Aggregation and micellization of sodium dodecyl sulfate in the presence of Ce(III) at different temperatures: A conductometric study

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ABSTRACT

Aggregation properties of sodium dodecyl sulfate (SDS) in the presence of cerium(III) chloride, at various temperatures (298.15–323.15 K) have been measured by the electrical conductance technique. The experimental data on aqueous solutions as a function of SDS concentration show the presence of two inflexion points indicating the presence of two distinct interaction mechanisms: the first, occurring at SDS concentrations below the critical micelle concentration of the pure surfactant, which can be explained by the formation of aggregates between dodecyl sulfate (DS^-) and Ce(III), while the second one, at SDS concentrations around the critical micelle concentration (cmc) of the pure surfactant which is due to the SDS micellization. The aggregation between DS^- and Ce(III) was confirmed by static light scattering. The binding ratio of DS^-/Ce(III) changes from 6 to 4, shows a slight dependence on the Ce(III) concentration and is independent of the temperature. The thermodynamic micellization parameters, Gibbs energy, enthalpy and entropy of micellization were calculated on the basis of the experimental data for the aggregation concentration, and the degree of counterion dissociation of the micelles. The SDS micellization is energetically favoured by increasing either the concentration of $CeCl_3$ or the temperature. Such behaviour is clearly dominated by a decrease of the micellization (exothermic) enthalpy. The entropy of micellization approaches zero as the cerium(III) chloride concentration and temperature increase.

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1. Introduction

Because of their unique electronic configuration and very similar chemical properties, lanthanide compounds have a wide variety of applications. Normally, lanthanide ions are in the trivalent (Ln(III)) oxidation state, and present sharp and well-defined luminescence resulting from the shielded $f \rightarrow f$ transitions. Transitions between these orbitals are strictly parity forbidden, and as a consequence have rather low molar absorption coefficients. In contrast, cerium(III) shows rather different behaviour since the lowest energy electronic band corresponds to the allowed $4f \rightarrow 5d$ transition, leading to reasonable molar absorption coefficients and broad absorption or emission bands. This property is important for a number of applications of cerium salts, including the production of luminescent materials [1–4]. In addition, cerium salts have been used as corrosion inhibitors [5,6], chemical precursors of organic [7,8] and inorganic [9] reactions, catalysts [10], and can also be used to induce hydrolysis of DNA [11]. Many of the Ce(III) applications occur in surfactant-containing media, such as the formation of cerium-containing nanoparticles [12] and nanowires in reverse micellar environment. It is known that the presence of surfactants can alter the chemical and physical properties of lanthanides, and that the interaction of lanthanides with surfactants leads to a decrease in the solubility and an increase in the Krafft temperature [13]. In addition, lanthanide ions have a marked effect on surfactant micelle properties [14], mainly resulting from electrostatic interactions between lanthanide ions and oppositely charged surfactant micelles [15]. Further, the fluorescent properties of these trivalent cations can be significantly changed in the presence of both cationic and anionic surfactants [16,17]. Recently [18,19] we have shown that the fluorescence intensity of a number of trivalent rare-earth ions (in particular, europium and terbium) is enhanced in the presence of sub-micellar concentrations of anionic surfactants. This can be justified by an exchange of hydrated water molecules of the cations by the surfactant. We have also reported that such aggregation does not significantly affect the critical micelle concentration of the surfactant.

We extend this to the study of the effect of Ce(III) on the micellization properties of sodium dodecyl sulfate at and below the micellar concentration region, at various temperatures ranging from 298.15 to 323.15 K. Alterations in the solution properties are monitored by variations in the electrical conductivity, which is shown to be a reliable technique for identifying changes in the ionic behaviour of these solutions. Experimental results are discussed on the basis of SDS aggregation and micellization parameters.

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2. Experimental

Cerium(III) chloride heptahydrate (stated purity greater than 98%) and sodium dodecyl sulfate were purchased from Fluka. These reagents were used without further purification. All solutions were prepared using Millipore-Q water.

