

Different Theoretical Approaches for the Study of the Mixed Tetraethylene Glycol Mono-*n*-dodecyl Ether/Hexadecyltrimethylammonium Bromide Micelles

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The mixed micelles C₁₂E₄ (tetraethylene glycol mono-*n*-dodecyl ether)/CTAB (hexadecyltrimethylammonium bromide) were studied. The surface tension results were analyzed on the basis of a treatment that considers the relation between the activity coefficients according to the Gibbs–Duhem equation. The results are compared with Rubingh's treatment, which considers the activity coefficients given by the regular solution theory. The conductivity of the samples and the aggregation numbers at low mixture concentration are also given.

Introduction

Mixed micelles that contain more than one type of surfactant are especially important because most of the commercially available surfactants are mixtures with better properties than pure surfactants. Some surfactant mixtures present synergistic properties. They produce a higher decrease in the surface tension and a lower critical micelle concentration (CMC) value than each pure surfactant does. Different mixtures formed by different surfactants such as nonionic/nonionic,^{1–3} nonionic/anionic,^{4–10} cationic/nonionic,^{11,12} anionic/biosurfactant,^{13,14} and anionic/cationic¹⁵ have been studied.

Different theoretical treatments have been used to explain the mixed micelles properties. The first one was given by Lange¹⁶ and used by Clint.³ This treatment is based on the phase separation model that considers the ideal behavior of each surfactant in the mixed micelle. This theory provides a suitable description for mixtures of surfactants with similar headgroups for which a nearly ideal mixture might be expected, but it fails to describe the properties of surfactant mixtures with different headgroups.

Rubingh¹⁷ divulged a treatment based on the regular solution theory for the mixed micelles that provides a way to deal with the enthalpy and entropy in mixed micelle formation. This treatment has been extensively used,^{2,9–11,18} but the treatment has been criticized from the thermodynamic point of view because it does not take into account the dissociation of ionic surfactants, and the surfactant adsorption on the surface. Other treatments based on the mass action model¹⁹ have been used, and Motomura^{20a,b} developed a thermodynamic treatment using the excess thermodynamic functions similar to those for the adsorbed film. Hoffmann and colleagues²¹ used a treatment based on the molar free enthalpy of nonideal binary mixtures, and they arrived at an equation that is a special case of Motomura's equation^{20a} for mixtures of two nonionic surfactants. Others treatments based on thermodynamics excess functions that are similar to Rubingh's equation have also been used.²²

In this paper we used a simple theoretical treatment based on Lange's model that uses the Gibbs–Duhem equation to relate the activity coefficients of the surfactant in the mixed micelles. This treatment is similar to that given by Hoffman²¹ that arrives to the same equation as Motomura's equation.^{20a} The results obtained for the system C₁₂E₄/CTAB (tetraethylene glycol mono-*n*-dodecyl ether/hexadecyltrimethylammonium bromide)

with this treatment are compared to the results obtained using Rubingh's method. The Rubingh and theoretical treatments are also used at surfactant concentrations higher than the CMC.

The conductivity values and the aggregation numbers obtained by fluorescence quenching measurements, at concentrations near the CMC, are also given.

Materials and Methods

Tetraethylene glycol mono-*n*-dodecyl ether (Nikkochemicals, 99+%) and hexadecyltrimethylammonium bromide (SigmaUltra) were used as supplied. The quencher, *N*-cetylpyridinium chloride (Merck), and the probe, pyrene (Merck), were recrystallized from methanol/diethyl ether mixtures.

The surface tensions of aqueous mixed surfactant solutions at various concentrations were determined as a function of the mole fraction of C₁₂E₄, using the ring method with a LAUDA TE-1C tensiometer. All measurements were carried out at 25.0 ± 0.1 °C. Each experiment was repeated several times until a good reproducibility was achieved. The CMC values were determined as the sharp break point in the surface tension against the logarithm of concentration curves at various mole fractions of C₁₂E₄.

