Colloid Polym Sci 274:662–668 (1996)
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ORIGINAL CONTRIBUTION

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Counterion specificity of the micelle surface and its implications on micellar catalysis

Received: 3 February 1995
Accepted: 20 November 1995

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Abstract Dodecyltrimethylammonium bromide – Dodecyltrimethylammonium hydroxide – water mixtures were studied with ion-selective electrodes, and the aggregation behavior, degree of ionization of the micelles and the distribution constants of bromide and hydroxide ions between water and micelles were found, showing that some suppositions about the interpretation of micellar catalysis are incorrect, and these interpretations must be revised. The results support the mass action model for the theoretical treatment of micellar catalysis.

Key words Micellar catalysis – critical micelle concentration – micellization – counterion distribution constants – hydroxide surfactants

Introduction

Alkyltrimethylammonium halide ($RTA^+ X^-$) surfactants have been extensively used to study the micellar catalysis on reactions with several hydroxyl compounds [1–4]. In these reactions, the hydroxyl concentration is commonly much larger than that of surfactant. The Stern layer of the micelle must contain both OH^- and halide (X^-) ions due to the ionic exchange [5, 6]. Since different counterions have different affinities for the micelle surface [2, 6–8], the properties of micelles alter when this counterion exchange occurs, thus affecting their catalytic properties.

The theoretical treatment of micellar catalysis has some problems [9]. It is easy to fit the experimental data to the theoretical equations, because they have many adjustable parameters, such as the degree of ionization (α), the constant of exchange of OH^- and X^- ions between water and micelle surface. K_X^{OH}, the reaction rate constant in the micellar pseudophase, and the amphiphile monomer concentration, which is usually taken as equal to the critical micelle concentration (CMC) [10]. The usual treatment is to assume reasonable values for some of these parameters such as α and the CMC, and then estimate the values of the others using experimental data for rate constants or equilibrium constants. These treatments depend critically upon the value of α, but they usually fit the data well. However, this treatment does not give unique values for the parameters; many other values may fit the data equally well [11].

In this work, we studied the system dodecyltrimethylammonium hydroxide (LTAOH) – dodecyltrimethylammonium bromide (LTAB) – water to test some of the suppositions used in the theoretical treatment of micellar catalysis and to obtain experimentally the values of some of the above mentioned parameters, e.g. α, CMC, and K_{Br}^{OH}. The study was mainly carried out with ion-selective electrodes, on systems having molar ratios LTAB: LTAOH of 1:0, 2:1, 1:1, 1:2 and 0:1.

Experimental

Preparation of the hydroxyl surfactant

Much care was taken to avoid contamination with atmospheric CO_2 in this work. Surfactant solution preparation and measurements were performed in a plastic sealed chamber, which had several dishes with NaOH concentrated solution inside. This chamber had two plastic gloves to work inside. After introduction of the material, a flow of N_2 was passed, which had been previously bubbled through NaOH solution. All work inside the chamber started 2 h after the chamber was closed and nitrogen was admitted. All runs were performed at $25.0 \pm 0.1\,^{\circ}C$, by thermostatted water circulation.

The water employed was double-distilled, boiled for 1 h to expel dissolved CO_2 and then left to cool in a N_2 atmosphere.

A concentrated LTAOH solution was prepared by passing LTAB (Sigma) solution through an ion-exchange column. This column was prepared using a 50 mL burette filled with Amberlite IRA-400 (OH) (Carlo Erba). The total elimination of Br^- was verified with $AgNO_3$ + HNO_3 solutions. The whole system was sealed to avoid CO_2 contamination.

The final LTAOH concentration was determined three times by conductimetric titration with HCl.

All runs were performed in duplicate.

