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Heat capacities and volumes of suspensions in the presence of surfactants

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Abstract

Density and heat capacity measurements of water–surfactant–silica ternary systems were carried out as functions of the silica and the surfactant concentrations at 298 K. From these properties, the apparent molar volume and heat capacity of the surfactant in a given water + silica mixture were evaluated. As well, the apparent specific volume and heat capacity of silica, at a given concentration, in the water + surfactant mixtures as functions of the surfactant concentration were calculated. Some surfactants (decyltrimethylammonium bromide, sodium octanoate, sodium decanoate, sodium dodecanoate, sodium decylsulfate, sodium perfluorooctanoate and *N*,*N*-dimethyldodecylamine-*N*-oxide) were chosen to study the effect of the head-group and the hydrophobicity. As a general feature, the hydrophobicity of the surfactant does not essentially play a role on the silica–surfactant interactions whereas the surfactant head-group does. Finally, the apparent specific properties of silica are very sensitive in detecting the destabilization of suspensions whereas the apparent molar properties of the surfactant are not.

Keywords: Surfactants; Colloidal silica; Volumes; Heat capacities

1. Introduction

The adsorption of surfactants onto solid surfaces has been extensively studied by means of several experimental techniques [1–5] since it plays a determinant role in many industrial processes such as detergency, wetting, flotation, adhesion and dispersion stability. This process is a very complex phenomenon influenced by a large number of parameters such as pH, electrolyte concentration, temperature, surfactant concentration, nature of both the solid and the surfactant, etc. Various solid surfaces have been studied; for instance, for a long time several kinds of negatively charged silica have been investigated as surface models for the adsorption of cationic surfactants [6–8]. It is accepted that the adsorption of surfactants from water to the hydrophilic solid particles depends on the surfactant content. At low concentration, individual molecules adsorb flat on the polar sites and at higher concentration aggregates are forming. Besides the mono- and bi-layers, ionic surfactants form aggregates called hemimicelles [9,10] and admicelles [10,11] whereas nonionic surfactants [12,13] form micelles-like aggregates.

The thermodynamic studies have mainly addressed the determination of the isotherms of adsorption [14–18]. The isosteric method [16] and microcalorimetry [14–16,18] have been used to evaluate the adsorption enthalpy. Apparent molar volumes of sodium dodecylsulfate on polystyrene were reported [19]. Flow techniques were also used to measure the density and the heat capacity of suspensions in the presence of various additives [20,21]. It turned out that the thermodynamic properties are very sensitive to the solid particle–surfactant interactions and also provide straightforward information on the state of the dispersion (whether it is flocculated or stable).

To give a contribution to the thermodynamics of suspensions, densities and heat capacities of colloidal silica in the presence of various surfactants were determined. The apparent molar volume and heat capacity of the surfactant in a given water + silica mixture were determined as functions of the surfactant concentration. Also, the apparent specific volume and heat capacity of silica, at a given concentration, in the water + surfactant mixture as functions of the surfactant concentration were calculated. In some cases, the silica concentration effect was analyzed. To study the effect

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of the head-group and the hydrophobicity of the surfactant, the following compounds were chosen: decyltrimethylammonium bromide, sodium octanoate (NaOct), sodium decanoate (NaDec), sodium dodecanoate (NaL), sodium decylsulfate (NaDeS), sodium perfluorooctanoate (NaPFO) and *N*,*N*-dimethyldodecylamine-*N*-oxide (DDAO).

2. Experimental

2.1. Materials

Ludox HS40 is a Dupont silica product provided by Aldrich. It is an aqueous dispersion 40% (w/w) having the pH = 9. The Karl Fisher method was used to determine the exact concentration of the aqueous suspension. Decyltrimethylammonium bromide (Kodak) was crystallized from an ethanol/ethyl acetate mixture (1:7 w/w) and dried in a vacuum oven for 4 days at 313 K. Sodium octanoate, sodium decanoate, sodium dodecanoate (Sigma products) and sodium decylsulfate (Kodak) were used as received. Perfluorooctanoic acid (Fluka) was crystallized from carbon tetrachloride and dried at 298 K; its sodium salt was obtained by neutralization with an aqueous sodium hydroxide solution. The product precipitated after cooling at 278 K and was recovered by filtration. *N*,*N*-dimethyldodecylamine-*N*-oxide was a 30% (w/w) aqueous mixture (Fluka). The original solution (ca. 0.8 mol kg^{-1}) was diluted and the correct molality of the stock solution was determined by means of density measurements as described elsewhere [22].

The standard partial molar volumes of the surfactants in water, evaluated from density measurements, agree with those reported in [22–25] confirming that the products are pure for our purpose.

The pH values of the water–surfactant–silica mixtures were between 8.5 and 9.0 with the exception that the dispersions containing sodium decylsulfate at concentrations larger than 0.1 mol kg^{-1} were pH = 7.

All solutions were prepared by mass.

2.2. Measurements of the suspensions

Special care was taken in determining the density and the heat capacity of the suspensions that were stirred by means of a magnetic apparatus until they were introduced into the equipment. The suspensions were prepared in poly(ethylene) bottles to avoid silica adsorption.

2.3. Equipment

2.3.1. Density

The solutions densities were measured at 298 K by using a vibrating tube flow densimeter (Model 03D, Sodev Inc.) sensitive to 3 ppm. The temperature was maintained constant within 0.001 K by using a closed loop temperature controller (Model CT-L, Sodev Inc.). The calibration of the densimeter was made with water ($d = 997.047\,\mathrm{kg\,m^{-3}}$) [26] and the aqueous sucrose solutions of known densities [27].

2.3.2. Heat capacity measurements

The relative differences in the heat capacities per unit volume ($\Delta\sigma/\sigma_0$) were determined with a Picker flow microcalorimeter [28] (Setaram) at 298.000 ± 0.001 K. Using a flow rate of about $1 \times 10^{-8}\,\mathrm{m^3\,s^{-1}}$ and a basic power of 19.7 mW, the temperature increment was ca. 0.5 K. The reproducibility of the specific heat capacity measurement is $1 \times 10^{-4}\,\mathrm{J\,K^{-1}\,g^{-1}}$.

The specific heat capacity (c_p) of a solution of density d is related to the excess heat capacity per unit volume ($\Delta\sigma/\sigma_0$) through the equation

$$c_p = c_{p_0}\left\{1 + \frac{\Delta\sigma}{\sigma_0}\right\}\frac{d_0}{d} \tag{1}$$

where c_{p_0} and d_0 correspond to the specific heat capacity and density of the reference solvent, which is water in our case. The c_{p_0} value used [29] is $4.1792\,\mathrm{J\,K^{-1}\,g^{-1}}$.

2.3.3. Conductivity

The specific conductivity measurements were performed at 298.0 ± 0.1 K (digital conductimeter analytical control 120) to determine the critical micellar concentration (cmc) of the surfactant and the degree of ionization of the micelles (β) in water and in water + silica mixture. The former corresponds to the intersection point of the straight lines (in the pre- and post-micellar regions) of the plot of specific conductivity versus surfactant concentration whereas β is given by the ratio of the slopes of these straight lines [30]. The obtained values, collected in Table 1, are not affected by the presence of silica.

