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ORIGINAL CONTRIBUTION

Micellization study and adsorption properties of an ionic surfactant synthesized from hydrogenated cardanol in air–water and in air–brine interfaces

T. N. Castro Dantas · T. Y. F. Vale · A. A. Dantas Neto · H. Scatena Jr. · M. C. P. A. Moura

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Abstract A sulfonate (2,4-sodium dissulfonate-5-*n*-pentadecylphenol) was synthesized from hydrogenated cardanol and the micellization study was carried out using temperature and electrolyte concentration as variables. The adsorption parameters were obtained using surface tension data by the Frumkin adsorption model and the Simplex Nelder–Mead method. Values of critical micelle concentration (cmc) and surface excess (Γ) were obtained in three different temperatures (303 K, 313 K, and 323 K) and two electrolyte concentrations (NaCl solution—0.1 M and 0.25 M). It was verified that cmc decreased with increasing electrolyte concentration and temperature. The Gibbs free energy showed that the micellization process was spontaneous for all studied systems and temperatures, and also that the presence of several CH_2 groups was significant for micelle formation.

Keywords Cardanol · Cashew-nut shell liquid · Ionic surfactant · Micellization process · Adsorption process

Abbreviations

C	concentration (mol L^{-1})
$\Re$	Universal gas constant (J K^{-1} mol^{-1})
T	temperature (K)
K_F	Frumkin adsorption constant (m^3 mol^{-1})
A	Interaction lateral parameter
^{1}H-NMR	hydrogen nuclear magnetic resonance
^{13}C-NMR	carbon nuclear magnetic resonance
DMSO	dimethyl sulfoxide
Γ	surface excess
$\Gamma_{\max}$	maximum surface excess (mol L^{-1})
$\Gamma_{\max,\ F}$	maximum surface excess in Frumkin model
γ	surface tension (mN m^{-1})
$\Delta_{\mathrm{mic}}G$	variation of Gibbs free energy of micelle formation (kJ mol^{-1})
θ	fractional adsorbed surface density
$\delta\gamma$	mean square average deviation in surface tension (mN m^{-1})
δ	chemical displacement

Introduction

Surfactants have been used thoroughly in processes such as flotation, detergency, paint formulations, lubricants, microelectronics, and enhanced oil recovery [1–4]. The study of surfactant adsorption at the air–aqueous solution interface has received much attention due to its great importance in both practical and theoretical points of view [5–7]. The micellization study of surfactants in solution is important because the performance of the surfactant in many interfacial processes depends on its concentration and orientation at the interface [8].

Micellization process begins when monomeric surfactant ions concentrate, spontaneously, at the fluid interface in a monolayer form, under a condition of apparent adsorption, until maximum adsorption capacity, when the surface excess remains constant. It is at this point, after interface saturation, starting from the critical micelle concentration (cmc), that micelle formation occurs, being an important property of surfactants [9]. The micelles are surfactant clusters inside the solution, with spherical form in most of

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T. N. Castro Dantas (✉) · T. Y. F. Vale · A. A. Dantas Neto · H. Scatena Jr. · M. C. P. A. Moura
Departamento de Engenharia Química,
Universidade Federal do Rio Grande do Norte,
Campus Universitário s/n,
59072-970 Natal, RN, Brazil
e-mail: tereza@eq.ufrn.br

the times. It is well known that the cmc value of ionic surfactants decreases with increasing temperature, reaching a minimum value and starts to increase again [10–15]. When electrolytes are introduced in a surfactant solution, the cmc value tends to decrease. In some studies, it was observed that the addition of electrolytes into a surfactant solution influences the cmc differently, more strongly in ionic>amphoteric>nonionic surfactant solutions [16–18].

In this work, the influence of temperature and electrolyte content on the cmc value was investigated with a compound derived from hydrogenated cardanol (one of the components of cashew-nut oil). The phenomena of micellization and surfactant adsorption at air–water and air–brine interfaces were also investigated because compounds derived from hydrogenated cardanol have been studied as antioxidant substances [19, 20]. However, these processes are not clearly understood.

