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Full Length Article

4-Aminoantipyrine derived cationic surfactants: Synthesis, characterization, surface activity and screening for potential antimicrobial activities ☆

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ABSTRACT

A series of cationic surfactants were synthesized from chemical modification of 4-aminoantipyrine. The chemical structures were confirmed using infrared (FTIR) and nuclear magnetic resonance (^{1}H NMR) spectroscopy. The surface activities of the surfactants in their solutions were determined by surface tension measurements. Increasing the hydrophobic chain length increased the surface activity of the surfactants in solutions. Measurements of interfacial tension between surfactants solutions and light paraffin oil showed that the interfacial tension values were decreased with increasing the alkyl chain length. The emulsion stability measurements showed the applicability of these surfactants as emulsifying agents. The foaming power measurements showed the synthesized surfactants have low ability for foam formation. Thermodynamic properties of adsorption and micellization processes showed their tendency towards adsorption at solution interface and micellization in their solutions. Results showed reasonable surface activities compared to conventional cationic surfactants. The antimicrobial activity of the synthesized surfactants against Gram-positive bacteria (*Bacillus subtilis* and *Staphyl. aureus*, Gram-negative bacteria (*Pseudomonas aeruginosa* and *E. coli.*) and Fungi (*Candida albicans* and *Aspergillus niger*) was evaluated. The efficiency of these compounds as antimicrobial agents) was increased with the hydrophobicity and carbon chain length.
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1. Introduction

Schiff bases of 4-aminoantipyrine are known for their variety of applications in the area of catalysis [1,2], clinical applications [3], and pharmacology [4]. New kinds of chemo-therapeutic agents containing Schiff bases have gained significant attention among biochemists [1] and of those aminopyrines are commonly administered intravenously to detect liver disease in clinical treatment [3].

The increasing microbial resistance to antibiotics in use nowadays necessitates the search for new compounds with potential effects against pathogenic bacteria and fungi. The most spectacular advances in medicinal chemistry have been made when heterocyclic compounds played an important role in regulating biological activities. Heterocyclic moieties can be found in a large number of compounds which display biological activity. Antipyrine (N-heterocyclic compound) and its derivatives exhibit a wide range of biological activities and applications [4–6]. Antipyrine is a marker in the study of transfer and biotransformations of drugs in the human body [7] antipyrine metabolites are reported to show a positive correlation with plasma fibronectin level in monitoring patients with chronic liver illness (HBC, HCV and alcohol-related disease) [8].

Cationic surfactant characterized by two different parts one part is water soluble and has hydrophilic character in solution where another part is oil soluble and has hydrophobic character. One of the most important properties in solution is the formation of the critical micelle concentration (CMC) at high concentration and above which, higher concentration, of this concentration in aqueous solution are known to form different micelle forms such as spherical, vesicular and rod-like [9–13]. Several factors effect on the microstructure of surfactant micelleization such as structure of the surfactants, concentration and temperature [14–17]. Cationic surfactant or Quaternary ammonium salts compound show antimicrobial activity against Gram positive and negative bacteria, yeast and fungi. Most of the quaternary ammonium salts have positive nitrogen atoms in their chemical structure. Nitrogen atom is hetero atom and carries positive charge which leads to the enhancement the antimicrobial activity [18]. Several works deal

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with the synthesis of different cationic surfactant compounds and study the relationship between surface activity and antimicrobial activity against wide strain of pathogenic bacteria, fungi and yeast, have been published [19–21].

The objective of this study was to synthesize cationic surfactants derived from 4-aminoantipyrine with different hydrocarbon chain length and investigate the influence of the structure on surface tension, interfacial tension, emulsification power, foaming power, thermodynamic parameters of adsorption/micellazation and antimicrobial activity of the synthesized cationic surfactants.

2. Materials and experimental

2.1. Chemicals

4-Aminoantipyrine (97%), octyl bromide (98%), dodecyl bromide (98%), hexadecyl bromide (98%) and 4-(dimethylamino) benzaldehyde (99%) were obtained from Sigma-Aldrich, Germany. Ethanol and paraffin oil were obtained from ADWIC chemicals company, Egypt. Paraffin oil is light paraffin oil (a mixture of hydrocarbons) with carbon chains of (16 + 18) are the main components (87%).

2.2. Synthesis

2.2.1. Synthesis of 4-formyl-N, N-dimethyl-N-alkylbenzenaminium bromide

The synthesized cationic surfactants were obtained by coupling reaction between alkyl halide namely: Octyl bromide, dodecyl bromide and hexadecyl bromide (0.1 mol.) and 4-(dimethylamino) benzaldehyde (0.1 mol.) in 50 ml ethanol. The reaction mixture was refluxed for 24 h and left for complete precipitation of the cationic compounds. The produced cationic surfactant were filtered off and recrystallized three times from ethanol to produce the desired cationic surfactants [22].

