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Synthesis and Evaluation of heterocyclic Cationic Surfactants

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Abstract

Benzoquinazoline derivatives were synthesized and chemically modified to enhance their antimicrobial activity and performance as cationic surfactants. The target compounds were obtained via conversion to *N*-(3-(dimethylamino)propyl)-3-(4-oxo-4*H*-benzo[*d*][1,3]oxazin-2-yl)propanamide, followed by quaternization with fatty alkyl 2-chloroacetates. The synthesized cationic surfactants possess a quaternary ammonium hydrophilic head and hydrophobic alkyl chains, The hydrophobic head of an organic heterocyclic surfactant component attracted polar water, whereas the hydrophobic tail attracted non-polar solvents like oil. As a result, they dissolve in both polar and nonpolar liquids and are amphipathic. This characteristic has been used to produce surfactants as emulsifiers, solubilizers, and wetting agents. giving them amphipathic character. They effectively decrease surface and interfacial tension, show low values of the critical micelle concentration (CMC). and demonstrate superior aggregation behavior at low concentrations. These properties make the prepared compounds economically attractive and suitable for industrial applications. In particular, they show favorable surface activity as effective emulsifying agents for cosmetic formulations.

Keywords: Benzoquinazolines, Cationic surfactants, Emulsifying agent.

1. Introduction

Quinazolines are a significant type of nitrogen-containing heterocyclic compounds consisting of two fused six-membered rings: a pyrimidine ring and a benzene ring. **(Bala, I.A *et al.*, 2024; Nandwana, N.K *et al.*,2024; Pal, R *et al.*,2023)**. Fusion with the The benzene ring considerably modifies the physicochemical properties of the pyrimidine moiety. The nature and position of substituents on either ring strongly influence the chemical and biological behavior of quinazoline derivatives **(Hassan RM *et al.*,2025; Krapf *et al.*,2017; Mravljak *et al.*,2021)**.

Quinazoline and its derivatives are widely recognized as important scaffolds in medicinal chemistry because of their wide range of biological activities, which include antiviral, anticancer, and antibacterial properties., anti-inflammatory, antidiabetic, and anticonvulsant properties **(Bala, I.A *et al.*, 2024; Ghoneim, Mohammed M., *et al.*2024)**.

Surfactants are amphiphilic substances made up of polar, hydrophilic head and a nonpolar, hydrophobic tail **(Shaban SM *et al*., 2020; Harif S *et al*., 2023; Sakač N *et al*., 2023)**. Surfactants are categorized as nonionic, anionic, cationic, or amphoteric based on the charge of the head group. **(Sakač N *et al*., 2023; Opoku Appau P *et al*., 2022; Belhaj AF *et al*., 2020; Farias CB *et al*., 2021; Brycki BE *et al*., 2017; Salman AA(2022))**.They play a vital role in environmental protection by facilitating the elimination of contaminants such as industrial wastes,

pesticides, heavy metals, dyes, pharmaceutical residues, and oil spills, which pose serious risks

to ecosystems and human health. The cost of traditional cleanup techniques is frequently a barrier., efficiency, or technological constraints, whereas surfactant-based approaches offer effective alternatives**(Saini, R. V *et al*., 2023)**. Heterocyclic structures are common in many pharmacologically active compounds and are increasingly exploited in cosmetic and industrial formulations **(Guin M, *et al*., 2022**). Surfactants are indispensable in numerous applications, including emulsification, dispersion, cleaning, wetting, foaming, and defoaming **(Sakač N *et al*., 2023; Guin M, *et al*., 2022)**. Natural surfactants are particularly attractive due to their low toxicity, biodegradability, environmental compatibility, and enhanced thermal stability compared with synthetic analogues **(Guin M, *et al*., 2022; Saxena N *et al*., 2018; Molnár-Kiss AV& Nagy R., 2023)**.Cationic surfactants are organic compounds capable of forming positively charged species in aqueous solutions, either permanently or in a pH-dependent manner. The most common cationic surfactants contain a quaternary ammonium group as the hydrophilic head linked to one or two hydrophobic alkyl chains. Functional groups that include nitrogen, such amines, quaternary ammonium, and imidazolium, and pyridinium moieties are central to their structure and activity **(Honciuc A.,2021)**. Despite their strong antimicrobial properties, conventional quaternary ammonium surfactants often exhibit low biodegradability and membrane-disruptive toxicity. Consequently, current research focuses on developing more selective and less toxic antimicrobial cationic surfactants, particularly through the incorporation of amide or amino acid functionalities into the polar head group **(Pérez L *et al*., 2022)**.

