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ORIGINAL CONTRIBUTION

Đ. Dragčević
M. Bujan
Ž. Grahek
N. Filipović-Vinceković

Adsorption at the air/water interface in dodecylammonium chloride/sodium dodecyl sulfate mixtures

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Đ. Dragčević · Z. Grahek
Ruđer Bošković Institute
Bijenička 54
10000 Zagreb, Croatia

M. Bujan
Faculty of Agriculture
Ruđer Bošković Institute
Bijenička 54
10000 Zagreb, Croatia

Dr. N. Filipović-Vinceković (✉)
Ruđer Bošković Institute
Department of Physical Chemistry
Bijenička 54
10000 Zagreb, Croatia

Abstract The composition and properties of the adsorption films of dodecylammonium chloride/sodium dodecyl sulfate at the air/water interface depend on interactions between the film molecules and equilibria in the bulk phase (monomer-micelle and/or monomer-precipitate equilibria).

The negative value of surface molecular interaction parameter β^{mon} calculated using the regular solution theory indicates strong attractive interactions between adsorbed molecules. Electrostatic interactions between oppositely charged ionic head groups enhance the adsorption of surfactants and decrease the minimum molar area of surfactant molecules at the air/water interface. The addition of an oppositely charged surfactant enhances packing at the air/water interface and transition from a liquid expanded to a liquid condensed state. Surface potential measurements reveal positive values for the mixtures investigated, implying the cationic surfactant ions are closer to the surface than the anionic ones.

Key words Cationic/anionic surfactant mixtures – air/water interface – surface potential

Introduction

Numerous investigations of the adsorbed films at the air/water interface in the mixtures of cationic and anionic surfactant solutions have been reported [1–7]. The adsorption at the interface in a mixture of surfactants results in the formation of a mixed monolayer. The forces tending to cause mixed monolayer formation are similar to those causing formation of mixed micelles in the bulk phase. The strong electrostatic attraction of oppositely charged hydrophilic groups adjacent to each other at the air/water interface causes a large negative deviation from ideal mixing. The composition of mixed adsorbed films depends on the concentration ratio of the surfactants, the difference in their surface activity and the concentration of electrolytes in the solution.

In previous papers we reported the phase behavior of cationic/anionic mixtures of surfactants of the same hydrocarbon chain length whose ionic groups are opposite in charge and different in size (sodium alkyl sulfate, RSO_4Na, and alkylammonium chloride, RNH_3Cl, [8–10]. Precipitation of the catanionic surfactant and formation of mixed micelles in the excess of one of the surfactants was observed. Liquid crystalline phase was found in the transition region from solid crystalline catanionic surfactant to mixed micelles. In the concentration range where monomer-micelle equilibrium prevailed, mixed micelles of different composition were formed.

The present paper provides additional information on the relation between cationic and anionic surfactant adsorption at the air/water interface and phase behavior in the adjacent bulk phase. By using the regular solution theory, the composition of the mixed monolayer, surface

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activity coefficients of the components and the surface molecular interaction parameter were determined. It has been shown that the mixed adsorbed films are in equilibrium with the phases formed in bulk solution. Measurements have revealed that the surface potential of the mixed films investigated has a positive value. Among the numerous investigations of mixed binary surfactant solutions, there are a few studies on the electrostatic interactions in mixed cationic/anionic films [5, 6]. At least a part of the missing information can be obtained by surface potential measurements.

Experimental

Materials

Dodecylammonium chloride (DDACl) was prepared by neutralization of dodecylamine with HCl (11). Commercially obtained sodium dodecyl sulfate (SDS) was purified by repeated recrystallization. Surfactant purity was checked by observing that the surface tension (γ) vs. concentration (c) curve has no minimum.

Systems were prepared either with a constant anionic and increasing cationic component or vice versa.

The experiments were carried out at 298 K, except for surface potential measurements which were performed at ambient temperature (from 293 to 296 K).

Methods

Surface tension (γ) was measured by an Interfacial Tensiometer (K8, Krüss, Hamburg) and Langmuir trough.