Solution electrical resistances were measured with a Wayne–Kerr model 4265 Automatic LCR meter operating at 1 kHz. A Shedlovsky-type conductance cell, with a cell constant of approximately 0.0802 cm^{-1} was used in the measurements [20]. Solutions were always studied within 12 h after preparation. In a typical experiment a cerium(III) chloride solution was placed in the conductivity cell; aliquots of the SDS solution were then added in a stepwise manner using a Methrom 765 dosimate micropipette. The conductance of the solution was measured and recorded after each addition when the electrical conductivity reached a stable value, i.e., when the solution electrical resistance does not change more than 0.2% in 1 min. In-house software was used both for programmed surfactant addition and acquisition of electrical resistance data after reaching the equilibrium. The solution specific conductance value presented here, κ, was calculated from the experimental specific conductance and corrected for the experimental specific conductance of the water used under the present conditions. Table 1A (Supplementary material) shows the critical micelle concentrations (cmc) and the degree of counterion dissociation of micelles ($\alpha =$ number of counterions dissociated from the micelle/total number of counterions) of SDS at different temperatures; the experimental values are in good agreement with those reported elsewhere [21,22].

Static light scattering measurements were carried out following the procedure of Mougán and co-workers [23] using a Spex Fluorog 3-22 spectrofluorimeter in 90° configuration with the excitation monochromator set at 400 nm, and the emission scanned between 380 and 420 nm; 0.5 nm excitation and emission slits were used.

pH measurements were carried out with a Radiometer pH meter PHM 240 using an Ingold U457-K7 pH conjugated electrode; pH was measured on fresh solutions, and the electrode was calibrated immediately before each experimental set of solutions using IUPAC-recommended pH 4 and 7 buffers.

3. Results and discussion

3.1. Effect of the presence of Ce(III) on the electrical conductance of SDS

Fig. 1 shows the effect of the presence of Ce(III) chloride on the electrical conductance of aqueous SDS solutions. In the absence of Ce(III), the SDS electrical conductance presents two different linear regimes as a function of surfactant concentration, corresponding to behaviour above and below the pure surfactant critical micelle concentration (cmc).

In the presence of CeCl$_3$ (at concentrations above 0.2 mM) a significant change in the trend of the specific electrical conductance is observed. Although the specific conductance of these solutions has a rather complex interpretation due to the different contributions to the whole conductance, which are dependent on concentration and nature of the species, e.g., unimer-unimer, unimer-micelle, CeCl$_3$-unimer, etc., it is possible to observe three phase transitions between the four different regimes of the system.

At Ce(III) concentrations above 0.2 mM, but within the premicellar region, there is a slight increase in the specific conductance of the solution upon SDS addition. However, above a certain SDS concentration (e.g., line II in Fig. 1), there is a change in the dependence of κ with the SDS concentration, with this showing a relevant decrease in the slope, which becomes more pronounced and covers a wider surfactant concentration range as
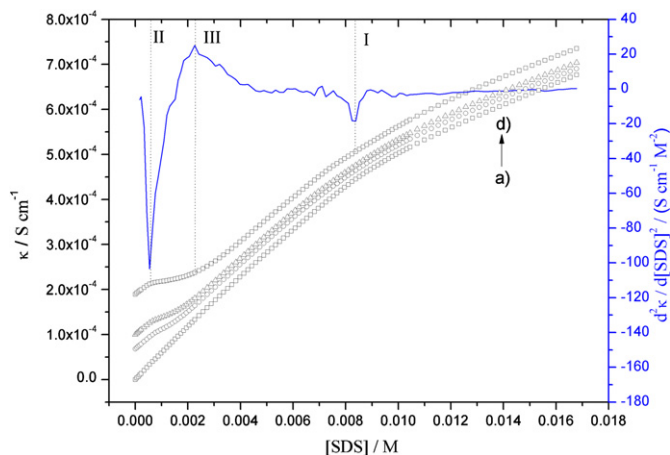

