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Apparent and partial molar volumes of long-chain alkyldimethylbenzylammonium chlorides and bromides in aqueous solutions at T = 15 °C and T = 25 °C

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Abstract

Density measurements of dodecyl- (C₁₂DBACl), tetradecyl- (C₁₄DBACl), hexadecyldimethylbenzylammonium chloride (C₁₆DBACl) and of decyl- (C₁₀DBABr) and dodecyldimethylbenzylammonium bromide (C₁₂DBABr) in aqueous solutions at T = 15 °C and T = 25 °C have been carried out. From these results, apparent and partial molar volumes were calculated. Positive deviations from the Debye–Hückel limiting law provide evidence for limited association at concentrations below the critical micelle concentration. The change of the apparent molar volume upon micellization was calculated. The relevant parameters have been presented in function of the alkyl chain length. Apparent molar volumes of the present compounds in the micellar phase, $V_{\phi}^{\rm m}$, and the change upon micellization, $\Delta V_{\phi}^{\rm m}$, have been discussed in terms of temperature and type of counterion.

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1. Introduction

The dualistic character of amphiphilic compounds having both hydrophilic and hydrophobic moieties is the basis of their relation to both external and internal interfaces in aqueous systems. Self-association of the amphiphilic compounds is a possible way of eliminating the energetically unfavourable contact between the nonpolar part and water while simultaneously retaining the polar part in an aqueous environment. The physical phenomenon responsible for such behaviour is referred to as the hydrophobic effect and arises from a subtle balance between intermolecular energies and entropies [1]. An equilibrium solution of amphiphiles in water corresponds to a system of aggregates, co-existing with a nearly constant concentration of monomers. Unlike in ordinary solutions, the micelles in amphiphile solutions can respond to variations on thermodynamic parameters (such as total concentration, or temperature, or ionic strength) by changing their size and shape distributions. This behaviour resembles that of a system governed by multiple chemical equilibrium [2]. The presence of changes in some physical properties of amphiphiles in aqueous solutions is assumed to be indicative of an aggregation state and the discontinuity was identified with the critical micelle concentration (cmc) of surfactant systems [3].

Alkyldimethylbenzylammonium chlorides and bromides are used in cosmetic products including hair conditioners and hair colouring preparations. Besides, being surfactant antistatic agents, the alkyldimethylbenzylammonium compounds work as biocides in various cosmetic and detergent products. Among others, the biocidal properties are used, when alkyldimethylbenzylammonium is added to all-purpose or specialized cleaning agents. The earliest studies on micellar properties of alkyldimethylbenzylammonium salts were reported by Walton *et al.* [4] and Cushman *et al.* [5]. A large number of scientific application reports arises from their well known germicidal activity as antiseptics and disinfectants agents [6]. More recently, a survey of literature on the fundamental micellar properties of this group of surfactants was reported by Del Castillo *et al.* [7]. Extensive studies on the thermodynamics of micellar solubilization of co-surfactants in mixed binary surfactant systems containing alkyldimethylbenzylammonium salts have been carried out by Treiner *et al.* [8–10]. Their conductance results revealed the existence of a second cmc for C_{14} DBACl and C_{16} DBACl [10,11].

In previous papers, we discussed changes in the micellar properties of alkyldimethylbenzylammonium chlorides as a function of alkyl chain length, type of counterion, and temperature [12–14]. In this paper, we report our results on apparent and partial molar volume measurements at T = 15 °C and T = 25 °C. Apparent molar volumes at infinite dilution and apparent molar volumes of the micelles are calculated and discussed in function of the alkyl chain length and the type of counterion.