LTA^+ ion-selective measurements

The LTA^+ ion-selective electrode was made by dissolving 0.3 g of carrier (dodecyltrimethylammonium dodecylsulphate) and 0.06 of dibutylphtalate (plastizicer) in 10 mL of tetrahydrofuran. 0.3 g of PVC were added, and the solution was allowed to evaporate in a Petri dish covered by a filter paper. The elastic film obtained was glued to a PVC tube with tetrahydrofuran. The inner electrode was Ag/AgCl, and the tube was filled with NaBr $0.001\ \mathrm{mol \cdot dm^{-3}}$ + LTAB $0.001\ \mathrm{mol \cdot dm^{-3}}$ solution. All measurements were performed against a calomel saturated electrode using a Tacussel millivoltmeter.

The carrier was prepared by mixing equimolar quantities of LTAB and SDS solutions; the precipitate was filtered, washed until no Br^- was detected, and dried.

The electrode was controlled by measurements on LTAB solutions, giving results in agreement with literature.

Counterion electrode measurements

OH^- determinations were made with an Orion glass electrode and pH-meter. Br^- determinations were made with an Br^--ion selective electrode (Orion) vs. SCE.

Computations based on electrode data

The activity coefficients γ were computed with the Davies' equation [12]:

$$\log \gamma = -0.5115\sqrt{I}/(1+\sqrt{I}) - 0.15I, \tag{1}$$

where I is the ionic strength. Only the unmicellized ions were considered to compute I. The unmicellized amphiphilic ion (LTA^+) and counterion (Br^- and OH^-) concentrations were computed from the E vs. total surfactants concentration C_T (= [LTAOH] + [LTAB]) curves using literature methods [13]. Then, the effective charge per micellized surfactant molecule, $\alpha = 1 - m/n$, was computed; m/n is the $OH^- + Br^-$ to LTA^+ ratio in the micelles:

$$m/n = \frac{C_T - [OH^-]_W - [Br^-]_W}{C_T - [LTA^+]_W} \tag{2}$$

where the brackets indicate molar concentration and the subscript W denotes water.

The counterion distribution constants between micelles and water were computed with [14–18]:

$$K_{Br}^{OH} = \frac{K'_{OH}}{K'_{Br}} = \frac{[OH^-]_M [Br^-]_W}{[OH^-]_W [Br^-]_M} \tag{3}$$

where the subscript M indicates micelles. K'_{OH} and K'_{Br} are the individual counterion distribution constants, given by:

$$K'_{OH} = \frac{[OH^-]_M}{[OH^-]_W \{[LTA^+]_M - [OH^-]_M - [Br^-]_M\}} \tag{4}$$

and

$$K'_{Br} = \frac{[Br^-]_M}{[Br^-]_W \{[LTA^+]_M - [OH^-]_M - [Br^-]_M\}} \tag{5}$$

Dye solubilization measurements

Sealed tubes with Sudan Black *B* and solutions of different surfactant concentration were left for 1 week in a thermostatied bath, with periodic stirring. Then the tubes were centrifuged and the supernatant absorbance was measured with a Spectronic-20 UV-Vis spectrophotometer at 600 nm.

Results

As an example, Fig. 1 shows the concentration of LTA^+, OH^- and Br^- as a function of the total surfactant concentration (C_T = [LTAB] + [LTAOH]) for the LTAB:LTAOH ratio = 2:1. The other systems gave similar plots.

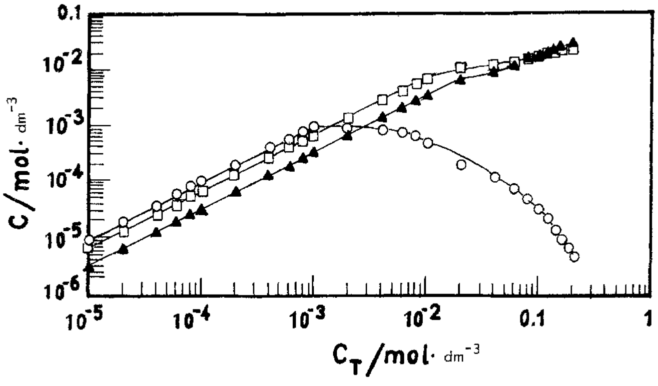

Fig. 1 Concentration of free ions in the system LTAB:LTAOH 2:1 vs. the total concentration. ○: [LTA$^+$], ▲: [OH$^-$], □: [Br$^-$]