Fig. 1. Effect of CeCl$_3$ on the specific conductance of CeCl$_3$/SDS mixtures, at 298.15 K. (a) [CeCl$_3$] = 0 M, (b) [CeCl$_3$] = 0.2 mM, (c) [CeCl$_3$] = 0.3 mM, (d) [CeCl$_3$] = 0.6 mM. Vertical lines show, as examples, breakpoints of $\kappa = f$([SDS]) corresponding to I: cmc or cmc^{ap}, II: cac$_1$, and III: mic (see text for meaning of abbreviations and further details). The second derivative used in calculation of cmc^{ap}, cac$_1$, and mic of SDS/Ce(III) 0.6 mM system is shown.
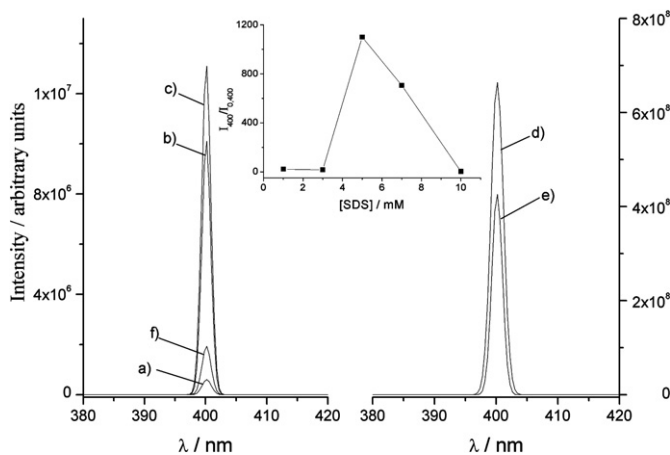

Fig. 2. Static light scattering spectra of 1 mM Ce(III) solutions at different SDS concentrations, at 298.15 K. (a) [SDS] = 0 M, (b) [SDS] = 1 mM, (c) [SDS] = 3 mM, (d) [SDS] = 5 mM, (e) [SDS] = 7 mM, and (f) [SDS] = 10 mM. Inset figure: maximum normalized light scattering intensity of Ce(III)/SDS solutions.

Ce(III) concentration increases. This strongly suggests that there is an alteration of the structural properties of the ionic conducting species, i.e., that addition of SDS to Ce(III) solution does not result in any apparent increase of ionic species in the whole solution. A possible justification is that upon the addition of SDS to Ce(III) salt, cerium(III) dodecyl sulfate aggregates are formed and, consequently, the ionic mobility, as well as the ionic conductance, will not increase. It is known that interactions between Ce(III) and SDS micelles are mainly electrostatic and weak, with cerium(III) lying close to the sulfate headgroups [24]; consequently, we should expect that such interactions also occurs between cerium(III) and dodecylsulfate ions as unimers. If such a hypothesis is correct, we should expect that after all the Ce(III) has been consumed in aggregate formation, further addition of SDS will result in an increase of specific conductance, in a similar way to that occurring in the absence of the cerium(III) electrolyte. This is observed above a particular SDS concentration, which we call the maximum interaction concentration (mic) (line III in Fig. 1). This explanation is supported by static light scattering studies. In Fig. 2 it is possible to observe that the addition of SDS to 1 mM Ce(III) solution results in a maximum scattering at ratio [SDS]/[Ce(III)] = 5. This value is fairly similar to that obtained for the mic, for the

Table 1
Effect of $CeCl_3$ concentration and temperature on the aggregation and micellization properties of sodium dodecyl sulfate

[Ce(III)] (mM)	0.2	0.3	0.4	0.6	0.8	1
			$T = 298.15$ K			
cac_1 (mM)	–	0.59 (0.05)[a]	0.54 (0.04)	0.51 (0.03)	0.51 (0.02)	0.49 (0.05)
mic (mM)	–	1.68 (0.05)	1.91 (0.06)	2.36 (0.04)	2.75 (0.09)	3.19 (0.11)
cmc^{ap} (mM)	–	8.80 (0.18)	8.15 (0.61)	8.32 (0.56)	8.05 (0.16)	8.16 (0.77)
			$T = 303.15$ K			
cac_1 (mM)	0.62 (0.02)	0.55 (0.02)	0.53 (0.03)	0.53 (0.04)	0.56 (0.03)	0.54 (0.03)
mic (mM)	1.28 (0.07)	1.68 (0.06)	1.76 (0.04)	2.55 (0.14)	2.88 (0.10)	3.41 (0.11)
cmc^{ap} (mM)	8.96 (0.25)	9.20 (0.22)	8.97 (0.54)	9.15 (0.47)	8.89 (0.14)	9.02 (0.61)
			$T = 313.15$ K			
cac_1 (mM)	1.01 (0.20)	0.73 (0.03)	0.62 (0.04)	0.61 (0.01)	0.61 (0.01)	0.56 (0.02)
mic (mM)	1.18 (0.07)	1.73 (0.06)	1.96 (0.05)	2.51 (0.06)	3.04 (0.04)	3.63 (0.03)
cmc^{ap} (mM)	9.52 (0.58)	8.89 (0.65)	9.85 (0.30)	9.94 (0.11)	9.96 (0.62)	10.15 (0.87)
			$T = 323.15$ K			
cac_1 (mM)	1.06 (0.08)	0.86 (0.03)	0.81 (0.02)	0.74 (0.02)	0.71 (0.02)	0.66 (0.15)
mic (mM)	1.12 (0.15)	1.94 (0.08)	2.02 (0.06)	2.55 (0.06)	3.49 (0.07)	4.31 (0.34)
cmc^{ap} (mM)	10.10 (0.51)	10.59 (0.41)	10.94 (0.58)	11.17 (0.85)	11.55 (0.47)	12.15 (0.41)

