Impact of Composition on the Performance of Sodium Linear Alkylbenzenesulfonate (NaLAS)[1]

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ABSTRACT: Like other commercial surfactants, commercial linear alkylbenzenesulfonate is not a single compound but a mixture of components. This mixture comprises different carbon-chain homologs, different phenyl isomers, and, in addition, the manufacturing co-product, dialkyltetralin sulfonate. Current manufacturing technology makes it possible to easily vary the average carbon chainlength. On the other hand, the phenyl isomer distribution and dialkyltetralin sulfonate content can be changed only within a certain range. Each of these variations in composition affects performance. This study reports on the effect of commercial alkylbenzenesulfonate composition on surface tension, solubility, viscosity, foam stability, and detergency. The study shows that average carbon chainlength affects all performance properties. Phenyl isomer distribution and dialkyltetralin sulfonate content affect only solubility and viscosity.
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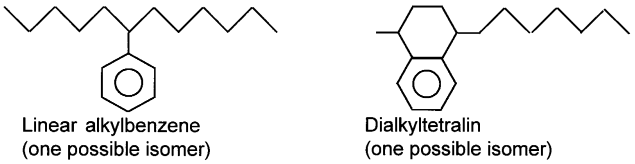
KEY WORDS: Detergency, dialkyltetralin, foam, linear alkylbenzenesulfonate, phenyl isomer, solubility, surface tension.

Performance of sodium linear alkylbenzenesulfonate (NaLAS) has been studied extensively for more than 30 years. Given this lengthy period, one would think every aspect of performance had been explored. That this is not the case is the result of both the increasing complexity of formulations and the increasing availability of different "types" of NaLAS. The complex interaction of NaLAS and other components in formulations is a developing area of study and will not be discussed in this paper. Instead, the focus will be on the variety of commercial NaLAS available and how this variety affects performance.

The variety of choices available for commercial NaLAS reflects the variety of choices available for the parent compound, linear alkylbenzene (LAB). The structure of LAB is often represented as an alkyl chain attached to a benzene ring at one of the interior carbon atoms (Scheme 1). This simple representation, however, obscures the complexity of the composition of commercial LAB. Both the length of the alkyl chain and the point of attachment of the alkyl chain to the benzene ring may vary in commercial LAB. Additionally, during manufacture of LAB, a side reaction occurs that yields the co-product, dialkyltetralin (structure shown in Scheme 1). In the manufacture of LAB only the average alkyl chainlength can be varied readily. On the other hand, phenyl isomer distribution and dialkyltetralin content are a consequence of the choice of alkylation chemistry.



SCHEME 1

The three catalysts used commercially to alkylate benzene to produce LAB are aluminum chloride, hydrofluoric acid, and a newly patented solid alkylation catalyst (1). A summary of these routes is shown in Scheme 2. Typical ranges of 2-phenyl isomer and dialkyltetralin content produced by each commercial process are illustrated in Table 1.

This paper reports on the effects of average alkyl chainlength, phenyl isomer distribution, and dialkyltetralin content on the surface chemistry, solubility, viscosity, foam stability, and detergency of NaLAS. Because commercial NaLAS always contains a distribution of carbon chainlengths, the term "carbon chainlength" will be used to mean the average carbon chainlength.

EXPERIMENTAL

The NaLAS used in the performance studies was produced by sulfonating commercial LAB with the three types of catalyst shown in Table 1. In addition to commercial LAB, some C_{12} narrow-distribution phenyl isomer samples were prepared for study. These were prepared by Grignard reaction of the appropriate bromoalkane and alkylphenyl ketone. The resulting alcohol was then hydrogenated to give the phenyl alkane. Subsequent sulfonation yielded the narrow-distribution phenyl isomer NaLAS. The phenyl isomer distribution of these products is given in Table 2.

A sample of dialkyltetralin was also prepared for surface tension studies. This was synthesized by dibromination of a

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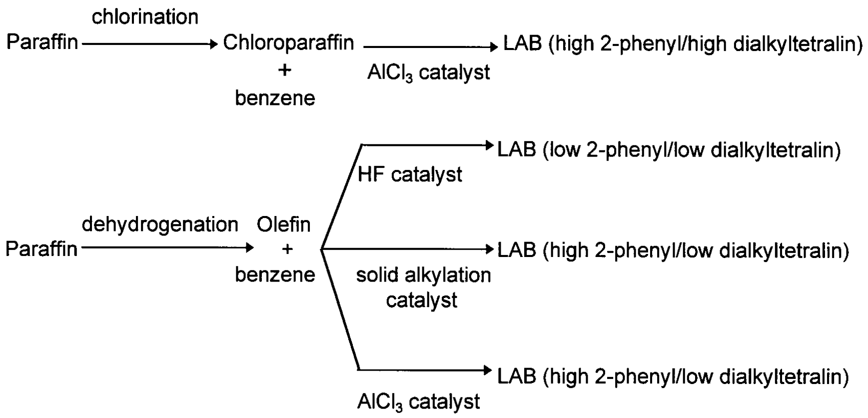

SCHEME 2

TABLE 1
Composition of Commercial Linear Alkylbenzenesulfonate

Property	$AlCl_3$-catalyzed alkylation[a]	HF-catalyzed alkylation[a]	Solid-catalyzed alkylation[b]
Average carbon chainlength	C_{11}–C_{13}	C_{11}–C_{13}	C_{12}–C_{13}
Percentage 2-phenyl isomer	27–30	15–20	27–30
Percentage dialkyltetralin	6–10	<1	<1

[a]Reference 1.
[b]Reference 2.

C_{12} internal olefin, followed by $AlCl_3$ alkylation of benzene. The product was 86.5% dialkyltetralin, 12% alkylbenzene and 1.5% unidentified reaction products. The dialkyltetralin was sulfonated to form dialkyltetralin sulfonate.