Specific conductivities were measured with a Crison 525 conductometer. The solution flask containing the conductivity cell (cell constant 0.1 cm⁻¹) was immersed in a water bath at 25.0 ± 0.1 °C.

The fluorescence steady-state quenching measurements were carried out in a Perkin-Elmer spectrofluorometer LS-5B at 25.0 ± 0.1 °C, using pyrene as a probe and *N*-cetylpyridinium chloride as a quencher. The fluorescence emission was integrated over 69 s, and the data reported are mean values from several measurements. Micellar aggregation numbers, *N*, were determined from the fluorescence intensity in the presence, *I*, and in the absence, *I*₀, of the quencher at 375 nm, according to the theoretical treatment given in the literature.^{23,24} The probe and quencher were considered to be located in the same environment as the surfactant molecules, and their concentrations were too small so that it could be safely assumed that they did not change the mixed micelle structure. By considering both the probe and the quencher to be distributed among the micelles according to Poisson statistics, with the quencher average occupation number given by the number of quencher molecules in a micelle, [Q]/*N*[Dn], where [Q] represents the quencher concentration in the

micelle, $[Dn]$ the micellized surfactant concentration, and N the aggregation number, one can easily deduced that

$$\ln I_0/I = [Q]N/[Dn] \quad (1)$$

Theoretical Treatment

Lange's treatment is based on the phase separation model with ideal behavior of surfactants in the mixed micelle. For a pure surfactant 1 at the critical micelle concentration, CMC_1

$$\mu_{M1} = \mu_{S1} = \mu_{S1}^0 + RT \ln(CMC)_1 = \mu_{M1}^0 \quad (2)$$

where μ_{M1} and μ_{S1} correspond to the chemical potentials of surfactant 1 in the micelle and in the solution, respectively, μ_{S1}^0 and μ_{M1}^0 the standard chemical potentials of surfactant 1 in the solution and in the micelle where the surfactant activity is 1. In the case of a mixture of surfactants in a solution with a composition given by α_1 , the mole fraction of surfactant 1 in the mixture that forms a mixed micelle

$$\begin{aligned} \mu_{M1} &= \mu_{M1}^0 + RT \ln \chi_1 = \mu_{S1}^0 + RT \ln(CMC)_1 + \\ &\quad RT \ln \chi_1 = \mu_{S1}^0 + RT \ln C_1 \\ \mu_{M2} &= \mu_{M2}^0 + RT \ln \chi_2 = \mu_{S2}^0 + RT \ln(CMC)_2 + \\ &\quad RT \ln \chi_2 = \mu_{S2}^0 + RT \ln C_2 \end{aligned} \quad (3)$$

where χ_1 and χ_2 are the mole fractions of surfactants 1 and 2 in the micelle, C_1 and C_2 are the surfactant concentrations in solution, and CMC^* is the critical micelle concentration for the mixture. So that when the first small amount of micelles are formed

$$\begin{aligned} C_1 &= \chi_1 CMC_1 = \alpha_1 CMC^* \\ C_2 &= \chi_2 CMC_2 = (1 - \chi_1) CMC_2 = (1 - \alpha_1) CMC^* \end{aligned} \quad (4)$$

it is easy to deduce the CMC^* as a function of CMC_1 , CMC_2 , and α_1

$$1/CMC^* = \alpha_1/CMC_1 + (1 - \alpha_1)/CMC_2 \quad (5)$$

In the case of nonideal behavior of the surfactant in the micelle and using the same treatment, we can write

$$\begin{aligned} C_1 &= \chi_1 (CMC_1) f_1 = \alpha_1 CMC^* \\ C_2 &= (1 - \chi_1) (CMC_2) f_2 = (1 - \alpha_1) CMC^* \end{aligned} \quad (6)$$