2.3.4. Apparent properties calculation

The apparent specific volume ($V_{\mathrm{sp},\phi}$) and heat capacity ($C_{\mathrm{sp},\phi}$) of silica in a given solvent was calculated as

$$V_{\mathrm{sp},\phi} = \frac{1}{d} - \frac{10^3(d - d_0)}{wdd_0} \tag{2}$$

$$C_{\mathrm{sp},\phi} = c_p + \frac{10^3(c_p - c_{p_0})}{w} \tag{3}$$

where d and c_p represent the density and the specific heat capacity of the silica suspension, respectively, whereas d_0

Table 1
Critical micellar concentration and degree of ionization of the micelles for some surfactants in water and in water + silica 1% (w/w) at 298 K

	$\mathrm{cmc_w}$ (mol kg^{-1})	β_w	$\mathrm{cmc_{w+Si}}$ (mol kg^{-1})	$\beta_\mathrm{w+Si}$
NaDeS	0.032	0.53	0.033	0.54
NaDec	0.103	0.63	0.098	0.59
NaL	0.025	0.38	0.026	0.41
NaPFO	0.03[a]	0.59[b]		

[a] From Ref. [24].

[b] From Ref. [38].

and c_{p_0} are the corresponding properties of the solvent; w is the concentration expressed as g of SiO_2/kg of solvent.

For the water–silica binary system, the solvent is water whereas for the water–silica–surfactant ternary system the solvent is the water–surfactant mixture whose density [22–25] and specific heat capacity [23–25] values were obtained from the apparent molar volumes and heat capacities of the surfactant in water available at several concentrations.

The $V_{\mathrm{sp},\phi}$ values of silica in water for the mixtures at $w = 4.02$ and 10.10 are 420×10^{-3} and $424 \times 10^{-3}\,\mathrm{m^3\,g^{-1}}$, respectively. They agree with the literature values [20]. The $C_{\mathrm{sp},\phi}$ value of silica at $w = 10.10$ is $0.766\,\mathrm{J\,K^{-1}\,g^{-1}}$.

The apparent molar volume ($V_{\phi,\mathrm{S}}$) and heat capacity ($C_{\phi,\mathrm{S}}$) of the surfactant in a given water + silica mixture was calculated as

$$V_{\phi,\mathrm{S}} = \frac{M}{d} - \frac{10^3(d - d'_0)}{m_\mathrm{S}^{\mathrm{w+Si}} d d'_0} \tag{4}$$

$$C_{\phi,\mathrm{S}} = Mc_p + \frac{10^3(c_p - c'_{p0})}{m_\mathrm{S}^{\mathrm{w+Si}}} \tag{5}$$

where M is the molecular weight of the surfactant, d assumes the same meaning as above and d'_0 is the density of the water + silica mixture; $m_\mathrm{S}^{\mathrm{w+Si}}$ stands for the molality of the surfactant in the water + silica mixture calculated as

$$m_\mathrm{S}^{\mathrm{w+Si}} = \frac{10^3 m_\mathrm{S}^{\mathrm{w}}}{10^3 + w'} \tag{6}$$

where $m_\mathrm{S}^{\mathrm{w}}$ is the molality of the surfactant in water and w' is the silica concentration expressed as g of SiO_2/kg of water. At a given w', d'_0 and c'_{p0} were calculated by introducing the previously obtained $V_{\mathrm{sp},\phi}$ and $C_{\mathrm{sp},\phi}$ values in Eqs. (2) and (3), respectively.

Heat capacity measurements of the water + DDAO binary system as functions of the surfactant concentrations were carried out as they are not available in the literature. Because of the low cmc value [31] ($2 \times 10^{-3}\,\mathrm{mol\,kg^{-1}}$), the pre-micellar region was not studied and, therefore, the standard partial molar heat capacity ($1172\,\mathrm{J\,K^{-1}\,mol^{-1}}$) was calculated on the basis of the additivity rule by using the data of the smaller homologues [31].

3. Results and discussion

3.1. Time effect

Silica suspensions are thermodynamically unstable systems and, then, time could be a variable to take into account in studying them. As Fig. 1 shows, the apparent molar volume of *N*,*N*-dimethyldodecylamine-*N*-oxide $0.7\,\mathrm{mol\,kg^{-1}}$ in the presence of SiO_2 1% (w/w) is independent of time within 5 h and it decreases by ca. 1% within 40 h, after that it is constant for the time of our investigation (up to 718 h). In the presence of silica 1% (w/w), $V_{\phi,\mathrm{S}}$ of sodium decylsulfate $0.7\,\mathrm{mol\,kg^{-1}}$ remains constant within 5 h after the suspension preparation and, then, it decreases by ca. 6%. In the case of sodium decanoate 0.69 and $0.88\,\mathrm{mol\,kg^{-1}}$ in silica 1% (w/w), $V_{\phi,\mathrm{S}}$ is constant for 20–30 h after that it decreases. $V_{\phi,\mathrm{S}}$ of sodium octanoate $0.37\,\mathrm{mol\,kg^{-1}}$ in the presence of silica 1% (w/w) does not change during the time of investigation (30 h).

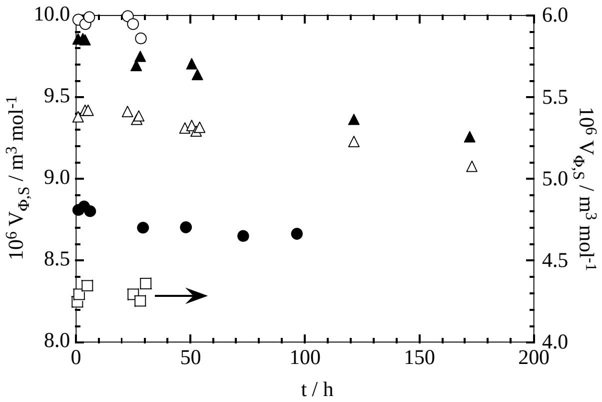

Fig. 1. Dependence on time of the apparent molar volume of surfactants in the water + silica mixture 1% (w/w) corrected for the standard value in water: (□) sodium octanoate 0.3676*m*; (●) *N*,*N*-dimethyldodecylamine-*N*-oxide 0.6932*m*; (△) sodium decanoate 0.6907*m*; (○) sodium decanoate 0.8814*m*; (▲) sodium decylsulfate 0.6970*m*. The error bars have size smaller than symbols.

Regardless of the surfactant nature and concentration, the volumetric property is constant within a few hours after the suspension preparation. Therefore, based on these evidences and by considering also that the study as a function of time is quite time consuming, we decided to perform the measurements on the suspensions 1 h after their preparation. This procedure assumes that the systems under study are kinetically stable. However, any destabilization of the suspensions can be evidenced by the experimental properties according to the literature findings [20].

