lactamide.[6] A mixture of lactic acid (0.11 mol) and *N,N*-dimethyl-*N*-propylamine (0.04 mol) was heated at 130 °C for 18 h in the presence of 4 Å molecular sieves**.** After completion (monitored by IR), the mixture was washed with diethyl ether, extracted with ethyl acetate**,** dried over Na_2SO_4**,** and concentrated under reduced pressure. The product was purified by vacuum distillation to give a pale yellow viscous liquid in 80–85% yield**.**

Synthesis of alkanoic acid, 2-{[3-(dimethylamino) propyl] amino} -1-methyl-2-oxoethyl ester derivatives, using the ester path.[7] To a dry toluene solution of *N,N*-dimethylamino-*N*-propyl-lactamide (0.05 mol)**,** methyl alkanoate (0.037 mol) was added slowly under a dry nitrogen atmosphere**.** Potassium carbonate (0.057 mol) was then added, and the mixture was heated to 130 °C for 15 h**.** Reaction progress was monitored by TLC, and water formed during esterification was collected using a dean–Stark trap**.** After completion (16–18 h), the reaction mixture was cooled, washed twice with 30 mL of water, and dried over anhydrous magnesium sulfate**.** The solvent was removed under reduced pressure using a rotary evaporator. The yields of the ester derivatives are summarized in Table 1. All synthesized intermediates were characterized by FT-IR spectra (see Table 3 and Fig. 2).

Synthesis of 1-propanaminium, N,N-dimethyl-N-benzyl-3-({1-oxo-2-[(1 oxoalkyl) oxy)] propyl}

amino)-chloride derivatives. To a solution of alkanoic acid, 2-{[3-(dimethylamino) propyl] amino} -1-methyl-2-oxoethyl ester (4a–e, 0.030 mol) in 30 mL diethyl ether**,** benzyl chloride (0.045 mol) was added dropwise with stirring. The reaction mixture was refluxed for 3–5 h and monitored by TLC. The resulting quaternary ammonium salts were isolated by fractional distillation. The products obtained were waxy or pasty in appearance. The yields are given in Table 2, and the FT-IR spectra of the final compounds are presented in Table 3 and (Fig. 3).

Table 1

Reaction parameters of different synthesized compounds

Compound Code	Amidoamine: Fatty Acid (Chain Length)	Reaction Time (h)	Yield (%)
4a	3:C10	15 h	75.5
4b	3:C11	15 h	70.8
4c	3:C13	15 h	80.0
4d	3:C15	15 h	83.6
4e	3:C17	15 h	85.5

Table 2

Reaction parameters of cationic surfactants derived from benzyl chloride

Compound Code	Fatty Amidoamine: Alkyl halide	Molar Ratio	Reaction Time	Yield (%)	Cationic content (%)
5a	4a:BCl	1:1.5	5 h	70.8	70.00
5b	4b:BCl	1:1.5	5 h	78.2	65.98
5c	4c:BCl	1:1.5	5 h	80.2	68.00
5d	4d:BCl	1:1.5	5 h	85.4	62.12
5e	4e:BCl	1:1.5	5 h	89.4	69.88

Table 3

Interpretation of FT-IR spectra of Synthesized Compounds

Functional Group	Ester (4a) (cm^{-1})	Esteramide Quaternary Compound (5e) (cm^{-1})
N-H stretching	3373.50	3356.14
N-H Deformation	1037.70	1037.22
C=O	1741.72	1741.70
Amide C=O	1654.92	1643.34
C-O	1165.00	1168.16
Tertiary N	2854.65,1537.27	2550.79
CH_2	2924.09	2920.23
Monosubstituted benzene ring		719.45,702.09, 1578
Quaternary N^+		1463.97