In the Langmuir trough, γ and surface potential (ΔV) were measured simultaneously under static conditions [12]. γ was measured with a carbon-coated platinum Wilhelmy slide, using a Cahn electrobalance. ΔV was measured directly as a difference between two identical Au-plated electrodes, one over the reference liquid (clean water), and another over the surfactant solution. The electrodes were connected to a high impedance electrometer (Keithley Model 604, Ohio, USA), and the resulting voltage difference fed to the recorder. Both electrodes referred to common electrical ground by large reversible Ag/AgCl electrodes immersed in the liquid. Two identical ^{241}Am α-emitting sources, each of 2.5×10^{10} Bq were used. The precision of γ measurements was from ± 0.1 mN m^{-1} for lower surfactant concentrations to ± 2 mN m^{-1} for higher surfactant concentrations. The diminished precision of the measurements was attributed to the carbon-coated platinium Wilhelmy slide. The ΔV precision was ± 10 mV for solutions of lower concentration and ± 50 mV for solutions of higher concentration. ΔV stability in the measured time period was ± 10 mV for DADCl while for SDS fluctuations within this time period were observed. The static values of γ and ΔV were recorded within 10 min of pouring new solution into the trough.

Interpretation of data

Using the regular solution theory [13–15] the composition of a mixed monolayer, the surface activity coefficient of the components and the strength of interaction between surfactants in the mixed monolayer can be determined. The basic equation is

$$1 = \frac{(X_{\mathrm{DDACl}})^2 \ln(\alpha_{\mathrm{DDACl}}\, C_{12}/C_{\mathrm{DDACl}}\, X_{\mathrm{DDACl}})}{(1 - X_{\mathrm{DDACl}})^2 \ln((1-\alpha_{\mathrm{DDACl}})\, C_{12}/(1 - X_{\mathrm{DDACl}})\, C_{\mathrm{SDS}})}, \tag{1}$$

where α_{DDACl} is the mole fraction of DDACl in the bulk phase. X_{DDACl} is the mole fraction of DDACl in the mixed monolayer, C_{DDACl}, C_{SDS} and C_{12} are solution phase molar concentrations of DDACl, SDS and their mixture, respectively, required to produce the same surface pressure (the surface pressure (Π) is defined by the equation $\Pi = \gamma_0 - \gamma$, where γ_0 is the surface tension at the pure water/air interface). The iterative solution of the above equation gives X_{DDACl}.

The surface molecular interaction parameter (β^{mon}) for the mixed monolayer and the surface activity coefficients for individual DDACl (f_{DDACl}) and SDS (f_{SDS}) in the mixed monolayer were calculated by the following equations

$$\beta^{\mathrm{mon}} = \frac{\ln(\alpha_{\mathrm{DDACl}}\, C_{12}/C_{\mathrm{DDACl}}\, X_{\mathrm{DDACl}})}{(1 - X_{\mathrm{DDACl}})^2} \tag{2}$$

$$f_{\mathrm{DDACl}} = \exp \beta^{\mathrm{mon}} (1 - X_{\mathrm{DDACl}})^2 \tag{3}$$

and

$$f_{\mathrm{SDS}} = \exp \beta^{\mathrm{mon}} X_{\mathrm{DDACl}}{}^2 \,. \tag{4}$$

The surface excess of either single surfactant molecules at the air/water interface was calculated (Gibbs adsorption equation) from the slope of γ vs. $\ln c_{\mathrm{DDACl}}$ or vs. $\ln c_{\mathrm{SDS}}$ when the concentration of the other surfactant was held constant. In the absence of electrolyte the surface excess of the DDACl (Γ_{DDACl}) and SDS (Γ_{SDS}), respectively, was calculated [16, 17] by the following equations

$$\Gamma_{\mathrm{DDACl}} = -1/2\, RT (d\gamma/d \ln c_{\mathrm{DDACl}})_{c_{\mathrm{SDS}}} \tag{5}$$

and

$$\Gamma_{\mathrm{SDS}} = -1/2\, RT (d\gamma/d \ln c_{\mathrm{SDS}})_{c_{\mathrm{DDACl}}}, \tag{6}$$

where c_{SDS} and c_{DDACl} are the concentrations of SDS and DDACl, respectively, R is the gas constant and T is the absolute temperature. The minimum area occupied by a surfactant molecule adsorbed at the interface (A/nm^2), when Γ is expressed in moles cm^{-2}, is

$$A = 10^{14}/\Gamma N_A \tag{7}$$

where N_A is Avogadro's number.

