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ORIGINAL CONTRIBUTION

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Apparent molar quantities of sodium octanoate in aqueous solutions

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Abstract Densities and sound velocities of aqueous solutions of sodium octanoate were determined in a range of molalities between 0.0352 and 0.8105 mol kg^{-1} at 25, 30, 35, 40 and 45 °C. The isotherms of molality dependence of both density and sound velocity were used to determine the cmcs. Apparent molar volumes and compressibilities were determined from measurements of ultrasound velocity and density. The values of apparent molar volumes and compressibilities at infinite dilution and the apparent molar quantities in the micellar range were obtained and studied as a function of temperature. Values of the critical micelle concentration and the apparent molar quantities in the premicellar and postmicellar range are discussed and compared with the values of the corresponding fluorinated compound.

Keywords Sodium octanoate · Apparent molar volumes · Compressibilities · Critical micelle concentration · Sodium perfluorooctanoate

Introduction

The self-assembly process of surfactants in aqueous solutions has been investigated in the last few years using different techniques and theories. One of the most important characteristics of surfactants is their capability to reduce the surface tension of a solution and form self-assembly structures (micelles) that appear at some critical concentration called the critical micelle concentration (cmc).

Micellar systems can be characterized by various molecular parameters, such as aggregation number, micellar radius and shape, using a wide variety of techniques, such as light scattering, neutron scattering, NMR, rheology and luminescence probing methods, and also by thermodynamic parameters [1].

The temperature dependence of the cmc can be used to estimate thermodynamic parameters of micellization by applying the charged pseudo-phase-separation model or the mass action model [2]. These parameters provide relevant information on the structural dependence of surfactants on micelle formation as well as on the Gibbs free energy, enthalpy and entropy of micellization.

Other methodologies can be used to obtain the thermodynamic parameters apparent molar volumes and compressibilities [3, 4] as well as heat capacities and enthalpies [5, 6]. Such parameters characterize the physical state of the micelle and can be calculated from density and sound velocity or microcalorimety, respectively. An interesting compilation of thermodynamic methods was provided by Desnoyers et al. [7].

The molar volumes of different electrolytes have been reviewed by Millero [8] and more recently by Lepori and Gianni [9]. Numerous papers have made important contributions to the general understanding of volumetric properties of surfactants in the micellar state. Concerning the apparent molar compressibility, from the earliest papers of Shigehara [10] and Vikingstad et al. [11], several interesting works have been developed. These have led to an understanding of factors contributing towards the total compressibility of a micellar solution. The contribution of head-group interactions at the micellar surface has been studied by Bloor et al. [12]. More recently, the study of the temperature dependence and influence of chain length on homologues of alkyltrimethylammonium bromide was reported by Zielinski et al. [13] and Kudryashov et al. [14].

In previous papers, attention was focused on the study of the thermodynamic process of micellization for sodium alkanoates and corresponding fluorinated compounds to understand changes in micellar properties with the substitution of the hydrogen of the hydrocarbon chain by fluorine atoms [15]. A survey of the literature was presented in a recent paper by González-Pérez et al. [15] on micellar properties of sodium octanoate in aqueous solution. The thermodynamic parameters derived from the knowledge of the temperature dependence of the cmc were reported according to the mass-action model and the results were discussed in comparison with those of the corresponding fluorinated compound. For more information on the micellization process of sodium octanoate, the temperature dependence of density and sound velocity above and below the cmc was studied. From these data apparent molar quantities such as volume and compressibility can be determined using the model suggested by Zielinski et al. [16].

The present work is a continuation of a previous paper on thermodynamic properties of sodium octanoate studied by conductivity [17]. Extensive studies on the micellization of sodium octanote in aqueous solutions using different techniques were reported previously by Ekwall and coworkers [18, 19, 20, 21, 22, 23, 24]. More recently, this compound was studied by D'Angelo et al. [25] using sound velocity to obtain the thermodynamics of micellization. Not much attention has been paid to the temperature dependence for volumetric and compressibility quantities of the present compound. To obtain more information on the apparent molar volumes and compressibilities and their temperature dependence, density and sound velocity were studied over a wide range of molalities at 25, 30, 35, 40 and 45 °C. The results were compared with those previously reported for sodium perfluorooctanoate.