Surface tension of aqueous solutions of NaLAS was measured by an automated Lauda TE 1C (Brinkmann, Westbury, NY) by the duNouy ring method. Temperature of the solutions was controlled to within ±0.1°C with a Brinkmann RCS waterbath. All solutions were prepared from water passed through a Nanopure water filter system (Barnstead, Dubuque, IA). The water had a conductance of 17 Mmho/cm (conductance meter, Yellow Springs Instrument, Yellow Springs, OH). To eliminate ionic strength effects, all solutions were made 0.01 M Na_2SO_4. Surface tension measurements of the 0.01 M Na_2SO_4 solution prior to addition of surfactant gave an average reading of 71.5 dynes/cm.

To measure solubility of commercial NaLAS solutions, the following procedure was used. First, all solutions were prepared with the same active level of NaLAS in deionized water that contained sufficient salt to equalize the ionic strength. Each of these solutions was then placed in a Fisher Isotemp incubator, fitted with a glass door (Fisher Scientific, Pittsburgh, PA). The temperature in the incubator was slowly lowered over several days. The temperature at which the solutions first turned cloudy was taken as the cloud point. The lower the cloud point, the higher the solubility. Clear points were obtained by warming the cloudy solutions in the incubator over several days and noting when the solution cleared.

TABLE 2
Phenyl Isomer Distribution of Narrow-Distribution C_{12} Sodium Linear Alkylbenzenesulfonate Samples

	Nominal phenyl isomer (%)				
	2	3	4	5	6
1-Phenyl	0.0	3.9	4.8	3	0.0
2-Phenyl	93.7	0.4	0.7	3	1.6
3-Phenyl	3.5	87.2	2.6	2.2	3.4
4-Phenyl	0.9	2.2	87.2	0.9	1.9
5-Phenyl	1.2	3.4	2.8	89.8	0.0
6-Phenyl	0.7	3.0	2.0	0	93.1

To determine the solubility of the C_{12} narrow-distribution phenyl isomers, a different procedure was used. A saturated solution of C_{12} phenyl isomer was prepared. This solution was then heated to 70°C for 0.5–3 h and then cooled to 5°C. After equilibrating at room temperature overnight, the cloudy mixture was centrifuged, and the clear supernatant was drawn off by a pipette, weighed, and dehydrated in a moisture balance to determine percentage solids. The percentage dissolved was then calculated from the weight lost to the supernatant.

Viscosity was determined in a Brookfield DV-II rheometer (Brookfield Engineering, Stoughton, MA) , fitted with a cone and plate configuration. Temperature was controlled to ±0.1°C with a Brinkmann RCS waterbath. Each of the solutions for which viscosity was measured was examined through a Nikon (Tokyo, Japan) Optiphot-2 polarizing light microscope, fitted with a Nikon 35-mm camera. The temperature was controlled to within 0.1°C with a Mettler 82 hot-stage attachment (Mettler Instrument, Hightstown, NJ), connected to a Mettler 80 controller. All photos were taken at 100×.

Foam stability was measured by using either a plate-washing test (3) or a soil titration test (4). In the plate-washing test, the formulation and water are introduced into a basin at 43°C. Foam is generated by blowing compressed air through a sintered glass disk gas dispersion tube inserted in the basin. Four basins are used, and the air pressure and time of bubbling are the same for each basin. Each operator washes two plates at each basin and then rotates to the next basin. Each plate contained 5 g of shortening. This procedure was repeated until there was a permanent break in the foam on the surface of the water in the basin. This break was taken as the endpoint, and the number of plates washed was recorded.

In the soil titration test, the formulations were added to a glass vessel thermostatted at 45°C. Foam was generated for 3 min by stirring with a glass stirring paddle, connected to a stirring motor. Soil was then added to the vessel as stirring continued. The soil was added from a preweighed syringe connected to a syringe pump. The soil consisted (by weight) of 15% shortening, 15% olive oil, 15% instant potato, 30% milk, and 25% water (soil no. 6 of Table 1 from Reference 4). The soil tolerance is reached when a rapid breaking of the foam occurs. At this point, the syringe was removed and reweighed, and the amount of soil necessary to cause the foam to dissipate was calculated. This test has shown excellent correlation with dishwashing tests, with greater soil consumption corresponding to greater numbers of plates washed.

The effect of NaLAS composition on detergency was measured with a Terg-O-Tometer (in-house manufacture). The test conditions are given in Table 3.

RESULTS AND DISCUSSION:

Surface activity. The impact of carbon chainlength, phenyl isomer distribution, and dialkyltetralin sulfonate content on surface tension is shown in Figures 1–3, respectively. The critical

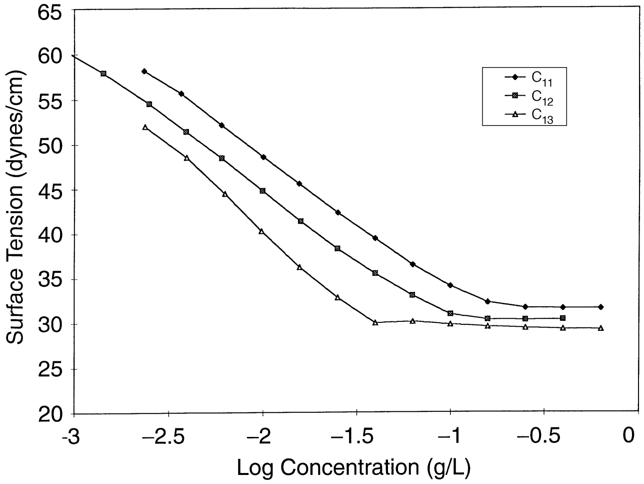

FIG. 1. Effect of average carbon chainlength on surface tension of low 2-phenyl, low dialkyltetralin sulfonate sodium linear alkylbenzene sulfonate; *T* = 25°C, 0.01 M Na_2SO_4.

