Variations of Interfacial Tension of the *n*-Butyl Acetate + Water System with Sodium Dodecyl Sulfate from (15 to 22) °C and pH between 6 and 9

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Interfacial tension of *n*-butyl acetate + water + sodium dodecyl sulfate is reported over the aqueous surfactant concentration range of (0 to $34.68 \cdot 10^{-5}$) $\text{mol} \cdot \text{L}^{-1}$, the temperature range of (15 to 22) °C, and the pH of 6 to 9. The measurements were performed by the drop-weight method, using a narrow capillary with very thin thickness. The interfacial tension values ranged from (15.8 to 4.5) $\text{mN} \cdot \text{m}^{-1}$, and the data show a good agreement with Szyszkowski's equation as the surfactant concentration varies. The variation of interface concentration and the area occupied by each molecule of surfactant shows that surface coverage decreases as the pH increases. The interfacial pressure, entropy, and enthalpy values are derived, and their variations are investigated versus appropriate parameters.

Introduction

Interfacial tension plays an important role in interphase mass and heat transfer. It is defined as the work to create a unit of new surface between two immiscible fluids.[1] This parameter affects the hydrodynamics and contact of phases for mass transfer purposes, and therefore one of the physical properties that must be known by chemical engineers for the design of liquid–liquid contactors is interfacial tension. Interfacial tension also plays an important role in improved oil recovery.

Contaminants are usually present to an unknown extent in industrial materials. They accumulate at the interface between phases, inhibit circulation within the drops, cause hydrodynamic and adsorptive barriers to transfer across the interface, and change the pattern of drop behavior. The interfacial tension data for pure (or clean) systems are rich in the literature; however, those with surfactants (as examples of impurity or contamination) are few.

On the other hand, the pH of aqueous solutions in contact with organic phases can alter this property. In liquid–liquid extraction, the pH of the water, used as the aqueous phase, and the solute transferred from one phase to another can provide alternatives in this case.

The temperature dependence of interfacial tension is also an important case to enhance the process efficiency or when thermal variations are present along an extraction column or during the process.

This study presents data on interfacial tension of the phases of *n*-butyl acetate + water, a recommended chemical system by the EFCE working party.[2] This chemical system is frequently used[3–6] as a system with intermediate interfacial tension in liquid–liquid extraction investigations. Solutions of surfactant sodium dodecyl sulfate (SDS), as simulating industrial contaminants,[7,8] are used in contact with *n*-butyl acetate, and the effects of temperature and pH are to be examined for each case.

The values of interfacial pressure for this system can also be obtained from interfacial tension data of the clean chemical system and that of surfactant solution.

Table 1. Interfacial Tension of Some Chemical Systems at 25 °C

chemical system	$\gamma/\text{mN} \cdot \text{m}^{-1}$ exptl	$\gamma/\text{mN} \cdot \text{m}^{-1}$ lit.
n-pentanol−water	5.6	5.5[a]
n-hexanol−water	7.2	6.8[b]
n-octanol–water	7.8	8.5[b]
dichloromethane−water	27.2	26.9[a]
chloroform−water	29.2	29.7[a]
toluene−water	35.7	36.1[b]

[a] Ref 18. [b] Ref 19.

Experimental Section

N-Butyl acetate and sodium dodecyl sulfate were Merck products with purities of more than 99.5 % and 99 %, respectively, and were used as received. Laboratory-distilled water was redistilled prior to experimentation.

The level of purity of sodium dodecyl sulfate was assessed by obtaining its critical micelle concentration in aqueous solution at 20 ± 0.1 °C, using the conductometric method.[9,10] The critical micelle concentration, obtained from the variation of specific conductivity against the concentration of SDS, was 0.00835 $\text{mol} \cdot \text{L}^{-1}$, close to the value reported in the literature[9] (0.00825 $\text{mol} \cdot \text{L}^{-1}$).

Aqueous surfactant (SDS) solutions were prepared by mass, using a METTLER AE-100 balance with an uncertainty of ± 0.1 mg. After preparing the main solution, the desired next solutions were obtained by successive dilutions. The uncertainty in concentration of SDS was estimated to be within $\pm\, 0.02 \cdot 10^{-5}$ $\text{mol} \cdot \text{L}^{-1}$.

