Solubilization in Mixed Micelles[1]

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Solubilization capacities for decane have been determined for a variety of mixed surfactant systems. Negative deviations from additivity are observed for nonionic/ionic mixtures where there are no attractive interactions between the surfactant headgroups. "Synergistic" positive deviations from additivity are observed for anionic/cationic mixtures, where strong attractive forces between the headgroups exist. The results are interpreted in terms of curvature effects using the Israelachvili, Mitchell and Ninham v/a_0l model and a model based on Laplace pressures. Data are also presented for the $C_{12}TAB/C_{12}EO_6$ system which clearly shows the differences in the solubilization sites for hexanol, which solubilizes in the palisade region, and decane, which solubilizes in the micellar core. The results indicate that any future modeling will have to include surfactant-solubilizate interactions as well as surfactant-surfactant interactions.

There has been a considerable amount of interest recently in mixed surfactant systems, both from practical and fundamental viewpoints (1-9). From the practical standpoint, virtually all current commercial cleaning products are mixtures of surfactants, be they simple chain homologues and isomers, or more complicated mixtures of dissimilar surfactants. In fact, the enhanced performance of many new formulations has been linked to "synergistic boosts" brought about by the interactions between the surfactant components (1,2). Much of the current literature has focused on theoretical modeling of mixed micelle formation in aqueous solutions (4-9). The regular solution theory approximation developed by Rubingh (4,5) is frequently used to model critical micellization concentrations for binary surfactant mixtures. The theory contains one adjustable parameter, W/RT, which is related to the degree of interaction between the two surfactants. Little work has been done to physically characterize mixed micelles in other areas.

One very interesting area that warrants study is solubilization. Solubilization is an important phenomenon in cleaning and detergency, especially for laundry applications where it is an important mechanism in oily soil removal. This area is becoming increasingly important because of the trend towards low temperature washing conditions imposed by the use of brightly colored synthetic fabrics and energy conservation considerations. At lower washwater temperatures, the roll-up mechanism for detergency is impaired, due to the increased viscosity of the soil adhered to the fabric (10). This is especially critical for oily soils which tend to adhere to synthetic fabrics in a stronger fashion (10).

This paper will examine the solubilization properties of the nonpolar oil, decane, in binary mixtures of surfactants. The results will be discussed in terms of the effects of surfactant composition on the curvature of the mixed micellar aggregate. Also, a comparison will be made between the solubilization properties of decane in the micellar core and hexanol which solubilizes into the palisade region of the micelle.

MATERIALS AND METHODS

Materials. Dodecyltrimethylammonium chloride ($C_{12}TAC$) and bromide ($C_{12}TAB$) were obtained from the Eastman Kodak company and purified by repeated recrystallization from anhydrous acetone and an acetone/ethanol solvent pair, respectively. Although $C_{12}TAB$ showed no minimum in the surface tension vs concentration plot, $C_{12}TAC$ did exhibit a small 1-2 dyne/cm minimum, indicative of the presence of a small amount of surface active impurity. Sodium dodecylsulfate (SDS) was obtained from Sigma and purified by recrystallization from ethanol, while hexaethyleneglycol mono n-dodecyl ether ($C_{12}EO_6$) was obtained from the Nikko chemical company and used as received. Decane (gold label) and hexanol were obtained from Aldrich.

Methods. Surface tensions were measured using a Wilhelmy plate technique with a Krüss model K10-PST tensiometer. The temperature was regulated at 25°C.

Quasielastic light scattering measurements were performed in Professor Kaler's laboratory at the University of Washington, and repeated here at the Clorox Technical Center using a Brookhaven Instruments Inc. system, equipped with a model BI-200SM goniometer, and BI-2030 digital correlator. A Spectra-Physics model 124B 35 mW helium-neon laser was used as the source, and photons were detected with an EMI 9865A photomultiplier tube. The sample was contained in a cylindrical tube, and immersed in an index matching fluid (dodecane, Aldrich). Signals were collected at a 90 degree scattering angle and analyzed by the method of cumulants. Diffusion coefficients were measured at a number of concentrations for the mixed systems, and the values extrapolated to zero concentration, D_0, were used in the calculation of the apparent hydrodynamic radii from the Stokes-Einstein relation. The diffusion coefficients were obtained in 0.3 M NaBr to reduce the electrostatic complications arising from intermicellar interactions (11).