3.2. Surfactants of different tail in silica suspensions

The curves of $V_{\phi,\mathrm{S}}$ and $C_{\phi,\mathrm{S}}$ versus $m_\mathrm{S}^{\mathrm{w+Si}}$, superimposed to those in water [24] (Fig. 2) and the unchanged cmc value (Table 1), do not indicate interactions between silica and sodium dodecanoate in both the monomeric and the micellized states. A similar behavior is exhibited by NaDec. The $V_{\phi,\mathrm{S}}$ and $C_{\phi,\mathrm{S}}$ versus $m_\mathrm{S}^{\mathrm{w+Si}}$ trends at the silica compositions studied, illustrated in Fig. 3, are coincident with those in water [25] but above 0.4 and $1.0\,\mathrm{mol\,kg^{-1}}$ the $C_{\phi,\mathrm{S}}$ and the $V_{\phi,\mathrm{S}}$ points, respectively, begin to diverge. Such a deviation may be ascribed to a change of the NaDec micellar structures. On the other hand, the water–sodium decanoate phase diagram [32] does not rule it out because it shows that a normal micellar solution phase is present in the range of the surfactant concentration analyzed by us. The $V_{\phi,\mathrm{S}}$ versus $m_\mathrm{S}^{\mathrm{w+Si}}$ curve for NaOct is moved slightly towards larger

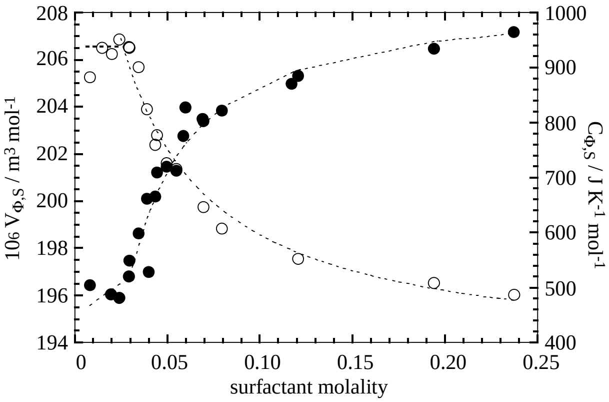

Fig. 2. Apparent molar volume (●) and heat capacity (○) of sodium dodecanoate in the water + silica mixture 1% (w/w) as functions of the surfactant concentration. Lines refer to the trends in water. The error bars have size smaller than symbols.

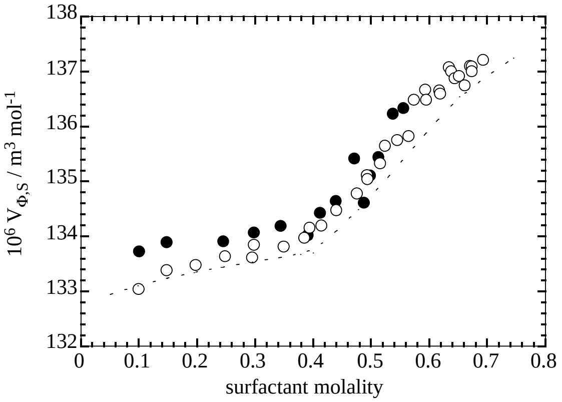

Fig. 4. Apparent molar volumes of sodium octanoate in water (----) and in water + silica mixtures 0.4% (w/w) (○) and 1% (w/w) (●) as functions of the surfactant concentration. The error bars have size smaller than symbols.

values with respect to that in water [25] upon the increase of the silica content (Fig. 4).

Sodium perfluorooctanoate and NaL exhibit comparable characteristics according to the findings [33,34] that the hydrophobicity of the perfluoromethylene group is 1.5 times that of the methylene group. The $V_{\phi,\mathrm{S}}$ data for NaPFO (Fig. 5) are not so well correlated as those for NaL. This behavior is not surprising. In fact, density values of perfluoroalcohols in water and in aqueous surfactant solutions exhibited a dependence on time, being stronger with smaller surfactant concentration [24]. Also, $V_{\phi,\mathrm{S}}$ of NaPFO in freshly prepared water + NaOct mixtures were reproducible, whereas those of solutions prepared a few days before the measurements were scattered [35].

Silica particles are hydrophilic in nature and at the pH of the experiments they are negatively charged. Therefore, hydrophobic forces between the surfactant alkyl chain and the particle surface are ruled out. Since the expected forces are electrostatic in nature, the only species that may interact with the particles are the counterions, which are identical for all the systems investigated.

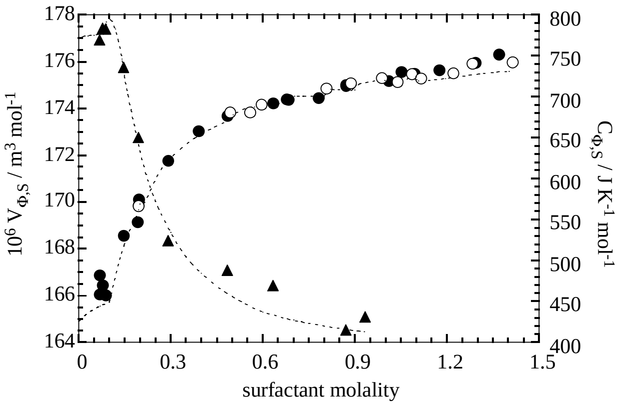

Fig. 3. Apparent molar volume (circles) and heat capacity (triangles) of sodium decanoate in the water + silica mixtures 0.4% (w/w) (open symbols) and 1% (w/w) (filled symbols) as functions of the surfactant concentration. Lines refer to the trends in water. The error bars have size smaller than symbols.

The $V_{\phi,\mathrm{S}}$ versus surfactant concentration curve may reflect not only the interactions between silica and surfactant but also those between the surfactant molecules. In addition, if the silica concentration tends to zero, $V_{\phi,\mathrm{S}}$ tends to the value in water. On the contrary, it is demonstrated that solute–solvent interactions are well detected by the standard (infinite dilution) partial property of the solute in a given solvent. In our case, we can calculate the apparent specific volume ($V_{\mathrm{sp},\phi}$) and heat capacity ($C_{\mathrm{sp},\phi}$) of silica, at a fixed composition, as functions of the surfactant concentration. Since, the silica content is low, the apparent specific property may be considered approaching the standard state and it is expected to be sensitive to the silica–solvent interactions in the suspensions. Thus, the apparent specific properties of silica may be more effective in indicating or detecting interactions present in the systems compared to the apparent molar properties of the surfactant.

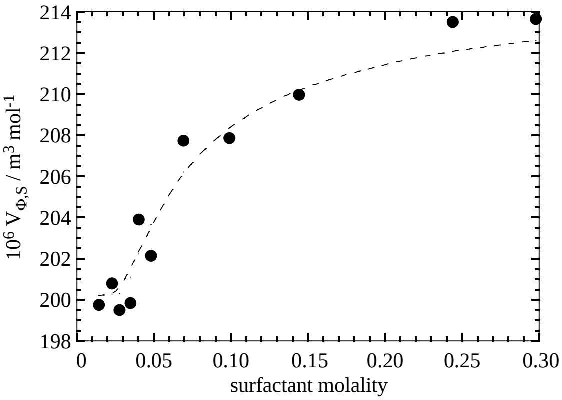

Fig. 5. Apparent molar volumes of sodium perfluorooctanoate in water (----) and in water + silica mixture 1% (w/w) (●) as functions of the surfactant concentration. The error bars have size smaller than symbols.

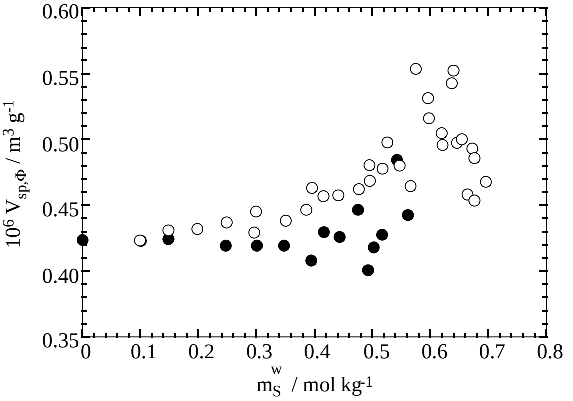

Fig. 6. Apparent specific volumes of silica 0.4% (w/w) (○) and 1% (w/w) (●) in aqueous solutions of sodium octanoate as functions of the surfactant concentration. The error bars have size smaller than symbols.