To adjust the pH, NaOH solutions (0.1 M), supplied by Merck, were used to reach the desired pH value in the aqueous phase, having the original pH value of 5.9. The measurements of pH values were performed using a CORNING-M140 pH meter, with an uncertainty of ± 0.01.

The drop-weight method which is a reproducible method and has been used by other investigators[11–14] was used to determine the interfacial tension of the samples. The drop-forming device and the procedure were similar to that described by our previous works.[10,13] A glass capillary (outer diameter of 0.65 mm) with a finely ground tip to give an angle of 90° between the ground

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10.1021/je700622n CCC: $40.75

Table 2. SDS Concentration C, Phase Densities ρ, and Interfacial Tension γ of System

				γ/mN·m^{-1}					
C/mol·L^{-1}	t/°C	ρ/kg·m^{-3}	ρ/kg·m^{-3}	pH = 6	pH = 6.5	pH = 7	pH = 7.5	pH = 8	pH = 9
0	15	998.97	887.95	15.8	15.6	15.5	15.4	15.2	15.0
	16	998.81	886.94	15.6	15.4	15.3	15.2	15.0	14.7
	17	998.63	885.92	15.4	15.2	15.1	14.9	14.7	14.4
	18	998.44	884.89	15.1	15.0	14.9	14.7	14.4	14.2
	20	998.04	882.84	14.7	14.5	14.2	14.1	14.0	13.7
	22	997.59	880.77	14.3	14.1	14.0	13.8	13.6	13.4
$2.17 \cdot 10^{-5}$	15	998.97	888.07	15.3	15.1	14.9	14.8	14.6	14.5
	16	998.80	887.06	15.0	14.8	14.7	14.6	14.4	14.2
	17	998.63	886.04	14.8	14.6	14.5	14.4	14.2	13.9
	18	998.44	885.02	14.6	14.4	14.3	14.2	14.0	13.7
	20	998.04	882.97	14.2	14.0	13.9	13.8	13.6	13.4
	22	997.59	880.92	13.9	13.7	13.6	13.5	13.3	13.1
$4.33 \cdot 10^{-5}$	15	998.97	888.11	14.4	14.3	14.2	14.0	13.9	13.6
	16	998.81	887.10	14.3	14.1	14.0	13.8	13.7	13.4
	17	998.64	886.08	14.1	13.9	13.7	13.6	13.4	13.2
	18	998.45	885.06	13.9	13.7	13.5	13.4	13.3	13.0
	20	998.04	883.01	13.6	13.4	13.3	13.2	13.0	12.7
	22	997.59	880.96	13.2	13.1	13.0	12.8	12.7	12.5
$8.67 \cdot 10^{-5}$	15	998.98	888.06	12.7	12.4	12.3	12.1	12.0	11.7
	16	998.82	887.03	12.4	12.2	12.1	11.8	11.7	11.4
	17	998.64	886.01	12.2	11.9	11.8	11.6	11.4	11.1
	18	998.45	884.98	11.9	11.7	11.4	11.3	11.1	10.8
	20	998.04	882.93	11.4	11.2	10.9	10.7	10.6	10.3
	22	997.60	880.88	11.0	10.7	10.6	10.4	10.1	9.8
$17.34 \cdot 10^{-5}$	15	998.98	888.07	10.3	10.2	10.0	9.9	9.7	9.4
	16	998.81	887.05	10.0	9.9	9.7	9.5	9.4	9.1
	17	998.64	886.02	9.8	9.6	9.4	9.2	9.0	8.8
	18	998.45	885.00	9.5	9.3	9.1	8.9	8.7	8.5
	20	998.05	882.95	9.1	8.9	8.7	8.5	8.3	8.0
	22	997.60	880.90	8.7	8.5	8.4	8.1	7.9	7.5
$34.68 \cdot 10^{-5}$	15	998.99	888.04	7.2	7.0	6.8	6.7	6.4	6.2
	16	998.82	887.02	6.9	6.8	6.5	6.4	6.1	5.8
	17	998.65	886.00	6.7	6.5	6.2	6.0	5.8	5.6
	18	998.46	884.97	6.5	6.2	6.0	5.8	5.6	5.3
	20	998.05	882.92	6.1	5.9	5.6	5.4	5.2	4.9
	22	997.61	880.87	5.7	5.5	5.2	5.0	4.8	4.5