Experimental

Materials

Sodium octanoate, with a purity of 97%, was purchased from Lancaster and was used without further purification. The different concentrations were prepared by weight using distilled water with conductivity below 3 μS cm^{-1} at 25 °C.

Densities and ultrasound velocity measurements

Densities and ultrasound velocities were continuously, simultaneously and automatically measured using a commercial apparatus (Anton Paar DSA 5000 densimeter and a sound velocity analyser, respectively). Because both the speed of sound and density are extremely sensitive to temperature, the Peltier method was used to keep the temperature constant to within $\pm 10^{-3}$ K. The reproducibilities of the density and ultrasound measurements were $\pm 10^{-6}$ g cm^{-3} and $\pm 10^{-2}$ m s^{-1}, respectively. Measurements of density and ultrasound velocity of aqueous solutions of sodium octanoate in the temperature range from 25 to 45 °C were taken as a function of concentration.

Methods

The thermodynamic methods for binary systems employed to study micellization have been described by Desnoyers et al. [7]. The following equations are used to determine the apparent molar volumes and compressibilities from density and sound velocity measurements.

From density measurements, apparent molar volumes can be calculated using the following equation [26]:

$$V_\phi = \frac{10^3(\rho_0 - \rho)}{m\rho\rho_0} + \frac{M}{\rho}, \tag{1}$$

where ρ is the density (expressed in grams per cubic centimetre) at the corresponding molality, ρ_0 is the density of pure water, and M and m are the molecular weight of the surfactant and the molality of the solution, respectively.

From sound velocity and density, the adiabatic compressibility of surfactant solutions can be calculated from the following relation:

$$\beta_\mathrm{S} = \frac{10^{-3}}{u^2\rho}, \tag{2}$$

where u is the sound velocity expressed in meters per second.

The apparent molar adiabatic compression of the solute was calculated by using the next relation [26]:

$$K_\phi = \frac{10^3(\beta_\mathrm{S} - \beta_{\mathrm{S},0})}{m\rho_0} + \beta_\mathrm{S} V_\phi, \tag{3}$$

where $\beta_{\mathrm{S},0}$ represents the adiabatic compressibility for pure water.

Results and discussion

The isotherms of density against molality in the form of $\rho - \rho_0$ at 25, 30, 35, 40, and 45 °C are shown in Fig. 1. The set of data shows breaks corresponding to the cmc.

The results of sound velocity against molality are shown in Fig. 2 in the form of $u - u_0$, where u_0 is the ultrasound velocity of pure water, at 25, 30, 35, 40 and 45 °C. Again the abrupt changes in the slopes represent the cmc.

From the linear segments above and below the cmc reported in Figs. 1 and 2, the cmc can be determined from the intercepts of both linear adjustments for every isotherm. The results of the cmc in the temperature range studied are shown in Table 1 together with values previously reported in the literature. The results are in

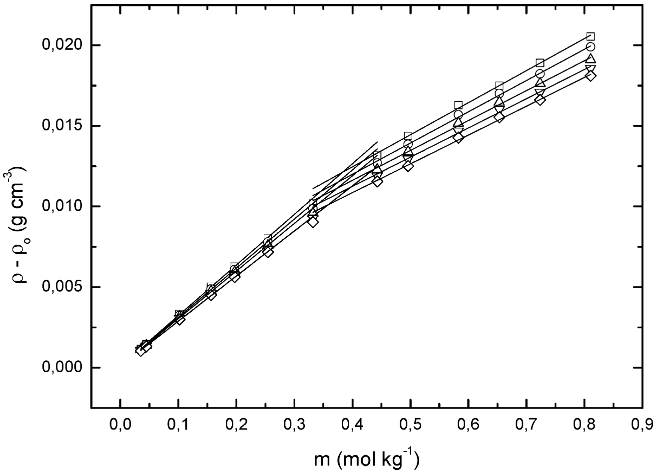

Fig. 1 Densities of aqueous solutions of sodium octanoate corrected for the density of the solvent, $\rho-\rho_0$, as a function of the molal concentration, m, at 25 °C (*squares*), 30 °C (*circles*), 35 °C (*up triangles*), 40 °C (*down triangles*) and 45 °C (*diamonds*)

good agreement with the data reported by D'Angelo et al. [25] and González-Pérez et al. [17]. The small differences are attributed to the different behaviour of the isotherms, depending on the technique used.