2. Materials and methods

 C_{12} DBACl was synthesized by quaternization of N, N-benzyldimethylamine with dodecyl chloride by applying a procedure similar to that used for the synthesis of the

TABLE 1

Densities, ρ , apparent molar volumes, V_{ϕ} , and partial molar volumes, V_2 , for C_nDBACl and C_nDBABr at T = 15 °C and T = 25 °C

т	ρ	V_{ϕ}	V_2
$(mol \cdot kg^{-1})$	$(g \cdot cm^{-3})$	$(cm^3 \cdot mol^{-1})$	$(cm^3 \cdot mol^{-1})$
		C ₁₂ DBACl	
		$T = 15 \ ^{\circ}\mathrm{C}$	
0	0.999099*		
0.0019	0.999106	335.9	336.9
0.0029	0.999108	336.7	337.6
0.0038	0.999110	337.0	338.2
0.0046	0.999110	337.6	338.8
0.0056	0.999113	337.5	339.5
0.0060	0.999113	337.7	339.8
0.0073	0.999115	337.9	340.7
0.0084	0.999113	338.5	341.4
0.0133	0.999085	341.3	344.2
0.0308	0.998951	345.1	349.0
0.0541	0.998771	346.5	348.3
0.0580	0.998765	346.2	347.6
0.0792	0.998578	346.8	347.8
0.0994	0.998449	347.1	347.7
0.1847	0.997917	347.1	347.9
0.2726	0.997398	347.1	347.9
		$T = 25 \ ^{\circ}\mathrm{C}$	
0	0.997043*		
0.0019	0.997041	342.0	342.6
0.0029	0.997039	342.4	343.2
0.0038	0.997037	342.6	343.6
0.0046	0.997034	342.9	344.1
0.0056	0.997030	343.3	344.6
0.0060	0.997028	343.5	344.8
0.0073	0.997026	343.3	345.5
0.0084	0.997020	343.7	346.0
0.0133	0.996975	346.1	348.1
0.0308	0.996801	348.9	350.7
0.0541	0.996591	349.5	351.4
0.0580	0.996562	349.4	351.0
0.0792	0.996360	349.9	350.5
0.0994	0.996176	350.1	350.7
0.1847	0.995453	350.2	350.5
0.2726	0.994727	350.3	350.7
		$T = 15 ^{\circ}\text{C}$	
0.0024	0 999094	369.9	383 1
0.0024	0.999089	371.4	384 7
0.0029	0.999079	373 3	382.3
0.0036	0.999079	373.3	380.0
0.0071	0.999040	376.6	380.2
0.0083	0.000076	377.1	379.3
0.0085	0.999020	377.6	379.2
0.0097	0.222002	577.0	519.4

m	ρ	V_{ϕ}	<i>V</i> ₂
$(mol \cdot kg^{-1})$	$(g \cdot cm^{-3})$	$(cm^3 \cdot mol^{-1})$	$(\text{cm}^3 \cdot \text{mol}^{-1})$
0.0113	0.998990	377.9	379.1
0.0140	0.998960	378.3	379.2
0.0150	0.998949	378.4	379.1
0.0171	0.998928	378.3	379.1
0.0191	0.998907	378.4	379.0
0.0218	0.998879	378.5	379.1
0.0235	0.998862	378.5	379.0
0.0255	0.998841	378.6	379.0
0.0266	0.998830	378.5	379.0
0.0312	0.998785	378.5	379.1
		$T = 25 \ ^{\circ}\mathrm{C}$	
0.0014	0 997042	369.8	377 1
0.0019	0.997038	371.8	380.2
0.0019	0.997032	373.8	381.1
0.0024	0.997032	374.9	381.1
0.0046	0.997004	377.6	382.7
0.0071	0.996071	379.4	383 3
0.0071	0.996955	379.9	382.8
0.0003	0.996935	380.4	382.0
0.0113	0.006012	380.8	382.0
0.0113	0.990912	381.3	383.1
0.0140	0.990875	281.4	282.0
0.0130	0.990801	281.6	382.9
0.0171	0.990833	281.6	282.0
0.0191	0.990807	281.0	282 1
0.0218	0.990772	201.0	282.0
0.0233	0.996730	381.9	383.0
0.0210	0.990132	501.9	505.1
		C_{16} DBACl $T = 15 \ ^{\circ}C$	
0.0060	0.999012	410.9	411.1
0.0094	0.998961	411.1	411.2
0.0100	0.998951	411.2	411.2
0.0149	0.998883	411.0	411.1
0.0185	0.99883	411.0	411.1
0.0187	0.998878	411.0	411.1
0.0235	0.998758	411.0	411.1
0.0235	0.220/20	411.1	411.1
0.0303	0.998659	411.2	411.1
0.0060	0.006047	$T = 25 {}^{\circ}\text{C}$	414.0
0.0000	0.99094/	415.5	414.8
0.0094	0.996882	414.6	415./
0.0100	0.9968/0	414./	415.3
0.0149	0.996783	414.9	415.5
0.0188	0.996/16	414.9	415.5
0.0235	0.996633	415.0	415.5
0.0296	0.996530	414.9	415.5
0.0303	0.996514	415.1	415.5