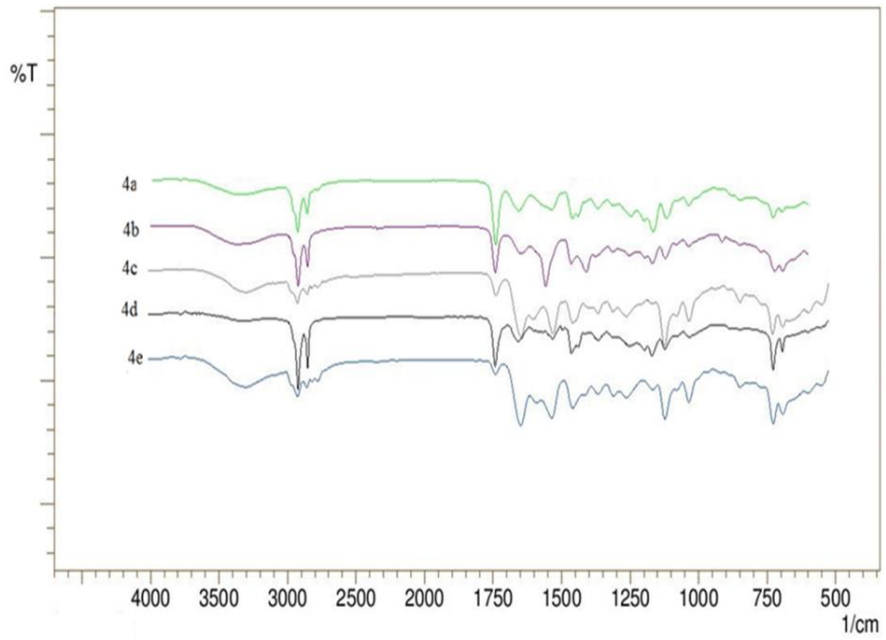

Fig. 2 – FTIR spectra of synthesized esteramides (4a–4e) showing characteristic absorption bands corresponding to N–H stretching, C=O, and C–O groups.

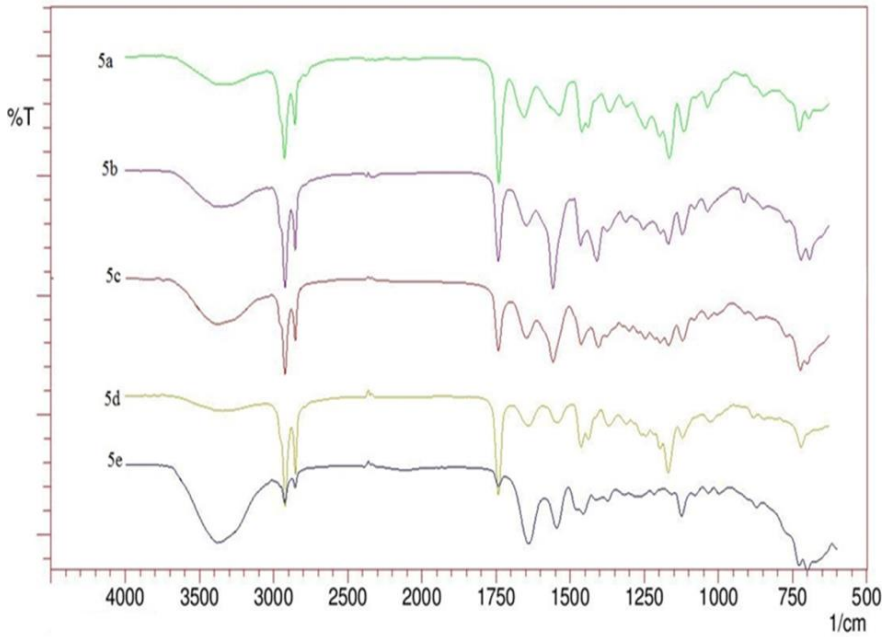

Fig. 3 – FTIR spectra of quaternary ammonium surfactants (5a–5e) derived from benzyl chloride.

Analysis of cationic surfactant

Determination of Cationic Content:[8] a) Chloroform (analytical grade); b) 0.004 M Sodium lauryl sulphate (SLS): Prepared by dissolving 1.464 g of SLS in 1000 mL distilled water; 0.003 % Methylene blue solution: Prepared by dissolving 0.003 g methylene blue, 30 g sodium sulphate, and 4.1 mL concentrated H_2SO_4 in distilled water and making up to 1000 mL; c) 0.004 M Surfactant solution: An accurately weighed quantity of the synthesized cationic surfactant was dissolved in 20–30 mL ethanol and diluted to 250 mL with distilled water.

Procedure:

To a 250 mL stoppered flask**,** 10 mL of 0.004 M SLS**,** 15 mL chloroform**,** and 25 mL methylene blue

solution were added and shaken thoroughly. After standing for phase separation, the organic layer (blue) was titrated with the 0.004 M cationic surfactant solution**.** Near the end point, the blue colour gradually shifted to the aqueous layer. The titration was stopped when both layers showed equal colour intensity. Results are summarized in Table 2.

