



Adsorption and micellization behavior of synthesized amidoamine cationic surfactants and their biological activity



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ABSTRACT

The adsorption and micellization behavior at aqueous surface of synthesized amido-amine cationic surfactants was studied using surface tension and conductometric measurements at three different temperatures of 25, 40 and 60 °C. The chemical structure of the synthesized cationic surfactants was confirmed using Fourier transform infrared and proton nuclear magnetic resonance spectroscopy. The synthetic route is simple and comprises two steps. The first is amidation between palmitic acid and dimethyl amino propyl amine, while the second step is quaternization of the first step product with different alkyl bromides. The study evaluated the effect of temperature and the hydrophobic chain length of the synthesized amidoamine cationic surfactants on the studied surface parameters. In aqueous system the adsorption tendency of the synthesized cationic surfactants is higher than micellization and both increase by increasing the hydrophobic character and the solution temperatures. The synthesized surfactants were found to have good antibiotic effect against gram positive and negative bacteria and fungi.

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1. Introduction

The adsorption and micellization of surfactants at the water–air interface have an effect on the properties of the water. The adsorption of the surface-active agent on the surface reduces the surface tension, which is the major factor for wider applications of surfactants [1–7]. Adsorption is the key factor for various applications like corrosion inhibitors (increasing adsorption, the inhibition efficiency, increase) and as capping agent in nanotechnology [8–10]. The opposing two parts of surfactants are the key factor for the adsorption at interfaces and aggregates in the bulk solution. Studying the physicochemical of the synthesized surfactants at the surface is very important in determining the best application, in which they may be used. Since most surfaces and natural colloid are often negatively charged, so the cationic surfactants provide a strengthened adsorption layer [11]. There are many papers that reported the biological activity of commercially available cationic surfactants like N-alkyltrimethyl ammonium halides (CTAB, TTAB, DTAB, CTACl) [12,13] and N-alkyl pyridinium such as cetyl or dodecyl pyridinium chloride (CPCI, DPCL) [14] and alkyl dimethyl benzyl ammonium surfactants (benzalkonium and benzethonium) are other examples of commercially produced surfactants investigated and widely used as disinfectants in hospitals [15]. Commercial twin-chain

surfactants, capable of forming vesicles, as dialkyl dimethyl ammonium halides (DDACl, DDAB and DODACl), were also studied [16]. In our study, we prepared cationic surfactants with twin chain and containing an amide functional group, which enhances its biodegradability, and reducing its aquatic toxicity. Intermediates produced in the degradation process of amide were found to be biodegradable and less toxic [17]. The microorganism gained self-immunity from conventional antibiotic, so the researchers focused on searching new antibiotic like surfactants. The research aimed to prepare cationic surfactant containing twin chain and amide group from the low price material. The physicochemical and thermodynamic of the aqueous solution of surfactants were determined from surface tension and conductance measurements at three different temperatures. The antibiotic effect of the synthesized cationic surfactants was determined using filter paper disk agar method against gram positive and negative bacteria and fungi.

2. Materials & experimental

2.1. Materials

Dimethylaminopropylamine, hexadecanoic acid and 1,3-dibromo propane were purchased from Sigma Aldrich Company. Decyl bromide, dodecyl bromide and hexadecyl bromide were purchased from Merck Company and used as it without any purification. All the used organic solvents were purchased from Algomhoria Chemical Company.

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2.2. Synthesis of amidoamine cationic surfactants

2.2.1. Synthesis of *N*-(3-(dimethylamino) propyl) palmitamide

The first step is amide formation through reaction of 0.03 mol from palmitic acid (7.7 g) with 0.1 mol from dimethylamino-1-propylamine (3.06 g) in 130 mL toluene. *p*-Toluene sulphonic acid (0.01%) was added as dehydrating agent to the reaction mixture. The reaction was stopped after complete removal of the reaction water (0.03 mol, 0.54 mL) using Dean–Stark system. The solvent was removed using vacuum rotary evaporator. The catalyst was extracted from the reaction medium using petroleum ether. Subsequent purification was done by means of vacuum distillation to remove the excess and residual materials [18].

2.2.2. Synthesis of *N*-(3-(dimethyl alkyl ammonio) propyl) palmitamide bromide derivatives

0.01 mol from the synthesized amide in the first step (3.41 g) was refluxed with 0.01 mol from the alkyl halides decyl bromide (2.21 g), dodecyl bromide (2.49 g), and hexadecyl bromide (3.05 g) separately in the presence absolute ethyl alcohol as a solvent for 25–30 h depending on the alkyl halide. After evaporating the absolute alcohol, the residual was purified with diethyl ether. The obtained product labeled DMOPP, DMDPP and DMHPP for cationic surfactant with decyl, dodecyl and hexadecyl alkyl chain respectively. The synthetic routes are represented in Scheme 1.