Rubingh's treatment considers the activity coefficients given by the regular solution theory

$$\ln f_1 = \beta(1 - \chi_1)^2; \quad \ln f_2 = \beta(1 - \chi_2)^2 = \beta\chi_1^2 \quad (7)$$

where the β parameter is $\beta = (E_{11} + E_{22} - 2E_{12})/RT$, which takes into account the interaction energy between the monomers of surfactant 1, surfactant 2, and monomers of surfactants 1 and 2 in the mixed micelle, respectively. From eqs 6 and 7 it is easy to deduce the following equation

$$\chi_1^2 \ln[(\alpha_1 CMC^*)/\chi_1 CMC_1] = (1 - \chi_1)^2 \ln[(1 - \alpha_1) CMC^*/(1 - \chi_1) CMC_2] \quad (8)$$

This equation can be solved by an iterative calculation method that gives χ_1 from the experimental value of the critical micelle concentration of the mixture (CMC^*). From this χ_1 value, which gives the mixed micelle composition, it is possible to obtain

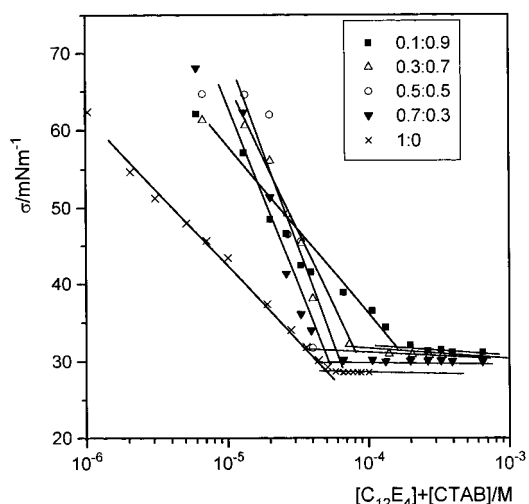


Figure 1. Surface tension versus total concentration natural logarithm for the mixed micelles at different compositions.

the values of f_1 and f_2 , the activity coefficient from eq 6, and the β value from eq 7.

This Rubingh's model has the restriction that the activity coefficients given by the regular theory of real solution eq 7 must be considered. However, it is possible to use another treatment without considering this restriction in the activity coefficients. This treatment is based on Lange's model and considers the Gibbs–Duhem equation to relate f_1 and f_2 . From eq 6 we can write

$$\ln CMC_1 + \ln \chi_1 f_1 - \ln \alpha_1 = \ln CMC_2 + \ln \chi_2 f_2 - \ln(1 - \alpha_1) \quad (9)$$

and differentiate to α_1

$$\begin{aligned} d \ln \chi_1 f_1 / d\alpha_1 - 1/\alpha_1 &= d \ln \chi_2 f_2 / d\alpha_1 + 1/(1 - \alpha_1) \\ (1/1 - \chi_1)(d\chi_1/d\alpha_1) + \chi_1 d \ln f_1 / d\alpha_1 &= (1 - \chi_2) d \ln f_2 / d\alpha_1 + \\ &\quad [\chi_2 / \alpha_1 (1 - \alpha_1)] \end{aligned} \quad (10)$$

so that taking into account the Gibbs–Duhem equation: $\chi_1 d \ln f_1 / d\alpha_1 + \chi_2 d \ln f_2 / d\alpha_1 = 0$, the eq 10 reduces to

$$\begin{aligned} -d \ln(1 - \chi_1) / d\alpha_1 &= d \ln f_2 / d\alpha_1 + [\chi_1 / \alpha_1 (1 - \alpha_1)] \\ -d \ln(\chi_2 f_2) / d\alpha_1 &= \chi_1 / \alpha_1 (1 - \alpha_1) \end{aligned} \quad (11)$$

and using eqs 6 and 11 it is reduced to

$$-d \ln(\chi_2 f_2) / d\alpha_1 = 1/(1 - \alpha_1) - d \ln CMC^* / d\alpha_1 = \chi_1 / \alpha_1 (1 - \alpha_1) \quad (12)$$

from which we finally obtain

$$\chi_1 = -(1 - \alpha_1) \alpha_1 d \ln CMC^* / d\alpha_1 + \alpha_1 \quad (13)$$

The same equation was given by Hoffman and colleagues²⁰ as a particular case of Motomura's equation.^{19a} This equation allows us to calculate χ_1 from $d \ln CMC^* / d\alpha_1$, and then to calculate the activity coefficients using eq 6 without the restrictions of the regular solution theory.

