Simultaneous influence of uni-univalent salt aqueous solutions and sodium dodecyl sulfate surfactant on interfacial tension of toluene-water

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(*Received 27 September 2012 • accepted 1 January 2013*)

Abstract−The simultaneous influence of NaCl, KCl and KI salts and well known sodium dodecyl sulfate (SDS) surfactant on the interfacial tension (IFT) of conventionally used chemical system of toluene-water was studied. The concentration range of salts was within (0.010 to 0.075) mol/dm³ and of surfactant within (1.7 to 26.0)×10⁻⁵ mol/dm³. SDS adsorption on interface is highly intensified in the presence of salts and IFT can reach to 67.1% of its initial value. Accordingly, the role of ions was investigated and the order of salts effectiveness was revealed as KI>>KCl>NaCl. The obtained data, with both effects, were nicely reproduced using an equation of state, based on Gibbs adsorption equation and the Langmuir isotherm. Two relevant important adsorption parameters exhibited reasonable variations. Furthermore, the general revealed linear variation of IFT with a previously defined "effective concentration" indicates the strong influence of the surfactant counterions.

Key words: Interfacial Tension, Uni-univalent Salts, Equations of State, Toluene-water, SDS

INTRODUCTION

Interfacial tension (IFT) is a basic concept in processes working with immiscible phases, such as extraction and emulsification. Many interfacial phenomena are relevant in chemical or petrochemical processes, such as separation by flotation and liquid-liquid extraction; some relevant processes are also based on the formation of emulsion [1]. Mass transfer in such processes can be significantly altered by IFT variation, mainly due to provided variations in the system hydrodynamics. In this regard, salts and surface active agents are the mostly the materials that individually and/or mutually can alter the IFT.

The unique properties of surfactants provide the means to apply surfactants in wettability adjustment, detergency and the displacement of liquid phases through porous media on one hand, and to stabilize dispersions (including foams, froths and emulsions), or to destabilize dispersions (including foams and emulsions) on the other hand. Phase contacting hydrodynamic and enhancing contact area with drop or bubble breakage/coalescence phenomena is highly altered in the presence of surfactants. Of course, the general deleterious effect of contaminants and surfactants in mass transfer phenomenon should also be regarded. These in turn leads to a vast array of practical application areas which are illustrated in terms of mineral and petroleum processing. The petroleum industry, for instance, makes extensive use of surfactants from the enhanced oil recovery techniques to the emulsion breaking in production operations [2]. The optimum surfactant formulation for an emulsion system, as part of enhanced oil recovery (EOR), is dependent on many variables such as salinity, pH, and temperature. To investigate all these variations, the IFT property has to be taken into account.

Despite the tendency of most surfactants to locate at the interface and lower IFT, some electrolyte ions exhibit different behaviors; depending on their charge and ion size, they stay in solutions or are adsorbed at the interface [3,4]. This contrasting behavior can be altered and some coordinate/synergistic effect may be revealed due to specific collaboration between electrolyte ions and ionic surfactants at the interface.

Although many works have been reported on the influence of surfactants and/or salts on the surface tension [5,6], and also individually at the interfaces [7,8], there are few reports focusing on simultaneous effect of surfactants and salts on liquid-liquid interfaces [9,10]. Recently, Kumar et al. [11] studied the adsorption of ionic surfactants at organic-water interfaces in the presence of small quantities of sodium bromide. The results show a sharp decrease in the IFT with increasing salt concentration in the presence of a surfactant, while the salt by itself exhibits little effect [11,12]. The stronger IFT reduction has been attributed to repulsion between the salt and ionic surfactant ions. It has been reported that non-ionic surfactants do not exhibit this sort of behavior [13].

Accordingly, a number of theoretical models have been proposed for description of surfactant adsorption in such systems. The Gibbs adsorption isotherm has usually been essential. In addition, an improved model of ionic surfactant adsorption has been developed by considering penetration of counterions (ions with surfactant headgroup opposite charge) into Stern layer, right next to the interface [14,15]. To accomplish the previous models of ionic surfactant adsorption, this improved model was allowed to account the strong dependency of adsorption on the type of counterions. In this regard, the specific electrolyte concentration effect was taken into account in a simple and consistent model to estimate IFT and adsorption parameters by Kumar et al. [11].