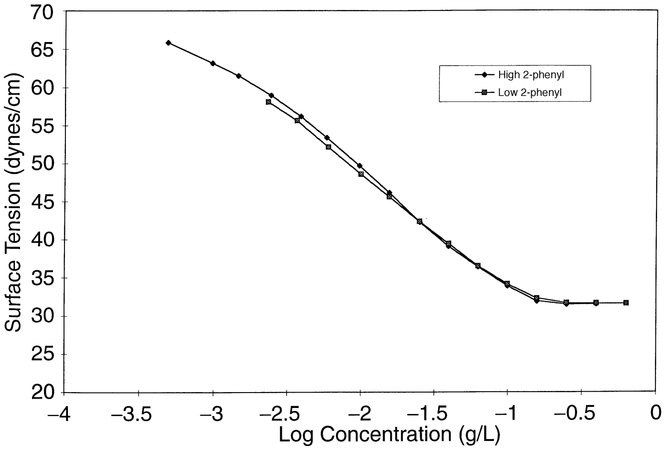

FIG. 2. Effect of phenyl isomer distribution on surface tension of C_{11} average, low dialkyltetralin sulfonate sodium linear alkylbenzene sulfonate; *T* = 25°C, 0.01 M Na_2SO_4.

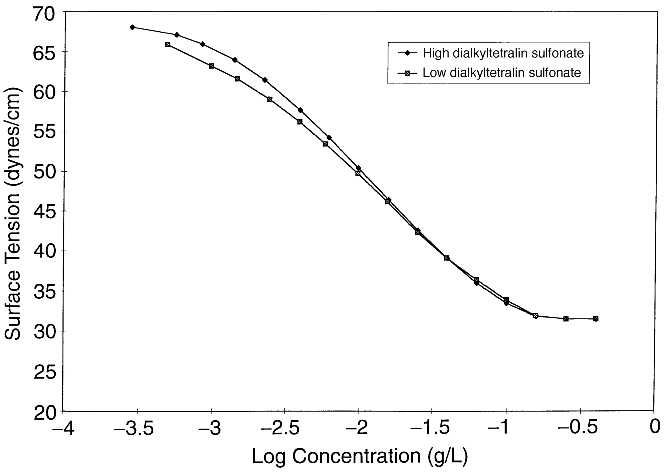

FIG. 3. Effect of dialkyltetralin sulfonate content on surface tension of C_{11} average, high 2-phenyl distribution sodium linear alkylbenzene sulfonate; *T* = 25°C, 0.01 M Na_2SO_4.

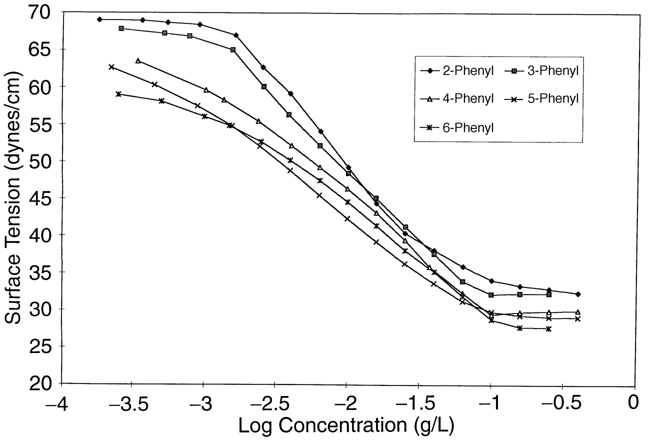

FIG. 4. Surface tension of individual phenyl isomers of C_{12} sodium linear alkylbenzene sulfonate; *T* = 25°C, 0.01 M Na_2SO_4.

TABLE 3
Detergency Test Materials and Procedures

Testing apparatus	Terg-O-Tometer
Wash cycle	10 min
Rinse cycle	5 min
Wash temperature	100°F (37.8°C)
Soil[a]/substrates	Sebum/cotton and sebum/permanent press—manufactured in-house with synthetic sebum; cotton (Test Fabrics S/419; Middles, NJ); permanent press (Test Fabrics S/7406—65% Dacron/35% cotton with permanent press finish)
	Mineral oil/permanent press—manufactured in-house from mineral oil with oil-soluble red dye to aid with detection; permanent press (Test Fabrics S/7406)
Number of soiled cloths	Three 4.5″ × 3.25″ swatches of sebum/cotton and sebum/permanent press cloths, washed together with three clean cotton cloths added for ballast; three 4.5″ × 3.25″ swatches of mineral oil/permanent press cloth washed with six clean cotton cloths added for ballast; three 4″ × 3″ swatches of clay/permanent press cloth, washed with six clean cloths added for ballast
Reflectance measuring device	Hunter D25 A colorimeter (Festoon, VA)

[a]Soil recipe consists of 67.6% synthetic sebum (below), 16.2% particulate dust, 10.8% triethanolamine, and 5.4% oleic acid. The sebum component is based on the recipe of Spangler *et al.* (5), as follows (all values in wt%): palmitic acid (Kodak, Rochester, NY), 10.0; stearic acid (Kodak), 5.0; coconut oil (Sargent-Welch, Skokie, IL), 15.0; paraffin wax (Paraseal), 10.0; spermaceti wax (Sargent-Welch), 15.0; olive oil (imported by Pompeian, Baltimore, MD), 20.0; squalene (Kodak), 5.0; cholesterol (Kodak), 5.0; oleic acid (Kodak), 10.0; and linoleic acid (Kodak), 5.0.

micelle concentration (CMC), surface tension at the CMC and area/molecule, derived from Figures 1–3, are shown in Table 4.

As seen in Figure 1, carbon chainlength significantly affects surface tension at a given concentration. Surface activity increases as average carbon chainlength increases when both phenyl isomer and dialkyltetralin sulfonate content are held constant.

Among the narrow-distribution phenyl isomer LAS samples, as the phenyl isomer number increases, the surface tension decreases (Fig. 4). The range of phenyl isomer distribution of commercial products, however, is not large enough to significantly alter the surface tension vs. log concentration plot (Fig. 2). It is interesting that the surface tension of a blend of equimolar C_{12} 2-phenyl NaLAS and C_{12} 6-phenyl NaLAS approximates the surface tension of C_{12} 4-phenyl NaLAS (Fig. 5).