TABLE 1 (continued)

m	ρ	V_{ϕ}	V ₂
$(mol \cdot kg^{-1})$	$(g \cdot cm^{-3})$	$(cm^3 \cdot mol^{-1})$	$(\text{cm}^3 \cdot \text{mol}^{-1})$
		C ₁₀ DBABr	
		$T = 15 \ ^{\circ}\mathrm{C}$	
0.0065	0.999380	313.1	313.6
0.0070	0.999399	313.2	313.7
0.0105	0.999550	313.4	314.2
0.0148	0.999728	313.8	314.7
0.0168	0.999812	313.9	315.0
0.0206	0.999970	314.0	315.5
0.0224	1.000040	314.3	315.8
0.0277	1.000250	314.6	319.2
0.0355	1.000540	315.5	320.6
0.0447	1.000850	316.9	320.7
0.0538	1.001170	317.4	320.7
0.0563	1.001254	317.6	320.3
0.0710	1.001760	318.3	320.2
0.1593	1.004770	319.2	321.0
0.2596	1.007880	320.0	320.7
0.2824	1.008590	320.0	320.5
0.3866	1.011690	320.0	320.5
0.5127	1.015170	320.2	320.6
		$T = 25 \circ C$	
0.0070	0.007218	1 = 25 C	217.6
0.0070	0.997318	217 4	217.6
0.0104	0.997434	317.4	217.6
0.0148	0.997620	317.0	317.6
0.0131	0.997043	317.4	217.6
0.0205	0.997850	217.5	217.6
0.0200	0.997833	317.5	317.0
0.0224	0.997927	317.5	222.0
0.0277	0.998131	318.7	323.0
0.0304	0.998200	210 4	323.4
0.0333	0.998570	319.4	323.0
0.0403	0.998558	320.0	323.5
0.0447	0.998080	320.0	323.4
0.0520	0.998920	320.4	323.0
0.0553	0.998980	320.5	323.4
0.0505	0.999070	321.1	323.5
0.0048	0.999540	321.1	323.5
0.0710	1.000320	321.4	323.2
0.0955	1.000320	321.9	323.5
0.2000	1.003730	322.7	323.0
0.5955	1.014050	525.2	323.9
		C ₁₂ DBABr	
	0.005.1.77	$T = 15 \ ^{\circ}\mathrm{C}$	
0.0017	0.999165	344.6	344.9
0.0021	0.999180	345.1	345.0
0.0028	0.999208	344.9	345.1
0.0035	0.999237	345.2	345.2
0.0045	0.999276	344.7	345.4

TABLE 1 (continued)

т	ρ	V_{ϕ}	V_2
$(\text{mol} \cdot \text{kg}^{-1})$	$(g \cdot cm^{-3})$	$(cm^3 \cdot mol^{-1})$	$(cm^3 \cdot mol^{-1})$
0.0056	0.999318	345.2	345.6
0.0068	0.999360	346.2	352.9
0.0078	0.999386	347.6	353.4
0.0098	0.999450	348.5	353.4
0.0107	0.999479	349.1	352.6
0.0124	0.999534	349.3	352.7
0.0133	0.999564	349.4	352.3
0.0151	0.999628	349.4	352.3
0.0172	0.999689	350.2	352.2
0.0202	0.999776	350.9	352.5
		$T = 25 \ ^{\circ}\mathrm{C}$	
0.0017	0.997103	349.8	349.7
0.0021	0.997117	349.7	349.7
0.0028	0.997142	349.6	349.7
0.0035	0.997168	349.9	349.7
0.0045	0.997202	349.7	349.7
0.0056	0.997242	349.7	357.0
0.0068	0.997274	351.5	358.8
0.0078	0.997299	352.4	358.0
0.0098	0.997352	353.6	358.4
0.0107	0.997382	353.7	357.9
0.0124	0.997430	354.0	358.1
0.0133	0.997450	354.6	357.8
0.0151	0.997499	355.0	357.8
0.0172	0.997558	355.3	357.8
0.0202	0.997639	355.7	358.1