Using the molality dependence of the density data reported in Fig. 1, the apparent molar volume in the premicellar and postmicellar range can be obtained using Eq. (1). The results are shown in Fig. 3. The first linear dependence corresponds to the surfactant in monomeric form and the increase upon micellization appears in surfactant solutions in the micellar state.

Knowing the density and sound velocity versus molality, we can then use Eq. (2) to determine the adiabatic compressibility of the present compound at every temperature. The apparent molar adiabatic compression of the solutions can be calculated using Eq. (3). The results are shown in Fig. 4. Similar behaviour to that reported in Fig. 3 was found, and the same explanation is valid. The linear behaviour corresponds to the monomeric form and the increase in compressibilities upon micellization came from the self-assembly process.

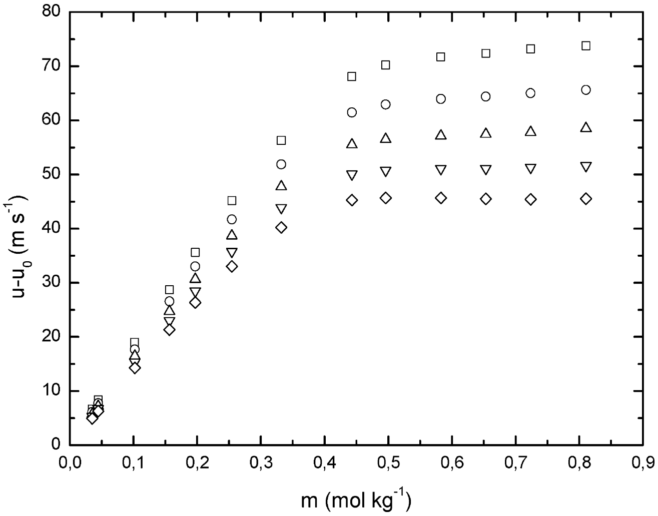

Fig. 2 Sound velocities of aqueous solutions of sodium octanoate corrected for the sound velocity of the solvent, $u-u_0$, as a function of m at 25 °C (*squares*), 30 °C (*circles*), 35 °C (*up triangles*), 40 °C (*down triangles*) and 45 °C (*diamonds*)

Apparent molar quantities at infinite dilution

It is well known that surfactant solutions below the cmc exhibit the classical behaviour of conventional aqueous

Table 1 Critical micelle concentrations (*cmcs*) of sodium octanoate in aqueous solution at different temperatures

Reference [25]		Reference [17]		Present work		
t (°C)	cmc (mol kg^{-1})	t (°C)	cmc (mol kg^{-1})	t (°C)	cmc (mol kg^{-1})^a	cmc (mol kg^{-1})^b
16	0.4014	25	0.3821	25	0.3828	0.3955
18	0.3969	27	0.3783	30	0.3760	0.3852
20	0.3919	30	0.3733	35	0.3747	0.3754
22	0.3879	32	0.3703	40	0.3678	0.3684
26	0.3846	35	0.3644	45	0.3589	0.3631
28	0.3812	40	0.3549			
30	0.3755	45	0.3508			
35	0.3705	50	0.3455			
40	0.3598	55	0.3433			
45	0.3531	60	0.3417			
50	0.3491	65	0.3426			
55	0.3452	70	0.3459			
60	0.3429					
65	0.3412					
70	0.3435					
75	0.3491					
80	0.3553					
85	0.3643					

[a] Data from density measurements
[b] Data from sound velocity measurements

salt solutions and only up to the cmc are remarkable changes found in their properties owing to the self-assembly of simple monomers, hence inducing changes in the general properties of the aqueous solutions.