Materials and methods

Synthesis of the sulfonated hydrogenated cardanol [20, 21]

The following reagents were used as obtained from commercial sources, without further purification: 3-*n*-pentadecylphenol (ACROS), concentrated sulfuric acid (Merck), *n*-butanol, diethyl-ether, and sodium hydroxide (VETEC). The IR spectrum was taken by using a Bio-Rad spectrometer (FTS 3000MX). The ^{1}H-NMR (500 MHz) and ^{13}C-NMR (125 MHz) spectra were recorded on a Brucker spectrometer (AC-500).

To a flask containing hydrogenated cardanol (0.20 mol) was added, slowly, concentrated sulfuric acid (0.90 mol, d=1.84 g/mL). The mixture was heated slowly up to 393 K under constant stirring (3 h). After, it was cooled to reach room temperature (301 K) and diluted using *n*-butanol, being transferred to a separation funnel and then washed with water under soft stirring. After aqueous phase decantation and separation, the solvent phase (*n*-butanol) containing the obtained product was evaporated by distillation. NaOH solution (0.45 mol/L) was added to the residue under stirring, at 323 K. The obtained product was washed with diethyl-ether and heated to dry (318 K). The surfactant (2,4-sodium dissulfonate-5-*n*-pentadecylphenol) was obtained with yield of 74% and used without further purification.

Surface tension measurements

Micellization study was carried out by surface tension measurements (SensaDyner Tensiometer) in three different systems: System I—using only water, System II—a 0.1 M NaCl solution, and System III—a 0.25 M NaCl solution, at three different temperatures: 303 K, 313 K, and 323 K. The maximum bubble pressure method, applied in this research, has been used extensively to measure surface tension of surfactant solutions [22, 23]. This method uses the Laplace–Young equation to determine the maximum pressure in a bubble [24].

Results and discussion

Surfactant characterization

After synthesis and purification [21], the characterization of the obtained surfactant was carried out. The synthesized compound (2,4-sodium dissulfonate-5-*n*-pentadecylphenol) identification was made by using IR, ^{1}H-NMR and ^{13}C-NMR spectroscopy. The obtained results were:

IR (ν cm^{-1}, KBr): 3,541 (OH stretching); 3,075 (C=C–H$_{Ar}$ stretching); 2,916 and 2,849 (C–H stretching); 1,601 and 1,466 (C=C$_{Ar}$ stretching); 1,400 (C–H deformation); 1,200 and 1,045 (SO_2 deformation).

^{1}H-NMR (δ (ppm), 300 MHz, DMSO): 7.93 (1H, s, Ar–*H*); 6.35 (1H, s, Ar–*H*); 2.91–2.86 (2H, t, CH_2–Ar); 1.55 (2H, b,–CH_2–CH$_2$–Ar); 1.24 (26 H, m, CH$_2$–CH_2–CH$_2$); 0.85 (3H, t, CH_3).

^{13}C-NMR (δ (ppm), 75 MHz, DMSO): 153.88; 144.27; 137.33; 127.38; 117.42; 32.84; 31.94; 30.94; 30.05; 29.72; 29.35; 22.73; 22.73; 14.55.

Critical micelle concentration (cmc) determination

Very diluted surfactant solutions contain only monomeric surfactant molecules. When the solution concentration is increased, the concentration at which the individual surfactant molecules start to aggregate to form structures called micelles is named cmc. The cmc of the developed surfactant was obtained in different conditions: I (water), II (0.1 M NaCl solution), and III (0.25 M NaCl solution), evaluating temperature and electrolyte concentration influence. Figures 1, 2, and 3 show the plots of surface tension (γ, in mN/m) versus the logarithm of surfactant concentration (ln C, in molar) at 303 K, 313 K, and 323 K, respectively.

The intercept of the two straight lines designates the cmc value (see Fig. 1—System III at 303 K, as example). For all the systems, this methodology was used. It is clear that surface tension decreases with increasing surfactant concentration until cmc value is reached and, above this value, surface tension is not affected by further increase in surfactant concentration.

