Mixed Sulfated Tallow Alkanolamides as Lime Soap Dispersing Agents

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ABSTRACT

Sulfated alkanolamides of hydrogenated tallow fatty acids have been shown to possess excellent lime soap dispersing and detergent properties. However the high melting points of the alkanolamides and their relative insolubility in organic solvents such as dichloroethane make sulfation on an industrial scale awkward. This difficulty has been overcome by the use of a eutectic mixture of the N-(2-hydroxypropyl)amide and N-(2-[2-hydroxyethoxy]ethyl)amide of unhydrogenated tallow fatty acids. The sulfation of such a mixture can be carried out at or slightly above room temperature, and only a small amount of a chlorinated solvent is required in order to keep the sulfation mixture fluid. The resulting sulfated mixed alkanolamide is an excellent lime soap dispersing agent, which is formulated readily with tallow soap and a glassy silicate into an effective heavy duty detergent.

The sodium salts of the sulfated hydrogenated tallow amides of various alkanolamines such as monoethanolamine(2-hydroxyethylamine), monoisopropanolamine (2-hydroxypropylamine) (1,2) and diglycolamine(N-[2-(2-hydroxyethoxy)ethyl]amine) (3) have been shown previously to possess excellent lime soap dispersing properties. Since the sulfated isopropanolamide of hydrogenated tallow fatty acid is somewhat more stable to alkaline hydrolysis than the corresponding ethanolamide (1), the former surfactant was utilized in an extensive study of soap-based detergent formulations (4). Unfortunately the sulfation of this amide on a large scale is awkward and industrially infeasible, because its high titer (74 C) makes sulfation at room temperature impossible. In addition, the amide is not very soluble in most organic solvents, so that it has to be dissolved in a 10-fold quantity of chloroform or 1,2-dichloroethane in order to yield a solution that is stirrable at room temperature.

The work reported here shows that the above difficulties may be overcome by a change from hydrogenated to unhydrogenated tallow fatty acid derivatives and, in addition, by the use of a eutectic mixture of about equal weights of the tallow isopropanolamide (IPAm) and diglycolamide (DGAm). This mixture has a titer of 46 C, which is 28 C below that of the isopropanolamide of hydrogenated tallow fatty acid. Samples of methyl ester of tallow fatty acid (TE 1618, Procter & Gamble Chemical Div. and Metholene 2203, Emery Industries, Inc.) were converted to the alkanolamides in the same manner as described in a previous publication (1). An industrial grade of diglycolamine (Jefferson Chemical Co.) was used.

TABLE I

Variation of Titer of Amide Mixtures with Composition[a]

% DGAm	% IPAm	Titer, C
100	0	58.0
90	10	55.5
80	20	52.0
70	30	49.5
60	40	47.0
50	50	46.0
40	60	48.0
30	70	49.0
20	80	50.0
10	90	52.5
0	100	54.5

[a]DGAm, N-(2-[2-hydroxyethoxy]ethyl)amide; IPAm, N-(2-hydroxypropyl)amide.

Sulfation with chlorosulfonic acid was carried out in the following manner. Mixed amide 109 g (.318 mol), consisting of equal weights of IPAm and DGAm, was dissolved in 25 ml 1,2-dichloroethane, and 46.3 g (.397 mol) chlorosulfonic acid was added slowly to the agitated amide solution. The reaction temperature was maintained below 30 C with an ice bath. After the addition of the chlorosulfonic acid was completed, agitation was continued for another 30 min. The sulfation mixture was then neutralized by adding it slowly to a well stirred mixture of ca. 32 ml 18 N sodium hydroxide and 200 g ice. The pH of the neutralized solution was adjusted to 8-9. The active anionic surfactant content of the solution was determined by titration with standard Hyamine 1622 (Rohm and Haas Co.) with dichlorofluorescein as indicator (5). The solutions were used in the performance test without further purification. All other sulfations with chlorosulfonic acid were carried out in an analogous manner.

The mixed amide (50% IPAm and 50% DGAm) was also sulfated with sulfur trioxide. Mixed amide, 109 g (.318 mol) was dissolved in 25 ml 1,2-dichloroethane. Sulfur trioxide, 31 g (.397 mol), was slowly vaporized and diluted with dry nitrogen. A stream of this gas mixture was introduced into the cold amide solution at such a rate that the temperature was maintained below 30 C with the aid of an ice bath. The sulfation mixture was neutralized as described above. This product was considerably darker in color than an analogous product sulfated with chlorosulfonic acid. Slight charring due to inadequate mixing could be observed.