To study surfactant solutions in monomeric form, the well-known equation of Redlich and Rosenfeld [27, 28] can be applied. To obtain the value of the apparent molar volume at infinite dilution, V_ϕ^0, it was assumed that sodium octanoate behaves as a 1:1 electrolyte in solution at concentrations below the cmc. Thus, at a given concentration the apparent molal volumes may be described in the premicellar region by the equation

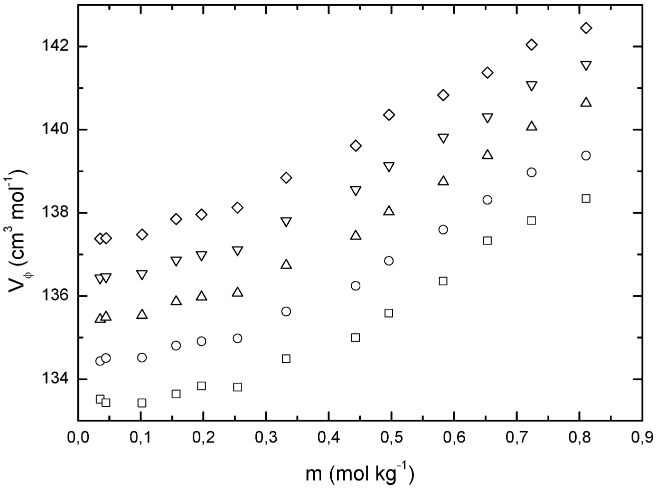

Fig. 3 Apparent molar volumes, V_ϕ, in the premicellar and postmicellar range as a function of m for aqueous solutions of sodium octanoate at 25 °C (*squares*), 30 °C (*circles*), 35 °C (*up triangles*), 40 °C (*down triangles*) and 45 °C (*diamonds*)

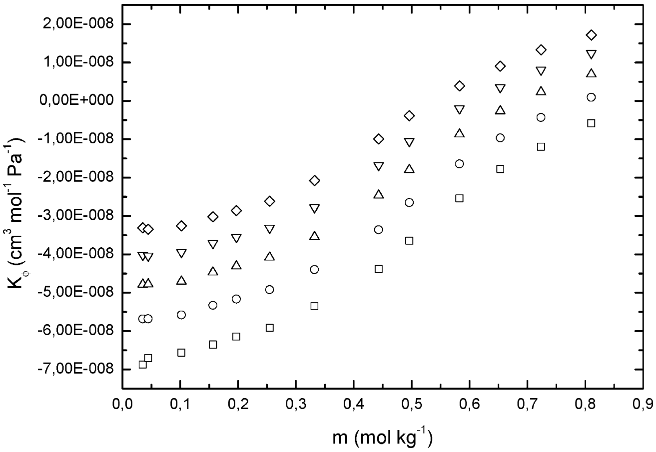

Fig. 4 Apparent molar compressibilities, K_ϕ, as a function of m for aqueous solutions of sodium octanoate at 25 °C (*squares*), 30 °C (*circles*), 35 °C (*up triangles*), 40 °C (*down triangles*) and 45 °C (*diamonds*)

$$V_\phi = V_\phi^0 + A_V m^{1/2} + B_V m, \tag{4}$$

where A_V is the Debye–Hückel limiting law coefficient and the values for the 1:1 electrolyte are 1.868, 1.955, 2.046, 2.138 and 2.234 cm^3 kg$^{1/2}$ mol$^{-3/2}$ at 25, 30, 35, 40 and 45 °C, respectively [8], and B_V is an adjustable parameter related to a pair interaction [29] and is equivalent to the second virial coefficient, which measures the deviation from the limiting law due to non-electrostatic solute–solute interactions. This coefficient is usually negative except for hydrogen-bonding interactions [29, 30]. More coefficients are not considered in the case of volume in Eq. (4).

The values of V_ϕ^0 and B_V for sodium octanoate at different temperatures were 133.1, 134.1, 135.0, 136.0 and 136.9 cm^3 mol^{-1} at 25, 30, 35, 40 and 45 °C, respectively. The results of V_ϕ^0 at 25 and 45°C are in excellent agreement with the data reported by Sakurai et al. [31] of 133.00 and 136.76 ml mol^{-1} at 25 and 45 °C, respectively. In the paper of Sakurai et al. a value of 127.85 ml mol^{-1} at 15 °C was reported, this value being below the Krafft point that was previously reported close to 25 °C [15, 17]. It is well known that the values of B_V decrease with increasing chain length but with longer chains the result goes in the opposite way. This unexpected trend was also reported by Leduc and Desnoyers [32], who proposed extra weak interactions between the solutes, apart from hydrophobic hydration. If fact this salt was proposed as being a structure-breaker in the scheme of Helper [33] and this was then confirmed by Sakurai et al. [31].