[a] Values inside brackets correspond to standard deviations.

same system, by electrical conductivity measurements (see Table 1).

3.2. Aggregation between SDS and $CeCl_3$

As described in the previous section, electrical conductivity measurements can be used to detect alterations in the structure of mixed $CeCl_3$/SDS solution properties. However, at 298.15 K, interactions between Ce(III) and SDS can only be observed at Ce(III) concentrations above 0.1 mM.

In order to quantify the different parameters, such as the critical aggregation concentration between SDS and $CeCl_3$ (cac_1) and the maximum interaction concentration (mic) between dodecyl sulfate and Ce(III) ions, we have used the following method: cac_1 and mic were calculated by fitting the raw data of specific conductance as a function of surfactant concentration to a simple nonlinear function obtained by direct integration of a Boltzmann type sigmoidal function [25]. Table 2A (Supplementary material) also shows the binding ratio (β), defined as equal to $\beta = (\text{mic})/[\text{Ce(III)}]$, which gives us information about the association stoichiometry between Ce(III) and dodecyl sulfate ions in the aggregates.

The onset of Ce(III)/SDS aggregation shows a dependence both on Ce(III) concentration and on temperature; it decreases upon either increasing Ce(III) concentration or decreasing temperature. These values (0.49–0.59 mM, at 298.15 K) occur in the same concentration interval of those found to other lanthanide trivalent cations [18,19,26]. With the exception of lowest Ce(III) concentration systems, it is possible to observe that cac_1 decreases as cerium(III) concentration increases and as temperature decreases. This behaviour can easily be explained by the fact that electrostatic interactions between Ce(III) and dodecyl sulfate ions are the main factors responsible for formation of aggregates. This is also in agreement with the fact that mic values increase upon increasing Ce(III) concentrations, which supports the hypothesis that Ce(III) can be considered as a limiting reactant for the aggregate formation.

Comparing these results with those found for the Eu(III) and Tb(III)/SDS systems [18,19], it is possible to observe that in the present case the stoichiometry of aggregate formation (a) only changes slightly with Ce(III) concentration (between 6 and 4, compared with $4 < \beta(\text{Eu/SDS}) < 20$ and $3 < \beta(\text{Tb/SDS}) < 6$), and (b) does not change with temperature. Two different aspects of the nature of the three lanthanide ions may be considered to explain these differences: Ce(III) has a lower charge density than the other trivalent lanthanide ions [27], and, secondly, hydrolysis is less sig-

nificant with $CeCl_3$ aqueous solutions than with the other trivalent lanthanides [28]. To check the importance of this second effect in the presence of SDS, pH measurements were carried out at different [SDS]/[CeCl$_3$] ratios, and showed differences of only 4.0% in the pH range 6.73 to 7.01 at concentration ratios of 0 and 28, respectively. These results show that the contribution of hydrolysis products to the conductivity and competitive processes can be completely neglected.

3.3. Micellization of SDS in the presence of Ce(III)/SDS aggregates

The effect of Ce(III) on the SDS structure within the pre-micellar region has an important effect on the surfactant micellization. Table 1 presents apparent critical micelle concentration—the critical micelle concentration in the presence of salt/aggregates, cmc^{ap} (line I—Fig. 1). The cmc^{ap} was calculated using the same method described above to calculate the cac_1. The degree of micellar counterion dissociation (α), for aqueous $CeCl_3$/SDS solutions, was calculated from the ratio between the slopes of the postmicellar and premicellar regions, in the plots of $\kappa = f(c)$ [20].