Samples for the determination of decane maximum additive concentrations (MAC) were prepared by tumbling 3.0 mL of surfactant solution with 0.3 mL of decane for 72 hr at 21°C. Some degree of emulsification was found to occur, especially with the pure nonionic solutions. For this reason the volume of decane was kept to a minimum in order to reduce the error created by surfactant solubilization into the organic layer. The samples were then centrifuged at 3000 rpm for 30 min at 21°C to separate the aqueous/oil layers. The aqueous layer was subsequently sampled, and the capacity was determined by a gas chromatographic method. A 3.0 mL aliquot of dodecane/isopropyl alcohol (0.3-2 mg dodecane/mL IPA) internal standard was added to 0.5 mL of the decane saturated micellar solution, and two microliters of this solution was injected into the sample port of a Varian model 3700 gas chromatograph equipped with a flame ionization detector. The column used was a 3% Supelco 2100, and the output signal was analyzed with a Varian

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4270 integrator. The measured peak areas were then translated into the amount of decane present using calibration curves developed for the internal dodecane standard. Hexanol capacities were determined using a turbidimetric method in which samples of hexanol plus surfactant solution were tumbled for 72 hr at 21°C. The samples were then freshly shaken, and the turbidity was measured visually and with a Brinkmann PC 800 probe colorimeter. The highest hexanol concentration at which a clear solution was obtained was presumed to be the hexanol MAC. The 95% confidence intervals were typically ±9% relative or less for both methods, based on triplicate analyses.

RESULTS

The regular solution theory (4,5) interaction parameters (W/RT) for the binary surfactant mixtures studied are presented in Table 1. Negative values of W/RT indicate a reduction in the free energy of micellization over that predicted by ideal solution theory. The larger the negative value, the greater the degree of synergistic surfactant-surfactant interactions. The W/RT parameters were determined from critical micellization concentration (CMC) plots for surfactant mixtures with no solubilizate present. Thus, they take into account surfactant-surfactant interactions only, and exclude the effect of solubilization upon the CMC. The mixtures range from strongly interacting anionic/cationic pairs with W/RT = −25 to weakly interacting ionic/nonionic systems with W/RT = −5. For the anionic/cationic surfactant pair (C_{12}TAC/SDS), the CMC's were measured in the composition range from X_{SDS} = 0.86–1.0, where no precipitation occurs. The solubilization data was obtained at 70°F (ca. 21°C). This is well below the cloud point for the $C_{12}EO_6$ (cloud point = 55°C) so that critical phenomena will not play a major role in the trends observed.

Measurements of the maximum additive concentration (MAC) were performed at various mole fraction ratios of the two surfactant components, with the total surfactant concentration held constant. The MAC is defined as:

$$\mathrm{MAC} = (C_{o,t} - C_{o,w})/(C_s - \mathrm{CMC}) \qquad [1]$$

where $C_{o,t}$ is the total moles of solubilizate dissolved in the aqueous phase; $C_{o,w}$ is the moles of solubilizate singly dispersed in the pure water phase. This is negligible since the water solubility for decane is 6×10^{-8} moles/liter (12). C_S is the total surfactant concentration, and CMC the critical micellization concentration of the mixture studied. More simply, the MAC is the total moles of solubilizate in the micelles divided by the total moles of surfactant in micelles. The total surfactant concentration was 50 mM or higher so that the overall surfactant composition is essentially equal to the mixed micellar composition.

Figures 1 and 2 present MAC vs mole fraction plots for decane solubilization in two sets of binary surfactant mixtures. These two surfactant mixtures give very diverse behavior in terms of their decane solubilization properties. It appears that the data can be divided into two groups: surfactant mixtures showing negative deviations from additivity or linear mixing, and those showing positive deviations, where additivity is defined as:

$$(\mathrm{MAC})_{1,2} = X_1\,(\mathrm{MAC})_1 + (1 - X_1)\,(\mathrm{MAC})_2 \qquad [2]$$

here the subscripts 1 and 2 and (1,2) refer to surfactants one and two, and their mixtures, respectively, and X denotes the mole fraction in the micelle.