TABLE 1 (continued)

* Reference [24].

decyl homologue [7]. The surfactant was purified by several recrystallizations from acetone. C_{14} DBACl and C_{16} DBACl with an assay of >99% and >97%, respectively, were from Fluka. Both compounds were used without further purification. C_{10} DBABr with a purity of 99% was synthesized by quaternization of equimolar amounts of *N*, *N*-benzyldimethylamine and decyl bromide in a double volume of ethyl alcohol. The reaction was carried out at 80 °C for 72 h. C_{12} DBABr with a purity of >99% was from Fluka. The five studied compounds were found to be in the form of monohydrates as determined by the Karl–Fischer method. Bidistilled water and solutions were properly degassed using an ultrasound unit.

The measurement of densities were carried out by using an Anton Paar DMA 60/ 602 vibrating tube densimeter operating under the static option. Solutions were prepared by mass using a Mettler H51 precision balance. The molalities of the solutions were prepared within an accuracy of $\pm 0.0001 \text{ mol} \cdot \text{kg}^{-1}$. The temperature inside the vibrating-tube cell was measured using Anton Paar DT 100-20 and DT 100-30 digital thermometers and was regulated better than ± 0.01 K using a HETO DBT thermostat. The apparatus was calibrated at the beginning of every set of experiments using water and air (at atmospheric pressure) as references (see table 1). Taking into

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account that the relative uncertainty in the difference between the densities of the sample and pure water is mainly due to the difference in the square of the corresponding oscillating periods, and that the relative uncertainty of a single period founded in our case was about 10^{-6} , then the uncertainty in the density is assumed to be less than $5 \cdot 10^{-6} \text{ g} \cdot \text{cm}^{-3}$.

3. Results and discussion

The values of densities, ρ , of aqueous solutions of C_nDBACl (n = 12, 14, and 16) and C_nDBABr (n = 10, 12) at T = 15 °C and T = 25 °C are listed in table 1. In an ionic surfactant system, below the cmc, the dependence of the density on molar concentration c can be expressed as

$$(\rho - \rho_0) = [(M_c + M_f) - (V_c + V_f)\rho_0]c,$$
(1)

where $M_{\rm f}$, $M_{\rm c}$, $V_{\rm f}$, and $V_{\rm c}$ are the molar masses and the apparent molar volumes of the free surfactants ions (f), and counterions (c), respectively, and ρ_0 is the density of pure water. If we assume that the apparent molar volumes are negligibly concentration-dependent, then equation (1) predicts a linear relation between density and concentration. However, this was found not to be true in the case of C₁₂DBACl at T = 15 °C, where a change in the slope of the density vs molality plot from positive to negative can be detected (see table 1). Such behaviour is not observed for the same surfactant at T = 25 °C and for all the other surfactants. This change in slope, indicates, according to equation (1), that the $V_{\rm c} + V_{\rm f}$ terms increase with temperature. This effect is related to the relaxation of structured water engaged in the solvation of the hydrocarbon chain, the headgroup, and the counterion upon increase in temperature.

The slopes of plots of density vs molality, for the bromides, are positive over the entire molality and temperature ranges studied; however, slopes for chlorides are negative over the whole set of data.