Molarity of surfactant $M_1=10M_2/ V_1$

where M_1= Molarity of surfactant; M_2= Molarity of sodium Lauryl sulphate; V_1= volume (mL) of surfactant solution used.

Cationic content was calculated using:

$$Cationic\ content\ = \frac{M_1 \times \text{mol weight of surfactant} \times 100}{\text{weight of sample} \times 4}$$

Determination of Surface tension and CMC. The surface-active properties of the synthesized cationic surfactants were evaluated using a Krüss tensiometer (K100) at 25 °C. Each compound showed good water solubility under the experimental conditions.

The critical micelle concentration (CMC) and surface tension at CMC (γcmc) values were determined and are summarized in Table 4. The CMC indicates the concentration at which surfactant molecules begin to form micelles. The obtained results confirmed that the amido-amine–based cationic surfactants possess low CMC values, which decrease with increasing hydrocarbon chain length (Tables 4 and 5).[9] In addition, all compounds exhibited surface tension values below 35 mN/m at a concentration of 0.1 g/L, indicating excellent surface activity.

Contact angle. The static equilibrium contact angle was measured on hydrophobic steel and glass plates to evaluate the wetting behaviour of the surfactants.[10] The difference in the advancing angles between the two surfaces reflects the surface homogeneity of the adsorbed film. The results of drop-spreading kinetics on both substrates are summarized in Table 4**.**

Wetting time. Circular canvas discs (1 inch diameter, 0.44 g average weight) were floated on 1% *w/v* surfactant solution and the wetting time was recorded using a stopwatch.[11] The time required for complete sinking of the disc was noted as the wetting time. Results are presented in Table 6.

Foamability. Foamability was evaluated using a 100 mL stoppered cylinder containing 20 mL of 1% (*w/v*) surfactant solution (5a–5e). The cylinder was shaken 50 times, and the foam volume was recorded at 0, 5, 10, and 30 minutes.[12] The foaming results are summarized in Table 6.

Antibacterial Activity of Synthesized Compounds. A gram-positive *Staphylococcus aureus* and a gram-negative strain *Escherichia coli* were used for antibacterial activity analysis of the cationic surfactant. An inoculum (1 mL) containing approximately 10^9 CFU mL^{-1} of each test organism was spread on nutrient agar plates in triplicate. Wells of 4 mm diameter were prepared using a sterile steel borer. One well served as a blank (water), while 70 µL of the surfactant solution (prepared in distilled water) was added to the others. Plates were incubated at 37 °C for 24 h, and the zones of inhibition were measured.[13] The mean inhibition diameter, corrected for the blank, represented the antibacterial activity. Results are presented in Table 7.

Table 4

Critical Micelle Concentration (cmc), Surface tension (γ),
Contact angle of amines 4a–4e determined by the Wilhelmy plate method (11) at 25 °C

Surfactant Code	CMC (mM/L^{-1})	Surface Tension (γ, mN m^{-1})	Θa	Θgp	Θsp
4a	0.004	25.49	–	54	53
4b	0.020	25.08	–	48	53
4c	0.009	25.00	–	44	56
4d	0.006	25.00	–	52	58
4e	0.002	25.56	–	41	62

Θa – advancing angle; Θgp – on glass plate; Θsp – on steel plate

Table 5

Critical Micelle Concentration (cmc), Surface tension (γ), Contact angle of amines 5a–5e determined by the Wilhelmy method at 25°C

Surfactant Code	CMC (mM/L^{-1})	Surface Tension (γ, mN m^{-1})	Θa	Θgp	Θsp
5a	0.002	35.49	–	32	57
5b	0.020	23.92	–	31	54
5c	0.0013	29.24	–	51	56
5d	0.0010	33.08	–	53	72
5e	0.004	25.97	–	30	56

Θa – advancing angle; Θgp – on glass plate; Θsp – on steel plate

Table 6

Foamability and wetting time of quaternary ammonium surfactants (5a–5e)