face and the internal bore at the end with the edges sharp was used. This thickness was very small, and the formation of a neck between the drop and the capillary tip was not observed under the conditions used in this study; otherwise, this phenomenon should be accounted for to obtain accurate values of interfacial tension.[15] The n-butyl acetate phase was held in a narrow glass syringe conducted by an adjustable syringe pump (Phoenix M-CP, French) and flowed through a rigid tube to the capillary in the stagnant aqueous phase. A very low flow rate (1 mL in 31.15 min, determined from the flow rate indication when calibrated by measuring the time of syringe piston movement) of the organic phase was conducted to the capillary, and drops were formed very slowly at the tip of capillary.

Each drop volume was obtained from at least three measurements of time where the maximum deviations from the average value were less than ± 0.3 %. The whole aqueous media and conducting tube were thermostatted with an uncertainty of ± 0.1 °C, using an adjustable safely thermostat (OPTIMA 740, Japan).

The aqueous phase with the known concentration of SDS and the value of pH in contact with the organic phase was kept in shaking at the appropriate temperatures in a thermostatted vessel during a period of at least four hours and then leaving at rest for an hour to obtain the equilibrium. Samples of the organic and the aqueous phases were withdrawn to measure the density and the interfacial tension.

The densities of the phases were measured at different temperatures using a density meter (Anton Paar DMA 4500, Austria) with an uncertainty of ± 0.05 kg·m^{-3}. The desired temperature is self-adjusted in this density meter with an uncertainty of ± 0.01 °C.

The experiments were carried out at different temperatures for each sample of aqueous solution, containing the corresponding pH value. New main SDS solutions were prepared for each set of experiments with a specified pH and SDS concentration and were used in successive experiments with different temperatures.

Calculation of Interfacial Tension. The details of calculations are given in our previous works.[10,13] The relationship between interfacial tension (γ) and the appropriate parameters is given by Harkins and Brown.[16]

$$\gamma = \frac{v \Delta \rho g}{r} \varphi \tag{1}$$

where $\Delta\rho$ is the density difference between the aqueous and organic liquids (ρ_a and ρ_o); r is the capillary radius; and φ is a constant which should be obtained from the tables of Harkins and Brown[16] and are correlated in an empirical equation by Drelich et al.[17]

$$\varphi = 0.167 + 0.193\left(\frac{r}{\sqrt[3]{v}}\right) - 0.0489\left(\frac{r}{\sqrt[3]{v}}\right)^2 - 0.0496\left(\frac{r}{\sqrt[3]{v}}\right)^3 \tag{2}$$

To examine the performance and reliability of the method, the interfacial tension of several pure chemical systems (Merck products with purities more than 99.5 %), saturated with water at 25 °C, were measured (without surfactant) and compared with

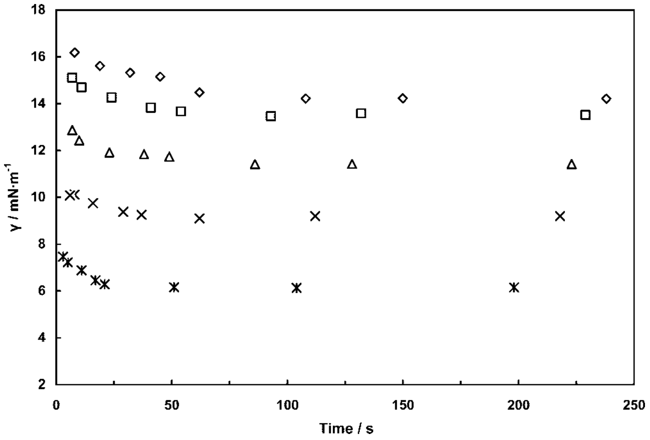

Figure 1. Interfacial tension of the system as a function of drop formation time at pH 6 and 20 °C, for different SDS concentrations: ◇, $2.17 \cdot 10^{-5}$ mol·L^{-1}; □, $4.33 \cdot 10^{-5}$ mol·L^{-1}; △, $8.67 \cdot 10^{-5}$ mol·L^{-1}; ×, $17.34 \cdot 10^{-5}$ mol·L^{-1}; ∗, $34.68 \cdot 10^{-5}$ mol·L^{-1}.