Results and discussion

An earlier published [8] phase diagram for DDACl/SDS mixtures served as an orientation to select systems for examination. Two cross-sections on the SDS-rich side, an equimolar cross-section and three cross-sections on the DDACl-rich side chosen for this investigations are indicated in Fig. 1. The results obtained may be conveniently divided into two parts. The first part comprises the results of surface tension and surface potential measurements for single DDACl, SDS and their equimolar solutions. The second part shows the results of surface tension and surface potential measurements for the cross-sections on the SDS-rich and DDACl-rich side.

Figure 2a shows typical γ vs. log$c$ curves for SDS, DDACl and their mixed solutions at an equimolar ratio. The fact that a much lower concentration for equimolar than that of the cationic or anionic surfactant alone is required to reach a constant surface tension value indicates strong synergism. The break point between the steeply descending and fairly constant branch of the γ-log$c$ curve for mixed solutions corresponds to the precipitation of the catanionic surfactant, dodecylammonium dodecyl sulfate (DDADS). In a narrow concentration range, close to the equimolar bulk composition soluble ion-pairs are formed in the bulk phase before DDADS precipitation [10].

By applying the regular solution theory, the composition of the mixed monolayer from equimolar mixtures was calculated (the values of C_{DDACl}, C_{SDS} and C_{12} were read from Fig. 2 for $\Pi = 32\ \mathrm{mN\,m^{-1}}$). The results obtained exhibit equimolar mixed monolayer formation with activity coefficient values much less than unity ($f_{\mathrm{DDACl}} = f_{\mathrm{SDS}} \approx 0.004$) indicating large deviations from ideality. The formation of equimolar adsorption films is in accord with the literature data, especially for surfactants of the same chain length [2, 7]. The relatively high negative value of β^{mon} (-22.1) indicates strong attractive interactions between DDA^+ and DS^- ions.

Surface potential was also recorded while compressing the monolayer. Figure 2b illustrates the change of ΔV vs. surfactant concentration. Comparison of the surface potential and surface tension measurements indicates that the former method is more sensitive for detecting changes at the air/water interface. The behavior of single components is typical of the cationic and anionic surfactant, respectively, DDACl, as a more surface active surfactant exhibits higher absolute surface potential values. The ΔV-value for SDS solutions increases up to $10^{-4}\ \mathrm{mol\,dm^{-3}}$ (at this particular concentration γ begins to decrease strongly) and then becomes constant, indicating that a saturated electrostatic layer of dodecyl sulfate ions is attained below the CMC for SDS. It may be explained by rearrangement of SDS molecules at the air/water interface as was shown by the radiotracer studies (two-dimensional micelle formation at the air/water interface) [18].

It is known that the surface potential of ionic surfactant is due to the rearrangement of dipoles at interfaces and a contribution of the electric double layer [19]. Consequently, the measured surface potential will depend on the electrostatic