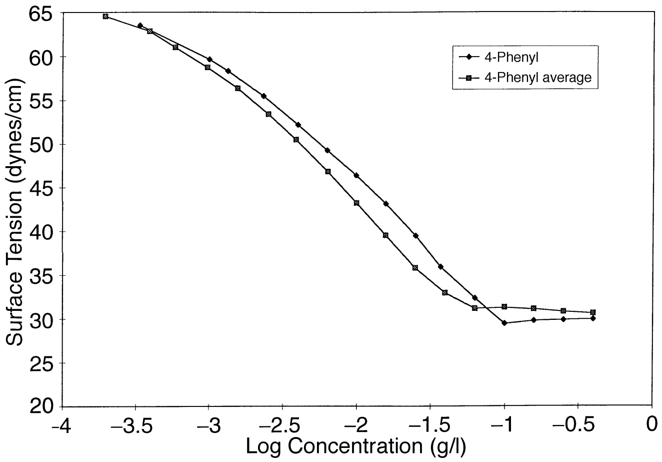

FIG. 5. Surface tension of 1:1 molar blend of C_{12} 6-phenyl sodium linear alkylbenzene sulfonate (NaLAS): C_{12} 2-phenyl NaLAS compared to C_{12} 4-phenyl sodium linear alkylbenzene sulfonate; *T* = 25°C, 0.01 M Na_2SO_4.

The surface tension vs. log concentration plot of "pure" C_{12} dialkyltetralin sulfonate and C_{12} NaLAS is shown in Figure 6.

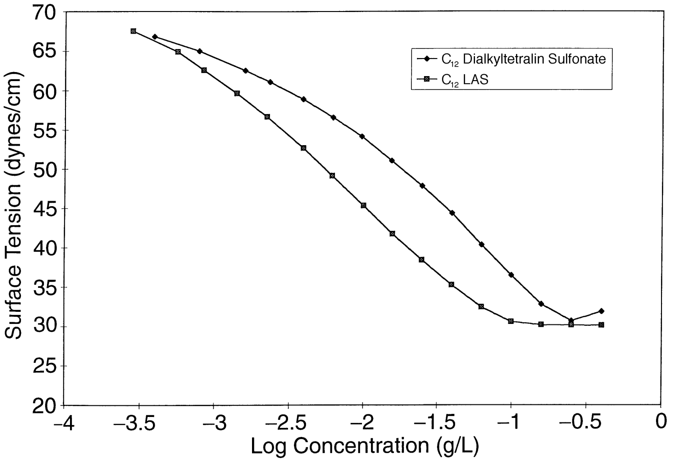

FIG. 6. Surface tension of high 2-phenyl, low dialkyltetralin sulfonate C_{12} sodium linear alkylbenzene sulfonate (NaLAS) and C_{12} dialkyltetralin sulfonate; *T* = 25°C, 0.01 M Na_2SO_4.

TABLE 4
Properties Derived from Surface Tension Measurements

	CMC (g/L)[a]	Surface tension at CMC (dynes/cm)	Area/molecule (Å^2/molecule)
C_{11} high 2-phenyl, low dialkyltetralinsulfonate	0.120	31.5	64
C_{11} high 2-phenyl, high dialkyltetralinsulfonate	0.120	31.8	64
C_{11} low 2-phenyl, low dialkyltetralinsulfonate	0.120	31.6	61
C_{12} low 2-phenyl, low dialkyltetralinsulfonate	0.105	30.3	59
C_{13} low 2-phenyl, low dialkyltetralinsulfonate	0.038	29.3	49

[a]CMC, critical micelle concentration.

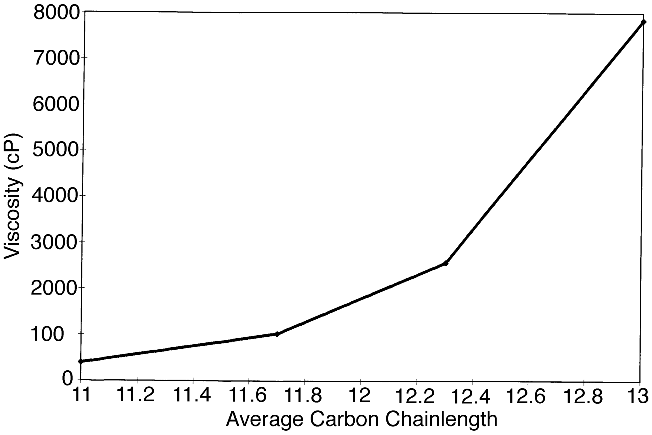

FIG. 7. Effect of average carbon chainlength on viscosity of 25% active low 2-phenyl, low dialkyltetralin sulfonate NaLAS; *T* = 30°C, shear rate = 5 s^{-1}. See Figure 6 for abbreviation.

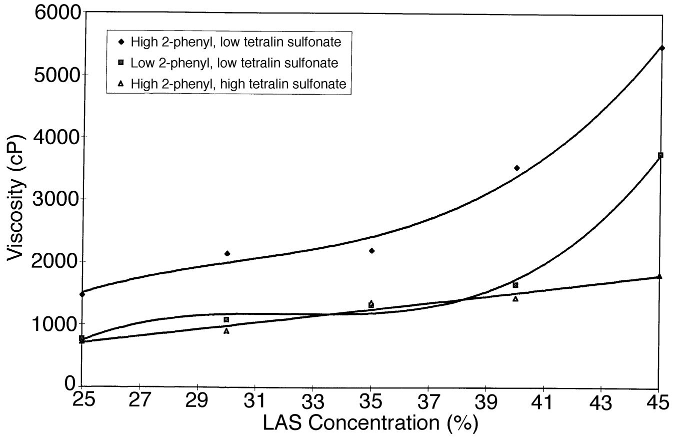

FIG. 9. Effect of phenyl isomer distribution and dialkyltetralin sulfonate level on viscosity of C_{11} average NaLAS; *T* = 25°C, shear rate = 5 s^{-1}. See Figure 6 for abbreviation.