those from the literature. The results are given in Table 1. The average deviations for N data points is given by

$$\%\mathrm{AD} = 100 \cdot \left[\sum_{i=1}^{N} \frac{|\gamma_{i,\mathrm{exptl}} - \gamma_{i,\mathrm{lit}}|/\gamma_{i,\mathrm{lit}}}{N} \right] \qquad (3)$$

where N, $\gamma_{i,\mathrm{exptl}}$, and $\gamma_{i,\mathrm{lit}}$ are, respectively, the number of data, the obtained interfacial tension, and the appropriate interfacial tension reported in the literature.[18,19] This value is 3.3 % for six data points, investigated in this work. The uncertainty in measurement results is estimated to be within ± 0.1 mN·m^{-1}.

Results and Discussion

Experimental Results. Six concentrations of SDS ranging from (0 to $34.68 \cdot 10^{-5}$) mol·L^{-1} were tested for six temperatures of (15 to 22) °C and six pH values of 6 to 9, within the estimated practical range of applications, resulting in 216 interfacial tension data. The interfacial tension data measured for the *n*-butyl acetate + water system at various temperatures, surfactant concentration, and pH are listed in Table 2 along with the corresponding density of phases. The values are in the range (15.8 to 13.4) mN·m^{-1} for a clean system and in the range (15.3 to 4.5) mN·m^{-1} for a contaminated system. Drop volumes within (107.1 to 142.8) mm^3 were generated.

To ensure the achievement of equilibrium interfacial tension, the variation with the drop formation time (obtained at different *n*-butyl acetate flow rates) was investigated. The results are shown by Figure 1 for different SDS concentrations at pH 6 and 20 °C, for instance. When the drops are formed very rapidly, the surfactant does not have sufficient time to be adsorbed at the interface. The interfacial tension is therefore reflecting an unsteady-state interface. As the time increases, the surfactant continues to accumulate and orientate itself at the interface. Finally, when the time of drop formation is very long (more than 90 s in this work) the interface becomes saturated and interfacial tension remains constant with no significant change for the further time. A similar behavior has been reported by Mollet et al.[20] The last point for each SDS concentration in Figure 1 corresponds to the measured value reported in this work. The range of drop formation time in this work was from (186 to 253) s.

Figure 2 shows the typical surfactant effects on the interfacial tension of system. At pH value of 7 and temperature of 20 °C, for instance, the interfacial tension decreases from (14.3 to 5.6) mN·m^{-1} by adding the surfactant. The trend of variation is nonlinear, in agreement with other works[10,13,21] and very low amounts of surfactant cause significant reduction in interfacial tension of the system.

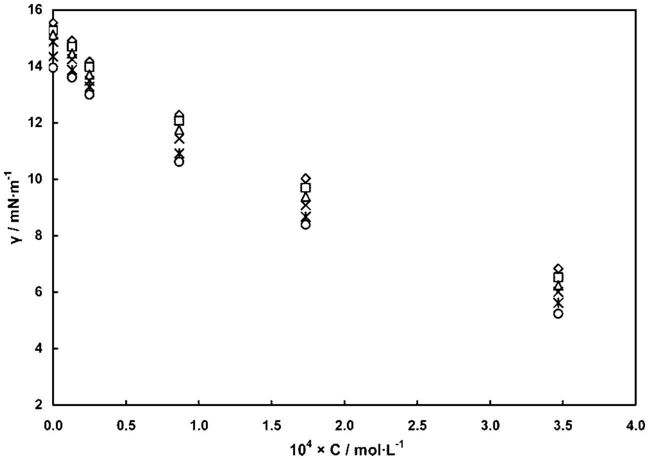

Figure 2. Interfacial tension of the system as a function of SDS concentration at pH 7 and different temperatures: ◇, 15 °C; □, 16 °C; △, 17 °C; ×, 18 °C; ∗, 20 °C; ○, 22 °C.