Some properties of 2,4-sodium dissulfonate-5-*n*-pentadecylphenol were determined for the three different systems tested at different temperatures, and the results are

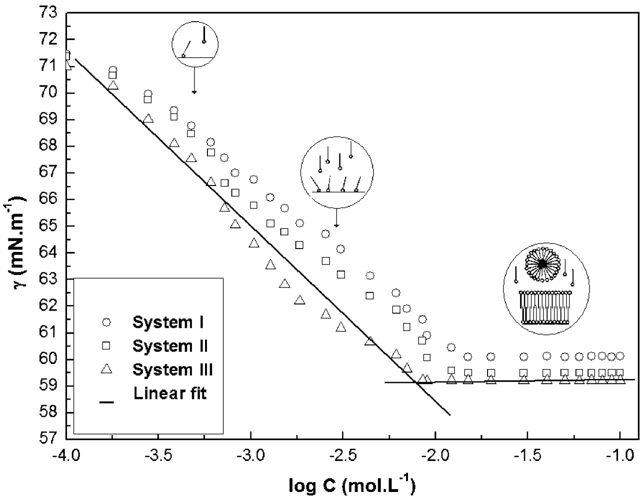

Fig. 1 Variation of surface tension as a function of log C for 2,4-sodium dissulfonate-5-*n*-pentadecylphenol in System I (water), System II (0.1 M NaCl) and System III (0.25 M NaCl) at 303 K

listed in Table 1.Together, with the results shown in Figs. 1, 2, 3, it can be observed that the cmc value decreases with increasing electrolyte concentration. In general, for ionic surfactants, the cmc value decreases with the addition of electrolytes because the interactions between the charged hydrophilic headgroups are weakened, thus favoring the formation of micelles. This occurs due to the electrolyte interference in micelle formation that neutralizes the charge at the micelle surface, reduces the thickness of the ionic atmosphere around the surfactant ionic heads and therefore the electrical repulsions between them, helping in this way the micelle formation [25].

When analyzing the temperature effect, it was verified that the cmc value decreased with temperature (303 to 313 K) and increased again (313 to 323 K) in the investigated range. It is well known that the critical micelle concentration (cmc) is influenced by temperature. For ionic surfactants in aqueous solution, the cmc first monotonically decreases to a certain minimum with increasing temperature and then increases with further increase in temperature, that is, a minimum in the curve of cmc vs. temperature is detected [26].

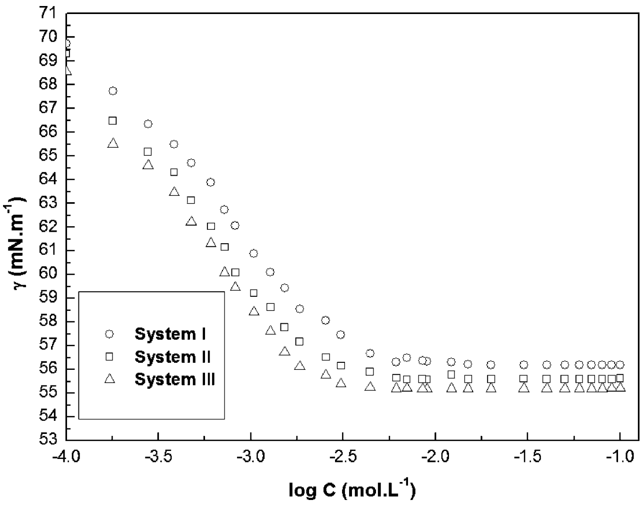

Fig. 2 Variation of surface tension as a function of log C for 2,4-sodium dissulfonate-5-*n*-pentadecylphenol in System I (water), System II (0.1 M NaCl) and System III (0.25 M NaCl) at 313 K

