

Synthesis and Foam Power of New Biodegradable Surfactant

R. Mousli, A. Tazerouti

Abstract—This work deals with the synthesis and the determination of some surface properties of a new anionic surfactant belonging to sulfonamide derivatives. The interest in this new surfactant is that its behavior in aqueous solution is interesting both from a fundamental and a practice point of view. Indeed, it is well known that this kind of surfactant leads to the formation of bilayer structures, and the microstructures obtained have applications in various fields, ranging from cosmetics to detergents, to biological systems such as cell membranes and bioreactors. The surfactant synthesized from pure *n*-alkane by photosulfochlorination and derivatized using *N*-ethanol amine is a mixture of position isomers. These compounds have been analyzed by Gas Chromatography coupled to Mass Spectrometry by Electron Impact mode (GC - MS/EI), and IR. The surface tension measurements were carried out, leading to the determination of the critical micelle concentration (CMC), surface excess and the area occupied per molecule at the interface. The foaming power has also been determined by Bartsch method, and the results have been compared to those of commercial surfactants. The stability of the foam formed has also been evaluated. These compounds show good foaming power characterized in most cases by dry foam.

Keywords—Non ionic surfactants, GC-MS, surface properties, CMC, foam power.

I. INTRODUCTION

THE synthesis of a new double stranded or bicatenary surfactant using alkanesulfonyl chlorides (RSO_2Cl) is developed. The latter used as starting materials are obtained by photochlorosulfonation [1] of a pure *n*-alkane, the *n*-tetradecane. The resultant tetradecanesulfonyl chlorides, a mixture of isomeric positions, are allowed to react with ethanolamine leading to double stranded or bicatenary nonionic surfactants of general formula $\text{R-SO}_2\text{-NR-CH}_2\text{-CH}_2\text{-OH}$. The synthesized compounds are characterized by the usual spectroscopic methods. Their physicochemical properties were investigated and compared to those of commercial surfactants.

II. EXPERIMENTAL AND MAIN RESULTS

A solution of 10 mmol of tetradecanesulfonyl chlorides in dry ether is added under magnetic stirring to a solution of 13 mmol of ethanolamine in dry ether. At the end of the addition, and after a purification step, a yellow product is obtained with a yield of about 84%. The compound is called:

I: secondary *N*-tetradecyl tetradecanesulfonamido ethanol.

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III. ANALYSIS OF SECONDARY *N*-TETRADECYL TETRADECANESULFONAMIDO ETHANOL

The product is analyzed by Infra Red and by GC-MS in electronic impact mode.

A. Analysis by Infra-Red

The IR analysis of the product was carried out in KBr pellet and shows the two absorption bands of the sulfonamide- $\text{SO}_2\text{-NH}$ group and the hydroxyl group OH (Table I):

Products	Characteristic bands	Functions
I	1384cm^{-1} 1141cm^{-1} 3315cm^{-1}	$\text{SO}_2\text{-NH}$ OH

B. Analysis by Gas Chromatography Coupled to Mass Spectrometry (GC-MS/EI) of Secondary *N*-tetradecyl Tetradecanesulfonamido Ethanol

The mass spectrum obtained by GC-MS analysis using EI mode (Fig. 1-2), shows the presence of the peak at m/z 320 which corresponds to the departure of $[\text{C}_{14}\text{H}_{29}]^+$, leading to the fragment $[\text{C}_{14}\text{H}_{29}\text{SO}_2\text{NCH}_2\text{CH}_2\text{OH}]^+$. It is also detected the peaks at m/z 97, 83 and 69 which respectively correspond to fragments $[\text{C}_7\text{H}_{13}]^+$, $[\text{C}_6\text{H}_{11}]^+$ and $[\text{C}_5\text{H}_9]^+$, thereby indicating the presence of the linear hydrocarbon chain in the molecule.