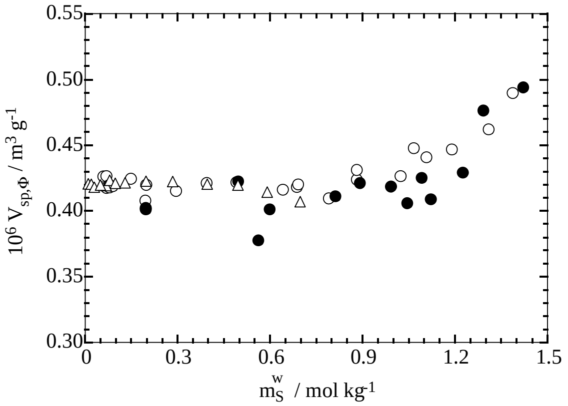

Fig. 8. Apparent specific volumes of silica 0.4% (w/w) (filled symbols) and 1% w/w (open symbols) in aqueous solution of sodium decanoate (circles) and sodium decylsulfate (triangles) as functions of the surfactant concentration. The error bars have size smaller than symbols.

The trend of $V_{sp,\phi}$ of silica at 0.4 and 1% (w/w) as a function of NaOct concentration exhibits a slight linear dependence on the surfactant concentration up to ca. 0.4 mol kg^{-1}, thereafter, the experimental points become scattered (Fig. 6). According to the cmc value of NaOct (0.45 mol kg^{-1}) [25], the $V_{sp,\phi}$ on m_S^w trend in the pre-micellar region confirms that no interactions between silica and dispersed surfactant are present. The scattering of the $V_{sp,\phi}$ points cannot be ascribed to experimental uncertainties. As shown earlier, $V_{\phi,S}$ of NaOct (0.37 mol kg^{-1}) in 1% (w/w) silica is independent of time according to the kinetic stability of the suspension. To verify whether the scattered points for $m_S^w >$ cmc are indications of the destabilization of the suspension, $V_{sp,\phi}$ of 0.4% (w/w) silica in two aqueous NaOct solutions, having close concentration values, were determined over several hours. $V_{sp,\phi}$ changes with time with a slope that is specific of the mixture (Fig. 7) according to the flocculation process which depends on the external variables (preparation of the mixtures, velocity and way of stirring, temperature, time, etc.) [20]. For silica in NaDec, a good correlation between the apparent specific properties and m_S^w was observed. In particular, $V_{sp,\phi}$ at 0.4 and 1% (w/w) are independent of m_S^w and superimpose up to 1 mol kg^{-1}; thereafter, they increase (Fig. 8). $C_{sp,\phi}$ displays a minimum at ca. 0.2 mol kg^{-1} (Fig. 9). Thus, these properties detect the same peculiarities evident in the apparent molar properties of the surfactant in the water + silica mixture. The heat capacity increase is consistent with the hydrophilic interactions which should allow the volume decrease; but that is not experimentally observed. Thus, the only reasonable explanation of this result is the occurrence of suspension destabilization. This is supported by the findings [20] that $V_{sp,\phi}$ at 0.4% (w/w) is independent of NaCl concentration up to 0.8 mol kg^{-1}, after which the volume increases as a consequence of the beginning of flocculation.

$V_{sp,\phi}$ in NaL decreases slightly with concentration (not shown) whereas $C_{sp,\phi}$ exhibits a small maximum (Fig. 10) at the cmc. Moreover, $V_{sp,\phi}$ values are coincident with those in the presence of NaPFO (not shown) up to 0.03 mol kg^{-1}, after which they tend to diverge. Also, the data in NaPFO

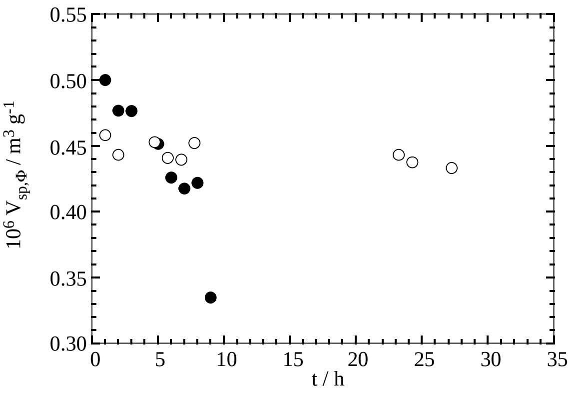

Fig. 7. Dependence on time of the apparent specific volume of silica 0.4% (w/w) in aqueous solutions of sodium octanoate 0.6536*m* (●) and 0.6636*m* (○). The error bars have size smaller than symbols.

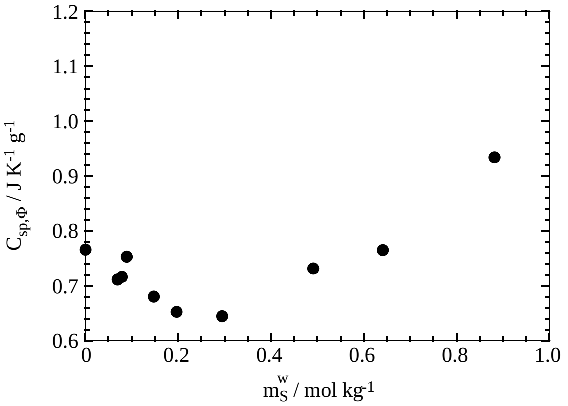

Fig. 9. Apparent specific heat capacity of silica 1% (w/w) in aqueous solution of sodium decanoate as a function of the surfactant concentration. The error bars have size smaller than symbols.

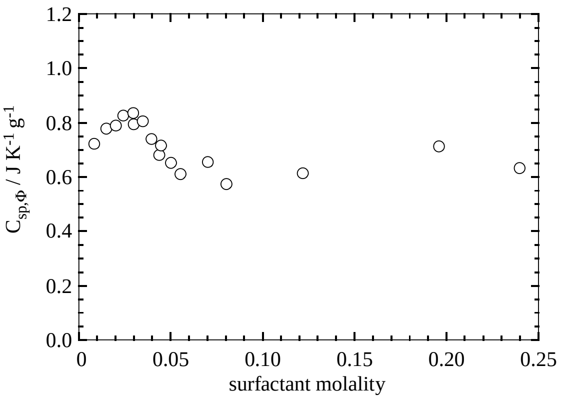

Fig. 10. Apparent specific heat capacity of silica 1% (w/w) in aqueous solution of sodium dodecanoate as a function of the surfactant concentration. The error bars have size smaller than symbols.