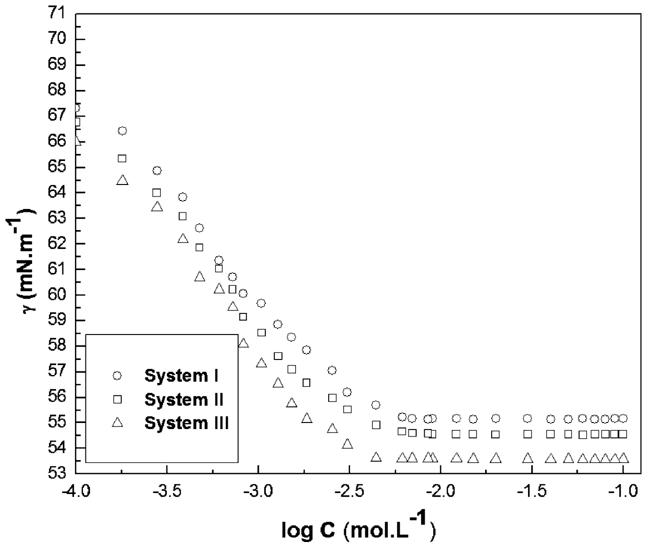

Fig. 3 Variation of surface tension as a function of log C for 2,4-sodium dissulfonate-5-*n*-pentadecylphenol in System I (water), System II (0.1 M NaCl) and System III (0.25 M NaCl) at 323 K

According to Varade [27], the effect of temperature on the cmc of surfactants in aqueous solutions is usually analyzed in terms of two opposite effects: first, as the temperature increases the degree of hydration of the hydrophilic group decreases, which favors micellization; second, an increase in temperature also causes the disruption of the water structure surrounding the hydrophilic group and this is unfavorable to micellization. It seems from the data showed in Table 1 that these two effects were observed in the studied range of temperature.

The determination of the cmc value makes possible the application of 2,4-sodium dissulfonate-5-*n*-pentadecylphenol as a corrosion inhibitor and in enhanced oil recovery.

The cmc is considered a key factor in determining the effectiveness of surfactants as corrosion inhibitors. The corrosion inhibition efficiency of a surfactant normally is enhanced by increasing the surfactant concentration (inhibitor) until the cmc value is reached, forming a protective layer that isolates the metal surface from the aggressive environment.

In this work, the influence of electrolytes and temperature in the cmc value was also verified, since we intend to apply the synthesized surfactant as a corrosion inhibitor in pipelines used in oil and gas production.

Due to their efficiency in lowering interfacial tension, surfactants have been employed in enhanced oil recovery.

Table 1 Some properties of 2,4-sodium dissulfonate-5-*n*-pentadecylphenol in three different systems: S I (water), S II (0.1 M NaCl) and S III (0.25 M NaCl), at different temperatures

Parameter	303±1 K			313±1 K			323.15±1 K		
	S I	S II	S III	S I	S II	S III	S I	S II	S III
cmc (mmol.L^{-1})	14.5	12.3	6.47	4.59	3.26	2.59	4.24	3.42	3.09
γ_{cmc} (mN m^{-1})	60.11	59.53	59.21	56.18	55.57	55.18	55.15	54.60	53.61
$\Delta_{\mathrm{mic}}G$ (kJ.mol^{-1})	−21.3	−22.2	−25.4	−28.0	−29.8	−31.0	−29.4	−30.5	−31.1

They can aggregate themselves at interfaces between fluids with different polarities, such as oil and water, leading to a reduction in the interfacial tension.

In surfactant flooding, it is very important to understand the adsorption of surfactants in the reservoir porous media. The loss of surfactant by adsorption reduces the efficiency of the process, making the reduction of interfacial tension between residual water and crude oil difficult, resulting in a technically unviable process. Studies reported in the literature [28, 29] suggest the use of surfactant concentrations at least above the cmc value for ionic surfactants [28] and around the cmc value for nonionic ones [29].

Determination of the Gibbs free energy of micelle formation

The value of cmc can be used to obtain the Gibbs free energy of micelle formation ($\Delta_{\mathrm{mic}}G$). The appropriate relation depends on the nature of the surfactant. Ionic surfactants are represented as dissociated molecules in solution, but not completely and necessarily at the surface or in the form of micelles. When adding electrolytes to the system and assuming the electroneutrality of the interfacial system in diluted solutions [24, 30], the Gibbs free energy equation for the ionic micellization process, considering only the formation of surfactant ions and when the concentration of generated ions is very low (diluted solutions), is:

$$\Delta_{\mathrm{mic}}G = 2\Re T \ln \mathrm{cmc} \tag{1}$$

where cmc is the critical micelle concentration, expressed as mole fraction units, $\Re$ is Universal gas constant (J K^{-1} mol^{-1}), and T is temperature (K).