The presence of an electrolyte in an ionic surfactant solution will affect the polarity of the ionic micellar surface by changing the electrostatic repulsions between the charged micelles and the counterions. An assessment of this effect can be made by analysing the degree of counterion dissociation (α). From the data shown in Table 2A (Supplementary material), α is independent of Ce(III) concentration and is only slightly dependent on the temperature, following an increase similar to that found with aqueous SDS solutions. Calculating an average degree of counterion dissociation of micelles (α_{av}) for each temperature we obtain α_{av} equal to 0.39 ($\pm$0.02), 0.38 ($\pm$0.02), 0.39 ($\pm$0.01), and 0.40 ($\pm$0.01) for 25, 30, 40, and 50 °C, respectively. However, comparing the degree of counterion dissociation of SDS micelles in the presence and absence of $CeCl_3$, α decreases ca. 7 to 3%, for 298.15 and 323.15 K respectively, in the presence of the Ce(III) salt. If we take data for the effect of NaCl (having the same anion as that used in the $CeCl_3$/SDS solutions) as a reference, and consider that the concentration of NaCl present in these systems can reach a maximum value of 1 mM, we would expect an increase in the α values [29,30]. The fact that the opposite occurs can only be justified by the presence of Ce(III)/DS$^-$ aggregates which contribute to an increase in the ionic strength, in particular as a consequence of the high valence of cerium [29], contributing to the binding of a greater number of counter ions to the micelle, and consequently leading to a decrease in the degree of counterion dissociation [31].

Looking at the effect of CeCl$_3$ concentration and temperature on cmc^{ap} (Table 1), we may conclude that the values are, in general, higher than the critical micelle concentration of SDS (Table 1A in Supplementary material): this cannot be justified on thermodynamic grounds. However, if we take into account the fact that the surfactant concentration necessary for complete association with all the cerium(III) chloride present in solution is given by the mic, we may use the following equation: cac$_2$ = cmc^{ap}-mic to obtain the accurate critical aggregation concentration of SDS (cac$_2$). The values of cac$_2$ are lower than the cmc of SDS, showing that the onset of SDS micellization responds to added electrolyte, which is in agreement with studies by Griffiths et al. [32]. A more detailed analysis of cac$_2$ as a function of [CeCl$_3$] shows that the SDS micellization depends on both the initial Ce(III) concentration and the temperature. Plotting cac$_2$ = f([CeCl$_3$]) and using a linear least-squares procedure to fit the experimental data, the following values were obtained for the slopes: -2.83 (± 0.45), -2.72 (± 0.10), -2.33 (± 0.05), and -1.41 (± 0.26) for 298, 303, 313, and 323 K, respectively. These values show that the dependence of cac$_2$ on the CeCl$_3$ concentration decreases upon increasing the temperature. At the same time, by comparing the SDS micellization in the absence and presence of CeCl$_3$, and taking the 1 mM CeCl$_3$ system as example, it is possible to calculate a decrease in Gibbs energy of micellization in the presence of the electrolyte of -2.0 kJ/mol at 298 K whilst at 323 K this decrease is only -1.0 kJ/mol; that is, increasing the temperature the micellization of SDS approaches that occurring in a salt-free solution. This shows that, increasing the temperature, the SDS micellization becomes less dependent on the presence of CeCl$_3$.

The standard Gibbs free energy and enthalpy of micellization (ΔG_m^0 and ΔH_m^0) of SDS in the absence and in the presence of the electrolyte were calculated using [22,33]

$$\Delta G_m^0 = (2 - \alpha)RT \ln X \qquad (1)$$

and

$$\Delta H_m^0 = -RT^2 \left[(2 - \alpha)\frac{d \ln X}{d(T)} + \ln X \frac{d(1 - \alpha)}{dT} \right], \qquad (2)$$

where X is the critical micelle concentration (cac$_2$ in this paper) in mole fraction units, and the other symbols are as previously indicated.

Since the dependence of $\ln X$ as a function of T is linear, we have used the straight line equation

$$\ln X = a + bT \qquad (3)$$

to fit these parameters, where a and b are constants. On the other hand $(1 - \alpha)$ is independent of the temperature, for each CeCl$_3$ initial concentration (see Table 2A in Supplementary material). After computation of the fitting parameters, Eq. (2) can be re-written as

$$\Delta H_m^0 = -RT^2 \left[(2 - \alpha)b \right]. \qquad (4)$$

Finally, the entropy of micellization was evaluated from

$$\Delta S_m^0 = \frac{\Delta H_m^0 - \Delta G_m^0}{T}. \qquad (5)$$

Equations (1)–(5) were solved using data shown in Table 2A in Supplementary material and the corresponding micellization thermodynamic data are compiled in Table 3A in Supplementary material.