From the density values, apparent molar volumes of surfactants in water were calculated according to the equation [15]

$$V_{\phi} = M/\rho - (\rho - \rho_0)/(m\rho\rho_0),$$
(2)

where M is the molar mass of the solute in and m its molality. The partial molar volumes V_2 were calculated from V_{ϕ} using the equation

$$V_2 = V_{\phi} + m(\partial V_{\phi}/\partial m)_{T,P}.$$
(3)

The values of partial molar volumes, V_2 , are also listed in table 1 together with the values of the apparent molar volumes.

In the premicellar region, the values of V_{ϕ} can be expressed by the relation

$$V_{\phi} = V_{\phi}^0 + A_{\rm V} m^{1/2} + B_{\rm V} m, \tag{4}$$

where V_{ϕ}^{0} is the value of apparent molar volume at infinite dilution, $A_{\rm V}$ is the Debye– Hückel limiting law coefficient which amounts to 1.696 cm³ · kg^{1/2} · mol^{-3/2} and 1.865

	$B_{\rm V}/({\rm cm}^3\cdot{\rm kg}\cdot{\rm mol}^{-2})$		
	$T = 15 \ ^{\circ}\mathrm{C}$	$T = 25 \ ^{\circ}\mathrm{C}$	
C ₁₂ DBACl	338 ± 20	340 ± 16	
C ₁₀ DBABr	59 ± 6	-2 ± 3	
C ₁₂ DBABr	52 ± 5	-7 ± 8	

TABLE 2 Values obtained for the adjustable parameter B_V of equation (4)

cm³ · kg^{1/2} · mol^{-3/2} at T = 15 °C and T = 25 °C, respectively, and B_V is an adjustable parameter which measures individual deviations from the limiting law. The fit of the V_{ϕ} values to this equation yields the limiting value V_{ϕ}^0 . The parameters B_V obtained for the studied systems are listed in table 2. It is instructive to observe those values. While for the chlorides B_V remains constant with the temperature, for the bromides the slope changes from positive to almost zero (the zero value is inside the error bars). If we observe the dependence with hydrophobic moiety of the molecule, there is no significant change for the bromides, with n = 10 and 12. However, for both dodecyl a great change can be observed. All these facts indicate that the counterions play the main role in the deviations from the limiting law. Finally, positive deviations from the Debye–Hückel limiting law provide evidence for limited association at concentrations below the cmc.

As Millero pointed out [15], the densities of aqueous solutions of the electrolytes can be determined from the relation

$$\rho = \rho_0 + \left(M - \rho_0 V_{\phi}^0 \right) c - 10^{-3} A_{\rm V} \rho_0 c^{3/2} - 10^{-3} B_{\rm V} \rho_0 c^2.$$
(5)

A more lengthy relation can be derived when concentration are expressed as molalities. Since $\partial \rho / \partial c$ is linear with *n*, it can be immediately seen that the linearity of density vs molarity plots in the low concentration range indicates that the second and third terms in equation (5) are negligibly small or cancel out. Increase of density with molarity indicates thus that the $M - \rho_0 V_{\phi}^0$ term in the first parameter in equation (5) is positive. This term appears to be negative when the molecular weight of the counterion in surfactants is low and its apparent molar volume is comparatively high, as found in the case of chlorides.

The values of V_{ϕ}^{0} for C₁DBACl from Boyd [16] and Owens *et al.* [17], for C₁₀DBACl from González-Pérez *et al.* [12], and for C₁₂DBACl from present results, together with the values for alkyltrimethylammonium chlorides (C_nTACl) for C₁TACl from Verrall and Conway [18], for C₈TACl and C₁₂TACl from Milioto *et al.* [19] at T = 25 °C are listed in table 2. The values of V_{ϕ}^{0} for C₁₀DBABr are 312.6 cm³ · mol⁻¹ and 317.4 cm³ · mol⁻¹ at T = 15 °C and T = 25 °C and for C₁₂DBABr are 344.6 cm³ · mol⁻¹ and 349.5 cm³ · mol⁻¹ at T = 15 °C and T = 25 °C, respectively. The values for the bromides confirm those reported earlier [20].