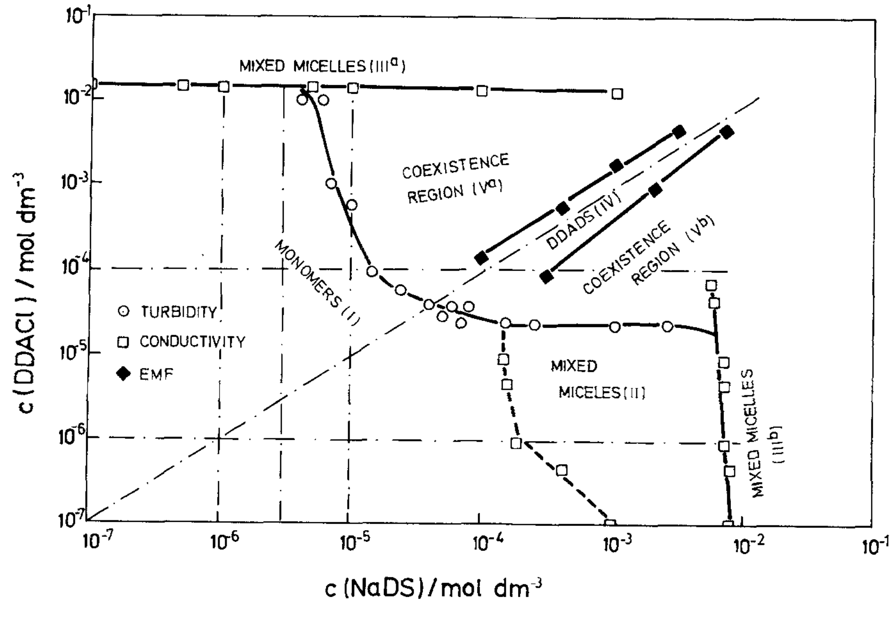


Fig. 1 Phase boundaries observed in (DDACl/SDS) systems: I. monomers and/or ionic pairs: II. mixed micelles with a moderate excess of SDS; III^a. mixed micelles with high DDACl content; III^b. mixed micelles with high content of SDS; IV. solid crystalline DDADS, V^a and V^b coexistence region of solid crystalline DDADS and liquid crystalline phases. The dashed lines indicate the investigated cross-sections (after ref. [8])

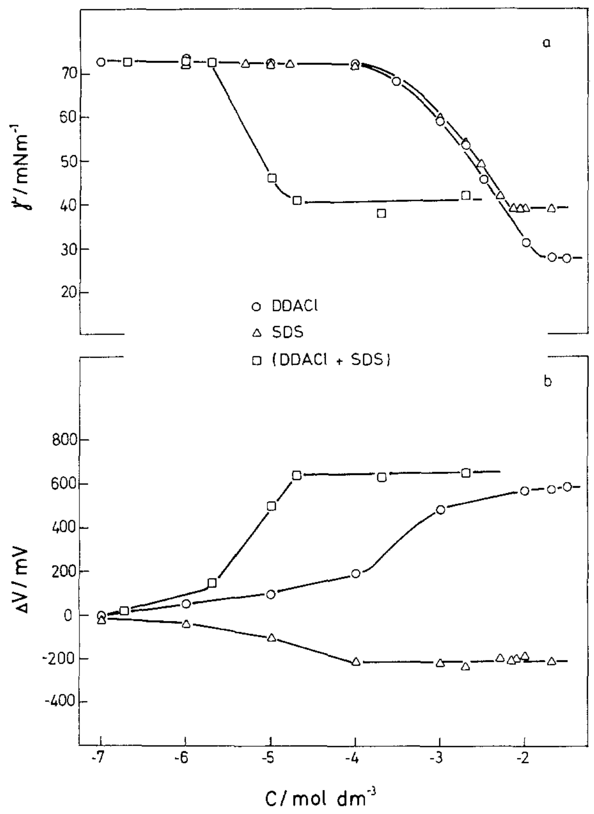


Fig. 2, a Surface tension (γ) of single SDS and DDACl solutions and of their equimolar mixtures vs. total surfactant concentration. **b** Change in surface potential (ΔV) with surfactant concentration (c) of single SDS and DDACl solutions and their equimolar mixtures. Temperature: from 293 to 296 K

potential due to the charge of the head groups and the corresponding dipole moments of each component (dipole moments of the sulfate and ammonium head groups, associated water and the terminal – $\mathrm{CH_3}$ groups). Assuming that the ratio of positive and negative headgroups in a mixed adsorption film is 1:1 one may conclude that the monolayer should behave as a monolayer of the nonionic surfactant.