Most natural water sources contain uni-univalent salts to some extent. Meantime, there is an inevitable level of contamination/impurity in the most industrial systems that reduce the rate of mass transfer. For investigating this matter, surfactants are usually used

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as the artificially added contaminant agents in testing systems. This study aims to find the effect of uni-univalent salts on IFT of toluene-water chemical system containing quantities of the quite well known surfactant, sodium dodecyl sulfate (SDS). European Federation of Chemical Engineers (EFCE) has recommended the toluene-water chemical system for liquid-liquid extraction investigations [16]. The results help to provide precise contactor design and to simulate a process under real conditions. In this regard, the important parameters of maximum adsorbed surfactant concentration and adsorption constants are determined and evaluated.

MATERIALS AND METHODS

1. Materials

Toluene, sodium dodecyl sulfate (SDS) and NaCl, KCl and KI salts were all Merck products with mass fraction purities of more than 99.5%, 99.5%, 99.8%, 99.5% and 99.5%, respectively. UV spectrum of pure SDS should represent no detectable absorbance above 200 nm [17]; thus, the UV spectrum was prepared by UV-visible spectrophotometer (JASCOV-630). It showed no detectable absorbance above 200 nm. The water used in all experiments was deionized with electrical conductivity value of 0.08 µS/cm.

2. Measurements

To achieve equilibrium conditions prior to experiments, toluene and water were mixed for at least 1 h, and then left at rest for another 1 h at 20.0 °C. The required aqueous solutions were prepared by mass using an Ohaus (Adventurer Pro, AV 264) balance with an uncertainty of ±0.1 mg. Six surfactant concentrations ranging from (0 to 26.0)×10^{-5} mol/dm^3, each with three salt concentrations of: 0.010, 0.025 and 0.075 mol/dm^3 were examined. The concentration range for surfactant is far from its CMC, which has been previously reported in the presence and absence of different salts [18, 19]. Estimated standard deviations for the lowest concentrations of NaCl and KCl were about ±(4.3 and 4.2)×10^{-6} mol/dm^3, respectively. This decreased to ±4.0×10^{-6} mol/dm^3 for KI solution due to higher molecular weight of the salt. For the lowest concentration of surfactant, the standard deviation reached to ±3.4×10^{-7} mol/dm^3.

The density of aqueous and organic phases was measured at desired temperature (20.0 °C) by means of an Anton Paar DMA 4500 densitometer with uncertainty of ±0.01 kg/m^3. The desired temperature was self-adjusted in this densitometer with an uncertainty of ±0.01 °C. Table 1 lists the density of different salt solutions. Data demonstrate that addition of salts would increase the density of water. The very low concentration of added surfactant did not show considerable effect on density.

To determine IFT values, the drop volume method was used. The experimental setup and procedure has been previously described [8]. The main part of the device was a very thin glass capillary with

outer diameter of 4.50 mm placed in a jacketed glass cell connected to a thermostat. The capillary tip had an angle of 90° between ground face and the internal with the edges sharp. An adjustable syringe pump (phoenix M-CP, French) injected the aqueous phase into toluene phase. A very low flow rate (1.0 mL in 29.50 min) was used for slow formation of drops at the capillary tip. Giving enough time (more than 140 s) in this work ensures reaching equilibrium IFT, for the aim of complete adsorption at interface when each drop formation accomplished. Values for surface tension variation versus drop formation time have shown that even with low concentration of salts, a chemical system containing SDS can attain equilibrium conditions rapidly [20]. All IFT experiments were performed at 20.0 °C, maintained constant by a thermostat (Optima740, Japan) with an uncertainty of ±0.01 °C.