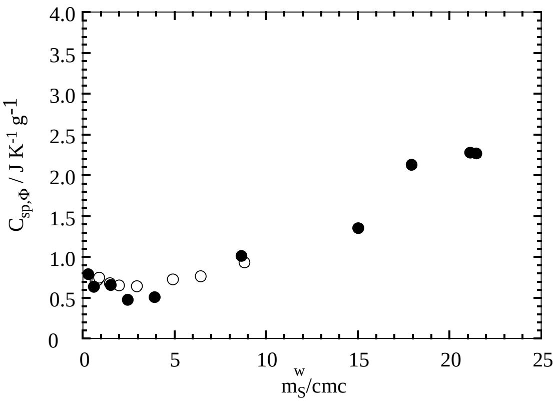

Fig. 11. Apparent specific heat capacities of silica 1% (w/w) in aqueous solution of sodium decanoate (○) and sodium decylsulfate (●) as functions of the surfactant concentration normalized with respect to the cmc. The error bars have size smaller than symbols.

are not well correlated. Since for both surfactants the cmc value is ca. 0.03 mol kg^{-1} (Table 1), the different behavior can be ascribed to the Na^{+} concentration. In fact, at a given $m_{\mathrm{S}}^{\mathrm{w+Si}}$ value, the micellized surfactant concentration of both NaPFO and NaL is practically equal but that of the free sodium ion (m_{Na^+}) is different. The latter is given by $m_{\mathrm{Na}^+} = \mathrm{cmc}_{\mathrm{w+Si}} + (m_{\mathrm{S}}^{\mathrm{w+Si}} - \mathrm{cmc}_{\mathrm{w+Si}})\beta_{\mathrm{w+Si}}$. Since the degree of ionization of the micelles for NaPFO is larger than that for NaL (Table 1), for a fixed $m_{\mathrm{S}}^{\mathrm{w+Si}}$, the m_{Na^+} value for the former is larger and NaPFO may destabilize more effectively than NaL.

3.3. Surfactants of different head-group in silica suspensions

The effect of the surfactant head-group was analyzed by studying decyltrimethylammonium bromide, NaDeS and DDAO.

The mechanism of interaction between negatively charged silica and cationic surfactant may be expressed [9] as: (1) the formation of a surfactant monolayer on the basic sites; (2) the surfactant aggregation as a consequence of the lateral interactions between the surfactant chains; (3) the saturation of the surface particles at $m_{\mathrm{S}}^{\mathrm{w}}$ close to the cmc. Optical reflectometry, atomic force microscopy and fluorescence spectroscopy studies [1,2] have shown that in the region close to the cmc, aggregates of hexadecyltrimethylammonium chloride [1] and bromide [2] adsorbed on negatively charged silica and alumina, respectively, are larger than the micelles in solution. It is also known [9] that small amount of a cationic surfactant added to the negatively charged suspension promotes the flocculation. In addition, for concentrations larger than the cmc, the stabilization is restored by the positive charge formed on the particle surface.

The cationic surfactant chosen in our study is decyltrimethylammonium bromide. The study of the suspension containing 0.4% (w/w) silica was impossible to carry out, as the dispersion did not achieve an acceptable state of homogeneity and the experimental points were not correlated at all. Dispersions of Ludox HS40 in the presence of dodecyl- and octyl-trimethylammonium bromides were studied [20]. It has been shown that at low concentration, large variations in $V_{\mathrm{sp},\phi}$ occur due to the flocculation. For surfactant concentrations larger than the cmc, the stabilization is restored and $V_{\mathrm{sp},\phi}$ is approximately the same as that in water.

In the range of $m_{\mathrm{S}}^{\mathrm{w}}$ studied here, $V_{\mathrm{sp},\phi}$ in NaDeS is independent of the surfactant concentration (Fig. 8) whereas $C_{\mathrm{sp},\phi}$ shows a minimum at ca. 0.08 mol kg^{-1}. To detect the head-group effect of the anionic surfactants, having very close alkyl chain lengths, we plotted the apparent specific properties of silica in NaDeS and NaDec as functions of the surfactant concentration normalized with respect to the cmc value. It turned out that the $C_{\mathrm{sp},\phi}$ versus $m_{\mathrm{S}}^{\mathrm{w}}$/cmc trends superimpose (Fig. 11) whereas the $V_{\mathrm{sp},\phi}$ versus $m_{\mathrm{S}}^{\mathrm{w}}$/cmc curves (not shown) are equal up to $m_{\mathrm{S}}^{\mathrm{w}}/\mathrm{cmc} \approx 9$ beyond which they diverge.

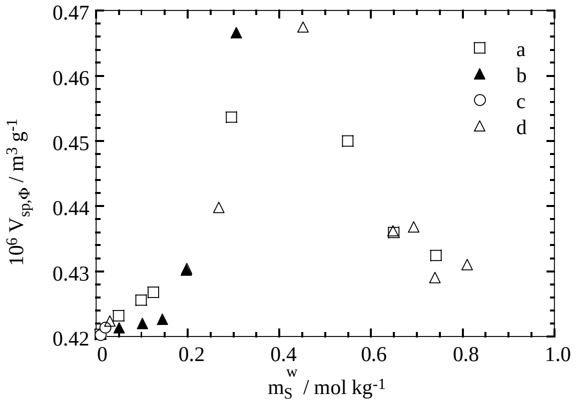

Fig. 12. Apparent specific volume of silica 1% (w/w) in aqueous solutions of *N*,*N*-dimethyldodecylamine-*N*-oxide as a function of the surfactant concentration. a, b, c and d stand for independent experiments. The error bars have size smaller than symbols.

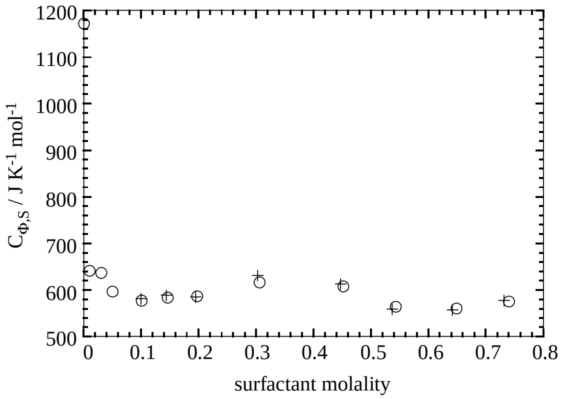

Fig. 13. Apparent molar heat capacities of *N*,*N*-dimethyldodecylamine-*N*-oxide in water (○) and in the water + silica 1% (w/w) mixture (+) as functions of the surfactant concentration. The error bars have size smaller than symbols.

Four independent series of density experiments were carried out for the suspensions in the presence of DDAO. Because of the low cmc, only the micellar region was studied. The experimental data are consistent. The $V_{\mathrm{sp},\phi}$ as a function of $m_{\mathrm{S}}^{\mathrm{w}}$ is a sigmoid shaped curve to 0.4 mol kg^{-1} and thereafter it decreases tending to a constant value slightly larger than that in water (Fig. 12). The S-shaped trend below the maximum may be ascribed to the adsorption of micelles onto the silica particles surface. In particular, the –NO group may interact with the undissociated basic sites of the particles because at the experimental pH the presence of DDAO in the protonated form is negligible [36]. The decrease of $V_{\mathrm{sp},\phi}$ beyond 0.4 mol kg^{-1} remains unclear unless micelle desorption is invoked. The heat capacity data of DDAO in water can clarify this point. Fig. 13 illustrates that in the range of 0.2–0.6 mol kg^{-1}, the $C_{\phi,\mathrm{S}}$ versus $m_{\mathrm{S}}^{\mathrm{w}}$ curve exhibits a broad maximum, which is usually ascribed to the micellar transition [37]. The latter is not influenced by the presence of silica as the experimental points indicate (Fig. 13). Therefore, the graph in Fig. 12 reflects the variation of the interactions between silica and micellized surfactant because the micellar aggregates undergo a structural transition.