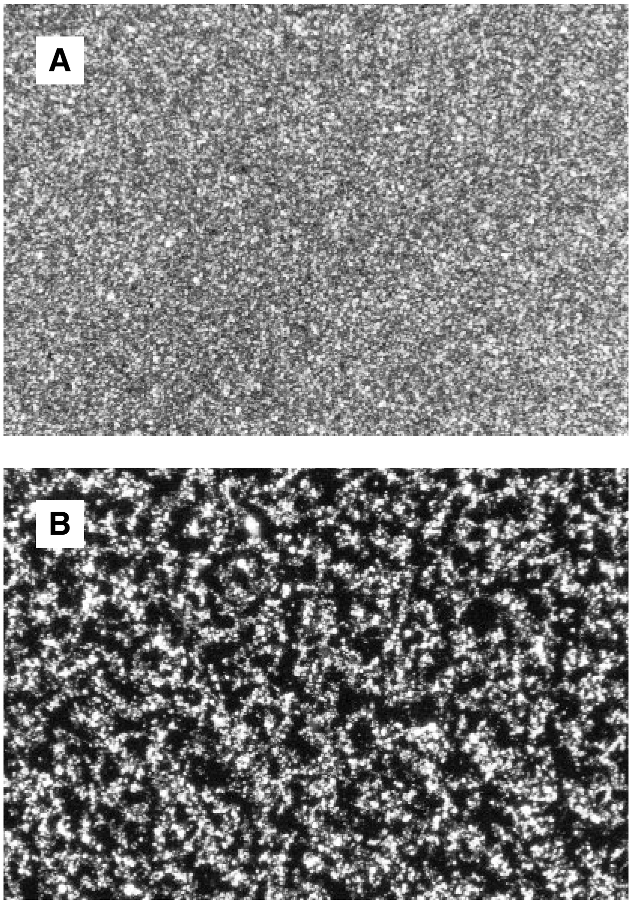

FIG. 8. Photomicrographs of $C_{12.3}$ average (A) and C_{13} average (B) low 2-phenyl, low dialkyltetralin sulfonate NaLAS; *T* = 30°C, 100×, polarized light. See Figure 6 for abbreviation.

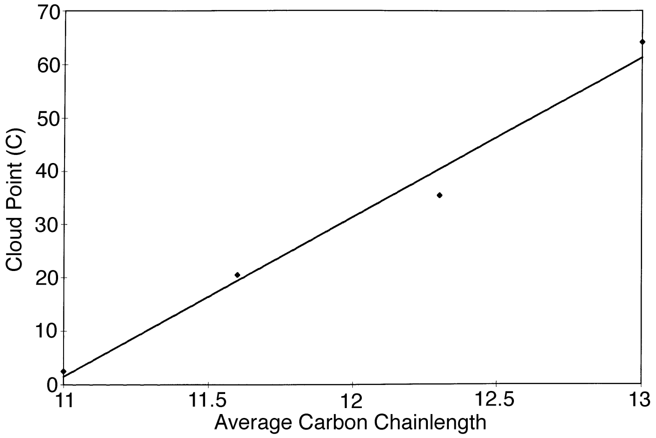

FIG. 10. Effect of average carbon chainlength on solubility of 15% active, low 2-phenyl, low dialkyltetralin sulfonate content NaLAS solutions. Sodium sulfate (1%) added. See Figure 6 for abbreviation.

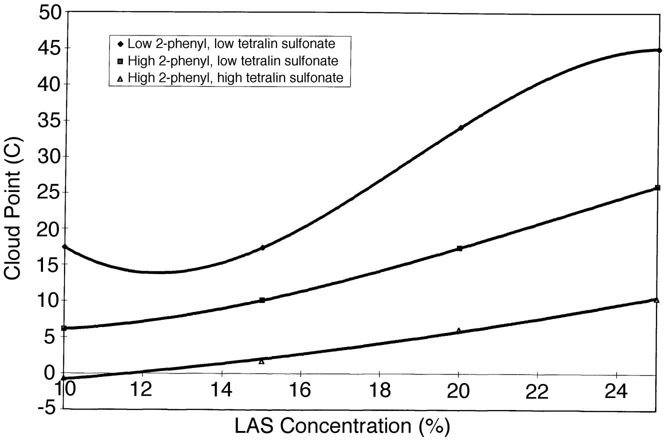

FIG. 11. Effect of phenyl isomer distribution and dialkyltetralin sulfonate content on solubility of C_{11} average NaLAS solutions. See Figure 6 for abbreviation.

It is interesting that dialkyltetralin sulfonate is surface-active, with a CMC approximately that of the corresponding C_{12} NaLAS. At concentrations above the CMC, the surface tension is approximately the same for C_{12} dialkyltetralin sulfonate and

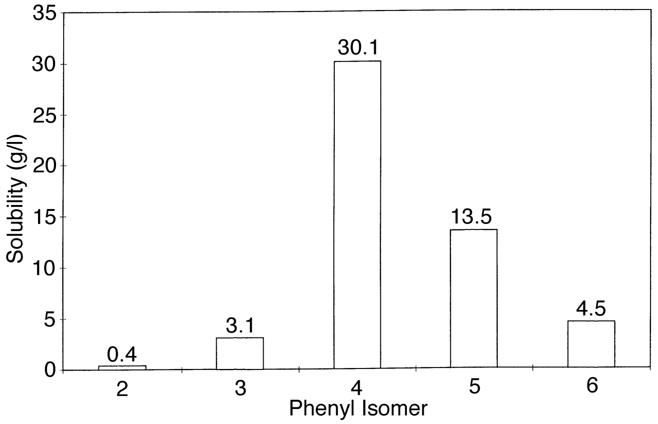

FIG. 12. Solubility of narrow phenyl isomer distribution C_{12} NaLAS at room temperature. See Figure 6 for abbreviation.

C_{12} NaLAS. Given the similar surface activity of dialkyltetralin sulfonate and C_{12} NaLAS, it is not surprising that commercial high dialkyltetralin sulfonate and low dialkyltetralin sulfonate NaLAS also have similar surface activity (Fig. 3).