The weakly nonideal system pictured in Figure 1 is a nonionic/ionic mixture, and the negative deviations from additivity observed are consistent with solubilization data for Yellow OB in nonionic/ionic mixtures observed previously by Nishikido (13). Nishikido attributed the lower mixture solubilization to a decrease in the compactness for the polyoxyethylene (POE) chain of the nonionic following insertion of ionic surfactant. For decane which solubilizes in the micelle core, changes in the compactness of the POE region would not be expected to have large effects on its solubilization (9). However, a similar trend is observed, which points to the need for a more general explanation for this process. Also plotted in Figure 1 are the hydrodynamic radii obtained from QLS measurements. A correlation is observed between the hydrodynamic radii and the decane MAC data as a function of mole fraction.

TABLE 1

Interaction Parameters in Mixed Micelles

Mixture	W/RT
$C_{12}TAB/C_{12}EO_6$	−5.3
$C_{12}TAC/SDS$	−25

$C_{12}TAB = C_{12}H_{25}N(CH_3)_3Br$

$C_{12}EO_6 = C_{12}H_{25}(CH_2CH_2O)_6H$

$SDS = C_{12}H_{25}SO_4Na$

$C_{12}TAC = C_{12}H_{25}N(CH_3)_3Cl$

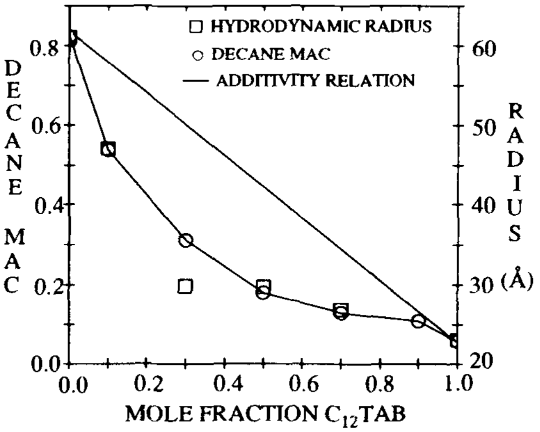

FIG. 1. Plot of the MAC for decane solubilization vs mole fraction C_{12}TAB in binary mixtures of C_{12}TAB and $C_{12}EO_6$. The total surfactant concentration is constant at 100 mM. Also plotted are the additivity relation, illustrating negative deviations from additivity, and the hydrodynamic radii, which correlate well with the MAC data.

Nishikido's model does not contain any compactness terms for ionic surfactants, and hence does not apply to the strong positive deviations from additivity observed for mixtures of anionic (SDS) and cationic ($C_{12}TAC$) surfactants (Fig. 2). Turbid solutions were observed between X_{SDS} = 0.22–0.86, due to ion-pair precipitation. The positive deviations from additivity observed for the strongly interacting anionic/cationic mixtures are consistent, however, with an apparently "anomolous" finding in 1949 by Lambert and Busse (14), who observed that a 1:2 mixture of cetyl pyridinium chloride and Igepon T (anionic surfactant) solubilized ca. 40% more Orange OT than either material separately. Figure 2 again shows a good correlation between the measured aggregation numbers (15) and the decane MAC data.

In Figures 3 and 4 a comparison is made between decane solubilization into the micelle core and hexanol solubilization into the palisade region of the mixed micelles. Figure 3 shows that there is an opposite trend in the hexanol vs decane solubilization data. The hexanol data are consistent with data published by Nguyen *et al.* (16) for hexanol solubilization into ionic/nonionic mixtures.