The Gibbs free energy of micelle formation ($\Delta_{\mathrm{mic}}G$) was calculated by using Eq. 1. Table 1 presents the results of γ_{CMC} and $\Delta_{\mathrm{mic}}G$ for all studied systems. It was noticed that the surface tension decreases with increasing surfactant concentration. However, the surface tension was not very low when compared to results obtained in a previous work using ionic surfactants derived from castor oil [24]. This fact can be attributed to the existence of a small amount of impurity present in the studied sample. More appropriately, this can be due to the localization of the sulfonate group being in an *ortho* position to the alkyl group. Previous researches showed that the surfactant with the sulfonate group in the *ortho* position with respect to the alkyl chain

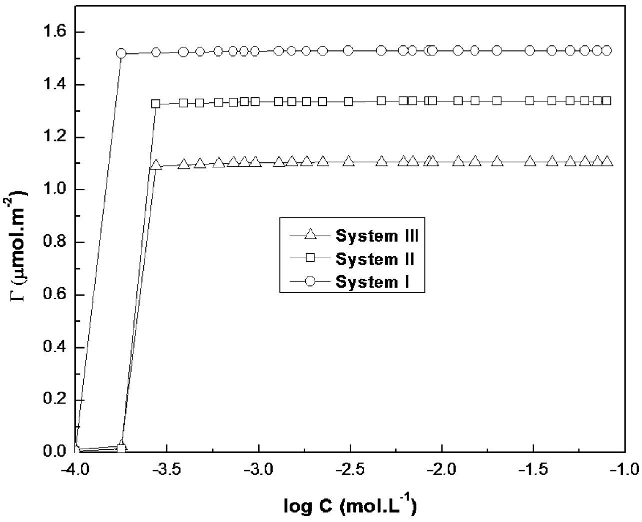

Fig. 4 Variation of surface excess as a function of log *C* for 2,4-sodium dissulfonate-5-*n*-pentadecylphenol in System I (water), System II (0.1 M NaCl) and System III (0.25 M NaCl) at 303 K

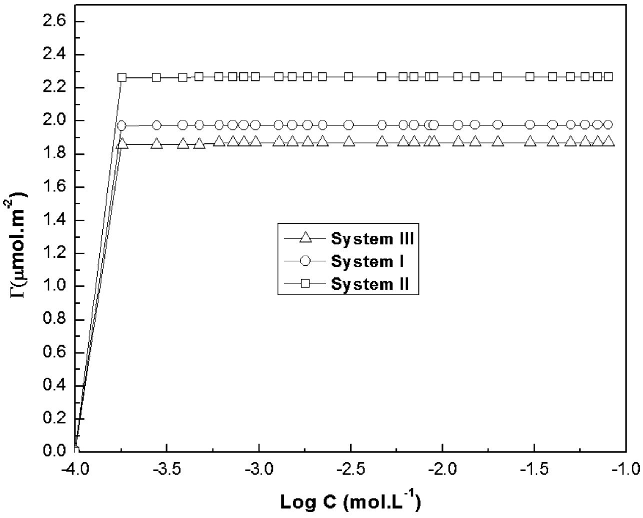

Fig. 5 Variation of surface excess as a function of log *C* for 2,4-sodium dissulfonate-5-*n*-pentadecylphenol in System I (water), System II (0.1 M NaCl) and System III (0.25 M NaCl) at 313 K

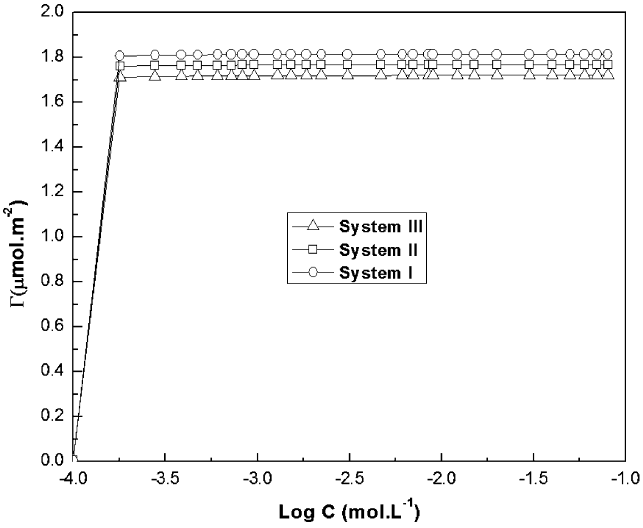

Fig. 6 Variation of surface excess as a function of log C for 2,4-sodium dissulfonate-5-*n*-pentadecylphenol in System I (water), System II (0.1 M NaCl) and System III (0.25 M NaCl) at 323 K

had lower optimal salinity, higher surface tension and lower solubility in water than surfactants with the sulfonate group in the *para* or *meta* positions [31, 32].