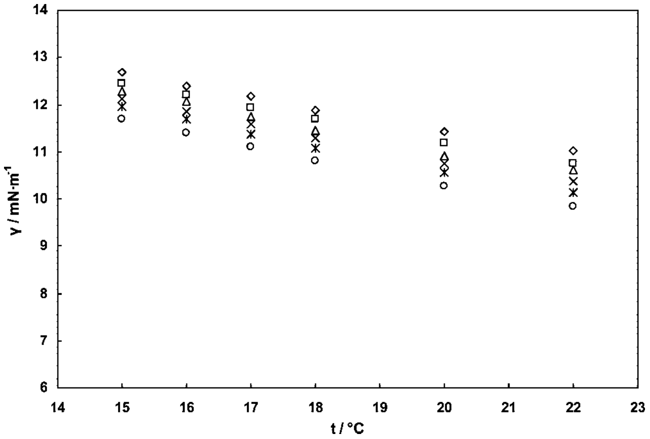

Figure 3. Interfacial tension of the system as a function of temperature at different pH values and SDS concentration of $8.67 \cdot 10^{-5}$ mol·L^{-1}. ◇, pH 6; □, pH 6.5; △, pH 7; ×, pH 7.5; ∗, pH 8; ○, pH 9.

The increase of temperature from (15 to 22) °C results in lowering of the interfacial tension when surfactant concentration and pH are fixed. Figure 3 shows that the trend is almost linear within the temperature range used. The interfacial tension finds an average decrease of 1.7 mN·m^{-1} in this temperature range with an SDS concentration of $8.67 \cdot 10^{-5}$ mol·L^{-1}, for instance.

The values show that interfacial tension decreases as the pH increases at a constant temperature and surfactant concentration. The change in interfacial tension is almost linear as a typical variation as shown by Figure 4. This variation can provide a large drop size produced in acidic aqueous solutions compared with basic solutions in contact with the *n*-butyl acetate phase. It is notable that the influence of pH is much less than that of the surfactant (maximum reduction of 1.3 mN·m^{-1} compared with 8.9 mN·m^{-1}, while other relevant parameters are constant for each variation) within the used variation ranges.

Modeling. The variation of obtained interfacial tension with SDS concentration follows the Szyszkowski equation very well. For surfactant concentrations below the CMC, the equilibrium surface concentration, Γ, on the interface can be estimated by the Gibbs adsorption equation

$$\Gamma = -\frac{1}{nRT}\left(\frac{d\gamma}{d\ln C}\right) \tag{4}$$

where R is the gas constant; T is the temperature; and C is the bulk concentration of surfactant. The factor n represents the number of species that consist of the surfactant and adsorb at the interface. For an ionic surfactant like SDS, $n = 2$. On the other hand, for the correlation between surface concentration and the bulk concentration, the Langmuir isotherm can be used

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

where Γ_m is the maximum surface concentration and K_L is the Langmuir equilibrium adsorption constant. The corresponding surface equation of state is the Szyszkowski equation which is obtained from the combination of eqs 4 and 5

$$\gamma = \gamma_o - nRT\Gamma_m \ln(1 + K_L C) \tag{6}$$

where γ_o is the interfacial tension of pure phases without surfactant. The experimental data were fitted to eq 6 by nonlinear regression, and the obtained Γ_m and K_L values are listed in Table 3. The difference between calculated and experimental data was obtained in terms of the so-called coefficient of determination $(R^2)^{22}$

$$R^2 = 1 - \frac{\sum_{i=1}^{N} (\gamma_{\text{calcd}} - \gamma_{\text{exptl}})^2}{\sum_{i=1}^{N} (\bar{\gamma} - \gamma_{\text{exptl}})^2} \tag{7}$$

where N, γ_{cald}, γ_{exptl}, and $\bar{\gamma}$ are, respectively, the number of data used in the fit, the interfacial tension calculated by the model, the experimental interfacial tension, and the average of all the appropriate experimental values in the fit. The values of R^2 (0.9833 to 0.9998) are also given in Table 3, showing the goodness of fitting.