Results and Discussion

Some of the surface tension results of the mixture at different compositions are given in Figure 1. The critical micelle

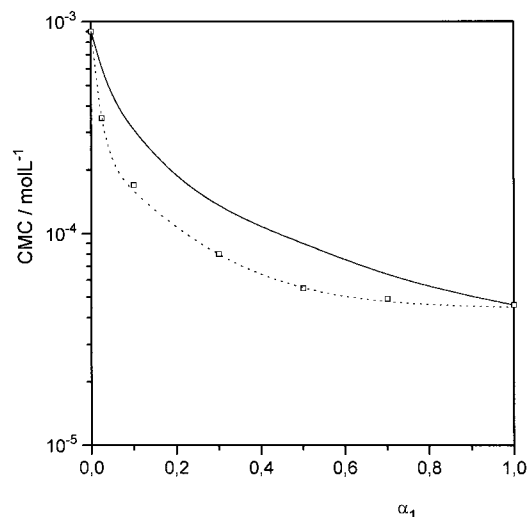


Figure 2. Critical micelle concentration variation at different mixture compositions: (□) experimental results; (solid line) calculated value according to the ideal behavior.

TABLE 1: Experimental Critical Micellar Concentration, Mole Fraction of Surfactant 1 in the Micelle According to the Ideal Behavior, χ_1^i , Rubingh's Model, χ_1^R , and the New Treatment, χ_1 , and the Value of β According to Rubingh's Treatment

α_1	CMC*, M	χ_1^i	χ_1^R	β	χ_1
0.025	3.5×10^{-4}	0.33	0.42	-2.37	0.68
0.1	1.7×10^{-4}	0.67	0.58	-2.62	0.49
0.3	1.1×10^{-4}	0.89	0.70	-3.22	0.69
0.5	5.5×10^{-5}	0.98	0.77	-3.25	0.78
0.7	4.9×10^{-5}	0.97	0.83	-3.46	0.79

concentration, CMC*, at different mole fractions of nonionic surfactant 1 in the mixture, α_1 , were obtained as the break point of both straight lines of Figure 1, and they are depicted by dots in Figure 2. The CMC* values obtained considering the ideal behavior of the surfactants in the micelle according to eq 5 are plotted in Figure 2 by a solid line, using CMC₁ = 4.6×10^{-5} M and CMC₂ = 9×10^{-4} M.²³ Similar results were obtained by another system that shows that the critical micelle concentration is lower than the predicted value according to the ideal behavior of surfactants in the mixed micelle. The mole fraction of surfactant 1 in the micelle by considering ideal behavior, χ_1^i , are given in Table 1. These values increase with the mole fraction of surfactants in solution, α_1 .

In a first approach we tried to adapt the CMC* values to Rubingh's treatment, eq 8. The equation was solved by an iterative calculation method obtaining χ_1^R , the mole fraction of surfactant 1 in the micelle, from the mole fraction of surfactant 1 in the mixture, α_1 , and the experimental value of critical micelle concentration, CMC*. The values of χ_1^R and the β parameter values obtained by using eq 8 are given in Table 1. According to the results, the value of β is not constant with the mixture composition, but it decreases with the amount of nonionic surfactants in the mixture, which means β becomes more negative as the amount of nonionic surfactant in the mixture increases. Similar results have been obtained with other surfactant mixtures,^{4,5,10,25} but in the presence of salt that screens the electrostatic interactions the β is constant with the salt concentration.²⁶ The errors in CMC measurements implies small changes in β values, but the variation of β with the micellar composition is of the same kind and it does not alter the following discussion of results. From the results in Table 1 it