Table 1 Concentrations at which the aggregation of the different ions start, vs. the mol fraction of LTAB in the surfactant

X_{LTAB}	Concentration (Mol·dm^{-3}) LTA$^+$	 Br$^-$	 OH$^-$	Literature CMC values
1	0.0011	0.0105	–	0.008 [20] 0.0175 [51] 0.011–0.014 [8] 0.0154 [52] 0.0146 [53]
0.6667	0.0010	0.016	0.024	
0.5	0.0026	0.020	0.020	
0.3333	0.0035	0.010	0.020	
0	0.0010	–	0.010	0.033 [20] 0.00077 [21,22] 0.0305–0.0295 [21]

The breaks indicating the beginning of aggregation of the different components in each system are summarized in Table 1, as a function of the LTAB molar fraction X_{LTAB} in the surfactant ($X_{LTAB} = [LTAB/\{[LTAB] + [LTAOH]\}$). The data are also shown in Fig. 2.

Figure 3 shows the α values vs. X_{LTAB}.

Figure 4 shows the values of K'_{OH} and K'_{Br} in the system LTAB: LTAOH 2:1. The average values of these constants (taken in the almost horizontal zone of the curves) and that of K^{OH}_{Br} are shown in Table 2 as a function of X_{LTAB}.

The ratio $[OH^-]_M/[Br^-]_M$, which is not used in the literature, remained almost independent of surfactant concentration (Fig. 5) but was dependent on X_{LTAB} (Fig. 6). The least squares fitting of the data in Fig. 6 is:

$$[OH^-]_M/[Br^-]_M = -(0.254774 \pm 0.000096) \log X_{LTAB} - 0.092773 \pm 0.000097$$

$$r = -0.99999$$

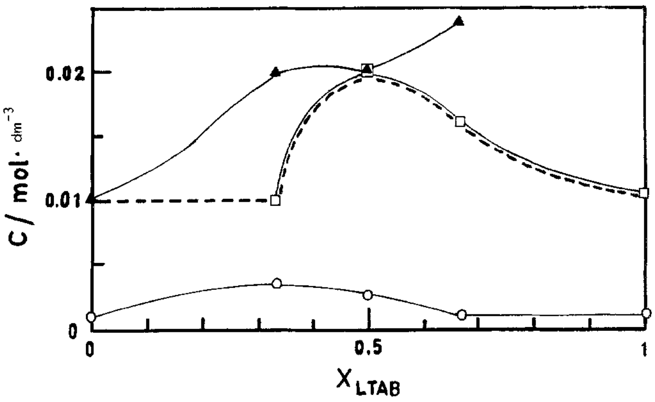

Fig. 2 Concentration at which the aggregation of different ions start vs. mole fraction of LTAB in the surfactant. ○: LTA$^+$, ▲: HO$^-$, □: Br$^-$ ---: CMC

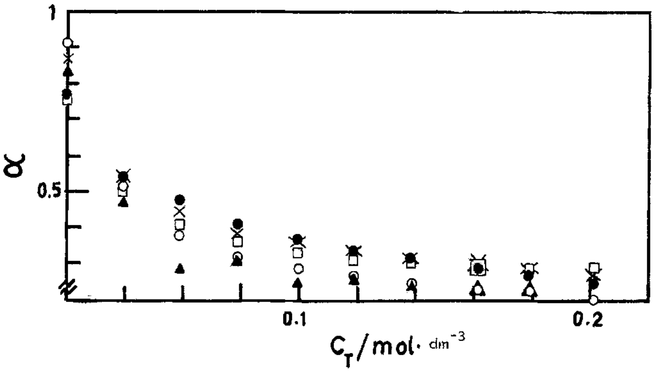

Fig. 3 Micelle ionization degree vs. total concentration. The LTAB: LTAOH ratio is: □ 1:0, × 2:1, ○ 1:1, ▲ 1:2 and ● 0:1