4. Conclusions

Thermodynamic data are useful to investigate colloidal suspensions. The hydrophobicity of the surfactant essentially does not play a role, whereas the head-group does according to the polarity of the surface silica particles. It was shown that for the kinetically stable suspensions, the sodium alkylcarboxylates do not exhibit specific affinity towards the silica particles whereas DDAO does.

Finally, the apparent specific properties of silica are very sensitive in detecting the destabilization of the system, in contrast to the apparent molar properties of the surfactant.

Acknowledgements

The authors are grateful to the MIUR for the financial support.

Appendix A

Apparent specific volume and heat capacity of silica in aqueous solutions of various surfactants at 298 K.

$m_{\mathrm{S}}^{\mathrm{w}}$ (mol kg^{-1})	$10w$ (mass%)	$10^{-3}d$ (kg m^{-3})	$10^{-3}d_0$ (kg m^{-3})	$10^6\ V_{\mathrm{sp},\phi}$ (m^3 g^{-1})	c_p (J K^{-1} g^{-1})	c_{p0} (J K^{-1} g^{-1})	$C_{\mathrm{sp},\phi}$ (J K^{-1} g^{-1})
Sodium octanoate							
0.09930	3.97	1.002625	1.000341	0.424			
0.1481	4.20	1.004287	1.001902	0.431			
0.1980	4.12	1.005799	1.003465	0.432			
0.2487	3.87	1.007199	1.005023	0.437			
0.2960	4.11	1.008794	1.006447	0.429			
0.2986	4.06	1.008781	1.006528	0.445			
0.3502	3.70	1.010126	1.008049	0.439			
0.3860	3.96	1.011282	1.009091	0.447			
0.3952	3.93	1.011468	1.009361	0.463			
0.4151	3.91	1.012016	1.009894	0.457			
0.4413	3.87	1.012634	1.010537	0.458			
0.4766	3.89	1.013508	1.011420	0.463			
0.4943	3.75	1.013775	1.011829	0.481			
0.4952	3.99	1.013967	1.011851	0.469			
0.5168	3.80	1.014314	1.012333	0.478			
0.5253	4.10	1.014564	1.012509	0.498			

Appendix A (*Continued*)

m_S^w (mol kg^{-1})	$10w$ (mass%)	$10^{-3}d$ (kg m^{-3})	$10^{-3}d_0$ (kg m^{-3})	$10^6\ V_{sp,\phi}$ (m^3 g^{-1})	c_p (J K^{-1} g^{-1})	c_{p0} (J K^{-1} g^{-1})	$C_{sp,\phi}$ (J K^{-1} g^{-1})
0.5469	3.88	1.014961	1.012948	0.480			
0.5659	3.78	1.015372	1.013347	0.465			
0.5749	3.81	1.015266	1.013577	0.554			
0.5954	4.17	1.015901	1.013959	0.532			
0.5967	3.77	1.015804	1.013989	0.516			
0.6193	4.03	1.016436	1.014448	0.505			
0.6207	3.90	1.016430	1.014470	0.496			
0.6366	4.38	1.016829	1.014840	0.543			
0.6397	3.38	1.016370	1.014867	0.552			
0.6456	3.89	1.016890	1.014941	0.498			
0.6536	3.76	1.016990	1.015115	0.500			
0.6636	4.01	1.017485	1.015314	0.458			
0.6718	3.67	1.017275	1.015421	0.493			
0.6755	4.00	1.017565	1.015514	0.486			
0.6755	4.01	1.017625	1.015437	0.454			
0.6950	3.95	1.017936	1.015836	0.468			
0.1005	9.70	1.005899	1.000326	0.423			
0.1484	9.08	1.007042	1.001834	0.425			
0.2471	9.95	1.010593	1.004836	0.420			
0.3006	9.63	1.011990	1.006415	0.420			
0.3471	9.31	1.013154	1.007758	0.420			
0.3941	9.72	1.014831	1.009088	0.408			
0.4158	9.51	1.015181	1.009769	0.430			
0.4434	9.92	1.016121	1.010440	0.426			
0.4750	9.36	1.016345	1.011179	0.447			
0.4925	10.34	1.017767	1.011576	0.401			
0.5021	9.34	1.017224	1.011792	0.418			
0.5167	8.98	1.017254	1.012123	0.428			
0.5423	9.49	1.017838	1.012969	0.485			
0.5612	9.92	1.018532	1.013012	0.443			
Sodium decanoate							
0.1964	4.09	1.004147	1.001706	0.402			
0.4957	4.80	1.009386	1.006616	0.423			
0.5612	3.79	1.009951	1.007587	0.378			
0.5978	3.97	1.010504	1.008124	0.402			
0.8105	3.59	1.013276	1.011156	0.411			
0.8902	4.15	1.014624	1.012217	0.421			
0.9919	4.62	1.016198	1.013507	0.419			
1.0436	5.21	1.017416	1.014313	0.406			
1.0913	4.82	1.017535	1.014762	0.425			
1.1205	4.55	1.017946	1.015253	0.409			
1.2252	4.08	1.018864	1.016532	0.429			
1.2914	6.37	1.020610	1.017284	0.477			
1.4202	4.61	1.021113	1.018786	0.494			
0.05893	9.61	1.004252	0.998760	0.426			
0.06908	9.40	1.004434	0.998984	0.418			
0.06954	9.12	1.004222	0.999014	0.427	4.1455	4.1768	0.712
0.07875	9.69	1.004843	0.999226	0.418	4.1443	4.1776	0.717
0.08882	10.11	1.005394	0.999546	0.419	4.1426	4.1768	0.753
0.1459	10.22	1.006711	1.000859	0.425	4.1340	4.1693	0.681
0.1963	9.75	1.007435	1.001743	0.408	4.1161	4.1498	0.653

Appendix A (*Continued*)