Studying the apparent thermal expansion coefficient $\left(1/V_\phi\right)\left(\partial V_\phi/\partial T\right)_p$ can provide information about solute–solute interactions [34]. A decrease was found for this coefficient with temperature (values of 1.46×10^{-3}, 1.44×10^{-3}, 1.42×10^{-3}, 1.37×10^{-3} and 1.32×10^{-3} at 25, 30, 35, 40 and 45 °C, respectively), and became more pronounced up to 35 °C. This general decrease with temperature was found previously by Sakurai et al. [31]; however, now it is confirmed for more temperatures. The more pronounced slope of the apparent thermal expansion coefficient found up to 35 °C suggests that the effect of thermal motion is stronger. This result is in accordance with the loosening of water molecules surrounding the Na$^+$ counterion suggested by González-Pérez et al. [15, 17].

The values of B_V increase with temperature, going from −0.87 to 0.42 cm^3 kg mol^{-1} in the temperature range studied. Such behaviour was observed in other amphiphilic systems and was attributed to the formation of premicellar aggregates [35].

The apparent molar compressibility at infinite dilution, K_ϕ^0, can be calculated using the following relation:

$$K_\phi = K_\phi^0 + A_K m^{1/2} + B_K m, \tag{5}$$

where the parameter A_K represents the Debye–Hückel limiting law coefficient and B_K is an adjustable parameter that represents the deviations from the limiting law. The parameter A_K reported by Desnoyers et al. [7] based on the previous work of Bradle and Pitzer [36] is inconsistent with the experimental data, as reported by Garnsey et al. [4] for electrolyte solutions. This is the reason for applying Eq. (5) with A_K and B_K as free parameters.

Fitting the data below the cmc reported in Fig. 4 to Eq. (5), we can obtain the relevant parameters, K_ϕ^0, A_K and B_K. In the present case, A_K was used as a free parameter in the fitting. The values of K_ϕ^0 obtained are negative at low temperatures, which can be interpreted as a result of higher resistance to the pressure of water structures around the surfactant in the monomeric form compared with that of water in the bulk, as suggested by Cabani et al. [37].

Apparent molar quantities in the micellar range

In the micellar range apparent molar volumes and compressibilities show an increase, rising to a plateau that corresponds with the apparent molar volume in the micellar state. Values of V_ϕ corresponding to the post-micellar region were fitted to equation

$$V_\phi = V_\phi^{\mathrm{cmc}} + \frac{\Delta V_\phi^m (m - \mathrm{cmc})}{[B + (m - \mathrm{cmc})]}, \qquad (6)$$

where B is an adjustable parameter without physical meaning, V_ϕ^{cmc} is the value of the apparent molar volume at the cmc and ΔV_ϕ^m is the difference between the limiting value of V_ϕ and V_ϕ^{cmc} and can be identified as the apparent molar volume upon micellization.

Values of V_ϕ^{cmc} and ΔV_ϕ^m at different temperatures are shown in Table 2. ΔV_ϕ^m decreases with increasing temperature. As suggested by Zielinski et al. [13], such effects indicate that the structure of the micelles is looser than that of the monomers at each temperature and the dehydration of the ionic group may be brought about with increased temperatures.

In the postmicellar region, an equation similar to Eq. (6) was applied. The K_ϕ values were fitted to equation

$$K_\phi = K_\phi^{\mathrm{cmc}} + \frac{\Delta K_\phi^m (m - \mathrm{cmc})}{[C + (m - \mathrm{cmc})]}, \qquad (7)$$

where C is an adjustable parameter without physical meaning, K_ϕ^{cmc} is the value of the apparent molar adiabatic compressibility at the cmc and ΔK_ϕ^m is the difference between the limiting value and K_ϕ^{cmc}, and can be identified with the apparent molar adiabatic compressibility upon micellization.