Surfactant	Concentration (% w/v)	Wetting time(s)	Foam Volume (mL)			
			0 min	5 min	10 min	30 min
5a	0.5	152	25	20	20	20
	0.25	21	20	–	–	–
	0.125	10	Almost no foam			
5b	0.5	165	23	22	22	21
	0.25	6	20	–	–	–
	0.125	6	Almost no foam	–	–	–
5c	0.5	165	22	21	20	20
	0.25	5	25	22	21	20
	0.125	30	22	–	–	–
5d	0.5	150	22	21	21	–
	0.25	13	22	22	22	21
	0.125	6	21	–	–	–
5e	0.5	130	65	54	45	36
	0.25	5	55	48	47	46
	0.125	8	25	23	23	22

Table 7

Result of antibacterial activity tested compounds (the values of inhibition zone are mentioned on the 'mm'scale)

Surfactant	Gram-negative	Gram-positive
	Escherichia coli (mm)	*Staphylococcus aureus*(mm)
5a	9	12
5b	9.5	13
5c	10	10
5d	11	15
5e	13	17

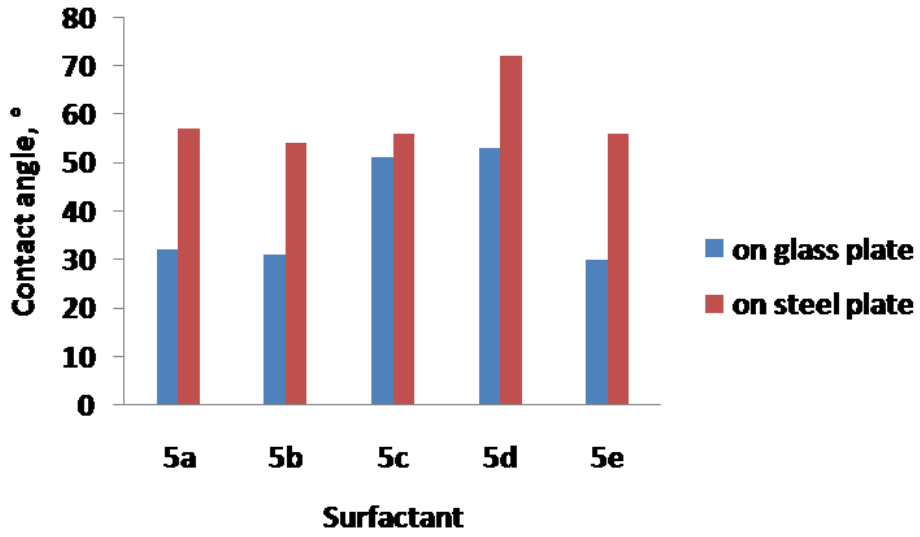

Fig. 4 – Comparison of contact angles (θ) for quaternary ammonium surfactants (5a–5e) measured on glass and steel plates at 25 °C.

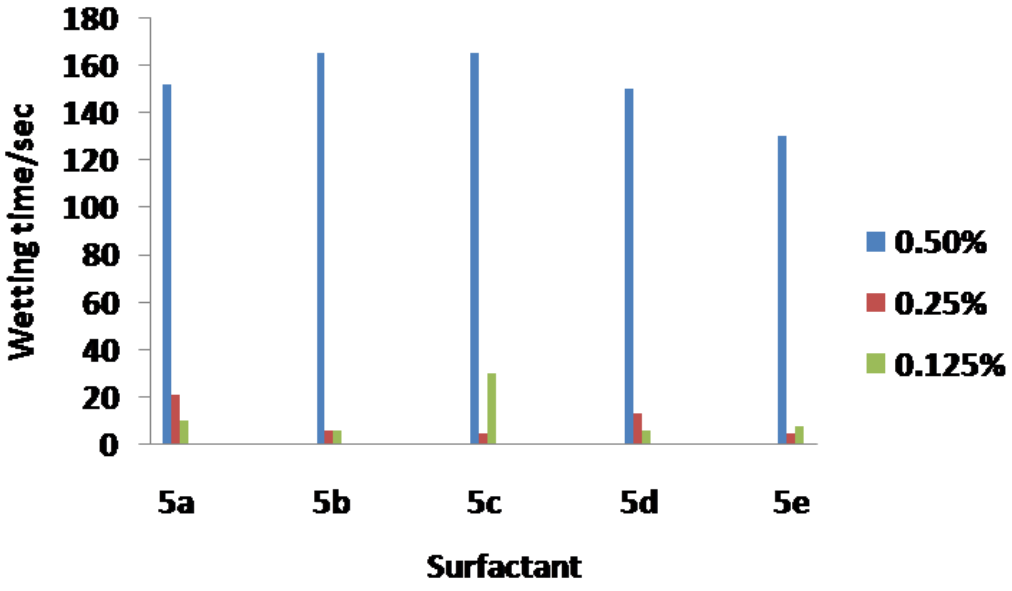

Fig. 5 – Wetting time of quaternary ammonium surfactant solutions (5a–5e) at different concentrations.