Using eq 6 provides a maximum deviation less than ± 0.7 % and an average deviation of ± 0.14 %. The results show that both the pH of the aqueous phase and the concentration of sodium dodecyl sulfate influence the relationship between interfacial tension and temperature.

The minimum area occupied by an SDS molecule (A) at interface was also calculated using[23]

$$A = \frac{1}{\Gamma_m N_{Av}} \tag{8}$$

where N_{AV} is Avogadro's number. The calculated A values are listed in Table 3. There is an average occupied area of $111.0 \cdot 10^{-20}$ m^2 for each molecule, within the conditions used. Increasing pH provides a reduction in this area.

The surface concentration (Γ) values were calculated by the Langmuir equation (eq 5). The Γ value increases with increasing surfactant concentration (C), tending to a constant value of the maximum surface concentration (Γ_m). Figure 5 shows the variation based on average Γ_m and K_L values, obtained at different corresponding temperatures. Comparison of variations shows that the surface concentration increases as the pH increases; however, the difference is very low at low surfactant concentrations.

The interfacial pressure of surfactant solutions is expressed as $\Pi = \gamma_o - \gamma$ for which the obtained values for this system are within (0.33 to 8.93) mN·m^{-1}. The results show that the interfacial pressure can be considered independent of pH and temperature, within the range used in this work, compared with the very significant influence of SDS concentration. Presented in Figure 6 is the variation of interfacial pressure of the system as a function of surfactant concentration for different temperature and pH values. The variation of this parameter can be clearly expressed by eq 6; however, irrespective of the influence of pH and temperature, the whole data can be expressed through the empirical equation

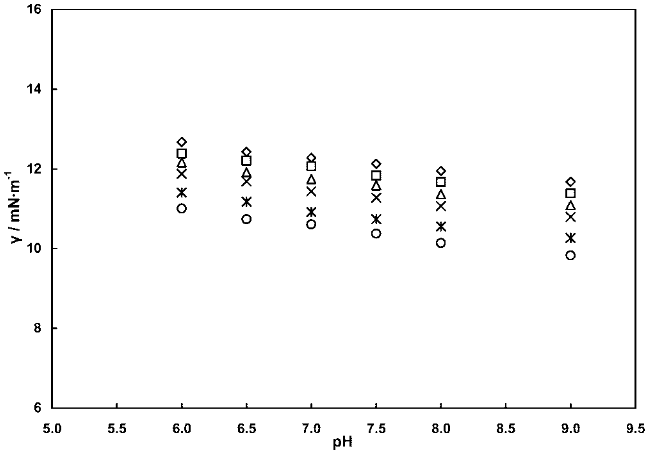

Figure 4. Interfacial tension of the system as a function of pH at different temperatures and SDS concentration of $8.67 \cdot 10^{-5}$ mol·L^{-1}: ◇, 15 °C; □, 16 °C; △, 17 °C; ×, 18 °C; ∗, 20 °C; ○, 22 °C.

Table 3. Interfacial Tension Parameters Γ_m and K_L (Equation 6), the Coefficients of Determination R^2, and the Area Occupied by an SDS Molecule A (Equation 8) for the System

pH	t / °C	$10^6 \cdot \Gamma_m$ / mol·m^{-2}	K_L / m^3·mol^{-1}	R^2	$10^{20} \cdot A$ / m^2
6	15	1.49	6.63	0.9833	111.3
	16	1.51	6.60	0.9990	110.0
	17	1.49	6.80	0.9992	111.7
	18	1.44	7.08	0.9993	115.5
	20	1.45	6.85	0.9996	114.6
	22	1.45	6.85	0.9995	114.9
6.5	15	1.50	6.62	0.9987	110.5
	16	1.48	6.88	0.9990	112.5
	17	1.44	7.24	0.9993	115.0
	18	1.43	7.34	0.9994	116.2
	20	1.45	6.93	0.9998	114.9
	22	1.47	6.67	0.9991	113.0
7	15	1.47	6.99	0.9869	113.0
	16	1.54	6.50	0.9992	107.7
	17	1.42	7.61	0.9990	116.8
	18	1.38	7.93	0.9994	120.2
	20	1.54	6.23	0.9987	107.6
	22	1.61	5.87	0.9985	103.4
7.5	15	1.50	6.77	0.9987	110.6
	16	1.43	7.45	0.9929	116.0
	17	1.51	6.88	0.9992	110.3
	18	1.45	7.30	0.9995	114.2
	20	1.52	6.47	0.9986	109.0
	22	1.51	6.65	0.9987	110.0
8	15	1.52	6.69	0.9989	109.0
	16	1.54	6.56	0.9993	107.6
	17	1.52	6.79	0.9996	109.1
	18	1.56	6.44	0.9992	106.6
	20	1.56	6.37	0.9984	106.8
	22	1.51	6.67	0.9979	109.7
9	15	1.55	6.52	0.9987	106.9
	16	1.58	6.39	0.9992	105.1
	17	1.57	6.42	0.9996	106.0
	18	1.53	6.73	0.9995	108.6
	20	1.52	6.66	0.9983	109.2
	22	1.49	7.03	0.9972	111.8