2.2.2. Synthesis of 4-aminoantipyrine derived cationic surfactants

A mixture of 4-formyl-N, N-dimethyl-N-alkylbenzenaminium bromide (0.15 mol.), 4-aminoantipyrine (0.15 mol.) and ethanol (150 mL) was added to a round flask and heated at 80 °C with continuous removal of water of the reaction [23]. The reaction was stopped after removal of water. The desired cationic surfactants obtained namely; (Z)-4-(((1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-yl)imino)methyl)-N,N-dimethyl-N-octylbenzenaminium bromide (APS-8); (E)-4-(((1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-yl)imino)methyl)-N-dodecyl-N,N-dimethylbenzenaminium bromide (APS-12) and (E)-4-(((1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-yl)imino)methyl)-N-hexadecyl-N,N-dimethylbenzenaminium bromide (APS-16), Fig. 1.

2.3. Measurements

2.3.1. Surface tension

The surface activity of this surfactant was determined from surface tension data and surface tension was measured by the platinum ring method using a Kruss K6 tensiometer, Kruss Co., Germany. The surface tension measurements were carried out with a concentration from 10^{-2} to 5×10^{-5} mole/L at 20, 40 and 60 °C. The solutions were placed in a clean cup made from Teflon with a diameter of 28 mm, and the surface tension measurements started after 2 h to ensure solution interface stabilization and surfactants molecules adsorption at the water/air interface. The surface tension values were considered as an average of three replicates [24].

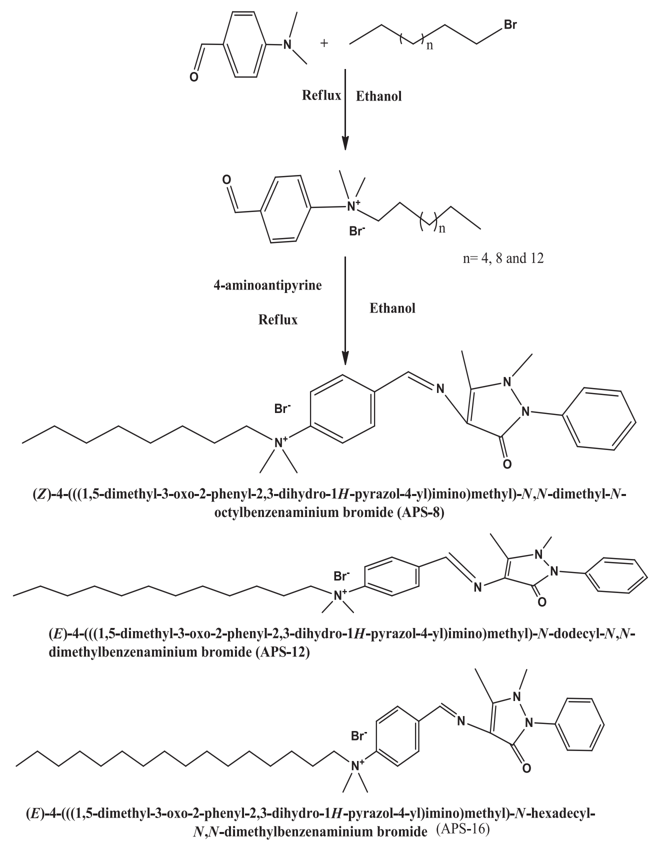

Fig. 1. Synthetic route of the synthesized cationic surfactants (APS-8, APS-12 and APS-16).

2.3.2. Interfacial tension

The interfacial tension of the synthesized cationic surfactants was measured between aqueous solutions of the synthesized surfactants and light paraffin oil (GC analysis of the used paraffin oil showed that C_{16} and C_{18} components consist about 87% of the total carbon distribution of the used oil) at concentration 0.1% by weight and 25 °C using the same procedures of the surface tension measurements [25].

2.3.3. Emulsion stability

The stability of the emulsions was determined as follows: 10 mL (0.5%) of aqueous solutions of the synthesized surfactant and 10 mL of paraffin oil mixed together in a closed graduated cylinder by vigorous stirring at 25 °C [19]. Then, the cylinder was allowed to stand for separation of the two layers of paraffin oil and aqueous surfactant solution. The steps were repeated three times, and the time of separating 9 mL of pure aqueous surfactant solution was counted. The emulsion stabilities of the surfactants were the average of the three readings [26].

2.3.4. Foaming power

Foaming properties of the synthesized quaternary ammonium salts was measured after shaking 100 mL of 0.1% of this compound solution in a graduated 250 mL cylinder at 25 °C. Foam production was measured by the foam height (in mL) and foam stability was measured by the time [27].

2.3.5. Antimicrobial measurements

Synthesized cationic surfactants (APS-8, APS-12 and APS-16) were screened for their antimicrobial activity against Gram-positive bacteria (*Bacillus subtilis* and *Staphyl. aureus*),

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