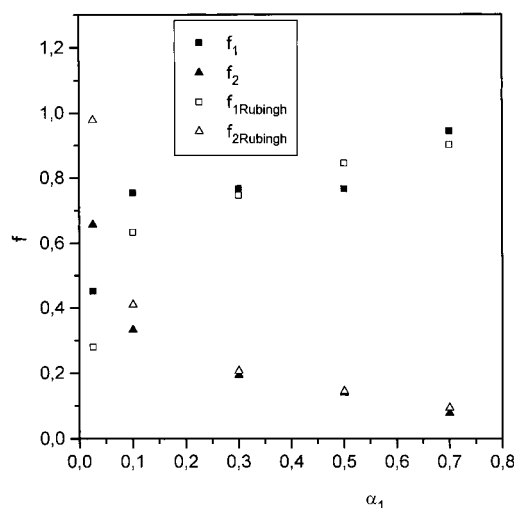


Figure 3. Activity coefficients for the samples with different compositions: empty dots according to Rubingh's treatment, full dots according to the new treatment.

is also deduced that Rubingh's treatment predicts smaller amounts of surfactant 1 in the mixed micelle than the ideal behavior.

To adapt the experimental CMC* values to the theoretical treatment, based on the Gibbs–Duhem equation, given by eq 13, we obtained the following empirical variation of CMC* with α_1 .

$$\ln \text{CMC}^* = 4 \times 10^{-5} + 6.52 \times 10^{-4} \exp(-\alpha_1/0.0154) + 2.05 \times 10^{-4} \exp(-\alpha_1/0.171) \quad (14)$$

from which the value of $d \ln \text{CMC}^*/d\alpha_1$ can be easily obtained.

The values of χ_1 , the mole fraction of surfactant 1 in the micelle obtained from eq 13, are also given in Table 1. From the results it is deduced that both treatments give similar values of χ_1 , at α_1 values higher than 0.1. The activity coefficients using Rubingh's treatment and the new treatment are plotted in Figure 3 showing that both theoretical treatments also give the same value at α_1 values higher than 0.1. With a small amount of C₁₂E₄ surfactant in the mixture, a big deviation between both treatments exists, although it is necessary to say that a small variation in the CMC*, even with the experimental error, could approach both calculated values.

From these results we can conclude that the treatment based directly on the Gibbs–Duhem equation, which does not introduce any restriction to the behavior of surfactant mixtures, gives the same results as Rubingh's treatment, which considers that mixed micelles behave as a regular solution, but with the restriction of considering that the β value parameter depends on the mixture composition. This is an interesting result that means that in the micelle this mixture of surfactants behaves as a regular solution, with a mixing entropy given by the ideal behavior.

According to these results, Figure 4 shows the pseudophase diagram of this system in which the equilibrium between monomers in solution and the mixed micelle can be seen. (In Figure 4 we have neglected the χ_1 calculated with the new treatment at $\alpha_1 = 0.025$.)

We have also calculated the surfactants 1 and 2 concentrations in the aqueous solution, C_1 and C_2 , at a surfactant concentration

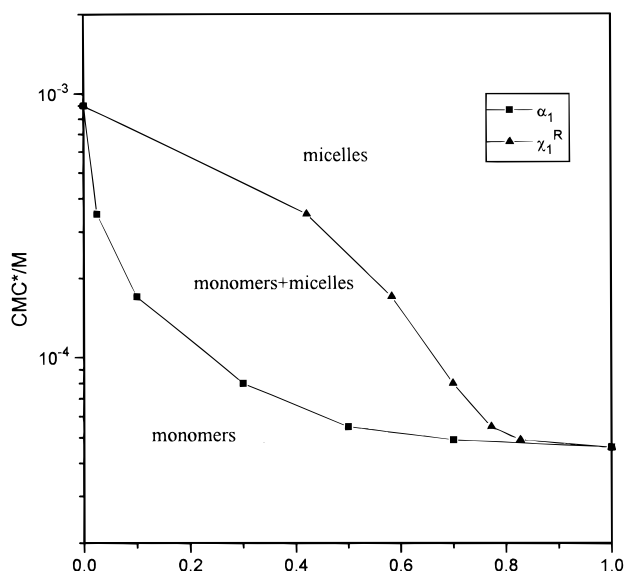


Figure 4. Pseudophase diagram of the mixed micelle $C_{12}E_4/CTAB$.