The results of K_ϕ^{cmc} and ΔK_ϕ^m at different temperatures are shown in Table 2. The values are positive, indicating the predominant role of the decrease in hydrophobic hydration in the association process.

Fluorocarbon–hydrocarbon comparison

In recent years surfactants containing fluorinated chains have attracted the attention of the scientific community because this kind of compound has interesting properties from a theoretical and practical point of view. The development of fluorine chemistry permits the synthesis of a wide variety of fluorinated surfactants as well as mixtures of both hydrogen and fluorine compounds in the same salt. Fluorinated surfactants show both hydrophobic and lipophobic behaviour and their hydrophobicity is higher than that of the corresponding hydrocarbonated compounds. This behaviour is evident from the fact that many physical properties of the shorter-chain fluorinated surfactants correspond to the longer-chain hydrogenated compounds. The cmc of perfluorocarbon surfactants is approximately 1.5 times higher than that of the corresponding hydrocarbon compound and this is attributed to the difference in transferring the Gibbs free energy of $–CF_2–$ and $–CH_2–$ from the aqueous to the micellar phase; hence, this response is transmitted to the enthalpy and entropy of micellization. Other thermodynamic properties, such as apparent molar volumes and compressibilities, show drastic changes with the substitution of hydrogens by fluorines in the same surfactant structure.

The dependence of apparent molar volumes on chain length has been studied by Tamaki et al. [38], Perron and Desnoyers [39], and more recently by De Lisi and coworkers [40, 41]. Not much attention has been given to the temperature dependence of such compounds and the differences between fluorocarbon and hydrocarbon compounds.

Taking into account previous data, the temperature dependence of apparent molar quantities in sodium perfluorooctanoate and sodium octanoate in the

Table 2 Apparent molar volumes and compressibilities at the cmc and the increase upon micellization estimated rom Eqs. (6) and (7)

t (°C)	V_ϕ^m (cm^3 mol^{-1})	ΔV_ϕ^m (cm^3 mol^{-1})	$10^8 K_\phi^m$ (cm^3 Pa^{-1} mol^{-1})	$10^7 K_\phi^m$ (cm^3 Pa^{-1} mol^{-1})
25	134.2	14.2	-5.2336×10^{-8}	1.16
30	135.4	13.8	-4.3623×10^{-8}	1.00
35	136.7	13.4	-3.5869×10^{-8}	0.91
40	137.9	13.1	-2.8506×10^{-8}	0.84
45	138.8	12.8	-2.1873×10^{-8}	0.78

premicellar and postmicellar range can be studied to determine the influence of a fluorocarbon chain substitution on the general properties of these surfactant solutions.

Apparent molar volumes and compressibilities at infinite dilution provide information on solute–solvent interactions. The temperature dependence for the apparent molar volume and compressibilities of sodium octanoate and perfluorooctanoate from Ref. [15] are shown in Fig. 5.

Linear behaviour was found for apparent molar volumes at infinite dilution. The slope was 0.19 for octanoate and 0.40 for perfluorooctanoate, taking into account only the values below 40 °C for perfluorooctanoate. As previously found, the values above 40 °C undergo a slight decrease as a consequence of loss of water molecules around the counterion.

The effect of substitution of the hydrogen by fluorine on the alkyl chain on the temperature dependence of sodium octanoate produces an increase in the apparent molar volume that follows the next relation:

$$\begin{aligned}\Delta V_\phi^0 &= V_\phi^0(F) - V_\phi^0(H) \\ &= 57 \pm 1 + (0.45 \pm 0.05)t - (0.0036 \pm 8.39E - 4)t^2,\end{aligned} \tag{8}$$

where t is given in centigrade.

The apparent molar compressibility of both fluorinated and hydrogenated compounds shows a sharp distinct behaviour. Linear dependence was found for the hydrogenated compound, which confirms the classical temperature response for short chains [13]. The fluorinated compound exhibits the sigmoidal response characteristic of longer chains (see dodecyltrimethylammonium and tetradecyltrimethylammonium bromides in Ref. [13]). This result is a direct consequence of the higher hydrophobicity of the fluorinated compound and shows that this contributes to dramatic changes in compressibility.