Analysis of enthalpy and entropy contributions for the micellization processes sheds light on the balance of forces involved in micelle formation. In the present case this assessment can be done in two different ways: checking the effect of temperature for a constant Ce(III) concentration or how thermodynamic parameters change as a function of CeCl$_3$ concentration at a certain given temperature.

In the absence of CeCl$_3$, SDS micellization is entropy-driven, whilst with an increase of temperature the SDS micellization clearly becomes enthalpy-driven, which is in good agreement with previous literature reports [21,22].

In the presence of CeCl$_3$ with the SDS solution, self-assembly becomes more exothermic and the Gibbs energy of micellization is slightly more negative with both the salt and the temperature. A direct reading of data shows that by increasing [CeCl$_3$] concentration, SDS micellization is mainly controlled by enthalpy changes. For different CeCl$_3$-containing solutions, the enthalpy and entropy of micellization decrease with temperature. This behaviour can be justified as follows: at low temperatures, the destruction of the hydrophobic hydration is responsible for the observed ΔS_m^0 increase. As the temperature increases, the structure and size of water molecules aggregates decrease and, consequently, ΔH_m^0 becomes more exothermic, and this effect is predominant [34]. What is relevant in CeCl$_3$-containing solutions is that, for example, at CeCl$_3$ 1 mM, the change in the entropy approaches zero: this can be justified by the predominant effect on the hydrophobic hydration being due to Ce(III)/dodecyl sulfate aggregates.

If we look at the data in a different way, by discussing the effect of CeCl$_3$ addition on the micellization properties of SDS at one temperature, it is also possible to observe a similar effect: for each temperature, ΔH_m^0 and ΔS_m^0 decrease by increasing CeCl$_3$ concentration in the solution (see Fig. 3A in Supplementary material). These data also show that the salt is not dissociated in solution, because if it was we would expect an increase in ΔS_m^0 with an increase of salt in solution [22]. Another possible and concomitant explanation is that in the presence of DS$^-$/Ce(III) aggregates (or even just CeCl$_3$), we may anticipate an increase in the aggregation number of the SDS micelles [28,34]. Consequently, this closer packing of surfactant molecules in the micelle results in a favourable enthalpy change, and the counterions present in solutions will act to decrease the repulsions between the ionic head-groups existing in the micelle [35].

4. Conclusions

Electrical conductometry is a useful technique for the determination of aggregation and micellization properties of sodium dodecyl sulfate in solutions containing cerium(III) chloride. The effects of CeCl$_3$ concentration and of temperature have been analysed. At [Ce(III)] greater than 0.2 mM it is possible to observe an alteration in the profile of $\kappa = f$[Ce(III)] which can be explained by the formation of large ionic species, suggested to be cerium(III) dodecyl sulfate aggregates, which have lower mobilities than that found from a simple addition of unimers and salt ions. The critical aggregation concentration for cerium(III) dodecyl sulfate formation (at concentrations around 0.5–1.0 mM) depends on both cerium concentration and temperature, as has also been confirmed by static light scattering.

The SDS:CeCl$_3$ binding ratio shows a slight dependence on the initial CeCl$_3$ concentration, changing from 6 to 4 when the Ce(III) concentration increases. Such a small alteration, in comparison with other lanthanide ions, can be justified by the low charge density of cerium(III) and consequently electrostatic interactions do not favour alteration in the shape of aggregate when salt concentration increases.

The Gibbs energy of micellization of SDS, in the presence of cerium chloride, decreases by increasing salt concentration and temperature. It is suggested that such a decrease results from the enthalpic contribution to the micellization; that is, in the presence of CeCl$_3$ and at different temperatures (in particular at temperatures greater than 303.15 K) $|\Delta H_m^0| > |T \Delta S_m^0|$ suggesting that DS$^-$/Ce(III) aggregates entropically stabilise the micellization process by promoting hydrophobic stabilization of water molecules.

These results provide the expectation that such cerium(III)/dodecyl sulfate aggregates may find applications in various fields, including catalysis and photoinduced polymerization.

Acknowledgments

Supplementary material

The online version of this article contains additional supplementary material.

Please visit DOI: 10.1016/j.jcis.2008.03.046.

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