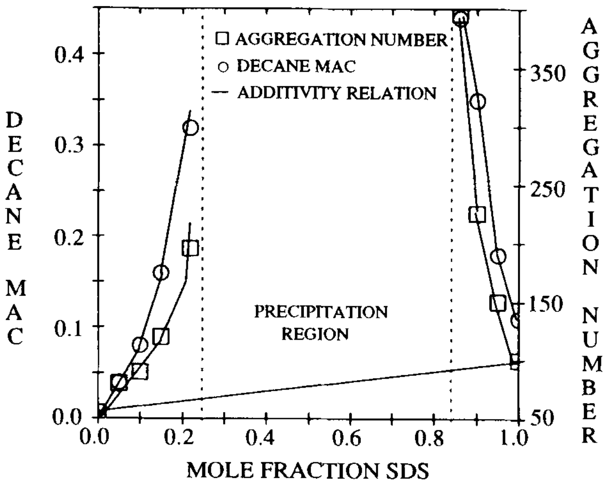

FIG. 2. Plot of the MAC for decane solubilization vs mole fraction SDS in binary mixtures of SDS and $C_{12}TAC$, illustrating the strong positive deviations from additivity observed. Also plotted are the aggregation numbers obtained by Malliaris *et al.* (15) for this system.

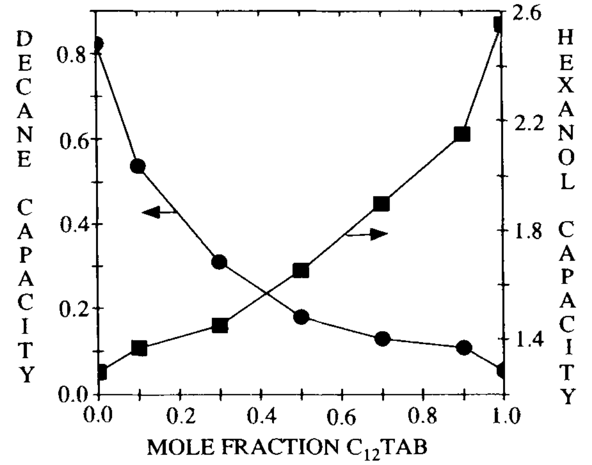

FIG. 3. Comparison of decane solubilization vs hexanol solubilization in binary mixtures of $C_{12}TAB$ and $C_{12}EO_6$. The total surfactant concentration is 100 mM.