$$\Pi/\text{mN}\cdot\text{m}^{-1} = 14.01\{1\ \exp(-2.87\cdot 10^{3}C/\text{mol}\cdot\text{L}^{-1})\} \tag{9}$$

Using this equation, the standard deviations were found to be less than 0.3 $\text{mN}\cdot\text{m}^{-1}$ for 180 data points and the mean deviation of within ± 6.7 %. The coefficient of determination is 0.9901, and the lower surfactant concentrations provide the higher deviations. Figure 6 shows the agreement between measured and predicted values.

Thermodynamic properties of the interface of the system can be obtained by the following equations. The excess interface entropy per unit area or variation of entropy per unit area due to interface formation[24,25] is

$$S^{\text{I}} = -\frac{\text{d}\gamma}{\text{d}T} \tag{10}$$

and the interfacial enthalpy is

$$H^{\text{I}} = \gamma - T\left(\frac{\text{d}\gamma}{\text{d}T}\right) \tag{11}$$

The interfacial entropies and the enthalpies of the system were calculated from eqs 10 and 11 and drawn in Figures 7 and 8 vs the temperature for the case of no surfactant present. The trend in both variations shows almost a linear decrease with temperature in the temperature range of (15 to 22) °C. The decrease in interfacial enthalpy means that the energy required for interface formation decreases as the temperature increases. This case is favorable in liquid–liquid operations with respect to energy consumption. The influence of pH in this regard is rather complicated as is presented in Figures 7 and 8.

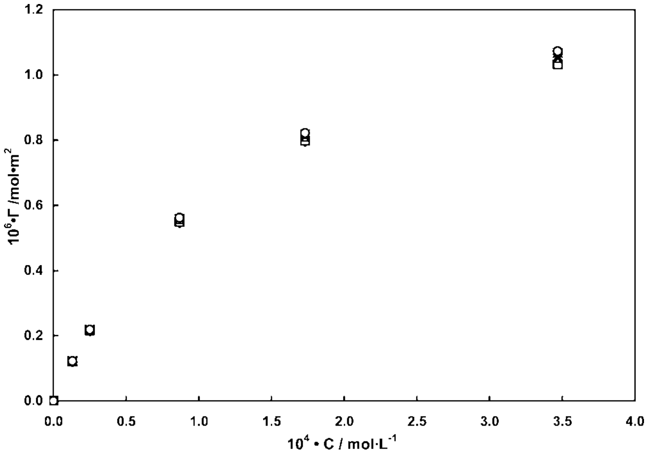

Figure 5. Interfacial concentration of SDS for the system as a function of its concentration at different pH values: ◇, pH 6; □, pH 6.5; △, pH 7; ×, pH 7.5; ∗, pH 8; ○, pH 9.