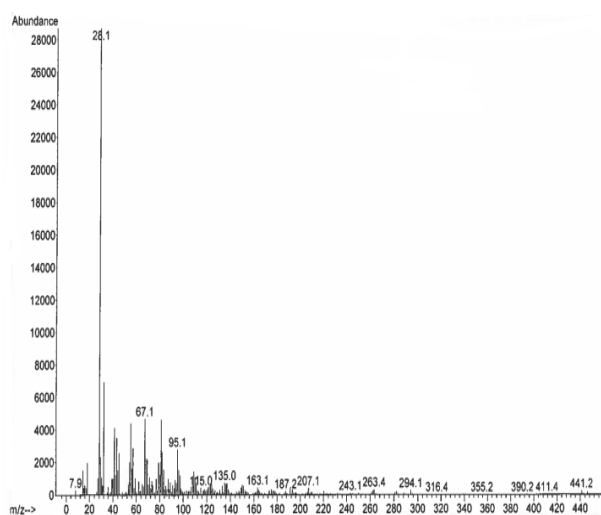


Fig. 1 Mass spectra (IE) of *N*-tetradecyl tetradecane-1-sulfonamido ethanol

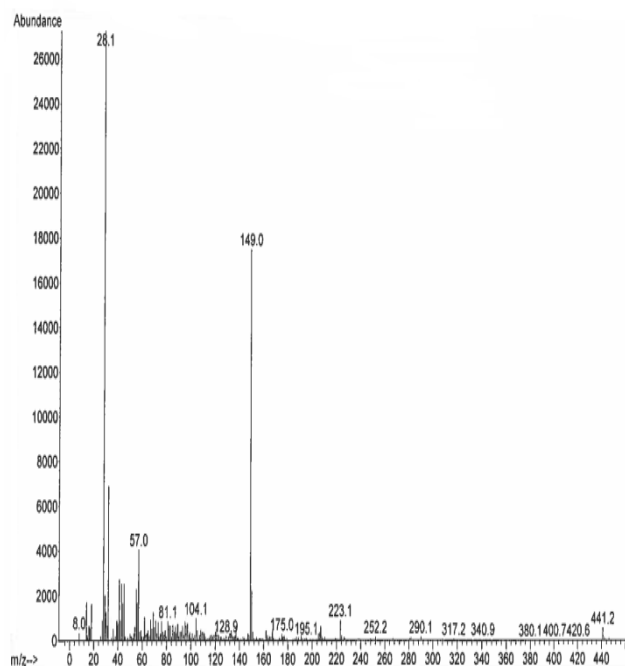


Fig. 2 Mass spectra (IE) of N-tetradecyl tetradecane-2-sulfonamido ethanol

IV. PHYSICO-CHEMICAL PROPERTIES

The results of the study of the micellar behavior of these compounds and their foaming power are presented below. The results are compared to those of two commercial samples SAS (Sodium Alkane Sulfonates) and SDS (Sodium Dodecyl Sulfate). The apparatus used is a tensiometer (Prolabo TD 2000) with a very fine and perfectly wettable platinum blade. A series of surfactant aqueous solutions at different concentrations ranging from 10^{-2} M to 10^{-5} M is prepared. A mother solution was prepared and dilutions were made by adding distilled water. To study the surface activity of these sulfonamides and determine their critical micelle concentration, monitoring the variation of surface tension versus the concentration of sulfonamide aqueous solutions was conducted. Each experiment to measure surface tension has been duplicated. The set of values of surface tension obtained is used to draw the γ isotherms versus the logarithm of the concentrations of each solution. The resulting curve is plotted in Fig. 3:

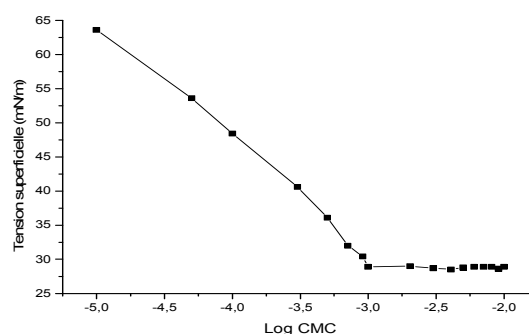


Fig. 3 Variation of the surface tension versus the concentration of secondary N-tetradecyl tetradecanesulfonamido ethanol at 25°C

It can be concluded from the graph obtained, that this compound behaves like conventional surfactants and lowers the interfacial tension between air and water in proportion to their concentration and have a distinct CMC. The absence of a minimum on the curve of surface tension in the region of the CMC proves that the product is pure. From this curve, the value of the CMC was determined. The surface excess (Γ) and molecular area at the interface (A) were calculated for all samples and those of commercial ones. The results are summarized in Table II.

TABLE II
PROPERTIES AND ADSORPTION PARAMETERS OF THE SULFONAMIDE
SURFACTANT IN PURE WATER

Compounds	T (°C)	CMC (mol/l)	γ_{CMC} (mN/m)	$\Gamma \times 10^{10}$ (mol/cm ²)	A (Å ² /molec)
I	25	$1.2 \cdot 10^{-3}$	28.29	5.34	31.04
SAS	45	$1.00 \cdot 10^{-3}$	29	2.49	66.67
SDS	45	$8.20 \cdot 10^{-3}$	/	3.40	49.00

These sulfonamides lower effectively the surface tension of water, from 73 mN / m to 30 mN. Comparing the two commercial surfactants SAS and SDS with compound I, it can be seen that their CMC are of the same order of magnitude (10^{-3} M).

V. FOAMING PROPERTIES

The study of the evolution of the initial foam height versus concentrations of secondary N-tetradecyl tetradecanesulfonamido ethanol and LAS showed that foam increases with the concentration, and even beyond the CMC. The results show that these surfactants have good foaming power which is well compared to that of LAS, showing the ability of these compounds to form foam.

The height h of the foam versus time t of N-tetradecyl tetradecanesulfonamido ethanol is reported in Fig. 4 in the concentration range studied for periods up to 250 minutes. As seen on Fig. 4, it brought good foam stability over time for each concentration [2]-[5]. The synthesized sulfonamides show stability in the region and beyond CMC. They give rise to the formation of stable foam as the calculated R_s are all above 60%, therefore the foams formed are considered as stable.

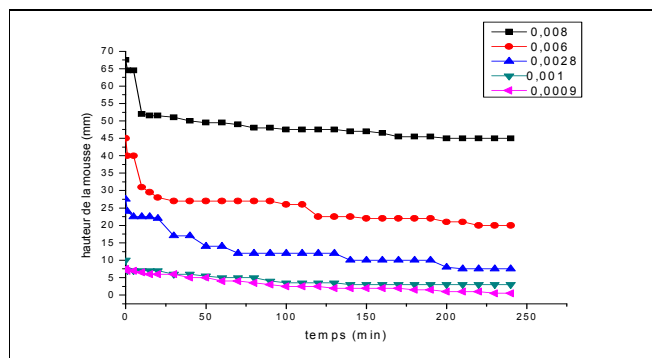


Fig. 4 Variation of the foam height versus time for tetradecyl tetradecanesulfonamido ethanol

VI. CONCLUSION

In this work, a new method has been developed for the preparation of a new non ionic surfactant from secondary tetradecanesulfonyl chlorides. The synthesized compounds obtained with good yields were analyzed by spectroscopic methods, namely, IR and mass spectrometry in electron impact mode.

Regarding the physico-chemical properties, the results show good surface activity at the water-air interface of sulfonamides, and are well compared to those of commercial samples SAS and SDS. The investigation shows that this surfactant possesses good foaming power when compared to those of SDS and LAS.

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