In this work, the synthesis of a novel quinazoline-based cationic surfactant is described. The prepared compound was characterized by FT-IR spectroscopy, and its surface-active properties were systematically investigated.

2. Experimental section

2.1. Materials and instruments

All chemical was used without further purification. succinic anhydride, anthranilic acid,1-octanol, and chloroacetic acid were purchased from Sigma-Aldrich., N,N-dimethyl propane diamine and We bought petroleum ether (40–60 °C) from Fisher Scientific UK.. *Para*-toluene sulphonic acid (PTSA) was obtained from Loba Chemie, anhydrous sodium sulphate, sodium carbonate Al-Nasr Chemical Company supplied acetic anhydride, glacial acetic acid, paraffin oil, and benzene; Biochem Company supplied tetrahydrofuran. The Nicolet is 10 FT-IR spectrophotometer was used to perform FT-IR (ATR or KBr disc).

2.2. Synthesis

Cationic surfactant was prepared and the synthetic procedures were explained in the following reactions through four main steps.

2.2.1. Synthesis of 3-(4-oxo-4H-benzo[*d*][1,3]oxazin-2-yl)propanoic acid (A).

A mixture of succinic anhydride (4 g, 0.04 mol) and anthranilic acid (5.4 g, 0.04 mol) in glacial acetic acid (GAA) (20 mL) was refluxed for five hours.

The residue was recrystallized in acetone after the solvent was evaporated, producing 2-(3carboxypropanamido) The crystal of benzoic acid 3 is yellow in color. The To obtain 3-(4-oxo-4H-benzo[*d*][1,3]oxazin2-yl), the resultant carboxylic acid 3 (2 g, 0.0084 mol) was heated in acetic anhydride (8 mL) for an hour at 130–140 ^{o}C propanoic acid.. The gas was then condensed using a vacuum pump (**Khodarahmi GA *et al*., 2022**)**.**

2.2.2. Synthesis of *N*-(3-(dimethylamino)propyl)-3-(4-oxo-4H-benzo[*d*][1,3]oxazin-2-yl)propenamide (B).

A mixture of previously prepared acid (1gm, 1equiv.) and N,N dimethyl propane diamine (0.5mL, 1equiv.) was dissolved in dry benzene. *p*-toluenesulphonic acid (PTSA, 1 mol%) used as a catalyst, After that, Dean-Stark refluxed the mixture while stirring it at a temperature of roughly 170 °C until one equivalent of water was formed. The synthesized amide was extracted using petroleum ether, the organic phases were dried over anhydrous Na_2SO_4, and the solvent was vacuum-evaporated until it was completely dry.**(Sabatini MT *et al*., 2017)**.

2.2.3. General procedure for the synthesis of fatty alkyl 2-chloroacetates (octyl 2chloroacetate) (C).

Alkyl 2-chloroacetates were produced, the corresponding fatty alcohol (1 equiv.) namely,1-octanol, was mixed separately to chloroacetic acid (1 equiv.)in drybenzene. *P*toluene sulphonic acid (PTSA, 1 mol%)used as a catalyst, After that, Dean-Stark refluxed the mixture while stirring it at a temperature of roughly 170 °C until one equivalent of water was formed. Petroleum ether was used to extract the produced fatty alkyl 2-chloro-acetate, the organic phases were dried over anhydrous Na_2SO_4, and the solvent was vacuum-evaporated until it was completely dry. (**Abo-Riya M *et al*., 2016**).

2.2.4. Synthesis of the target cationic surfactants (3,3-dimethyl-1-(octyloxy)-1-oxo-6-(3-(4oxo-4H-benzo[*d*][1,3]oxazin-2-yl)propanamido)hexan-2-aminium chloride) (D).

A mixture of obtained amide (1gm, 0.01 mol) was taken to reflux with a suitable amount of fatty esters (0.9 mL, 0.01 mol) produced from the third step using tetrahydrofuran (THF) as a solvent for a long time nearly 90 hours at 70 °C with continuous stirring to yield the targeted cationic surfactant. This reaction is put in a round-bottom flask, After the reaction was finished, sample was saved till the evolution of THF then, washed by diethyl ether many times for further purification of the final synthesized cationic surfactant. FT-IR analysis have been utilized to verify the synthetic cationic surfactants' structures **(Shaban SM *et al*., 2020)**.