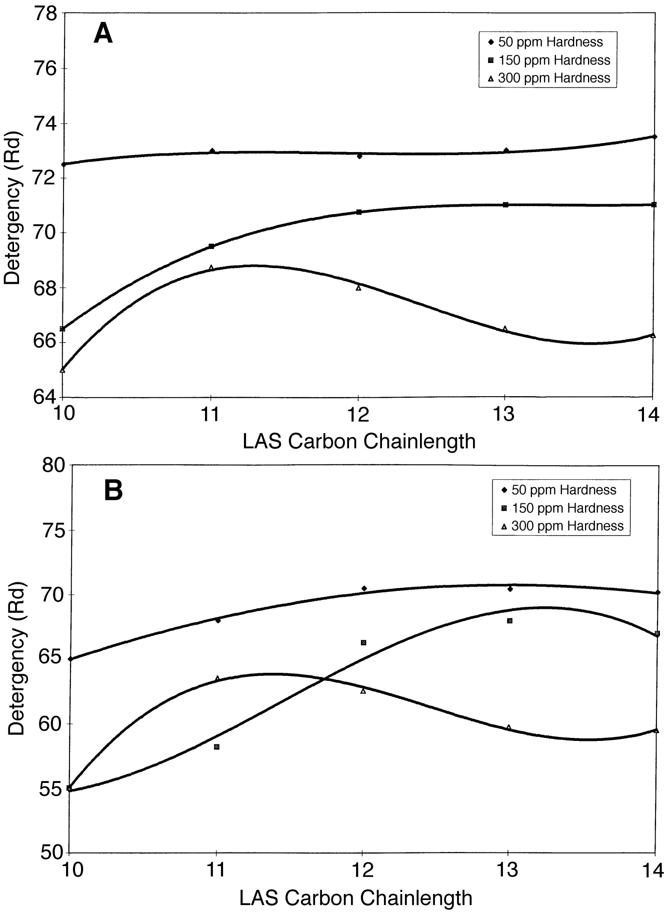

FIG. 14. Effect of carbon chainlength on (A) dust-sebum/cotton detergency and (B) dust-sebum/polyester cotton detergency of high 2-phenyl, high dialkyltetralin sulfonate NaLAS (16% NaLAS/30% sodium tripolyphosphate/10% silicate formulation; 0.15% use level, T = 100°F). See Figure 6 for abbreviation.

Viscosity. The impact of average molecular weight on viscosity was examined by measuring the viscosity of 25% active solutions at 30°C. The results are shown in Figure 7. Increasing the average carbon chainlength of NaLAS increases viscosity at the same phenyl isomer and dialkyltetralin sulfonate content. The increase in viscosity with increasing carbon chainlength is the result of changes occurring in the structure of the solution. The solutions of C_{11} and C_{12} NaLAS are isotropic, and the viscosity most likely depends on the interaction of anisotropic micelles (either rod- or disk-like). Supporting this suggestion is the observation of flow birefringence of the C_{11} and C_{12} NaLAS solutions. Because they can align in flow, anisotropic micelles are likely to show flow birefringence (6). Above 25% active concentration, NaLAS of higher carbon chainlength forms solutions that are dispersions of lamellar liquid crystals. Figure 8 contains photographs of $C_{12.3}$ and C_{13} average NaLAS solutions, taken through a polarizing light microscope, and shows lamellar liquid crystals. The dispersion of smaller lamellar liquid crystals seen in the solution of C_{13} NaLAS corresponds to the highest viscosity shown in Figure 7.

Increasing the 2-phenyl isomer content of a 25 wt% solution of C_{11} NaLAS with low dialkyltetralin sulfonate content increases viscosity (Fig. 9). Both of these solutions show flow birefringence, so the difference in viscosity may reflect differences in the rate of rearrangement between micelles.

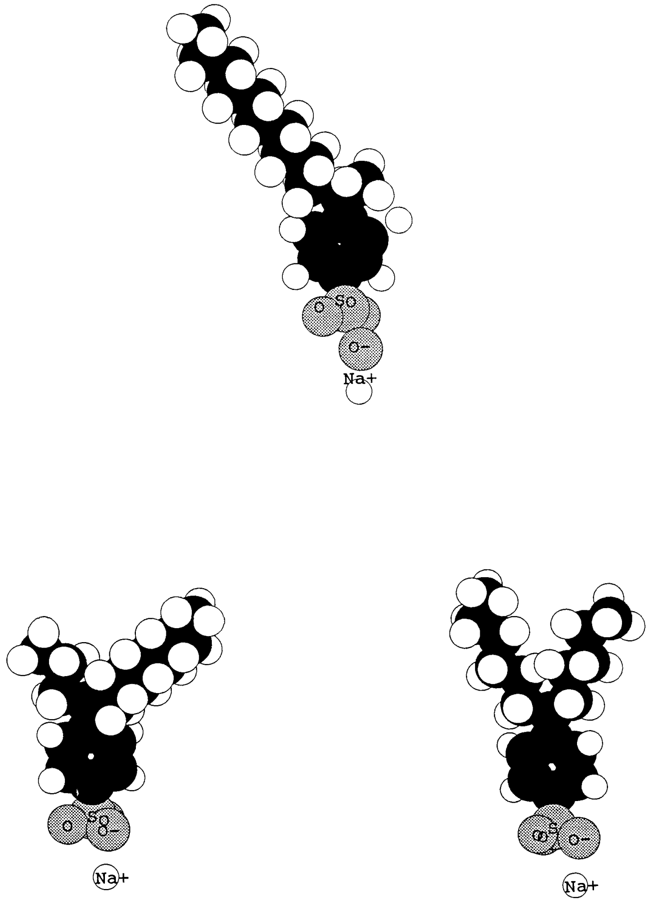

FIG. 13. Structure of phenyl isomers of NaLAS. See Figure 6 for abbreviation.

Increasing dialkyltetralin sulfonate content decreases viscosity (Fig. 9). As described below, dialkyltetralin sulfonate acts as a hydrotrope and interferes with the formation of surfactant structure in solution. If the flow units in the isotropic solutions are anisotropic micelles as presumed, then dialkyltetralin sulfonate may inhibit their formation and so lower the viscosity.