Instrumental Analysis. FT-IR spectra of synthesized cationic surfactant and cationic content were obtained. FT-IR spectra were obtained by using a model Miracle 10. DRS plate of the sample was subjected to analysis with 30 scan.

RESULTS AND DISCUSSION

Synthesis of cationic surfactant

Lactic acid-based cationic surfactants were synthesized in three steps (Fig. 1). The first step involved the amidation of lactic acid with *N,N*-dimethyl-*N*-propylamine, followed by esterification with fatty acids and quaternization with benzyl chloride. The best yield and cationic content were obtained using a 1:1.5 molar ratio of benzyl chloride to ester derivatives (Table 2). The products showed good solubility in water due to the presence of polar amide and ester groups. All synthesized compounds were obtained in moderate to good yield and confirmed by FT-IR (Table 3).

FT-IR Analysis

FT-IR spectra confirmed ester and amide formation by characteristic bands at 3373, 1641, and 1738 cm^{-1}. The quaternized surfactants showed additional peaks at 1578, 719, and 702 cm^{-1} due to aromatic C=C and C–H deformation, along with a band at 1464 cm^{-1} attributed to methyl stretching of the quaternary ammonium group.[14, 15]

Surface active properties of synthesized compound

Surface tension decreased with increasing alkyl chain length (C_8–C_{18}), while CMC values were low for all surfactants (5a–5e), indicating strong surface activity (Tables 4 and 5). The high molecular weight surfactants formed micelles rapidly and are suitable for mild formulations.

Contact angle and Wetting action

Contact angle studies showed that compound 5e spread rapidly on glass and steel surfaces, giving the lowest surface tension (26.5 mN/m) and best wetting performance. Canvas disc tests confirmed that 5b, 5c, and 5e exhibited excellent wetting efficiency (Tables 4–6, Fig. 5).

Foamability

Foaming properties of the synthesized surfactants were evaluated at varying concentrations. As shown in Fig. 6 and Table 6, surfactants 5b, 5c, and 5d displayed lower foaming power, whereas 5e exhibited the highest and most stable foam. Foam stability increased with the length of the hydrocarbon chain, and the overall foaming ability followed the trend: 5e>5d>5b>5c>5a, consistent with the surface tension data. The superior foaming and stability of compound 5e make it suitable for formulations such as shampoos and liquid soaps, where rich and stable foam is desirable.

Antibacterial Activity of Synthesized Compounds

The antibacterial activity of the synthesized cationic surfactants was evaluated against Gram-positive (Staphylococcus aureus) and Gram-negative (Escherichia coli) bacteria, and results are presented in Table 7.[16] Compound 5e exhibited the strongest antibacterial activity, particularly against *S. aureus*. The enhanced activity is attributed to hydrogen bonding interactions and the longer alkyl chain**,** which enhances membrane permeability and disruption.[17]

Overall, the synthesized cationic surfactants showed higher antibacterial activity against Gram-positive bacteria than Gram-negative bacteria. This difference arises from the outer membrane structure of Gram-negative bacteria, which is rich in lipopolysaccharides and proteins that restrict the penetration of amphiphilic molecules.[18] The antibacterial efficiency increased with increasing hydrocarbon chain length, following the order: 5e>5d>5b>5c>5a, as summarized in Table 7.[19]

CONCLUSION

Lactic acid-based long-chain cationic surfactants were successfully synthesized in three steps and exhibited remarkable surface-active and antibacterial properties. The synthesized quaternary ammonium salts showed low surface tension, low CMC values, good wetting ability, and high foam stability. Among all, compound 5e demonstrated the best overall performance, showing strong wetting, stable foam formation, and excellent

antibacterial activity. The antibacterial tests revealed higher activity against Gram-positive bacteria, consistent with the known structural differences in bacterial membranes. The developed cationic surfactants can thus be considered eco-friendly, biodegradable, and effective candidates for use in personal care products, detergents, and antimicrobial formulations.

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