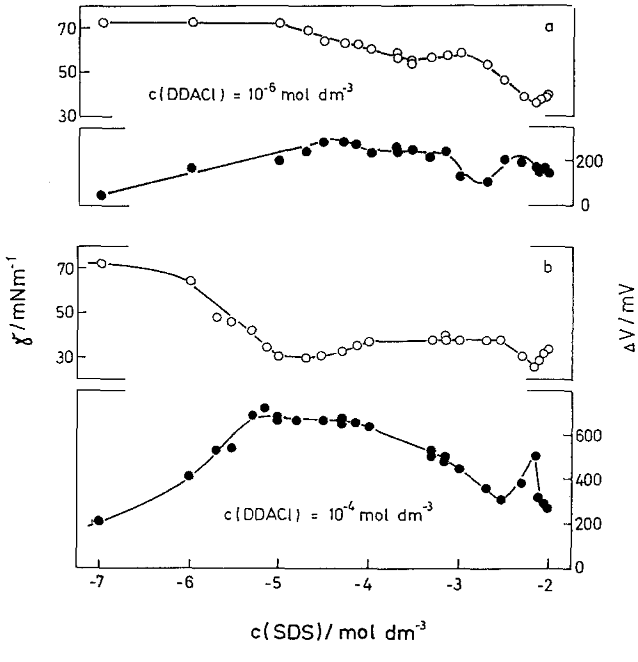
The surface potential of an equimolar DDACl/SDS bulk mixture has even higher positive values than that of the simple DDACl film. This result is rather surprising and difficult to explain only by dipole moment contributions. Obviously, the negative groups embedded in the bulk phase are effectively screened, so they do not dominate surface potential. This implies a specific orientation of the molecules adsorbed at the air/water interface; on the average, $\mathrm{DDA^+}$ ions are closer to the surface than $\mathrm{DS^-}$ ions. Such behavior may be explained by a strong tendency of the negative sulfate group to orient itself deeper into water compared to the positive ammonium group due to the differences in their geometry and size, solubility and/or binding of the counterions in the underlying layer.

Investigations of phase behavior [8] revealed asymmetry in the SDS-rich systems; a more pronounced shift of the $\mathrm{CMC_{SDS}}$ with DDACl addition, higher decrease in surface charge density at the mixed micelle/solution interface and two regions of the mixed micelle formation. At lower SDS concentrations mixed micelles with a small excess of the anionic component and close to the $\mathrm{CMC_{SDS}}$ mixed micelles with SDS in high excess were formed. The mixtures prepared on the DDACl-rich side showed mixed micelles formation only in the region of the $\mathrm{CMC_{DDACl}}$.

Changes of surface tension and surface potential in two cross-sections on the SDS-rich side (one cross-section corresponding to the isotropic region ($c(\mathrm{DDACl}) = 1 \times 10^{-6}\ \mathrm{mol\,dm^{-3}}$) and the other extending into the coexistence region (coexistence of solid crystalline DDADS and liquid crystalline phase, $c(\mathrm{DDACl}) = 1 \times 10^{-4}\ \mathrm{mol\,dm^{-3}}$) are presented in Figs. 3a and 3b. The examination of surface tension curve (Fig. 3a) exhibits the first decrease in γ ends at the SDS concentration where first mixed micelles are formed. The second γ decrease ends in the vicinity of conventional critical micelle concentration of SDS.

At higher DDACl concentrations, the first broad minimum in the surface tension curve (Fig. 3b) corresponds to transition from clear to turbid solutions. The increase in surface tension at concentrations just below equimolar, indicates that the enrichment with the anionic surfactant partially extracts the cationic surfactant from the solution

Fig. 3 Surface tension (γ) and surface potential (ΔV) vs. log SDS concentration in mixtures of DDACl/SDS. Constant DDACl concentrations are indicated. Temperature: from 293 to 296 K



and consequently from the air/solution interface due to incorporation into the precipitate. With SDS increasing concentration no change in surface tension occurs in the concentration region where solid crystalline DDADS and liquid crystalline phase coexist. It seems that with an increase in SDS concentration, the desorption of DDACl from the air/solution interface is followed by SDS adsorption to such an extent that the surface tension remains constant (it is in accord with ΔV decrease, Fig. 3b).