higher than the CMC^* , taking into account that

$$\chi_1 = \alpha_1(C - C_1)/(C - C_1 - C_2) \quad (15)$$

where C is the total surfactant concentration in the solution. Using C_1 and C_2 according to eq 6 we obtain

$$\chi_1^2(CMC_1f_1 - CMC_2f_2) - \chi_1(C - CMC_1f_1 - CMC_2f_2) + \alpha_1C = 0 \quad (16)$$

As both treatments give the same f_1 and f_2 values, we consider that they are well given by Rubingh's eq 7 with the β variation given in Table 1. The equation was solved by an iterative calculation method. The C_1 , C_2 , and χ_1 calculated results are plotted in Figure 5. They show that for a total high surfactant concentration the mixed micelles have the same composition as the mixture, $\chi_1 = \alpha_1$. It is also possible with the activity coefficient given in Figure 3 to calculate the mixing thermodynamic functions by considering the regular theory treatment that explains these results. For these solutions $\Delta S_M = \Delta S_M^{ideal}$, which means the excess entropy function is $S^E = 0$ and $G^E = \Delta H_M$, where $G^E = RT \sum \chi_i \ln f_i$ and $\Delta G^M = RT \sum \chi_i \ln \chi_i f_i$. The calculated results are given in Table 2. The results show that the mixed micelle formation is more favorable with a small amount of nonionic surfactant in the micelle according to the χ_1 value given in Table 2. We have also found that the excess free energy of micellization ($G^E = \Delta H_M$) is not a symmetrical distribution with respect to the composition in the micelles, as occurs for anionic/zwitterionic mixtures.²⁷ Small variations in the experimental CMC values between the experimental error produces small changes in all the parameters and in the thermodynamic functions, but this variation does not change the discussion of the results.

The specific conductivity of the mixture at different compositions versus the amount of CTAB is plotted in Figure 6. The specific conductivity is not sensitive enough to determine the critical micelle concentration in the system, as the CTAB concentration in the CMC^* is very small. Above the CMC^* , the conductivity of the solution can be considered independent of the composition of the mixture at the small surfactant concentrations used in this study.

To understand the conductivity results, we can consider that only the ionized surfactant in water and the micelle contribute

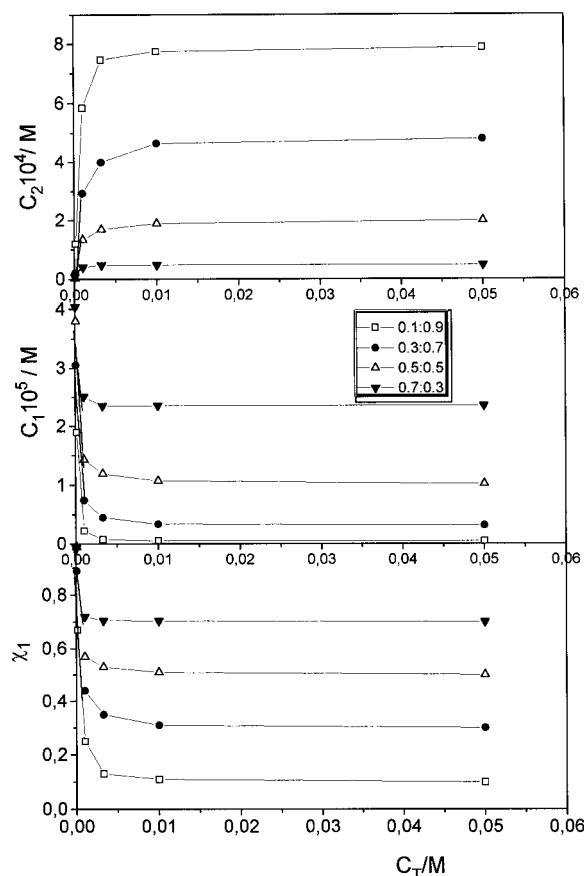


Figure 5. Cationic surfactant concentration, nonionic surfactant concentration in solution, and the mole fraction of surfactant 1 in the micelle versus total concentration according to Rubingh's treatment for different compositions.