Each drop volume was obtained from at last eight measurements of time to be used for drop volume calculations. IFT (γ) was related to volume of drops by Harkins and Brown equation [21] as

$$\gamma = \frac{v \Delta \rho g}{2 \pi r f\left(\frac{r}{\sqrt[3]{v}}\right)} \tag{1}$$

where $\Delta \rho$ is difference between aqueous and organic densities, r and v are capillary radius and volume of drop, respectively, and f(r/$\sqrt[3]{v}$) is a constant that can be obtained by polynomial fit equation proposed by Lee et al. [22],

$$f\left(\frac{r}{\sqrt[3]{v}}\right) = 1.000 - 0.9121\left(\frac{r}{\sqrt[3]{v}}\right) - 2.109\left(\frac{r}{\sqrt[3]{v}}\right)^2 + 13.38\left(\frac{r}{\sqrt[3]{v}}\right)^3$$
$$- 27.29\left(\frac{r}{\sqrt[3]{v}}\right)^4 + 27.53\left(\frac{r}{\sqrt[3]{v}}\right)^5 - 13.58\left(\frac{r}{\sqrt[3]{v}}\right)^6 + 2.593\left(\frac{r}{\sqrt[3]{v}}\right)^7 \tag{2}$$

Here, the range of dimensionless constant f(r/$\sqrt[3]{v}$) was between 0.34-0.52. IFT measurements had a relative uncertainty of about ±0.2 mN/m.

To examine reliability of the method, IFT of water and toluene at 20.0 °C was measured (36.50 mN/m) and compared with reported values in the literature (36.1, 36.9) mN/m [23,24]. Also, surface tension of pure water at 25.0 °C was measured, 72.2 mN/m, close to 72.04 mN/m reported in the literature [25].

RESULTS AND DISCUSSION

1. Influence of Uni-univalent Salts

The results of interfacial tension measurements for different surfactant and salt concentrations are listed in Table 2. The variation of system IFT with different concentrations of KCl salt, for instance, is presented in Fig. 1. As is observed, in all cases, IFT envisages a sharp reduction with low SDS concentrations, which is due to high tendency of surfactant molecules for adsorption at the interface. After that, saturation of interface, under higher surfactant concentrations, causes a rather mild variation of IFT.

IFT variations are compared in Fig. 2 for the three salts used. With the highest salt concentration of 0.075 mol/dm^3, the amount of IFT reaches to 15.5 and 15.0 mN/m with NaCl and KCl, respectively; whereas, this value reaches to about 12.0 mN/m with the same concentration of KI. Note that the presence of just the used salts, within the used concentration range, provides no sensible change in IFT when compared with the significant effect of the surfactant.

Table 1. Density (kg/m^3) of different aqueous salt solutions at 20.0 °C

Salt	C$_s$ (mol/dm^3)		
	0.010	0.025	0.075
NaCl	998.64	999.26	1001.38
KCl	998.70	999.94	1001.78
KI	999.44	1001.24	1007.28