m_S^w (mol kg^{-1})	$10w$ (mass%)	$10^{-3}d$ (kg m^{-3})	$10^{-3}d_0$ (kg m^{-3})	$10^6\ V_{sp,\phi}$ (m^3 g^{-1})	c_p (J K^{-1} g^{-1})	c_{p0} (J K^{-1} g^{-1})	$C_{sp,\phi}$ (J K^{-1} g^{-1})
0.1981	10.14	1.007625	1.001768	0.420			
0.2949	9.70	1.009101	1.003447	0.415	4.0670	4.1002	0.645
0.3943	9.89	1.010783	1.005079	0.421			
0.4904	10.27	1.012514	1.006595	0.422	4.0011	4.0347	0.732
0.6410	9.77	1.014544	1.008853	0.416	3.9530	3.9842	0.765
0.6858	9.61	1.015090	1.009507	0.418			
0.6907	9.44	1.015086	1.009622	0.420			
0.7897	9.57	1.016686	1.011041	0.410			
0.8813	9.70	1.017787	1.012209	0.424	3.8509	3.8792	0.935
0.8817	10.51	1.018226	1.012261	0.431			
1.0228	9.92	1.019793	1.014113	0.427			
1.0653	10.55	1.020403	1.014593	0.448			
1.1067	9.24	1.020277	1.015120	0.441			
1.1898	9.95	1.021645	1.016152	0.447			
1.3091	9.73	1.022713	1.017491	0.462			
1.3860	9.79	1.023296	1.018323	0.490			
Sodium dodecanoate							
0.00816	9.49	1.002740	0.997251	0.419	4.1468	4.1792	0.724
0.01486	9.61				4.1468	4.1792	0.779
0.01962	10.30	1.003517	0.997521	0.415			
0.02013	10.34				4.1442	4.1789	0.790
0.02417	10.31	1.003648	0.997633	0.414	4.1449	4.1791	0.827
0.02951	9.56	1.003331	0.997762	0.415	4.1472	4.1788	0.837
0.02969	10.26	1.003720	0.997762	0.416	4.1447	4.1791	0.795
0.03477	9.41	1.003319	0.997823	0.413	4.1464	4.1779	0.806
0.03935	9.80	1.003597	0.997903	0.416	4.1420	4.1754	0.740
0.04022	10.23	1.003988	0.997900	0.402			
0.04382	10.24	1.003946	0.997961	0.413	4.1373	4.1727	0.681
0.04475	9.89	1.003720	0.997961	0.415	4.1391	4.1730	0.718
0.05015	9.83	1.003785	0.998043	0.413	4.1361	4.1703	0.652
0.05527	10.85	1.004491	0.998060	0.404	4.1311	4.1692	0.612
0.05901	9.68	1.003810	0.998172	0.415			
0.06026	10.45	1.004207	0.998175	0.420			
0.06948	9.73	1.003996	0.998315	0.413			
0.07006	10.62	1.004527	0.998308	0.412	4.1240	4.1609	0.657
0.08030	10.87	1.004831	0.998433	0.409	4.1174	4.1559	0.575
0.1182	10.11	1.004958	0.998937	0.402			
0.1218	9.87	1.004841	0.998960	0.401	4.1016	4.1361	0.615
0.1959	9.40	1.005578	0.999910	0.394	4.0693	4.1008	0.713
0.2397	10.74	1.006852	1.000433	0.400	4.0434	4.0800	0.633
Sodium decylsulfate							
0.00988	10.19	1.003469	0.997594	0.421	4.1427	4.1768	0.795
0.01982	10.70	1.004304	0.998135	0.420	4.1370	4.1744	0.640
0.02992	9.30	1.004065	0.998678	0.418			
0.04993	9.74	1.005254	0.999628	0.420	4.1268	4.1606	0.658
0.06908	10.47	1.006539	1.000484	0.419			
0.08039	9.96	1.006708	1.000989	0.423	4.1054	4.1415	0.479
0.09833	10.38	1.007759	1.001770	0.421			
0.1293	10.34	1.009099	1.003140	0.422	4.0736	4.1104	0.510
0.1969	9.53	1.011508	1.006022	0.423			

Appendix A (*Continued*)

m_S^w (mol kg^{-1})	$10w$ (mass%)	$10^{-3}d$ (kg m^{-3})	$10^{-3}d_0$ (kg m^{-3})	$10^6\ V_{sp,\phi}$ (m^3 g^{-1})	c_p (J K^{-1} g^{-1})	c_{p0} (J K^{-1} g^{-1})	$C_{sp,\phi}$ (J K^{-1} g^{-1})
0.2850	10.19	1.015543	1.009669	0.422	3.9848	4.0150	1.019
0.3960	10.24	1.019932	1.014002	0.420			
0.4957	10.14	1.023671	1.017782	0.420	3.8708	3.8963	1.358
0.5917	9.94	1.027091	1.021259	0.414	3.8290	3.8459	2.134
0.6970	9.96	1.030529	1.024607	0.407	3.7786	3.7930	2.281
0.7077	10.05				3.7726	3.7878	2.269
Sodium perfluorooctanoate							
0.01429	9.37	1.005791	1.000405	0.419			
0.02303	10.85	1.008646	1.002449	0.421			
0.02787	9.79	1.009195	1.003578	0.418			
0.03519	11.24	1.011700	1.005253	0.416			
0.04048	10.54	1.012357	1.006433	0.426			
0.04858	9.91	1.013915	1.008222	0.413			
0.06965	10.62	1.018690	1.012823	0.430			
0.1000	9.97	1.024932	1.019336	0.415			
0.1456	10.80	1.034902	1.028957	0.417			
0.2464	10.77	1.054819	1.049490	0.453			
0.3009	10.80	1.065530	1.060253	0.449			
N,*N*-dimethyldodecylamine-*N*-oxide							
Experiment (a)							
0.009819	10.12	1.002643	0.996805	0.420			
0.04905	10.73	1.001984	0.995828	0.423			
0.09868	10.66	1.000690	0.994606	0.426			
0.1248	9.65	0.999477	0.993975	0.427			
0.1962	9.40	0.997526	0.992296	0.441			
0.2958	9.86	0.995399	0.990047	0.454			
0.5497	8.86	0.989611	0.984772	0.450			
0.6495	8.32	0.987516	0.982860	0.436			
0.7412	8.21	0.985799	0.981176	0.432			
Experiment (b)							
0.01015	10.20	1.002682	0.996801	0.421			
0.05006	11.57	1.002456	0.995802	0.421			
0.1009	9.55	1.000029	0.994542	0.422			
0.1455	12.91	1.000882	0.993481	0.423			
0.1971	10.92	0.998455	0.992275	0.430			
0.3055	9.78	0.995024	0.989839	0.467			
Experiment (c)							
0.009877	10.10	1.002635	0.996808	0.420			
0.01969	9.68	1.002133	0.996562	0.421			
Experiment (d)							
0.03004	10.23	1.002180	0.996304	0.422			
0.1982	10.81	0.998369	0.992251	0.430			
0.2676	8.30	0.995301	0.990675	0.440			
0.4516	9.75	0.991896	0.986738	0.467			
0.6484	10.60	0.988833	0.982910	0.436			
0.6932	9.81	0.987523	0.982048	0.437			
0.7394	11.20	0.987564	0.981230	0.429			
0.8094	8.92	0.984995	0.979964	0.431			

Appendix B

Apparent specific volume of silica in aqueous solutions of various surfactants as a function of time at 298 K.

Time (h)	$10^{-3}d$ (kg m^{-3})	$10^{6}V_{\mathrm{sp},\phi}$ (m^3 g^{-1})
N,*N*-dimethyldodecylamine-*N*-oxide: $d_0 = 0.982048$; $w = 0.980\%$; $m_S^w = 0.6932m$		
1	0.987580	0.431
3	0.987568	0.432
6	0.987585	0.430
29	0.987643	0.424
48	0.987641	0.424
73	0.987671	0.421
97	0.987664	0.422
504	0.987669	0.421
574	0.987690	0.419
694	0.987660	0.422
742	0.987646	0.424
718	0.987648	0.424
Sodium decanoate: $d_0 = 1.009622$; $w = 0.944\%$; $m_S^w = 0.6907m$		
1	1.015086	0.420
4	1.015060	0.423
5	1.015060	0.423
22	1.015065	0.423
26	1.015095	0.419
27	1.015081	0.421
47	1.015128	0.416
50	1.015119	0.417
52	1.015140	0.415
54	1.015126	0.416
121	1.015180	0.411
173	1.015273	0.401
Sodium decylsulfate: $d_0 = 1.024607$; $w = 0.996\%$; $m_S^w = 0.6970m$		
1	1.030529	0.407
3	1.030526	0.408
4	1.030531	0.407
26	1.030630	0.398
28	1.030594	0.401
50	1.030623	0.398
53	1.030665	0.394
121	1.030834	0.378
172	1.030900	0.372
Sodium decanoate: $d_0 = 1.012209$; $w = 0.970\%$; $m_S^w = 0.8814m$		
1	1.017787	0.424
4	1.017807	0.422
6	1.017775	0.425
22	1.017770	0.426
25	1.017807	0.422
28	1.017876	0.415
Sodium octanoate: $d_0 = 1.007758$; $w = 0.990\%$; $m_S^w = 0.3676m$		
0.5	1.013154	0.453
1.3	1.013138	0.455
5.0	1.013119	0.457
25.0	1.013138	0.455
28.0	1.013152	0.453
30.5	1.013114	0.457
Sodium octanoate: $d_0 = 1.015314$; $w = 0.401\%$; $m_S^w = 0.6636m$		
1	1.017485	0.458
2	1.017547	0.443
4.7	1.017508	0.453
5.7	1.017557	0.441
6.7	1.017562	0.440
7.7	1.017510	0.452
23.3	1.017547	0.443
24.2	1.017571	0.438
27.2	1.017589	0.433
Sodium octanoate: $d_0 = 1.015115$; $w = 0.376\%$; $m_S^w = 0.6536m$		
1	1.016990	0.500
2	1.017080	0.477
3	1.017082	0.477
5	1.017178	0.452
6	1.017277	0.426
7	1.017310	0.418
8	1.017293	0.422
9	1.017630	0.335