Figure 4 shows changes in surface tension for the three cross-sections on the DDACl-rich side (two corresponding to the isotropic region ($c(\mathrm{SDS}) = 1 \times 10^{-6}$ and 3×10^{-6} mol dm^{-3}) and one extending into the coexistence region ($c(\mathrm{SDS}) = 1 \times 10^{-5}$ mol dm^{-3})). Addition of a constant amount of SDS shifts the surface tension isotherms towards lower DDACl concentrations.

In the vicinity of the CMC for SDS or DDACl, respectively all surface tension (Figs. 3 and 4) isotherms exhibit minimum which is usually attributed to the presence of more surface active impurities. In these particular cases we can treat SDS or DDACl, respectively as impurity. The rise in γ close to the CMC indicates that more surface active material is lost, presumably due to solubilization into mixed micelles.

From the γ-log c plots the surface composition, surface activity coefficients and surface interaction parameters were calculated for the cross-sections extending into the isotropic region (Table 1 a, b). The surface activity coefficient values far below unity indicate large deviations from ideal mixing and this deviation is greater for component for which the surface mol fraction is smaller. Large negative values of the surface interaction parameter indicate strong attractive interactions between adsorbed molecules.

At constant DDACl concentration the surface mole fraction of DDACl decreases with increasing SDS bulk concentration (Table 1a). The decrease of absolute β^{mon} with surface pressure indicates a weaker interaction in the mixed monolayer. Despite a possible great error in surface interaction parameter determination (the accuracy of determination is diminished by high asymmetry in bulk phase composition (20)), the decrease of β^{mon} absolute value with increasing surface pressure does not appear to be an experimental error. Since the interaction measured here is due to the electrostatic attraction between oppositely charged surfactant headgroups, the smaller absolute value of β^{mon} is attributable to the change in the composition of the mixed monolayer. The increase in composition

Table 1 Surface mol fraction of DDACl (X_{DDACl}), activity coefficients of DDACl (f_{DDACl}) and SDS (f_{SDS}), the surface monolayer interaction parameter (β^{mon}) in DDACl/SDS mixtures. Systems were prepared with (a) constant DDACl and increasing SDS concentration and (b) with constant SDS and increasing DDACl concentration. C_{12}/mol dm^{-3} is solution phase molar concentrations of DDACl/SDS mixture required to produce the same surface pressure (Π/mN m^{-1}) as a solution containing only DDACl or SDS, respectively

Π	C_{12}	α_{DDACl}	X_{DDACl}	f_{DDACl}	f_{SDS}	β^{mon}
a) $c(\mathrm{DDACl}) = 1 \times 10^{-6}$ mol dm^{-3}						
17	0.0003	0.0033	0.32	0.002	0.276	− 12.2
32	0.0050	0.0002	0.15	0.002	0.818	− 8.8
b) $c(\mathrm{SDS}) = 1 \times 10^{-6}$ mol dm^{-3}						
32	0.00316	0.9997	0.84	0.787	0.001	− 9.5
$c(\mathrm{SDS}) = 3 \times 10^{-6}$ mol dm^{-3}						
32	0.00239	0.9988	0.79	0.640	0.003	− 9.7

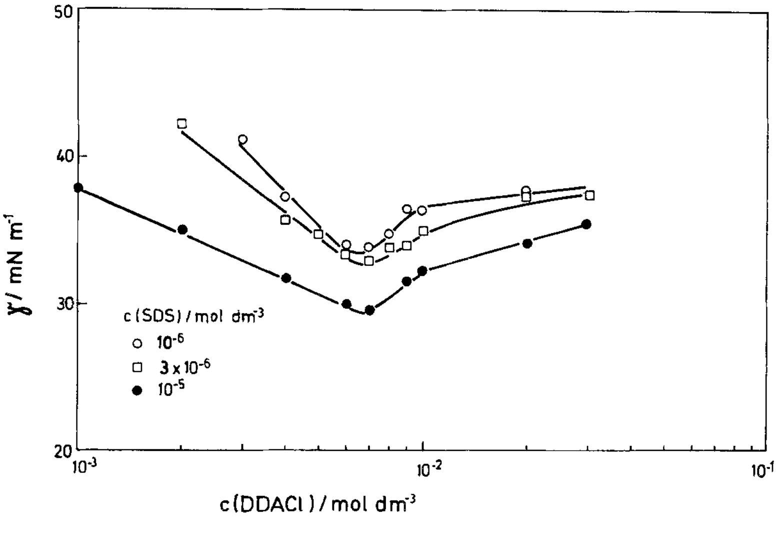


Fig. 4 Plots of surface tension γ vs. logDDACl concentration for DDACl/SDS mixtures. Constant SDS concentrations are indicated