Table 2. IFT of different solutions at 20.0 °C

$C \times 10^5$ (mol/dm^3)	γ (mN/m)									
	No salt	C_{NaCl} (mol/dm^3)			C_{KCl} (mol/dm^3)			C_{KI} (mol/dm^3)		
		0.010	0.025	0.075	0.010	0.025	0.075	0.010	0.025	0.075
0.0	36.5	36.5	36.5	36.5	36.5	36.5	36.5	36.5	36.5	36.5
1.7	35.1	31.4	30.7	28.6	31.1	29.7	28.2	31.6	30.7	28.0
3.5	34.2	29.2	27.2	25.9	28.9	27.0	24.8	29.2	27.5	24.7
5.2	33.4	27.4	25.7	23.7	27.0	25.3	23.1	27.2	24.8	21.5
8.7	31.5	25.7	24.0	22.2	25.5	23.8	21.3	25.0	22.4	19.8
17.3	27.6	23.7	21.0	20.5	23.1	20.7	18.5	22.2	19.3	14.4
26.0	26.4	20.9	19.3	15.5	20.6	18.3	15.0	20.3	16.7	12.0
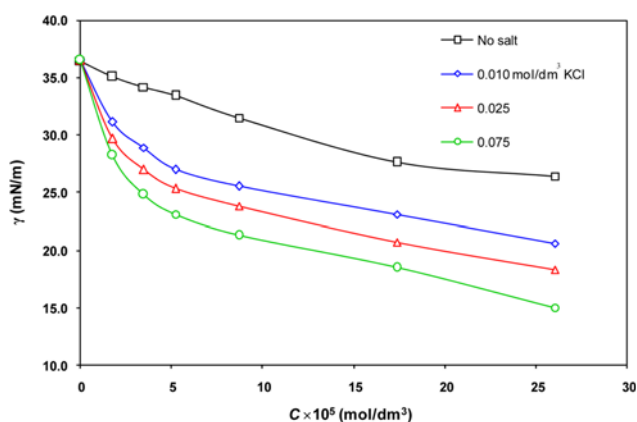
Fig. 1. IFT variations versus SDS concentration with different concentrations of KCl.
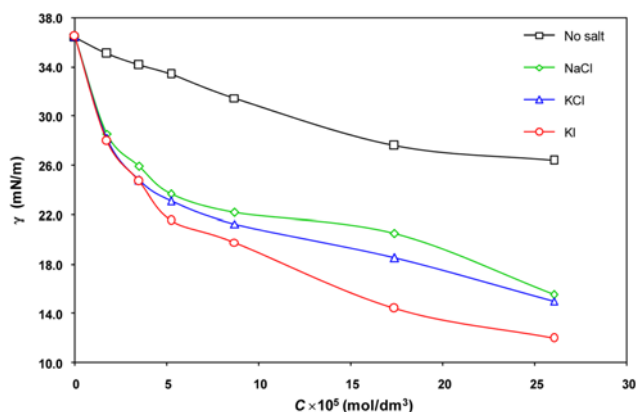
Fig. 2. IFT variations versus SDS concentration with constant salt concentration of 0.075 mol/dm^3.

Fig. 3. IFT variations versus the salt concentration with constant surfactant typical concentration of 26×10^{-5} mol/dm^3.

Addition of salts has generally caused significant intensification of SDS effect on IFT. This influence is significant with dilute solutions of SDS, to about 1.0×10^{-4} mol/dm^3. Meantime, as presented typically in Fig. 3, IFT is also sensible to low amounts of the salts (less than 0.025 mol/dm^3). These imply that water sources, which contain low quantities of salts, are highly prone to IFT reduction in the presence of ionic surfactants.

To interpret the IFT graphs in the presence of different salts, the interaction between salt ions and head group of the surfactant should be considered [26]. Experimental reports beside theoretical simula-

tion such as molecular dynamic (MD) indicate that reduction of electrostatic repulsion between surfactant headgroups can be the main reason for significant reduction of surface tension or IFT [13].

To describe how salts would facilitate surfactant adsorption at the interface, the concept of electric double layer (EDL) has to be considered. In this regard, EDL is formed when an ionic surfactant is adsorbed at the interface. The interface zone includes the Stern layer where adsorbed surfactant headgroups are placed, and diffuse layer through which ions and headgroups have to pass before reaching the Stern layer. Negative or positive charge of headgroups placed in the Stern layer can be compensated by the charge of counterions, since these can penetrate the Stern layer and provide a neutral charge media. The molecular dynamic simulations and X-ray reflectivity measurements of the thin films highly support this projection [27]. On the other hand, penetration of counterions to Stern layer leads to the compression of EDL [6]. This in turn makes the surfactant molecules prone to more adsorptions at the interface [8,11,13]. Thus, IFT reaches lower values in the presence of higher salt concentrations. A scheme of the field surfactant molecules and ions distribution at the interface is presented in Fig. 4. In the presence of salts, surfactant headgroups are closely adsorbed at the interface in conjunction with the counterions, providing a compact adsorptive barrier.