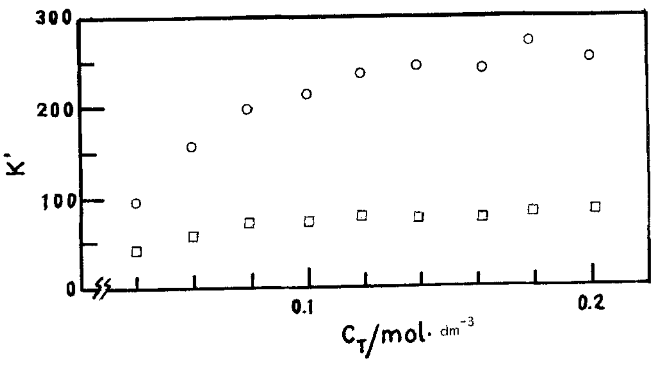

Fig. 4 Individual distribution constants in the system LTAB: LTAOH 2:1 □: K'_{OH}, ○: K'_{Br}

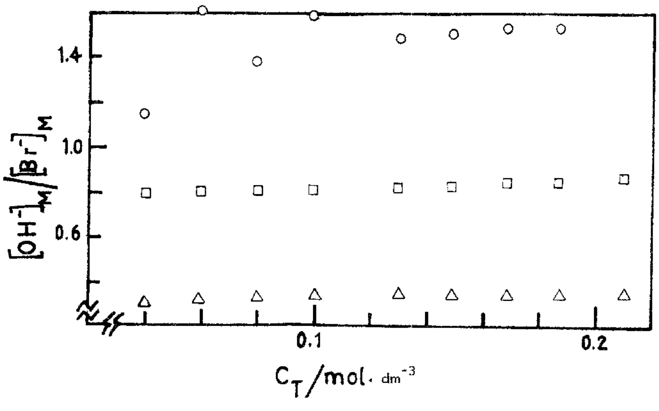

Fig. 5 $[OH^-]_M/[Br^-]_M$ vs. total concentration of surfactant. The LTAB: LTAOH ratio is: △ 2:1, □ 1:1 and ○ 1:2

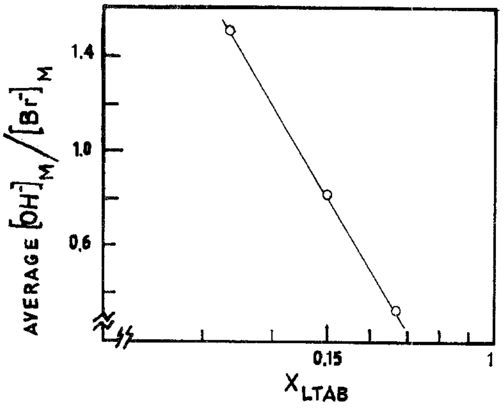

Fig. 6 $[OH^-]_M/[Br^-]_M$ vs. the mole fraction of LTAB in the surfactant

Table 2 Distribution constants as a function of the surfactant composition

LTAB:LTAOH	K'_{Br}	K'_{OH}	K_{Br}^{OH}
1:0	49.6 ± 7.3		
2:1	242 ± 19	70.3 ± 8.7	0.325 ± 0.015
1:1	142.2 ± 5.4	64.3 ± 3.2	0.471 ± 0.020
1:2	296 ± 11	66.2 ± 9.6	0.2485 ± 0.0042
0:1		16.8 ± 6.8	
Average (*)	176.3 ± 4.7	66.6 ± 2.7	0.2625 ± 0.0040

(*) Average of the values in 2:1, 1:1 and 1:2 mixtures.