PHYSICAL PROPERTIES, SURFACE ACTIVE TEST METHOD

The titers of the amides and amide mixtures were determined according to the AOAC method (6) and tabulated in Table I. The lime soap dispersant requirement (lsdr) of the sulfated compounds was determined according to the method of Borghetty and Bergman (7) and recorded in Table II. The lsdr refers to the number of grams of test compound required to be added to 100 g sodium oleate in order to prevent precipitation of 330 ppm water hardness.

In order to ascertain that the sulfated eutectic mixture was equivalent in detergency to the sulfated isopropanolamide of hydrogenated tallow fatty acid, it was incorporated into the same ternary compound detergent formulation previously described by workers from this laboratory for the isopropanolamide (2,4). Detergency evaluations were carried out in a tergotometer, as described in a previous publication (8). Detergent formulations were prepared consisting of 20% sodium salt of sulfated alkanolamide, 64% tallow soap, 15% sodium silicate Na_2O/SiO_2 1:1.6 and 1% carboxymethylcellulose. Detergency was measured in terms of reflectance increase (ΔR) after washing, and the data were recorded in Table II.

Since the objective of this study was to prepare detergent formulations that might find industrial utili-

TABLE II

Surface Active Properties of Sulfated Tallow Alkanolamides

Surfactant type[a]	Sulfating agent	Sulfating agent/amide, mol ratio	Lime soap dispersant requirement (lsdr)	ΔR Detergency of formulated detergent[b] at 0.2% 120F, 300 ppm water hardness		
				Testfabrics cotton-polyester	U.S. Testing cotton	EMPA 101 cotton
Sulfated IPAm, sodium salt	HSO_3Cl	1.3:1	5	17.8	7.1	33.7
Sulfated DGAm, sodium salt	HSO_3Cl	1.3:1	4	18.4	8.2	33.3
Sulfated 50% IPAm + 50% DGAm	HSO_3Cl	1.0:1	5	25.3	8.7	28.8
	HSO_3Cl	1.23:1	4	19.0	9.2	33.9
	HSO_3Cl	1.3:1	4	17.3	7.4	31.3
	SO_3	1.23:1	5	15.4	9.2	29.9
Control A				27.7	8.2	24.7
Control B				21.6	7.9	17.6

[a] IPAm, N-(2-hydroxypropyl)amide; DGAm, N-(2-{2-hydroxyethoxy}ethyl)amide.

[b] Formulation composition: 20% sulfated amide, 15% sodium silicate (Na_2O/SiO_2 1:1.6), 64% tallow soap, 1% carboxymethylcellulose.

zation, we employed two commercial household detergents as controls in the detergency evaluations. Control A was a phosphate-built anionic detergent and control B a sodium carbonate-built nonionic detergent.

The titer values of Table I indicate that the two types of tallowamide IPAm and DGAm form a eutectic mixture containing approximately equal weights of the two amides. The use of this eutectic mixture of unhydrogenated tallow alkanolamides should make sulfation on an industrial scale feasible and economical. Conceivably the small percentage of solvent could be eliminated if the agitation of the sulfation vessel is sufficiently powerful. Sulfation with sulfur trioxide also appears feasible, although undoubtedly a continuous process would be required in order to obtain a cleaner and lighter colored product than that prepared by the batch process described above.

It has been pointed out by Weil and coworkers (3) that the double bonds of the fatty alkyl chain are largely retained under mild sulfation conditions with chlorosulfonic acid as the sulfating agent. Since it was anticipated that a certain amount of attack on the double bonds would occur during sulfation, the reaction was carried out with different mole ratios of reactants in an effort to arrive at a ratio resulting in optimum surface active properties. However the data in Table II indicated that neither lsdr nor detergency were greatly affected by the mole ratio.

The lsdr of 4-5 for the preparations listed in Table II agreed well with a previously observed value of 4 for the sulfated diglycolamides of oleic or stearic acids (3) and an lsdr of 5 for the sulfated isopropanolamide of stearic acid (1). The detergency data obtained from soap-based detergent formulations using the test preparations as lime soap dispersing agents agree with previously published data (4) on analogous soap-based detergent formulations, in which the sulfated isopropanolamide of hydrogenated tallow fatty acid was used as the lime soap dispersing agent. The cotton detergency performance of the formulations of this study as shown in Table II is comparable to or slightly superior to that of the commercial controls A (phosphate-built anionic) and B (sodium carbonate-built nonionic). The detergency performance on cotton-polyester fabric is about equal to that of control B but inferior to that of control A. Thus it appears that the sulfated mixed amides of tallow fatty acid are suitable lime soap dispersing agents for tallow soap-based heavy duty detergent formulations.

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