TABLE 2: Mixing Thermodynamics Functions According to the New Treatment

α_1	χ_1^R	ΔH_M , kJ mol ⁻¹	ΔS_M , JK ⁻¹ mol ⁻¹	ΔG_M , kJ mol ⁻¹
0.025	0.42	-1.45	5.67	-3.14
0.1	0.58	-1.58	5.64	-3.26
0.3	0.70	-1.69	5.08	-3.20
0.5	0.77	-1.41	4.46	-2.74
0.7	0.83	-1.01	3.89	-2.17

to the conductivity. So that

$$\kappa = (\lambda_M + \lambda_{Br^-})(C_T - C_2^m) + (C_2^m \alpha) \lambda_m + (C_2^m \alpha) \lambda_{Br^-} \quad (17)$$

where λ_M is the molar conductivity of the CTA^+ in aqueous solution, λ_m is the molar conductivity of CTA^+ micellized, λ_{Br^-} is the molar conductivity of Br^- ions, α is the micelle ionization degree, C_T is the total CTAB concentration in solution, and C_2^m corresponds to the CTAB concentration in the micelle.

According to the conductivity results, Figure 6, the conductivity of the solution is the same for each CTAB concentration, independent of the mixture composition. So that we can reorganize eq 17 to read

$$\alpha = cte/C_2^m + cte' \quad (18)$$

where $cte = [\kappa - (\lambda_M + \lambda_{Br^-})C_T]/(\lambda_m + \lambda_{Br^-})$ and $cte' = (\lambda_M + \lambda_{Br^-})/(\lambda_m + \lambda_{Br^-})$. That means there is a linear variation between α and $1/C_2^m$ for a constant CTAB concentration. We cannot calculate the micellar ionization degree, α , but taking into account that according to the surface tension results, if α_1

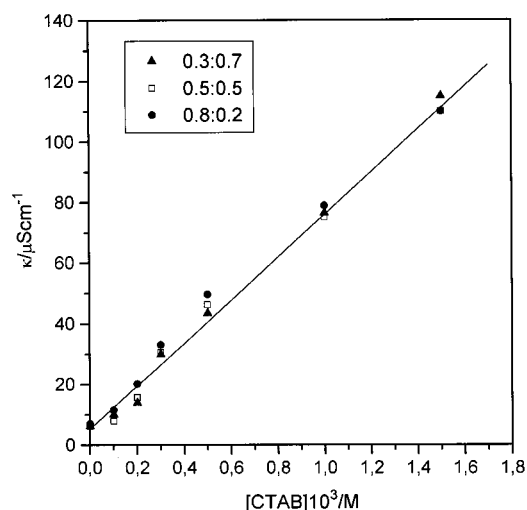


Figure 6. Specific conductivity of the samples at different cationic surfactant concentrations.