Discussion

The aggregation

The CMC of LTAOH is still an open question. Several authors found different values. Lianos and Zana [19] found that the CMC value of RTAOH is about twice that of RTAB. Ninham et al. [20] found the LTAOH CMC $\approx 3.3\times10^{-2}$ mol·dm^{-3}, but other authors found 7.7×10^{-4} mol·dm^{-3} [21,22]. We studied this problem exhaustively [23] and found that LTAOH follows a stepwise aggregation: the LTA$^+$ ions form small premicellar aggregates at $C_T = 0.001300 \pm 0.000041$ mol·dm^{-3}, which grow with concentration. At $C_T = 0.01108 \pm 0.00010$ mol·dm^{-3} until they become true micelles and then the counterions adsorb to the micelle surface. Some modification of the micelle structure occurs at $C_T = 0.0302 \pm 0.0028$ mol·dm^{-3}. This is in accordance with Bunton et al. [9], who found that Cetyltrimethylammonium hydroxide (CTAOH) and fluoride (CTAF) solutions show a wide range of concentrations in which only small, fully ionized aggregates exist, but increasing the concentration, these aggregates grow to form true micelles.

The formation of true micelles coincides with the aggregation of counterions. We verified this by dye solubilization experiments. The aggregation of LTA$^+$ ions to form small premicellar aggregates is almost independent of X_{LTAB} (Fig. 2). The CMC follows the minimal concentration at which one of the counterions joins the micelles. It is seen that, when bromide ions are present, they join the micelles at smaller concentrations than do hydroxide ions. This was also found by Bunton et al. [24], by measurement of kinetic of reactions catalyzed by micelles of CTAB, and indicates that the Br$^-$ ions preferentially adsorb onto the micelle surface, when compared with OH$^-$ ions.

The CMC of LTAB and LTAOH are almost equal, but it is about twice this value at $X_{LTAB} = 0.5$ (LTAB:LTAOH 1:1), showing a positive deviation from ideal mixtures.

The small, fully ionized aggregates existing between the aggregation of LTA$^+$ ions and that of counterions grow with concentration. Eventually, these aggregates may dissolve some hydrophobic molecules, but because of their small surface charge density, they are not able to attract counterions or catalyse reactions [25].

The concentration of micellised surfactant

It is commonly supposed that the concentration of micellized surfactant molecules (on a monomer basis) is given by

$$[RTA^+]_M = C_T - CMC\,. \qquad (6)$$

As may be seen in Fig. 1, (similar plots were found in the other systems, and may be seen in the literature [26]) Eq. (6) is incorrect in the systems studied here. $[LTA^+]_W$ falls very fast after the break in the concentration–concentration curve, and almost all LTA$^+$ molecules form aggregates. If the CMC value is small, the error when Eq. (6) is used may be small, too, but in other situations the difference may be significant and it may be the origin of lack of fit between theory and experiment.

Micelle ionization

In general, the counterion concentration in the micelle Stern layer grows with the intermicellar counterion concentration, especially with hydrophilic counterions, e.g. OH^- [12, 27–29]. This means that α diminishes under the same conditions, as may be seen in Fig. 3.

It is commonly accepted that different counterions have different affinities for the micelle surface. This arises from considering both the coulombic and the specific interactions between micelles and counterions. The co-ion and counter-ion distribution around a charged colloidal particle may be predicted by classical electrostatics [30, 31], but the observed ionic specificity suggests that an attractive term must be included in the computations. This term will be small for small, hydrophilic ions, but large for large, highly polarizable ions [32–36]. The polarizability for Br^- (4.99) is larger than that of OH^- (2.74) [37]. This means that α for LTAB micelles must be smaller than that of LTAOH micelles, and this value must be intermediate in LTAB + LTAOH mixtures. As may be seen in Fig. 3, the former is true, but the latter is not, indicating a non-ideal behavior of the mixtures.