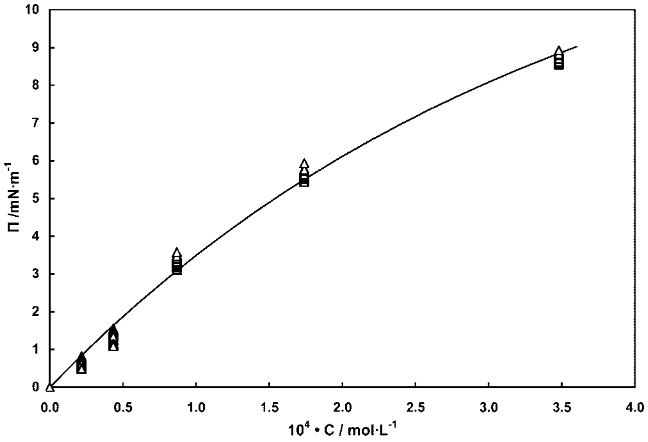

Figure 6. Variation of interfacial pressure of the system as a function of SDS concentration for different temperature and pH values. Line represents eq 9.

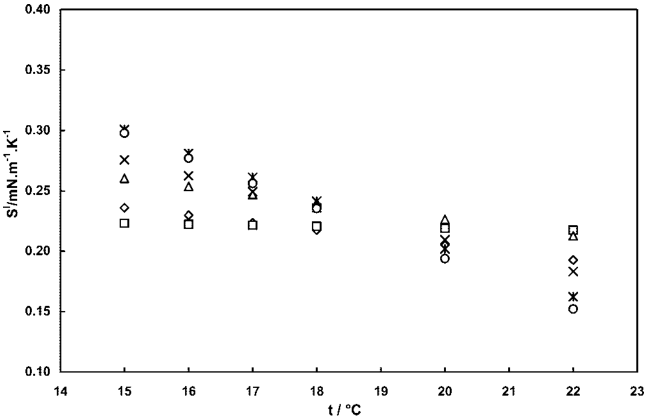

Figure 7. Interfacial entropy of the system as a function of temperature at different pH values, without surfactant: ◇, pH 6; □, pH 6.5; △, pH 7; ×, pH 7.5; ∗, pH 8; ○, pH 9.

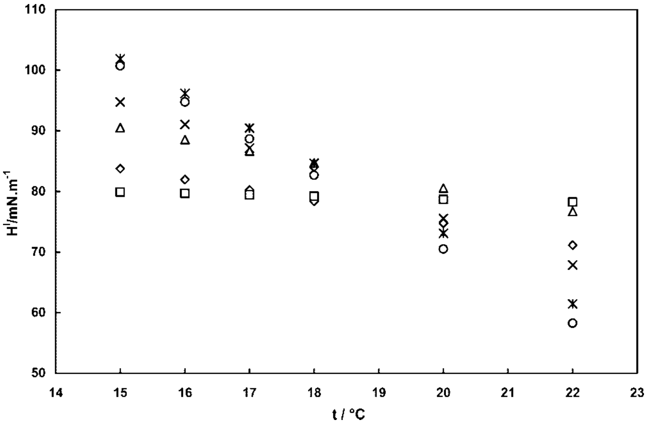

Figure 8. Interfacial enthalpy of the system as a function of temperature at different pH values, without surfactant: ◇, pH 6; □, pH 6.5; △, pH 7; ×, pH 7.5; ∗, pH 8; ○, pH 9.

Conclusions

New data for the interfacial tension of the chemical system of *n*-butyl acetate + water + sodium dodecyl sulfate were determined in the temperature range of (15 to 22) °C, pH 6 to 9, and surfactant concentration range of $(0 \text{ to } 34.68\cdot 10^{-5})$ $\text{mol}\cdot\text{L}^{-1}$. The experimental apparatus was based on the drop weight method, using a narrow capillary with very thin thickness. The increase of all investigated parameters caused reduction in interfacial tension with the superior influence of surfactant concentration. The generated data can be correlated very well with the Szyszkowski equation (a maximum deviation less than ± 0.7 % and an average deviation of ± 0.14 %). The appropriate parameters for the maximum surface concentration and adsorption constant were obtained with fitting to each series of experimental data under constant pH and temperature. Investigations on the surface concentration show that the surface concentration increases as the pH increases; however, this influence is very low under low surfactant concentrations. The interfacial pressure data were derived and correlated adequately with an empirical equation. With thermodynamic investigations, a general linear reduction in interfacial entropy and enthalpy was observed, within the range of temperatures.

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Received for review October 25, 2007. Accepted December 28, 2007. We express thanks to the university authorities for providing the financial support to carry out this work.

JE700622N