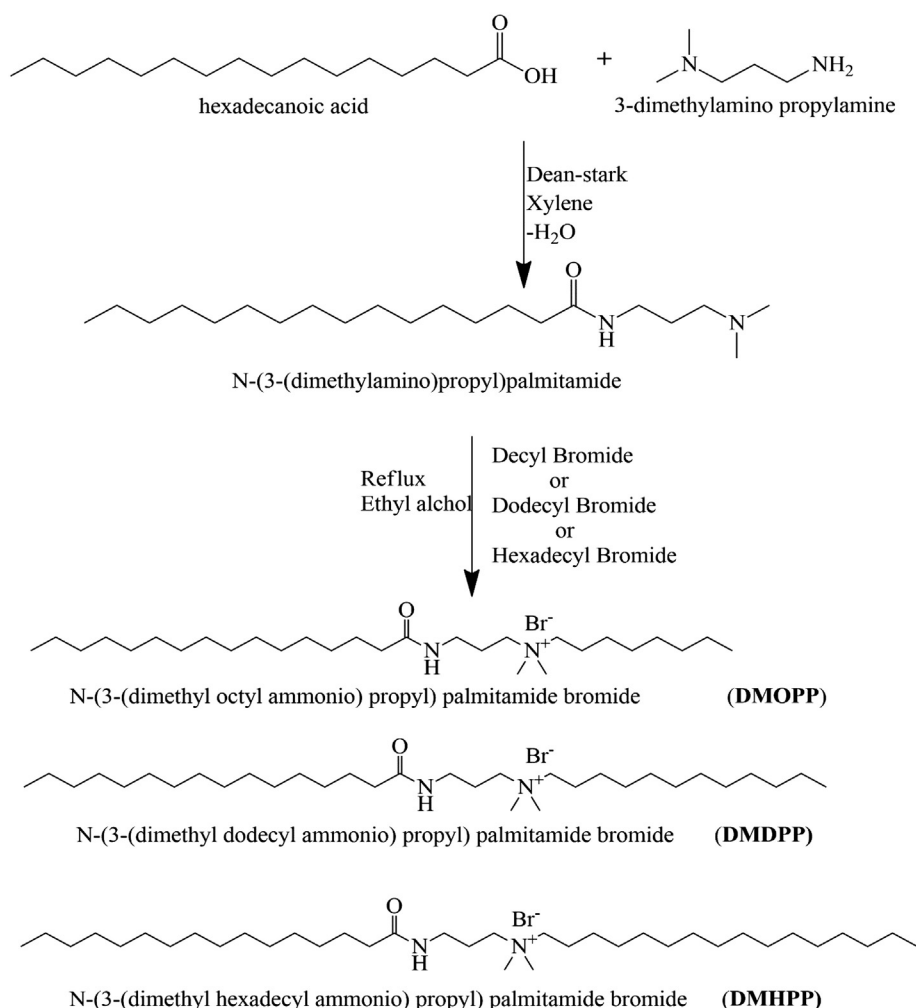
2.3. Structure confirmation

The synthetic routes of novel cationic surfactants were trappable by Fourier transform infrared (FTIR) and proton nuclear magnetic resonance spectroscopy ($^1\text{H NMR}$). The FTIR analysis was done in Egyptian Petroleum Research Institute using ATI Mattsonm Infinity Series™, Bench top 961 controlled by Win First™ V2.01 Software while $^1\text{H NMR}$ was done in National Research Institute using GEMINI 200 (^1H 200 MHz) in DMSO-d_6 .

2.4. Measurements

2.4.1. Surface tension measurements (γ)

The surface tension of aqueous solution of the novel amidoamine twin chain cationic surfactant were measured by a platinum ring detachment method using a K6 Krüss (Hamburg, Germany) tensiometer at three different temperatures 25, 40 and 60 ± 0.1 °C. The accuracy of the measurements was ± 0.5 mN·m $^{-1}$. The platinum ring was cleaned before each measurement with diluted chromic acid mixture solution and washed with double distilled water. Each concentration was measured three times and the average was recorded and used without correction. The critical micelle concentration (CMC) was determined from the break point in surface tension (γ) versus $[\log c]$ plots [19].



Scheme 1. Synthetic route of novel cationic amidoamine surfactants.

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