Brown fatty sample, yield=60% , The predicted functional groups FT-IR (ATR) spectra, which show bands at 3364 cm^{-1} (NH&NH2), 2925 cm^{-1} (CH, aliphatic fatty chain), 1744 cm^{-1} (C=O ester), 1630 cm^{-1} (C=O Amide), and 1210, 1112 cm^{-1} (C-O, C-N), confirm the produced surfactants.

^{1}H-NMR (400 MHz, DMSO-d6) spectra revealed signals at δ = 8.49 ppm. (NH, exchangeable by D_2O), (8,26-6,46) ppm (Aromatic H of benzo[1,3] oxazine ring, 4,82 ppm ($CH\text{-}NH_3^+$), 4.06 ppm (CH_2 -O), 3,71 ppm (CH_2-NH), 3.36 ppm (CH-C=O), 1,81 ppm (CH_2), 1,78 ppm

(dimethyl 2CH_3), 0.86 -ppm (CH_2 Alkyl chain -CH_3).

2.3. Surface properties

a) Surface tension measurements

One crucial characteristic of liquids is surface tension. Water's surface tension can be reduced using surfactants., which is 72 mN.m-1 for distilled water. Surface tension values of 0.1 % (wt.) of surfactant solutions were determined using capillarity method. the obtained values were measured at room temperature **(Asadov ZH *et al*., 2020)**. the values of surface tension are tabulated at Table-1.

b) Emulsion stability

Powerful shaking of 10 mL solution of surfactant (0.1% wt.) with 5 mL of paraffin oil mixed in 25-mL graduated cylinder for two min at 25℃ then, measuring the consumed time to separate the aquas phase (nearly 9 mL) from the emulsion and determined as the emulsion stability. Increasing the time that is required to separate the water indicates the stability of the emulsion formed (**El-Dougdoug WI *et al*., 2015; Alazrak SM *et al*., 2021; De S, Malik S *et al*., 2015; El-Dougdoug WI,(2020**)).

c) Foam Height

A solution containing 01% by weight of a surfactant was prepared. A 500 mL graduated cylinder was filled with 100 mL of this solution. At a temperature of 25 °C, the cylinder was briefly shook. After shaking, the height of the foam that developed was measured in millimeters. (**Belhaj AF *et al*., 2020;El-Dougdoug WI *et al*., 2015; Alazrak SM *et al*., 2021**).

(d) Stability to hydrolysis

Ten milliliters of sulfuric acid 2N or 0.05N were combined with ten milliliters of surfactant solution (0.1 weight percent). of sodium hydroxide placed in a thermostat system at 40 ℃. The solution's stability against hydrolysis was measured by the duration before it turned turbid or opaque (**Belhaj AF *et al*., 2020; El-Dougdoug WI,(2020**)).

Critical micelle concentration (CMC)

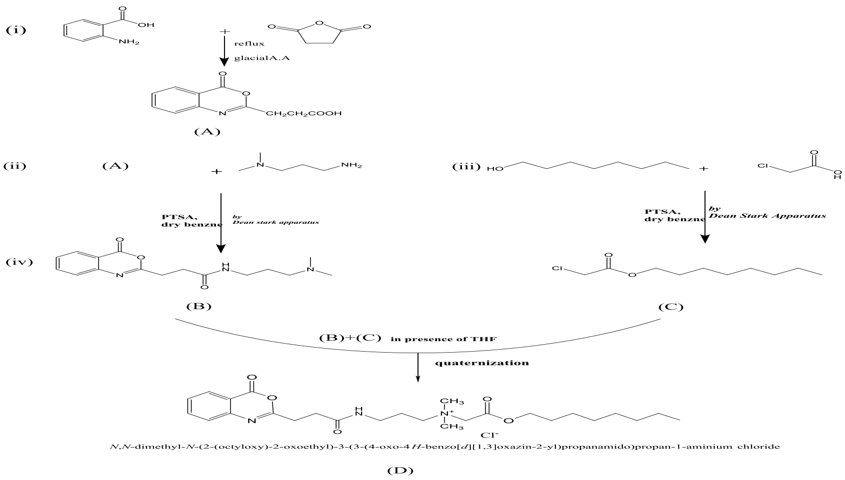
A freshly prepared surfactant solution of the synthesized Gemini cationic surfactant using distilled water was prepared many times with various molar concentrations at 25℃ to determine CMC of surfactants using surface tension method **(Alazrak SM *et al.*, 2021)**.