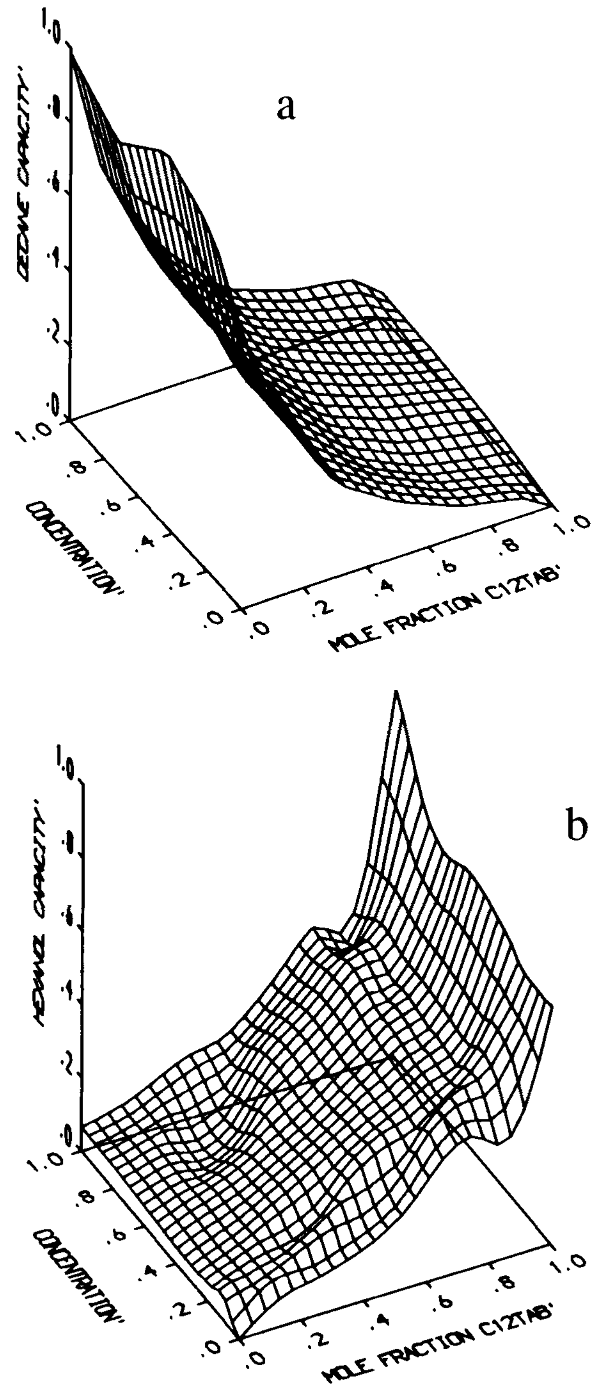

FIG. 4. Three-dimensional plots of the molar solubilization capacity vs mole fraction $C_{12}TAB$, and total surfactant concentration, for binary mixtures of $C_{12}TAB$ with $C_{12}EO_6$. In (a) the decane capacity is shown. For the three normalized axes the values range from: (decane capacity: 0–0.86; mole fraction 0–1.0; concentration 5–100 mM). In (b) the hexanol capacity is shown. The normalized values range from (hexanol capacity: 1.20–2.98; mole fraction 0–1.0; concentration 1–100 mM).

Figure 4 presents the solubilization data in a slightly different manner. Here, the surfactant concentration dependence is shown explicitly in three-dimensional plots of total surfactant concentration vs mole fraction vs molar solubilization capacity (moles oil solubilized in micelles/total moles surfactant). This "capacity" term differs from the MAC in that the CMC has not been subtracted in this case. This was done because MAC values can become artificially large when the total surfactant concentration is near the CMC, because the denominator (C_S − CMC) goes to zero. The concentration dependence near the CMC was studied because the composition of the monomer and micelle phases can actually be very different from the bulk compositions. That is, the micelle phase will be enriched in the more surface active component. For the ionic/nonionic surfactant mixtures near the CMC, most of the surfactant in micelle form will be nonionic, while most of the monomer will be made up of ionic surfactant. Therefore, it might be expected that the solubilization capacity would vary with concentration because the curvature of the micelle aggregate may also vary. While examining Figure 4, it is once again observed that decane and hexanol exhibit opposite trends in their solubilization properties. It should also be noted from Figure 4 that there appears to be no explicit concentration dependence on the observed solubilization properties: at low total surfactant concentrations the solubilization capacity does not deviate appreciably from other mixtures at higher concentrations and of the same bulk composition. Therefore, the solubilization properties are not able to show the predicted changes in micelle composition as a function of concentration.

DISCUSSION

Decane solubilization in mixed micelles. In a review on solubilization by solutions of surfactants Rosen (17) states that: "For hydrocarbons that are solubilized in the interior of the micelle, the amount of material solubilized generally increases with an increase in the size of the micelle. Therefore, any factor that causes an increase in either the diameter of the micelle or its aggregation number can be expected to produce increased solubilization for this type of material." Mixing surfactants is a very efficient way of changing micelle size and curvature (11,15).

The geometric aspects governing amphiphile aggregation into micelles have been modeled by Israelachvili *et al.* (18,19). The model contains three adjustable parameters: v, the volume of the hydrophobic chain; a_o, the effective headgroup area; and l, the length of the hydrophobic chain. When $v/a_ol \leqslant 1/3$, spherical micelles result. When $1/3 \leqslant v/a_ol \leqslant 1/2$, the spherical micelles deform into oblate ellipsoids, and eventually into infinite rods at $v/a_ol = 1/2$. Thus, in terms of core solubilization, larger values of v/a_ol will lead to enhanced solubilization. In this communication, variations in the a_o parameter achieved by mixing surfactants will be presented, although similar trends may also be observed by varying the v and l parameters as well. The tail group of the surfactants is kept constant at C_{12} so that the v and l parameters are similar in each case. The forces which hold micelles together and determine a_o are not strong covalent bonds, but rather weak electrostatic forces, hydrogen bonding, and hydrophobic interactions. Consequently, subtle changes in electrolyte concentration or solution pH may have large effects both on the inter- and intramicellar interactions.