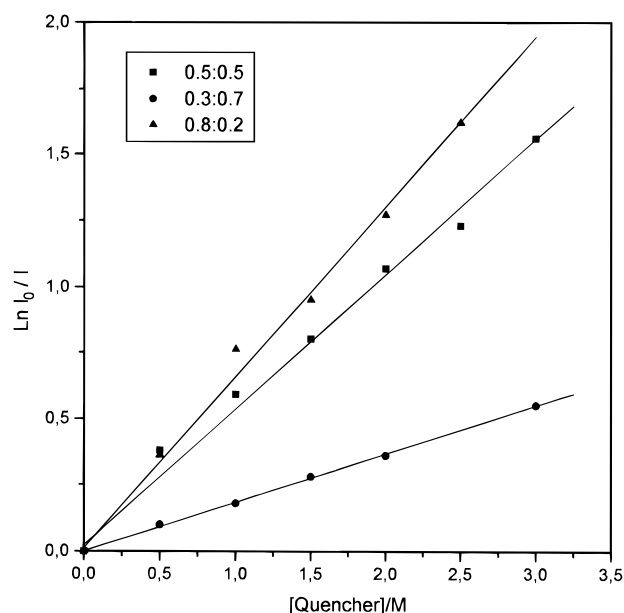


Figure 7. Logarithm of pyrene fluorescence intensity ratio in the absence and presence of quencher versus quencher concentration.

(the mole fraction of surfactant 1 in the mixture) increases, C_2^m near the cmc^* increases and the micellar ionization degree α decreases according to eq 18. That means that the ionization of cationic surfactant CTAB in the mixed micelle increases with the content of the nonionic surfactant, α_1 . Otherwise, as α_1 increases, β becomes more negative, eq 14, which means that E_{12} increases and we can finally conclude that the interaction energy between surfactants 1 and 2 in the mixed micelle increases with α_1 when the cationic surfactant ionization in the mixed micelle increases. This is the reason the regular theory of mixtures with a β constant value does not fit the results in this system, nonionic/cationic surfactant, where the ionization degree of the micelle increases with the amount of nonionic surfactant in the mixture, which influences the interaction of both surfactants in the mixed micelle.

The fluorescence quenching measurements adapt to the Poisson distribution, the $\ln I_0/I$ versus quencher concentrations fit a straight line (Figure 7), and the aggregation numbers, N , are given in Table 3, by considering all the quencher concentrations in the micellar phase. From the results it is deduced that at low surfactant concentrations, the aggregation number is

TABLE 3: Aggregation Numbers for the System C₁₂E₄/CTAB at Different Compositions at Total Concentration

α_1	[C ₁₂ E ₄ + CTAB], M	N	N_1	N_2
0.3	2×10^{-3}	34 ± 1	12 ± 2	22 ± 5
	3.3×10^{-3}	38 ± 7	11 ± 5	27 ± 8
0.5	1.2×10^{-3}	58 ± 6	29 ± 3	29 ± 3
	4×10^{-3}	61 ± 4	31 ± 4	30 ± 4
0.8	7.5×10^{-4}	44 ± 5	36 ± 7	9 ± 2

nearly independent of α_1 (the mixture composition) and of surfactant concentration. So that the mixed micelle should be spherical due to the small aggregation number.

Taking into account the values of χ_1 calculated from the new theoretical treatment, it is possible to obtain N_1 and N_2 , the monomer numbers of surfactants 1 and 2 in the mixed micelle. The values are given in Table 3. It can be seen that at small surfactant concentrations, depending on the mixture composition, micelles with similar aggregation numbers but different ratios of nonionic/cationic surfactant are formed, nonionic surfactant rich micelles or cationic surfactant rich micelles.

Conclusion

From all of these results we can conclude the theoretical treatment that does not introduce any restrictions on the activity coefficient of the surfactants in the mixed micelle reaches the same results as Rubingh's treatment, which considers that mixed micelles behave as a regular solution. That means the mixing entropy, ΔS_M , is well given as an ideal solution with a positive value. The ΔG_M and ΔH_M are more favorable to the mixed micelle formation when the micelle contains a small amount of nonionic surfactant. From the conductivity results we conclude that the cationic surfactant in the micelle is more ionized as the amount of nonionic surfactant increases in the mixture. With a low surfactant mixture concentration the aggregation numbers can be considered independent of the mixture composition, which means that mixed micelles are formed with the cationic surfactant more ionized as the nonionic surfactant increases in the mixed micelle. When the micelle is more ionized, the interaction energy between surfactants 1 and 2 is larger and the mixed micelle more separated from the ideal behavior.

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