Owing to easier penetration of K$^+$ counterions in comparison to Na$^+$, due to lower hydrated radii [28], in addition to higher mobil-
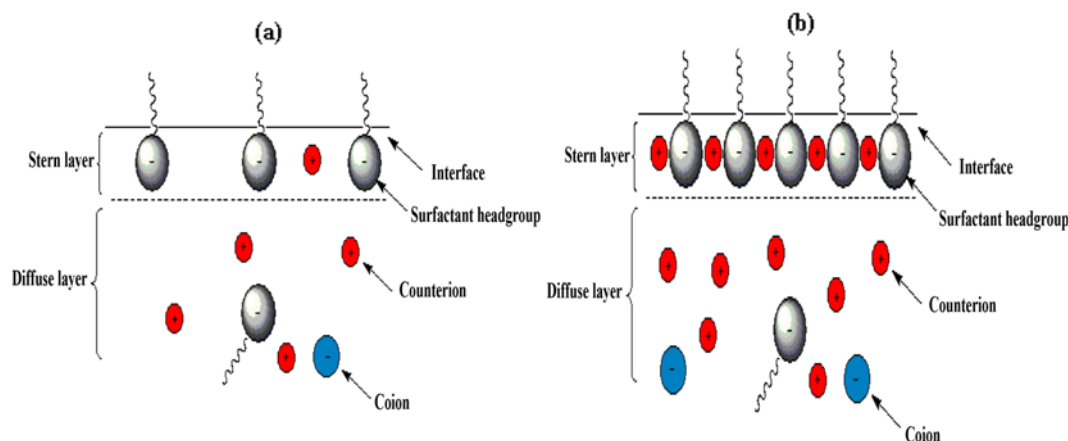
Fig. 4. Schematic presentation of the species distribution at the interface zone in absence (a) and presence (b) of a salt.

ity of this ion (ion mobility of K^+ is 3.1 and of Na^+ 2.7 cm²/V·s) [29], KCl acts somewhat stronger than NaCl.

An interesting revealed result is that KI behaves as the most effective salt, especially at higher concentrations. The IFT reduction reaches to 67.1% when 0.075 mol/dm³ of KI is added to a 26.0× 10^{-5} mol/dm³ of SDS solution. This strong effect stemmed from inner properties of KI. Considering the lack of salt anion adsorption in the presence of anionic surfactants [30], the reason for this case can be attributed to the less tendency of I^- (compared with Cl^-) for the ion-pair formation with K^+. This in fact stems from difference between hardness and softness properties of I^- and Cl^-, which leads to much freedom of K^+ from KI [31]. Lattice energy of KI is 650 kJ/mol, while that of KCl is 717 kJ/mol [32]. Thus, more surfactant molecules can be adsorbed and a sharp reduction of IFT appear in the presence of KI. The order of used salts effectiveness is reasonably appears as: KI>>KCl>NaCl.

Previous investigations on mass transfer, without salt, show significant decrease in the overall mass transfer coefficient due to surfactants adsorption [33,34]. In the presence of both SDS and KI, resistance against a solute mass transfer across the interface seems to be high due to lack of enough space for solute transfer across the interface. So, a significant decrease in the rate of mass transfer is expected in this regard. Further investigation is required to assess this subject.

2. Modeling the Surfactant Adsorption in the Presence of Salts

There are theoretical models that describe the surfactants behavior in the presence of electrolytes [10,14]. Most of them suggest relations which are based on the concept of chemical potential. In this regard, Gibbs adsorption equation is usually utilized for determination of adsorption parameters due to its simplicity and exact outcomes.

Here, the solutions have been assumed to be ideal, because of the low surfactant and salt concentrations. The Gibbs adsorption equation provides the relationship between the change in IFT and the changes in the bulk solution composition as

$$d\gamma = -RT \sum_i \Gamma_i d\ln C_i \tag{3}$$

where R is the gas constant, T is absolute temperature, Γ is equilibrium surface concentration (surface excess), C is the bulk concen-

tration of a species and i is the number of solute species that change of their bulk concentration would alter their concentration at the interface [35].

Ionic surfactants such as SDS can be presented as AR that can dissociate completely to A^+R^-, and in the presence of electrolyte B^+X^- (with relative high concentration) one can introduce the surfactant concentration as $C = C_{R^-}$ and salt concentration as $C_S = C_{X^-}$. Considering negligible adsorption of electrolyte coion X^- at interface [28,30] and that in this work, A^+ concentration is much less than B^+ (about three orders of magnitude); also with respect to the consistent charge balance between anions and cations everywhere, either in solution bulk or at interface, the relation $\Gamma_{R^-} = \Gamma_{B^+} = \Gamma$ is valid, and the equation below can be obtained:

$$d\gamma = -RTT[d\ln(C + C_S) + d\ln C] \tag{4}$$

On the other hand, surface excess, Γ, can be related to the surfactant bulk concentration by Langmuir isotherm:

$$\Gamma = \Gamma_m \frac{K_L C}{1 + K_L C} \tag{5}$$

where Γ_m is the highest interfacial concentration attainable and relates to the level of decrease in IFT; and K_L is the Langmuir equilibrium adsorption constant. Introducing Γ to Eq. (4) and then integrating, gives the following applicable surface equation of state (EOS) [11]:

$$\gamma = \gamma_0 - \left(\frac{RT\Gamma_m}{K_L C_s 1}\right) \times \left[(K_L C_s - 2)\ln(K_L C_s + 1) + K_L C_s \ln\left(\frac{C + C_s}{C_s}\right)\right] \tag{6}$$

Having Γ_m, one can calculate minimum area occupied by a molecule at interface, A_m, as:

$$A_m = \frac{1}{\Gamma_m N_A} \tag{7}$$

where N_A is Avogadro's number.

Also, The relevant Gibbs free energy of adsorption, ΔG_{ads}, can be obtained which reflects the adsorption tendency for each condition [11,36]:

$$\Delta G_{ads} = -RT \ln K_L \tag{8}$$

The experimental IFT data were fitted to Eq. (6) with nonlinear regression using Table 2D software. The results together with the

Table 3. Adsorption parameters at different used salt concentrations

Salt	C_s (mol/dm^3)	K_L (m^3/mol)	$\Gamma_m \times 10^6$ (mol/m^2)	A_m (nm^2)	ΔG_{ads} (kJ/mol)	R^2
No salt	0.000	9.76	1.70	0.98	−5.55	0.9915
NaCl	0.010	138.52	1.72	0.97	−12.02	0.9951
	0.025	190.86	1.80	0.92	−12.80	0.9941
	0.075	244.36	1.93	0.86	−13.40	0.9801
KCl	0.010	150.81	1.73	0.96	−12.23	0.9969
	0.025	207.47	1.83	0.91	−13.00	0.9970
	0.075	264.96	2.01	0.82	−13.60	0.9900
KI	0.010	102.18	2.00	0.83	−11.28	0.9992
	0.025	109.86	2.40	0.69	−11.45	0.9972
	0.075	145.23	2.74	0.61	−12.13	0.9970
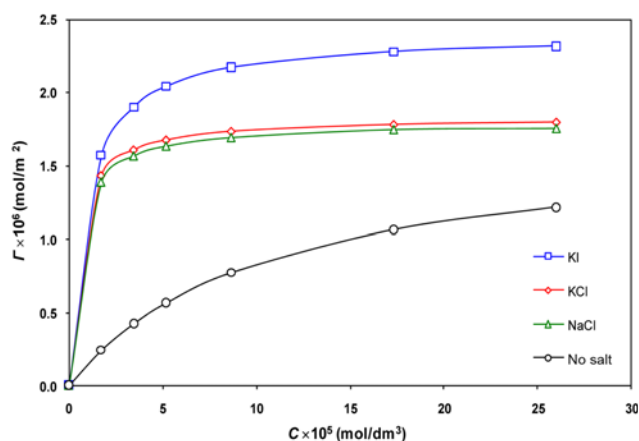
Fig. 5. The surface excess as a function of SDS concentration at constant concentration of 0.075 mol/dm^3 of different salts.

Fig. 6. Variation of IFT versus for adsorption of SDS with different concentrations of NaCl and KCl.