For the $C_{12}EO_6/C_{12}TAB$ mixture (Fig. 1), the introduction of the nonionic surfactant into the charged ionic surfactant results in a decrease of the electrostatic repulsions in the headgroup region and decreased a_o values. Typically, a_o ranges from 60–80 Å^2/molecule for pure ionics, to 40–50 Å^2/molecule for nonionics (19). This leads to v/a_ol values ranging from ca. 0.33 (spheres) to 0.43 (rods). The negative deviations from ideality, as evidenced by a negative regular solution theory parameter, are not due to any specific interactions between the ionic and nonionic headgroups, but rather to an attenuation of the electrostatic repulsions due to the presence of the uncharged nonionics. Gúering *et al.* (20) have shown that the introduction of charged dodecylsulfate anions into $C_{12}EO_5$ leads to an increase in the diffusion coefficients or a decrease in the micellar size. Similar interactions are operative in $C_{12}EO_6/C_{12}TAB$ mixtures and lead to the hydrodynamic radii presented in Figure 1. The decreasing micelle size leads to decreased decane solubilization.

In the $C_{12}TAC/SDS$ system the large negative interaction parameter ($W/RT = -25$) is indicative of a strong electrostatic attraction between the oppositely charged headgroups. This leads to dramatic reductions in the a_o value from ca. 70 Å^2/molecule for the pure ionics to ca. 30 Å^2/molecule for the mixtures. In fact, the decyltrimethylammonium-decylsulfate ion pair has a measured a_o value of 30 Å^2/molecule (21). This large reduction in the a_o value leads to the formation of long rod-shaped micelles with increased aggregation numbers. Malliaris *et al.* (15) have measured the aggregation numbers for the $C_{12}TAC/SDS$ system, and the trend mirrors the solubilization results presented here (Fig. 2). This correlation supports the curvature model for core solubilization. It is interesting to note that in previous studies (13,22) where synergisms in solubilization behavior have been observed, they have been attributed to attractive interactions between dissimilar headgroups. Tokiwa (22) found that attractive interactions between a benzene sulfonate group on an anionic surfactant and the polyoxyethylene portion of a nonionic surfactant lead to synergisms in solubilization behavior, while Nishikido (13) observed synergisms in solubilization behavior in nonionic/anionic surfactant mixtures when the POE length was long enough to form slightly positively charged oxonium ions. As Scamehorn points out (9), negative deviations from ideal solution theory do not lead to increased solubilization unless there is a specific attraction between the hydrophilic groups of the dissimilar surfactants. In both of these systems, reductions in the a_o value are probably responsible for the enhanced solubilization observed.

Laplace pressure effects. Another way to look at the effects of curvature variation in mixed micelles and its effect on solubilization properties is by using the concept of Laplace pressure. Laplace pressures arise from the curvature of the micellar interface and are given by:

$$\Delta P = 2\sigma/r \quad \text{(spheres)} \qquad [3]$$

where σ is the micelle core/water interfacial tension, and r is the core radius. Qualitatively, this pressure is greater than the pressure of the bulk solution and opposes the

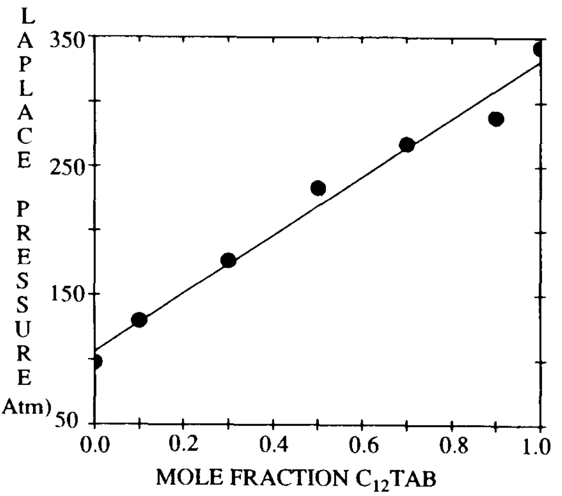

FIG. 5. Plot of the Laplace pressure vs mole fraction $C_{12}TAB$ in $C_{12}TAB/C_{12}EO_6$ mixtures. Laplace pressure data extracted from Figure 1 using equation [4].

entry of solubilizates into the micellar core. Mukerjee (23,24) has used the Laplace pressure effect to describe differences in the solubilizing power of homologous series of solubilizates into the micellar core. He finds that the Laplace pressure is less for octane solubilization into $C_{10}EO_{10}$ than for decane solubilization. In the present study, only one solubilizate, decane, is used, and the curvature of the micelle interface is varied by mixing dissimilar surfactants. If it is assumed that the decane mixes ideally, then the Laplace pressure can be calculated from measurable parameters using

$$\Delta P = -RT \ln X/V \qquad [4]$$

where R is the molar gas constant, T the absolute temperature, V the partial molar volume of the solubilizate, and X is the mole fraction of the solubilizate in the micelle.