3.Result and Discussion

3.1. Synthesis

Due to their diverse applications and valuable physicochemical properties, cationic surfactants are highly important compounds. In this work, a cationic surfactant was synthesized from a 1-octanol fatty ester (C) according to the chemical sequence shown in Scheme 1. Initially, 1-octanol was reacted with chloroacetic acid to yield the corresponding fatty acid derivative (A) (**Abo-Riya M *et al.*, 2016**). The resulting acid was then converted into amide (B) by reaction with *N,N-dimethylpropyldiamine* (**Sabatini MT *et al.*, 2017**). In the final step, fatty ester (C) was quaternized with *N-(3-(dimethylamino)propyl)-3-(4-oxo-4H-benzo[d][1,3]oxazin-2-yl)propanamide* (B) to afford the target cationic surfactant (D) (**Shaban SM *et al.*, 2020**).

FT-IR and ^{1}H NMR were used to confirm the synthesized surfactant's structure spectroscopy, and further verified by D_2O exchange through characteristic absorption signals.



Scheme.1. Synthetic rout of cationic surfactant of 1-ocetanol.

3.2. Surface properties

Table 1 demonstrates that the synthesized cationic surfactant exhibits pronounced surfaceactive properties, as evidenced by its capacity to lower water's surface tension. As surfactant molecules move from the bulk solution to the air-water mixture, this decrease takes place interface, where the hydrophobic tails face away from the water phase..

Table.1- surface properties of the produced cationic surfactant:

Compound No.	Foam h. 0.1% (mm)	Emu. Stab. 10 mmole (min:sec)	Stab. to hyd. acid (min:sec)	Stab. to hyd. base (min:sec)	CMC*10^{-3} (mol/L)
CS1	250	6	40:00	20:00	0.025

Emulsion stability

Emulsion stability, described as the capacity of a surfactant to efficiently form and maintain a stable dispersion of immiscible phases over time, is a critical parameter for its use as an emulsifying agent in various formulations **(Saxena N *et al.*, 2018)**. This property reflects the surfactant's capacity to reduce interfacial tension and prevent droplet coalescence. The synthesized surfactant exhibits strong emulsifying efficiency and good emulsion stability, indicating its potential for incorporation into cosmetic formulations, where long-term stability, safety, and performance are essential.

Foam Height

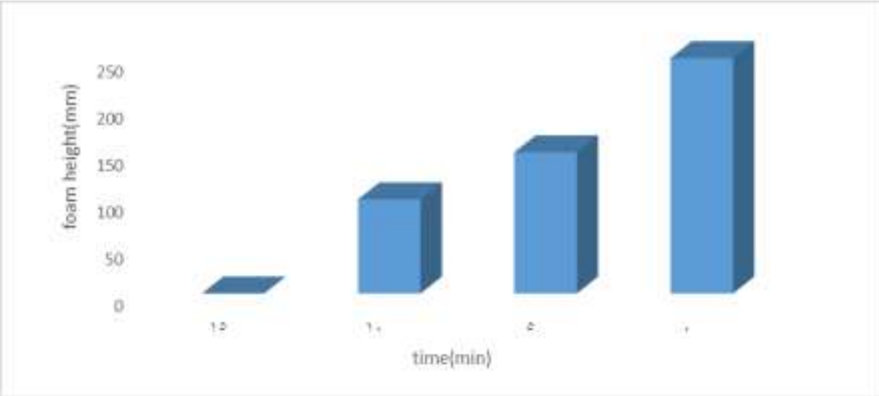


Fig.1 Foam stability for cationic surfactant.

The synthesized cationic surfactant demonstrates a high foaming height, reflecting its strong foaming capacity and excellent air–liquid interfacial activity. Such behavior arises from Surfactant molecules' adsorption at the air-water interface, where the hydrophobic tails stabilize air bubbles and prevent their rapid collapse. This property is particularly important for applications in detergents, washing formulations, and personal care products, where consistent foam formation enhances cleaning efficiency, product performance, and user perception. The high foaming ability, combined with its low critical micelle concentration and surface activity, underscores the surfactant's potential for diverse industrial and commercial applications.