asymmetry diminishes the electrostatic interaction at the air/solution interface. One could expect that the strength of electrostatic interaction reaches a maximum value in the equimolar mixed film. The very high negative value of β^{mon} (-22.1) obtained for the equimolar adsorption film supports this conclusion.

Two cross-sections on DDACl-rich side show that the surface molar fraction of DDACl slightly decreases with increasing SDS concentration. Surface interaction parameter β^{mon} exhibits almost the same value for $\Pi = 32\ \mathrm{mN\ m^{-1}}$.

Data of the surface excess concentrations and the minimum area per molecule of each surfactant by itself as obtained from the slope of its surface tension-concentration curve are listed in Table 2 a,b. The smaller Γ and consequently higher A for SDS compared DDACl are due to a smaller surface activity of SDS. The size of the polar head group is directly reflected in the mean area per molecule; the smaller size of the ammonium relative to the sulfate headgroup allows the cationic surfactant to be densely packed at the air/water interface.

The first mixed micelles formed in the systems with $c(\mathrm{DDACl}) = 1 \times 10^{-6}\ \mathrm{mol\ dm^{-3}}$ (Table 2a, column I) are in equilibrium with the monolayer showing a smaller surface excess of SDS the the monolayer in equilibrium with the second mixed micelles (column II). The addition of the oppositely charged surfactant increases the SDS surface excess (column II) and consequently decreases the minimum area which SDS molecules occupied at the air/water interface.

Table 2b presents the surface excess changes of DDACl with SDS addition (the cross-sections with constant $c(\mathrm{SDS}) = 1 \times 10^{-6}$, 3×10^{-6} and $1 \times 10^{-5}\ \mathrm{mol\ dm^{-3}}$). There is a significant variation in DDACl adsorption as a function of SDS concentration. Addition of lower SDS concentrations increases the surface excess of DDACl at the air/water interface. The decrease of Γ_{SDS} and Γ_{DDACl} at higher concentration of DDACl or SDS can be explained

Table 2 The surface excess of DDACl (Γ_{DDACl}) and SDS (Γ_{SDS}) and minimum area per surfactant molecule (A) at the air/solution interface in either single solutions and their mixtures. (a) mixtures with constant DDACl and increasing SDS concentration and (b) with constant SDS and increasing DDACl concentration

a) $c(\mathrm{DDACl})/\mathrm{mol\ dm^{-3}}$	$(\Gamma_{\mathrm{SDS}}/\mathrm{mol\ cm^{-2}}) \times 10^{10}$ I	II	$A_{\mathrm{SDS}}/\mathrm{nm^2}$ I	II
0		2.23		0.75
1×10^{-6}	1.01	2.75	1.65	0.60
1×10^{-4}		2.40		0.69
b) $c(SDS)/\mathrm{mol\ dm^{-3}}$	$(\Gamma_{\mathrm{DDACl}}/\mathrm{mol\ cm^{-2}}) \times 10^{10}$		$A_{\mathrm{DDACl}}/\mathrm{nm^2}$	
0		2.47		0.67
1×10^{-6}		2.91		0.57
3×10^{-6}		2.77		0.59
1×10^{-5}		0.84		1.98