Appendix C

Specific conductivities of various surfactants in water and in water + silica 1% (w/w) mixture at 298 K.

NaDeS in water		NaDeS in water + silica		NaDec in water	
$10^3 m_S^w$ (mol kg^{-1})	10χ (S m^{-1})	$10^3 m_S^{w+Si}$ (mol kg^{-1})	10χ (S m^{-1})	$10^3 m_S^w$ (mol kg^{-1})	10χ (S m^{-1})
0	0.004	0	0.21	0	0.008
3.46	0.27	3.40	0.42	6.00	0.39

Appendix C (*Continued*)

NaDeS in water		NaDeS in water + silica		NaDec in water	
$10^3 m_S^w$ (mol kg^{-1})	10χ (S m^{-1})	$10^3 m_S^{w+Si}$ (mol kg^{-1})	10χ (S m^{-1})	$10^3 m_S^w$ (mol kg^{-1})	10χ (S m^{-1})
6.82	0.50	6.71	0.65	11.86	0.73
10.10	0.73	9.94	0.87	17.57	1.05
13.30	0.94	13.08	1.07	23.14	1.37
16.41	1.15	16.14	1.26	28.58	1.67
19.45	1.35	19.13	1.44	33.89	1.98
22.41	1.54	22.04	1.63	39.07	2.28
25.30	1.73	24.87	1.83	44.14	2.56
28.11	1.93	27.64	2.00	49.09	2.83
30.87	2.10	30.35	2.17	53.92	3.11
33.55	2.26	32.98	2.31	58.65	3.36
36.18	2.38	35.56	2.42	63.28	3.61
38.74	2.49	38.08	2.52	67.80	3.84
41.24	2.59	40.54	2.60	72.23	4.09
43.69	2.68	42.94	2.68	76.56	4.32
46.09	2.76	45.29	2.76	80.79	4.54
48.43	2.86	47.59	2.84	84.94	4.75
50.72	2.93	49.84	2.92	89.01	4.97
52.96	3.01	52.04	3.00	92.99	5.18
55.15	3.08	54.19	3.07	96.89	5.38
57.30	3.16	56.30	3.14	100.71	5.58
59.40	3.24	58.36	3.21	104.45	5.74
61.46	3.32	60.38	3.27	108.12	5.90
63.48	3.39	62.36	3.34	111.72	6.09
65.46	3.47	64.29	3.41	115.25	6.21
67.39	3.55	66.19	3.48	118.71	6.37
69.29	3.61	68.05	3.55	122.11	6.50
71.15	3.69			125.44	6.62
				128.71	6.73
				131.92	6.84
				135.07	6.94
				138.17	7.05
				141.20	7.18
				144.19	7.26
				147.12	7.36
				150.00	7.45
				152.83	7.55
				155.61	7.64
				158.35	7.73
				161.04	7.82
				163.68	7.93

NaDec in water + silica		NaL in water		NaL in water + silica	
0	0.22	0	0.004	0	0.11
6.25	0.59	2.29	0.07	2.33	0.17
12.35	0.95	4.52	0.13	4.60	0.23
18.29	1.28	6.70	0.19	6.81	0.29
24.09	1.60	8.82	0.25	8.96	0.34
29.74	1.94	10.89	0.30	11.06	0.40
35.26	2.23	12.90	0.34	13.11	0.45
40.65	2.54	14.87	0.39	15.10	0.50

Appendix C (*Continued*)

NaDec in water + silica		NaL in water		NaL in water + silica	
45.92	2.84	16.79	0.44	17.05	0.55
51.06	3.12	18.67	0.48	18.95	0.60
56.08	3.40	20.50	0.52	20.80	0.64
60.99	3.65	22.30	0.56	22.62	0.68
65.79	3.92	24.05	0.61	24.38	0.71
70.48	4.19	25.76	0.64	26.11	0.75
75.07	4.45	27.43	0.68	27.80	0.77
79.57	4.68	29.06	0.71	29.45	0.79
83.96	4.90	30.66	0.74	31.07	0.80
88.26	5.17	32.23	0.75	32.65	0.82
92.47	5.39	33.76	0.77	34.19	0.84
96.60	5.61	35.26	0.79	35.71	0.85
100.64	5.81	36.73	0.81	37.19	0.87
104.59	6.00	38.17	0.82	38.63	0.88
108.47	6.16	39.58	0.84	40.05	0.90
112.27	6.31	40.96	0.85	41.44	0.91
116.00	6.47	42.31	0.87	42.80	0.93
119.65	6.60	43.64	0.88	44.14	0.94
123.23	6.76	44.94	0.89	45.44	0.95
126.74	6.90	46.21	0.90	46.72	0.96
130.19	7.00	47.46	0.92	47.98	0.97
133.57	7.16	48.69	0.93	49.21	0.99
136.89	7.26	49.89	0.94	50.42	1.00
140.15	7.37				
143.34	7.47				
146.48	7.58				
149.57	7.69				
152.59	7.80				
155.57	7.90				
158.49	8.02				
161.36	8.10				
164.19	8.20				

Appendix D

Apparent molar heat capacities of N,N-dimethyldodecylamine-N-oxide in water and in water+silica 1% mixture at 298 K

Appendix D (*Continued*)

$m^{w;S}$	cp	cp_o	$C_{\phi,S}$
		Water	
0.01015	4.1760	4.1792	642
0.03004	4.1696		637
0.05005	4.1613		597
0.10089	4.1416		577
0.14550	4.1265		584
0.19711	4.1090		587
0.30553	4.0816		617
0.45159	4.0357		608
0.54291	3.9891		565
0.64840	3.9547		561
0.73944	3.9367		575
		Water+Silica 1 mass %	
0..9994	4.1101	4.1462	582
0.14365	4.0842	4.1343	589
0.19498	4.0725	4.1407	584
0.30257	4.0535	4.1439	631
0.44717	4.0056	4.1423	613
0.53706	3.9510	4.1379	558
0.64160	3.9185	4.1381	556
0.73125	3.9023	4.1351	577

Units are: cp and cp_o, $J\,K^{-1}\,g^{-1}$; $C_{\phi,S}$, $J\,K^{-1}\,mol^{-1}$.

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