Solubility. Solubility was measured by cooling solutions until they formed a visible cloud and then heating them until the cloudiness cleared. The effects of average molecular weight, phenyl isomer distribution, and dialkyltetralin sulfonate content on solubility are shown in Figures 10 and 11.

As shown in Figure 10, increasing the average alkyl chainlength decreases solubility. This is expected because the Krafft temperature of the mixture is determined by the component with the highest Krafft temperature. The Krafft temperature of NaLAS homologs increases with increasing alkyl chainlength (7).

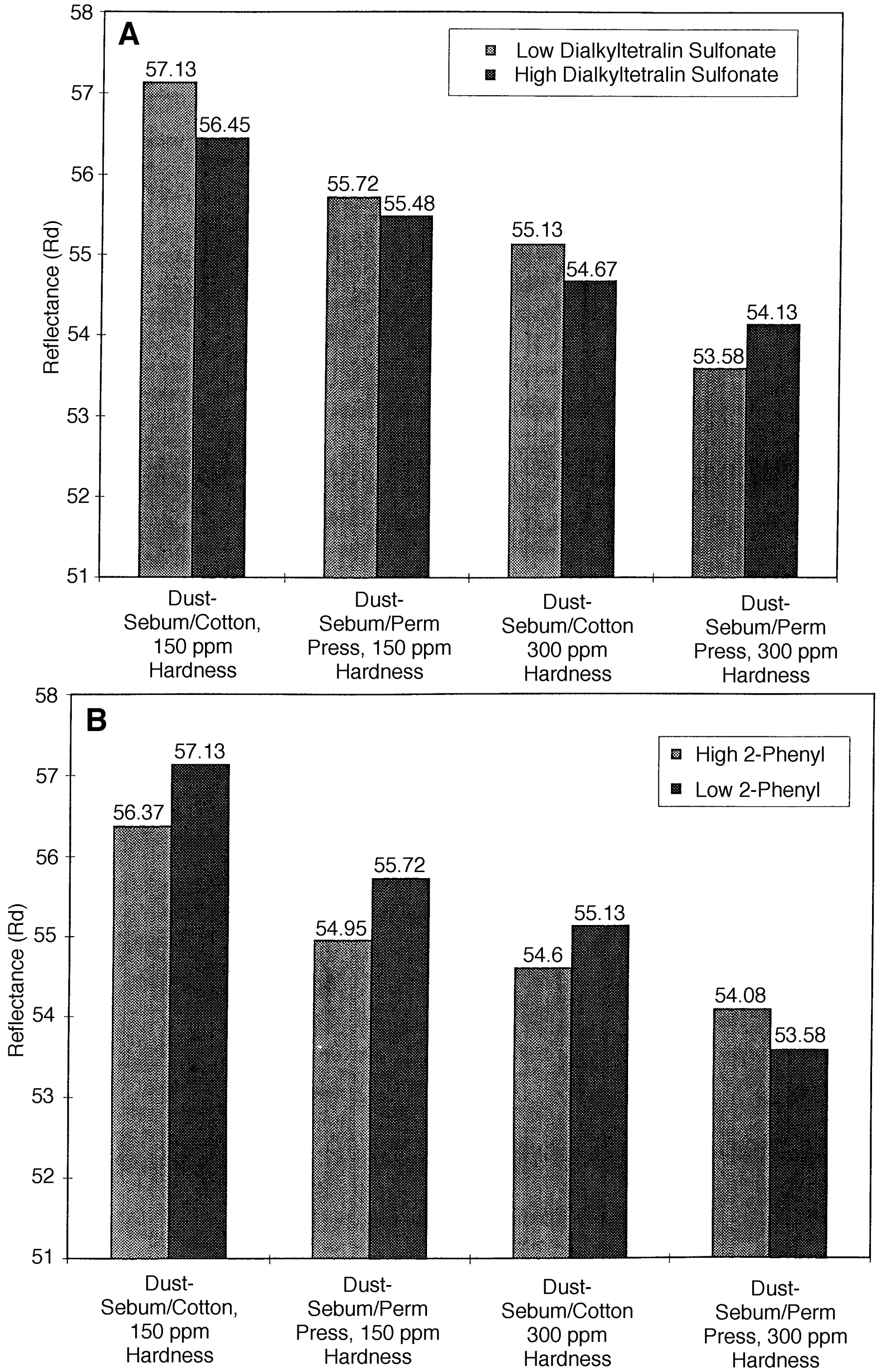

FIG. 15. Effect of (A) dialkyltetralin sulfonate content on detergency of C_{11} average, high 2-phenyl distribution NaLAS, and (B) phenyl isomer distribution on detergency of C_{11} average, low dialkyltetralin sulfonate content NaLAS; use level = 600 ppm surfactant, T = 100°F. See Figure 6 for abbreviation.

The effect of phenyl isomer distribution on solubility is shown in Figure 11. Increasing the 2-phenyl isomer distribution at the same average carbon chainlength and dialkyltetralin sulfonate level increases solubility. Increasing tetralin content at the same average carbon chainlength and phenyl isomer distribution also increases solubility.

The solubility of individual C_{12} narrow-distribution phenyl isomers is shown in Figure 12. C_{12} LAS phenyl isomers show a maximum solubility at approximately the 4-phenyl isomer position. The trend in solubility for the single phenyl isomers may be explained by the following. The Krafft temperature of a single homolog, single phenyl isomer surfactant depends on the energy of the crystal lattice (6). Strong interactions between molecules will promote crystallization and a high Krafft temperature. Strong interactions will be favored by a straight alkyl chain. A high-energy lattice will be found for those surfactants with a long straight alkyl chain. To the degree that the alkyl chain departs from a symmetrical distribution in space, interaction will decrease and Krafft temperature will decrease. The idea is illustrated in Figure 13. A 2-phenyl isomer will have one short chain that only slightly influences the distribution in space of the atoms in the remainder of the alkyl chain. Because the longer chain is not restricted in any direction, it should occupy a symmetrically shaped volume beyond the benzene ring. This "symmetrical" molecule should readily pack into a lattice and so show a high Krafft temperature (low solubility). As the "short" chain grows in length (and the other chain decreases), the effect on the space that the longer chain can occupy will increase. More of the space will be restricted by the presence of the other chain. Consequently, the occupied space beyond the benzene ring will become less symmetrical. As a result, the molecules will pack less readily together, and the solubility will increase. However, as the "shorter" chain begins to approach the length of the "longer" chain, the two chains will influence each equally, and the space will become symmetrical again, with two "lobes." Again, the solubility would be expected to decrease as the more symmetrical molecule can readily pack into a lattice.