Linear fitting of the values of V_{ϕ}^0 at T = 25 °C (collected in table 3) in function of the number of carbon atoms in the alkyl chain (C_n) yields slopes of 15.7 ± 0.1 cm³·mol⁻¹ and 15.8 ± 0.1 cm³·mol⁻¹ for the C_nDBACl and C_nTACl salts, respectively. These values are in satisfactory agreement with the result of 16.0 ± 0.2

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TABLE 3

Apparent molar volumes at infinite dilution, V_{ϕ}^0 , for C_nDBACl (T = 15 °C and T = 25 °C) and C_nTACl (T = 25 °C)

n	$V_{\phi}^0/(\mathrm{cm}^3\cdot\mathrm{mol}^{-1})$			
	C _n DBACl		C _n TAC1	
	$T = 15 \ ^{\circ}\mathrm{C}$	$T = 25 \ ^{\circ}\mathrm{C}$	$T = 25 \ ^{\circ}\mathrm{C}$	
1		168.9 ^{<i>b</i>,<i>c</i>}	107.3^{d}	
8			217.2^{e}	
10	307.2 ^{<i>a</i>}	310.7 ^a		
12	335.6	341.6	280.9 ^e	

^aReference [7].

^bReference [16].

^c Reference [17].

^dReference [18].

^e Reference [19].

 $\text{cm}^3 \cdot \text{mol}^{-1}$ reported by Tutaj *et al.* [21] for short-chain alkyldimethylbenzylammonium bromides C_nDBABr (n = 1, 4, and 6).

In the post-micellar region, the values of V_{ϕ} have been fitted to the asymptotic function

$$V_{\phi} = V_{\phi}^{\text{cmc}} + \left[\Delta V_{\phi}^{\text{m}}(m - M_{\text{cmc}}) / \{B + (m - M_{\text{cmc}})\} \right], \tag{6}$$

where V_{ϕ}^{cmc} is the value of the apparent molar volume at the cmc, $\Delta V_{\phi}^{\text{m}}$ is the difference between the limiting value at high concentration of surfactant, V_{ϕ}^{m} , and V_{ϕ}^{cmc} . The $\Delta V_{\phi}^{\text{m}}$ can be identified with the change in the apparent molar volume upon micellization. In the case of C₁₄DBACl and C₁₆DBACl the reported values of V_{ϕ} are restricted to the post-micellar region only because of the low cmc values. For all the studied compounds the values of V_{ϕ}^{m} and $\Delta V_{\phi}^{\text{m}}$ are listed in table 4. De Lisi *et al.* [22]

n	$V_{\phi}^{\mathrm{m}}/(\mathrm{cm}^3\cdot\mathrm{mol}^{-1})$		$\Delta V_{\phi}^{ m m}/(m cm^3\cdot m mol$	$\Delta V_{\phi}^{\rm m}/({\rm cm}^3\cdot{ m mol}^{-1})$	
$T = 15 \ ^{\circ}\mathrm{C}$		$T = 25 \ ^{\circ}\mathrm{C}$	$T = 15 \ ^{\circ}\mathrm{C}$	$T = 25 \ ^{\circ}\mathrm{C}$	
		C _n DBAC1			
10	314.2 ^{<i>a</i>}	317 ^a	7.0^{a}	5.7 ^a	
12	347.75	350.6	9.35	6.6	
14	379	383.1	15.0	10.6	
16	411.0	414.85	18.7	11.2	
		C_n DBABr			
10	313.7	317.1	6.8	6.3	
12	344.9	349.8	7.7	8.1	

Limiting apparent molar volumes of micelles of C_n DBACl, V_{ϕ}^m , and the change upon micellization, ΔV_{ϕ}^m , at T = 15 °C and T = 25 °C

^a Reference [7].

TABLE 4

found negative slopes of $\Delta V_{\phi}^{\rm m}$ of sodium octyl, decyl, and dodecyl sulphates in water against temperature in the temperature range from 25 °C to 130 °C at different pressures. This negative slope value is attributed to the expansibility of the surfactant in the micellar state being smaller than that in the aqueous phase. Atwood *et al.* [23] found this decrease with temperature for tricyclic drugs, and the effect has been attributed to the dehydration of the ionic head group.

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