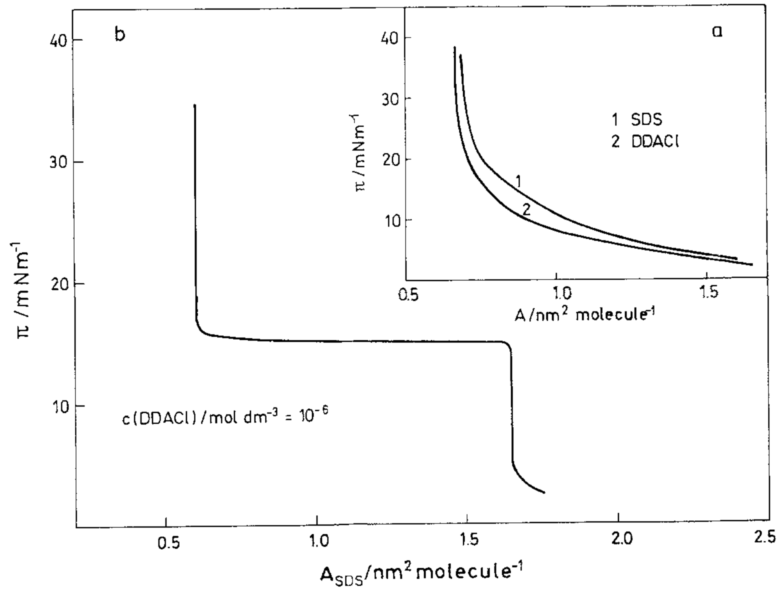


Fig. 5 Surface pressure (Π) vs. area per molecule (A) isotherms of single DDACl and SDS (a) and of their mixtures with varying SDS and constant DDACl concentrations as indicated (b) Temperature: 298 K

by solid crystalline DDADS and liquid crystalline phase formation.

The changes in the γ vs. log c isotherm are in accordance with changes in the corresponding surface potentials. The ΔV vs. log c curves (Fig. 2 a, b) consist of two maxima: the first broad maximum at lower SDS concentration and the second maximum close to the $\mathrm{CMC_{SDS}}$. A higher surface potential in the region of DDADS precipitation (Fig. 2b) than that of DDACl alone indicates the preferential position of the cationic surfactant at the air/water interface. Up to approximately equimolar concentrations DDACl is in excess and SDS is exhausted from the solution by DDADS precipitate formation. The decrease of surface potential with increasing SDS concentrations points to a change in surface layer composition. As the concentration of SDS increases, a point is reached where not only DDACl contributes to ΔV. A significant contribution is then expected from the increased concentration of SDS as well; however, the surface still remains positively charged.

The surface pressure (Π) vs. mean area per adsorbed molecule curves for single DDACl and SDS solutions (Fig. 5a) and their mixtures with constant DDACl concentrations (Fig. 5b) are useful to describe the state of adsorbed films at the air/water interface. The curves for DDACl and SDS are found to be quite similar to the Π vs. A curves for expanded films (21). The area occupied by DDACl molecules compared to that occupied by SDS molecules is smaller. Fig. 5b shows that the isotherm for mixtures exhibits two linear portion and a wide plateau ($1.65 - 0.60\ \mathrm{nm^2/molecule}$) of coexistence at $\approx 15.5\ \mathrm{mN\ m^{-1}}$ between an liquid expanded and liquid condensed phase. The addition of an oppositely charged surfactant results in monolayer condensation.

Conclusions

The present study shows that the composition and properties of the adsorption films of dodecylammonium chloride/sodium dodecyl sulfate at the air/water interface strongly depend on interactions between the film molecules and equilibria in the bulk phase (monomer-micelle and/or monomer-precipitate equilibria).

The nature and strength of interaction between DDACl and SDS are measured by the surface interaction parameter β^{mon}, which indicates strong attractive interaction. β^{mon} depends on the composition of the mixed monolayer.

The total surface excess of a surfactant in mixed cationic/anionic adsorption films is influenced by the interfacial area per mole of each surfactant and the electrostatic interactions between them. The increase of surface excess and the decrease of the minimum area per surfactant molecule are caused by strong electrostatic attraction. Single components occupy a greater minimum area per molecule due to charge repulsion. The presence of oppositely charged surfactants at the interface reduces repulsion and the minimum area per molecule decreases.

The addition of an oppositely charged surfactant enhances packing at the air/water interface and causes transition from a liquid expanded to a liquid condensed state. On the average, $\mathrm{DDA^+}$ ions are closer to the surface than $\mathrm{DS^-}$ ions and in all mixtures the surface potential exhibits a positive value.

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