Analyzing the free energy of micellization data (Table 1), one can conclude that the micellization process was spontaneous ($\Delta_{\mathrm{mic}}G<0$). With regard to the electrolyte addition, it can be noticed that with increasing electrolyte concentration the surface tension and $\Delta_{\mathrm{mic}}G$ are decreased. This was expected, as explained above, because with increasing electrolyte concentration the cmc value decreases due to the fact that interactions between the charged hydrophilic headgroups are weakened, favoring micelle formation.

Adsorption model

This work uses the Frumkin adsorption model [33] to obtain the relationships between surface adsorption and surfactant concentration in solution, based on the Gibbs adsorption equation for an ionic surfactant.

Frumkin adsorption isotherm is derived from the Langmuir tension model taking into consideration the effect of interactions that occur between surfactant molecules that are in direct contact with each other on the adsorbed layer [24, 33]. In a previous paper [24], the evolution from Langmuir model to Frumkin model is described in details. Frumkin adsorption model is given by:

$$\theta = \frac{\Gamma}{\Gamma_{\max,F}} = \frac{K_F C \exp(-A\theta)}{1 + K_F C \exp(-A\theta)} \tag{2}$$

where Γ is the surface excess, $\Gamma_{\max,\mathrm{F}}$ is the maximal surface excess in the Frumkin model, θ is the surface layer coverage, K_{F} is an adsorption equilibrium constant, and A is a parameter accounting for the lateral interaction.

The A parameter is related to the variations in the adsorption energy of the covered surface. When analyzing Eq. 2, it can be observed that when $A=0$, the Frumkin equation is reduced to the Langmuir equation. If $A>0$, a decrease in the effective adsorption constant occurs, which is caused by the repulsive interactions between the surfactant adsorbed monolayer and the molecules into the bulk solution. When $A<0$, there is a high compatibility between the bulk solution and the adsorbed monolayer [30].

The Frumkin isotherm for ionic surfactants is given by [24]:

$$\gamma = \gamma_0 + 2\Re T\Gamma_{\max,F}\left[\ln(1-\theta) - \frac{A}{2}\theta^2\right] + \Re T\Gamma_{\max,F}\left[K_F C_S \int_0^{\theta} \frac{1 + A\theta(1-\theta)}{\theta e^{A\theta} + K_F C_S(1-\theta)}\,d\theta\right] \tag{3}$$

In order to produce the best parameter adjustment for the assessed model, the mean square average deviation, denoted $\delta\gamma$, between the measured or experimental data

Table 2 Best fitted parameters of Frumkin model for 2,4-sodium dissulfonate-5-n-pentadecylphenol surfactant in systems I (SI—water), II (S II—0.1 M NaCl) and III (S III—0.25 M NaCl), at different temperatures

Parameter	Temperature								
	303±1 K			313±1 K			323±1 K		
	S I	S II	S III	S I	S II	S III	S I	S II	S III
$\Gamma_{\mathrm{m,F}}$ (mol/L)×10^6	1.53	1.337	1.108	1.977	2.267	1.867	1.815	1.768	1.721
K_{F} (m^3/mol)	41.96	54.71	107.8	45.07	27.95	41.61	33.43	43.91	37.03
A	−9.88	−9.02	−7.81	−10.6	−11.3	−10.3	−10.6	−10.4	−10.4
$\delta\gamma$ (mN/m)	0.30	0.36	0.31	0.56	0.46	0.37	0.50	0.37	0.31
Quality of fit	Good	Good	Good	Good	Good	Good	Good	Good	Good