coefficients of determination (R^2) are listed in Table 3. The high R^2 values (0.9801-0.9992) indicate nice agreement of Eq. (6) with the experimental data. The values of Γ_m and K_L for no salt system are 1.70×10^{-6} mol/m^2 and 9.76 m^3/mol, close to the previously reported values of 1.78×10^{-6} mol/m^2 and 9.9 m^3/mol, respectively [11]. These values rise drastic (more than 10 times) when salts are present, confirming compatibility of the used EOS with experimental data with respect to the above discussion. The highest obtained Γ_m values for the case of KI, for instance, is due to the lowest reached level of IFT with this salt.
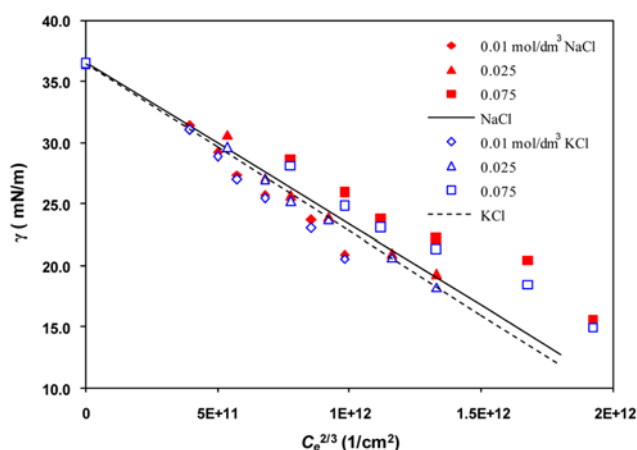
To have more sight into salt effect, relevant Γ values were obtained from Langmuir Eq. (3) and versus surfactant concentration is depicted in Fig. 5, typically for the maximum salt concentration of 0.075 mol/dm^3. The order of surface excess follows the same order of salts effectiveness. Same variations are also relevant when the salt concentration increases, according to the aforesaid discussions.

Flat zones of Γ curves represent the interface concentration near saturation by surfactant molecules. As is obvious, surface saturation occurs at a lower surfactant concentration when salts are present.

The effectiveness of a counterion on the surfactant adsorption can also be studied by the proposed adsorption isotherm by Ivanov et al. [28], which relates IFT to an effective concentration via equation:

$$\gamma = \text{const} - 3k_B T K C_e^{2/3} \tag{9}$$

valid for low coverage. In this equation, k_B, T and K are Boltzmann

constant, temperature and adsorption constant (independent of counterion concentration), respectively. The constant in this equation is close to, but not exactly equal to the IFT value of pure liquids (without surfactant and salt). Also, C_e is the effective concentration, introduced by

$$C_e = \sqrt{C(C + C_s)} \tag{10}$$

Eq. (9) together with Eq. (10) helps to analyze a system in the presence of both surfactant and salt. The value of K can be obtained from the linear dependency of IFT values to $C_e^{2/3}$. The higher slope indicates the higher ability of a counterion to affect surfactant adsorption at the interface. The IFT data in this work are presented in Fig. 6, according to Eq. (9). Data points are located around two distinct linear trend lines with R^2 values of 0.9835 and 0.9916, for NaCl and KCl, respectively. The corresponding obtained K values are 108.8 and 112.9 (dimensionless), respectively. The relevant intercept values (constant in Eq. (9)) are also 36.6 and 36.5 mN/m, respectively. As expected, the adsorption constant relevant to KCl is higher, in agreement with the general order of K_L values, given in Table 3.

CONCLUSIONS

The IFT variation of toluene-water system in the presence of both SDS surfactant and each of uni-univalent salts, NaCl, KCl and KI,

was investigated. It was found that the presence of these salts can severely enhance the SDS action in reducing interfacial tension. This influence is profound with low surfactant and salt dosages. The order of salts influencing relative effectiveness on IFT reduction appeared as KI>>KCl>NaCl. This behavior can be attributed to innate properties of salts in terms of lattice energy and the tendency of salt ions for making ion-pairs. Accordingly, these findings imply that mass transfer in liquid-liquid extraction process is significantly hindered when a contaminated system is accompanied with a level of aqueous phase salt concentration.

The combined equation of the Gibbs adsorption equation and the Langmuir isotherm can nicely be used for modeling the IFT variations, while the salt concentration is considered at the same time. A high coefficient of determination was accordingly achieved by adjusting the two relevant parameters. The maximum interfacial concentration (Γ_m) obeys reasonably the same order of salts effectiveness, and the Langmuir adsorption constant (K_L) increases with increase of salt concentration. The more effectiveness of K^+ counterion in comparison to Na^+ was also consistent in demonstrating the slope of general linear variation of interfacial tension versus an effective concentration according to the model developed by Ivanov et al. [28].

ACKNOWLEDGEMENTS

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