In the micellar range the increment upon micellization should decrease with temperature, following the dependence on chain length that goes from a very small slope for short chains to a higher slopes for longer chains. The increment in apparent molar volume and compressibility upon micellization can be seen in Table 2 for both perfluorooctanoate from Ref. [15], and octanoate (present result). The increase upon micellization for the apparent molar volume shows the classical behaviour and decreases with rising temperature as a consequence of the dehydration of the ionic head group. Zielinski et al. [13] observed a linear dependence of this parameter with temperature and the slope of such behaviour increased with chain length, indicating that there is some dependence on the hydrophobic part of the micelle. Such dependence can be observed in the different slopes of the present compound and the corresponding fluorinated one. The pronounced slope of the fluorinated compound is an indication that the decrease corresponds to the longer hydrocarbon tail, in the present case being an indication that the hydrophobicity of the fluorinated compound is in effect higher than that of the corresponding hydrogenated compound. This result is in concordance with that reported by Zielinski et al. [13], who found small slopes for short chains, octyltrimethylammonium bromide, and high slopes for long chains, tetradecyltrimethylammonium bromide.

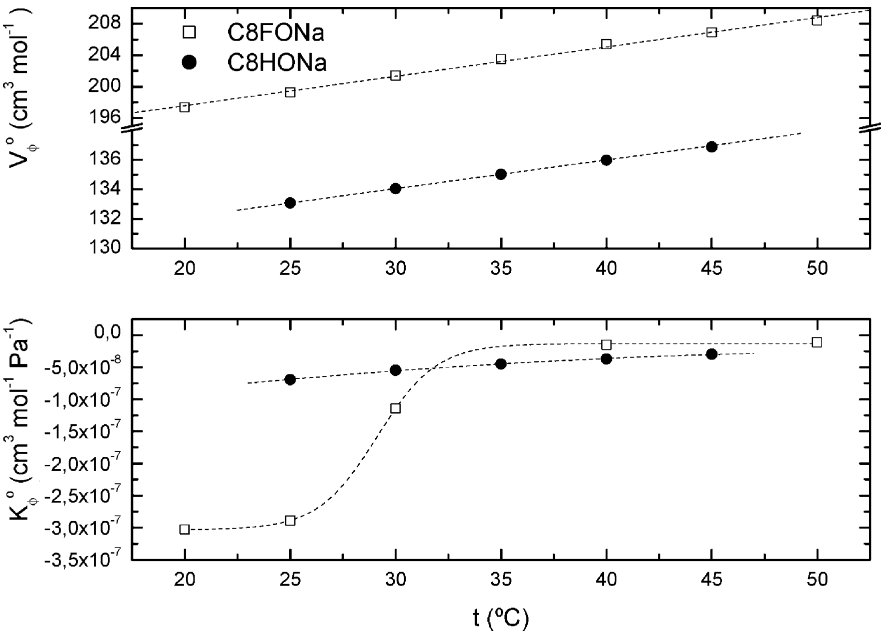

Fig. 5 V_ϕ and K_ϕ^0 for sodium octanoate (*circles*) and sodium perfluorooctanoate (*squares*) as a function of temperature

Similar behaviour can be observed for the apparent molar compressibility when this parameter decreases with temperature and the slope for the fluorinated compound is slightly higher than that for the corresponding hydrogenated one.

Conclusions

The cmc of sodium octanoate at different temperatures has been determined and the results confirm previous data reported by D'Angelo et al. [25] and Gonzalez-Pérez at al. [17] The apparent molar volumes and compressibilities at infinite dilution show linear dependence on temperature. Relevant parameters to study solute–solvent interactions have been determined. In the micellar range, the temperature dependence of the increase in apparent molar quantities has been reported and shows a slight decrease corresponding to the short chain, as reported by Zielinski et al. [13] with octyltrimethylammonium bromide. Finally, the apparent molar quantities reported for the present compound have been compared with the corresponding fluorinated compound and show that the behaviour of the latter corresponds with that expected for a longer hydrogenated compound and confirms that the behaviour of the fluorinated compound is strongly affected by its high hydrophobicity, close to that of a dodecyl-hydrogenated compound.

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