(exp) and the ones predicted by the model (model), is minimized. Thus:

$$\delta\gamma = \sqrt{\frac{\sum\left(\gamma_{\exp_i} - \gamma_{\mathrm{model}_i}\right)^2}{N}} \tag{4}$$

where N is the number of data points. The model is validated when the values of $\delta\gamma$ are compared to the error estimated for surface tension measurements, considered to be as low as 1.0 mN m^{-1}. The method is considered “good” if $\delta\gamma <$ 1.0 mN m^{-1}, “poor” if 1.0 mN m$^{-1} < \delta\gamma <$ 3.0 mN m^{-1}, and “very poor” if $\delta\gamma >$ 3.0 mN m^{-1}. The BASIC Software package can be used to carry out the direct minimization of Eq. 4, since it comprehends a built-in Nelder–Mead type Simplex search method for optimization routines [24, 30].

For 2,4-sodium dissulfonate-5-*n*-pentadecylphenol, at the three different systems tested, the data of surface excess by Frumkin model are presented in Figs. 4, 5, and 6 (surface excess versus the logarithm of surfactant concentration—ln C, in molar) at 303 K, 313 K, and 323 K, respectively. In all studied systems the surface excess increased with increasing surfactant concentration until the cmc value was reached and then it remains at a constant level. However, at 323 K, it was observed that there is not significant difference between the surface excesses. At 303 K and 313 K, in Systems II (0.1 M NaCl) and III (0.25 M NaCl) a larger surface excess was observed when compared to System I (water), suggesting a change in micelle structure.

It is well known that surfactants in solution can form spherical, cylindrical, or lamellar micelles in shape, fact directly dependent of the geometry of the monomeric units and their molecular environment. According to Rosen and Dahanayake [34] the cross-sectional area occupied by the hydrophilic group at the interface often varies with the molecular environment (i.e. pH, ionic strength of the solution, temperature, presence of additives, concentration of surfactant). Thus, adding electrolytes and changing the temperature it is possible to change the micelle shape. For ionic surfactants, the cross-sectional area decreases with increasing surfactant concentration, ionic strength of the solution and temperature, allowing the formation of more complex structures (see Fig. 1 as example).

Analyzing the temperature effect, it is also observed that from 303 K until 313 K, there is an increase in surface excess values in all systems. However, from 313 K until 323 K the surface excess decreased. It was expected that an increase in temperature causes a decrease of the surface excess; however, this behavior suggests that between 303 K and 313 K a change in the micelle aggregation occurs, that can be caused by the high surfactant concentrations.

Table 2 presents the $\Gamma_{\max,\mathrm{F}}$, K_{F}, and A parameters of the Frumkin model for 2,4-sodium dissulfonate-5-*n*-pentadecylphenol surfactant, in systems I (SI—water), II (S II—0.1 M NaCl), and III (S III—0.25 M NaCl), at different temperatures, calculated by using Eq. 3. Such parameters were determined by minimizing the mean-square average deviation in surface tension ($\delta\gamma$), according to Eq. 4. It can be observed that all lateral interaction values (A) were negative, which is an evidence of the lateral interaction between the CH_2 groups present in the carbonic chain of surfactant molecules. This phenomenon, when compared to the repulsive forces caused by equal-charged surfactant hydrophilic headgroup, is of great relevance for micelle formation, because the long carbonic chain (15 atoms) makes difficult the formation of monolayers.

Conclusion

The accomplishment of this research showed that the adsorption at the air–water or air–aqueous solution interfaces and the micellization process of the 2,4-sodium dissulfonate-5-*n*-pentadecylphenol, synthesized from the hydrogenated cardanol, is spontaneous for all studied systems, at three different temperatures (303 K, 313 K, and 323 K).

The Frumkin adsorption model was used to describe the surfactant adsorption process at the air–water and air–water solution interfaces and showed a good fit for all studied systems.

For the obtained surfactant, at a given temperature, it was verified that cmc decreased with increasing electrolyte concentration and the surface excess, parameter also studied, decreased when the electrolyte concentration increased. An exception was observed for surface excess at 313 K, suggesting a change in micelle aggregation.

The development of this study, as well as the obtained results, allowed us to plan the application of the obtained compound in processes such as enhanced oil recovery, antioxidants, and corrosion inhibitors.

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