Figure 3 indicates that the commonly used supposition of constancy of α with concentration [9, 30] is not true. α diminishes with increasing counterion concentration, as was also found by other authors [38, 39].

The value of α at the CMC is very high ($\approx$0.9) for LTAOH, and higher than that for LTAB, as expected from previous papers [19, 40, 41]. The values of α for RTAOH from the literature are 0.7 [12, 19], 0.48 [40] and 0.50 [41].

Our results indicate a slight preference of Br^- ions for the micelle surface, when compared with OH^- ions, but the dependence with concentration is almost the same. α tends to values of 0.3 to 0.2 at high concentrations, irrespective of the counterion nature.

The distribution of counterions between micelles and water

The pseudophase model for the ionic exchange (PIE) presumes that the OH^- and Br^- ions compete for the micelle surface as if it were a ion-exchange resin [5, 14, 15–18]. The PIE model uses Eq. (3) and suposses that α is constant [14, 42–44]. The $[OH^-]_M$ values may be computed in terms of Eq. (3) and the total concentration of surfactant. In general, the PIE model fails to explain micellar catalysis when $[OH^-]$ is increased [45–48].

Table 3 Literature values of the CTA^+ distribution constants

K'_{OH}	K'_{Br}	K^{OH}_{Br}
55 [46, 9]	2000 [46]	0.071 [46]
	> 1000 [9]	0.024 [54]
		0.048 [55]
		0.080 [5, 6]
		0.1–0.025 [5, 56]

An alternative model describes the micelle-counterion interaction with equations of the form of the Langmuir adsorption isotherm, which is equivalent to a mass-action model (MAM) [45, 47–49]. Equations (4) and (5), arising from this model, predict that the counterion concentration at the surface of micelles will grow with total concentration, whereas the PIE model in its usual form requires its constancy [14, 17, 18]. Since the value of α for the same system varies with the measurement method [12–14], there is no objective evidence for its constancy. Moreover, the appropriate value of α for kinetic experiments may be different from that obtained from other experiments, as mobility [38] or quasielastic light scattering [39].

The kinetic data for CTAOH were fitted with $K'_{OH} = 55$, although slightly different values were successfully used, too [48]. The values of K'_x are larger for ions such as Br^- [45, 47].

The MAM model does not imply constancy of α, but it may be fitted to this condition [42a, 50].

In this work we found that the individual selectivity constants K'_{OH} and K'_{Br} are dependent on the total surfactant concentration (Fig. 4). The average values are in Table 2. For comparison, literature values for CTA^+ micelles are shown in Table 3.

Our K'_{OH} average value (66.6 ± 2.7) is of the same order of magnitude as that in the literature, but the K'_{Br} average value (176.3 ± 4.7) is one order of magnitude smaller, and that of K^{OH}_{Br} (0.2625 ± 0.0040) one order of magnitude larger, than the literature value. This indicates that the relative affinity for the micelle surface of Br^- ions with respect to OH^- ions has been overestimated in the kinetic computations in literature.

The ratio $[OH^-]_M/[Br^-]_M$ is almost independent of concentration but dependent on the LTAB:LTAOH ratio (Figs. 5 and 6). This means that the concentration of OH^- ions is a constant fraction of the total counterion concentration in the micelle Stern layer for each X_{LTAB}.

Conclusions

The study of the LTAB–LTAOH–water systems showed that some suppositions in the interpretation of micellar catalysis are incorrect, and these interpretations must be revised.

The ionization degree of micelles is not constant and varies with total surfactant concentration. Its dependence on the counterion nature is less than supposed in literature.

The concentration of micellized surfactant molecules is not equal to $C_T - \mathrm{CMC}$, and may be almost equal to C_T at high concentration.

The preference for the micelle surface of Br^- ions when compared with OH^- ions may have been overestimated in several works on micellar catalysis.

The present work supports the mass action model for the theoretical treatment of micellar catalysis, against the PIE model.

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