The seemingly contradictory observation that a high 2-phenyl single isomer NaLAS has the lowest solubility but a commercial NaLAS with a high 2-phenyl distribution has the greatest solubility has been previously noted (8). This contradiction is resolved, however, if one considers the effect of mixed micelles on the concentration of monomers in solution (9). When mixed micelles form, the concentration of the monomer of each component is determined by the monomer mole fraction in solution and the CMC of the mixture. Once the monomer concentration and counter-ion concentration are known, their product can be computed and compared to the K_{sp}. The temperature at which the smallest K_{sp} is exceeded marks the precipitation of the least soluble component of the solution and also the Krafft point of the solution. Mixed micelle formation is not possible of course when only a single phenyl isomer is present, as in Figure 12.

Detergency. The impact of average carbon chainlength, phenyl isomer distribution, and dialkyltetralin sulfonate content on detergency is shown in Figures 14 and 15. The effect of average carbon chainlength on dust-sebum/cotton detergency is shown in Figure 14A and for dust-sebum/permanent press detergency Figure 14B. Highest detergency performance is obtained with a higher average carbon chainlength under low hardness conditions, while a lower average carbon chainlength is best under high water hardness conditions. Both the surface activity of the surfactant and its concentration affect detergency performance. Though higher average alkyl chainlength NaLAS has greater surface activity, it also has greater hard water sensitivity, which reduces its concentration under high water hardness conditions (10).

The effect of dialkyltetralin sulfonate level on detergency is shown in Figure 15A. The differences in the figure are not statistically significant at the 95% confidence level. Therefore, over the range of dialkyltetralin sulfonate concentration of commercial NaLAS, no difference is found in detergency.

The effect of phenyl isomer distribution on detergency is shown in Figure 15B. Again, the differences are not statistically significant at the 95% confidence level. Over the range of distributions of commercial NaLAS, no difference is found in detergency.

Foam stability. The impact on foam stability of average molecular weight, phenyl isomer distribution, and dialkyltetralin sulfonate content, as measured in dishwashing tests and soil titration tests is shown in Figures 16 and 17. Figure 16 shows that, in soft water, optimal performance is obtained with a high-molecular-weight NaLAS. In hard water, optimal performance is obtained with lower-average-molecular-weight NaLAS. Contrary to what is observed in detergency, overall foam performance increases with increasing water hardness. This may be because water hardness ions help stabilize the foam lamella (11).

Figure 17 shows the weight of soil necessary to break the foam generated in a stirred reactor vessel. The values are averages of three measurements and are statistically the same at the 95% confidence limit. Consequently, at the same average

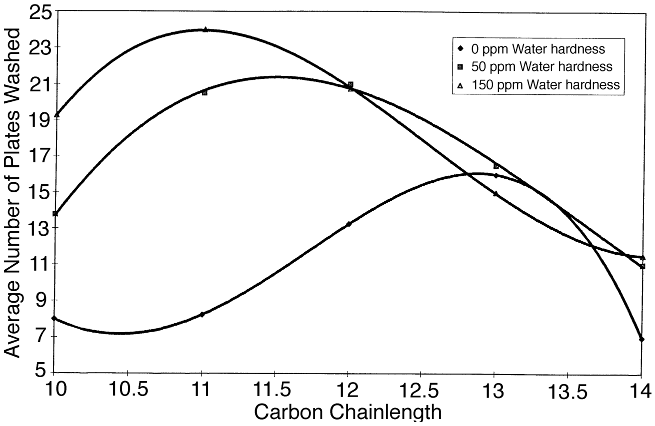

FIG. 16. Effect of carbon chainlength on foam stability of C_{11}, low 2-phenyl, low dialkyltetralin sulfonate content NaLAS. 24% NaLAS/6% alcohol ether sulfate/2% lauryl myristyl monoethanolamide, use level = 0.05%, vegetable shortening soil. T = 115°F. See Figure 6 for abbreviation.

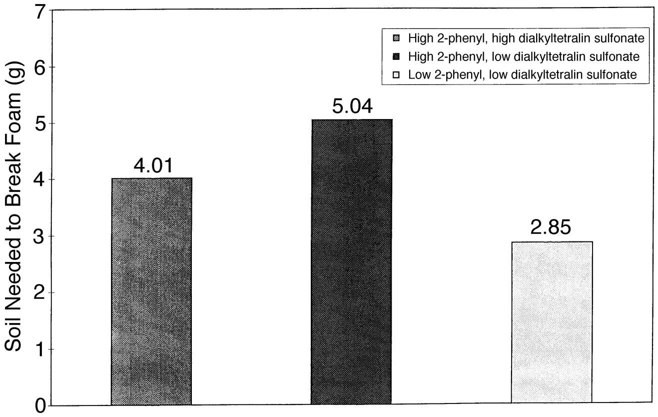

FIG. 17. Effect of phenyl isomer distribution and dialkyltetralin sulfonate content on foam stability of C_{11} average NaLAS. T = 45°C, use level = 400 ppm surfactant, water hardness =150 ppm.

carbon chainlength, foam stability is not affected by phenyl isomer distribution and dialkyltetralin sulfonate content over the range found in commercial NaLAS.

In conclusion, carbon chainlength is the most important factor in performance of NaLAS. All features of performance studied are affected by carbon chainlength. On the other hand, phenyl isomer distribution and dialkyltetralin sulfonate content affect only solubility and viscosity.

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