A plot of the calculated Laplace pressure vs mole fraction $C_{12}TAB$ for $C_{12}TAB/C_{12}EO_6$ binary mixtures is given in Figure 5. The Laplace pressure data can be fit to a straight line with a correlation coefficient of .991. Recall that the MAC vs mole fraction plot (Fig. 1) shows negative deviations from additivity. From Figure 5, it can be inferred that these negative deviations in solubilization behavior are related to changes in the curvature of the mixed micellar aggregate, and although the MAC does not vary linearly with mole fraction, the curvature does change in a linear fashion. Using Figure 5, it is possible to predict a solubilization capacity for any binary mixture of $C_{12}TAB/C_{12}EO_6$. Note that this model appears to work well for mixtures where there are not significant attractions between the headgroups (i.e. weakly interacting systems, with negative deviations from solubilization additivity). The nonlinear variation in curvature for systems with positive deviations does follow the correct qualitative trend, however. As the anionic and cationic surfactants are mixed, the curvature of the micelle decreases and the Laplace pressure decreases, leading to the enhanced solubilization observed.

Core solubilization vs palisade solubilization. The introduction of ionic surfactant into a nonionic surfactant results in mixed micelles with increased hydrophilic character. Consequently, the area per headgroup increases (from ca. 40 Å^2/molecule to 70 Å^2/molecule as discussed earlier), and the space available for solubilization between the surfactant molecules in the palisade layer increases. Thus hexanol solubilization, which occurs in the palisade layer, increases as more ionic surfactant is introduced (Figs. 3,4). On the other hand, the increased hydrophilic character leads to reductions in the aggregation numbers for the mixed aggregates. This leads to lower solubilization capacities for nonpolar substances such as decane, which solubilize in the micellar core (Figs. 3,4). The mixed micelle data show clearly that at least two sites for solubilizates exist, and that any future modeling should take into account not only surfactant-surfactant interactions, but also surfactant-solubilizate interactions.

Treiner *et al.* (25) have attempted to model mixed micelle solubilization in terms of surfactant-surfactant interactions only, hypothesizing that deviations from additivity in solubilization behavior are due to nonideality in the surfactant-surfactant interactions. They include a term into the additivity equation based on Rubingh's model (4,5) for nonideal surfactant interactions. Treiner's model works surprisingly well for solubilizates such as pentanol, which solubilize between the surfactant headgroups, where they are able to sense the surfactant-surfactant interactions. Treiner's model predicts that if the regular solution theory interaction parameter for a surfactant mixture is negative, then the solubilization capacity for a nonpolar solubilizate should be less in the mixture than what is predicted from normal additivity arguments. Thus, it would be impossible to get synergistic solubilization behavior for systems with negative W/RT. There are already many exceptions to this rule, the most glaring being the $C_{12}TAC/SDS$ mixture (Fig. 2), where W/RT = −25 and large synergisms from additivity are observed. Just as Nishikido's model fails to predict large changes for solubilization into the micelle core, so too does the Treiner model fail to account for the large synergisms observed in core solubilization. These models fail to take into account the roles of curvature and surfactant-solubilizate interactions in these mixed systems.

Unfortunately no sound quantitative model for mixed micellar solubilization exists today, although this study shows that curvature effects play a large role for solubilization into the micelle core. This study also shows that the role of curvature can be qualitatively predicted using the v/a_0l approach of Israelachvili *et al.* (18,19), and quantitatively, in some cases, using the Laplace pressure concept (23,24). Any future models for core solubilization must take into account curvature effects, and as the hexanol vs decane plots point out, surfactant-solubilizate interactions as well.

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