Stability toward hydrolysis:

The stability to hydrolysis of the synthesized cationic surfactant was investigated under both acidic and alkaline conditions to assess its chemical robustness and potential applicability in various formulations. As summarized in Table 1, the surfactant undergoes rapid hydrolysis in basic media, which is primarily due to the cleavage of the ester linkage in its molecular structure. This base-catalyzed hydrolysis results in the breakdown of the surfactant into its constituent acid and alcohol derivatives, limiting its long-term stability under strongly alkaline conditions. In contrast, the surfactant demonstrates considerable stability in acidic media, maintaining its structural integrity and functionality. The observed acid resistance indicates that the cationic surfactant is suitable for applications where exposure to mildly acidic environments is expected, such as in cosmetic and pharmaceutical formulations.

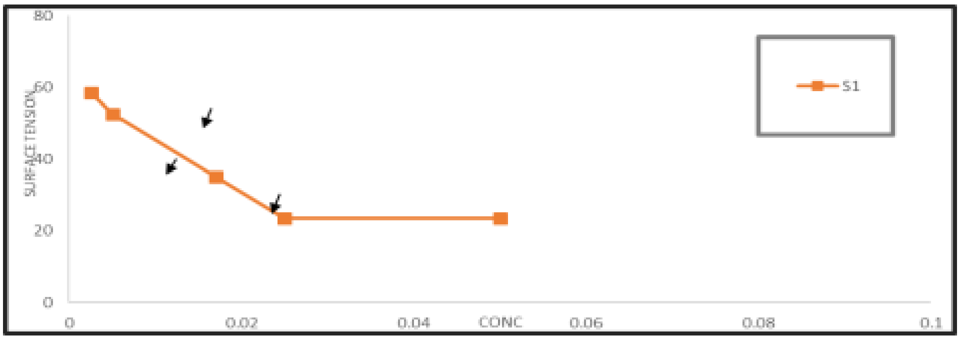


fig .2 Relation of surface tension with cationic surfactant concentration in aquas solution at 298 K.

Critical micelle concentration (CMC)

One crucial factor is For the purpose of describing surfactant solutions, the critical micelle concentration (CMC) defines the concentration at which Surfactant molecules go to the interface from the bulk solution, saturating it, and begin to self-assemble into micelles. This aggregation significantly influences The capacity of the surfactant help stabilize emulsions, dissolve hydrophobic materials, and reduce surface and interfacial tension.. The CMC can be determined experimentally from the breakpoint in the Surface tension vs surfactant concentration ($mol \cdot L^{-1}$) plot where the slope changes sharply, indicating the onset of micelle formation. A low CMC is highly desirable because it indicates that the surfactant can achieve effective surface activity at minimal concentrations, enhancing economic efficiency and reducing environmental impact. As shown in Table 1, the synthesized cationic surfactant exhibits a low CMC,highlighting its strong tendency to form micelles even at low concentrations. This property not only ensures efficient reduction of surface tension but also contributes to its excellent foaming capacity, emulsion stabilization, and potential antimicrobial activity. Such behavior makes the surfactant highly suitable for a wide range of industrial, cosmetic, and pharmaceutical applications, where minimal surfactant usage and maximal performance are critica .

4. Conclusion

Anovel cationic surfactant was successfully synthesized using 1-octanol, a naturally occurring fatty alcohol with eight carbon atoms, as the initial source. The surfactant's chemical structure was verified using FT-IR spectroscopy. Its surface-active properties were systematically evaluated, including surface and interfacial tension reduction, foam height, emulsion stability, and hydrolytic resistance under acidic and basic conditions.

The findings show that, in contrast to micelle production in the bulk solution, the surfactant exhibits substantial adsorption at the air–water interface and increased interfacial activity.. It possesses excellent foaming properties, making it suitable for industrial applications such as detergents and washing formulations. Furthermore, the surfactant shows high emulsifying capacity, highlighting its potential for cosmetic and personal care products. Hydrolytic studies reveal that the surfactant is more stable under acidic conditions, taking longer to hydrolyze, whereas it undergoes rapid hydrolysis in basic media due to the presence of an ester functional group.

Overall, the synthesized cationic surfactant combines high surface activity, strong emulsifying and foaming abilities, and selective hydrolytic stability, making it a promising candidate for a wide range of industrial, cosmetic, and pharmaceutical applications.

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