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Mass Transfer and Zeta Potential in Solvent Extraction

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ABSTRACT

The initial mass transfer rate of acetic acid from the aqueous to the organic phase with tri-*n*-octylamine and toluene or isododecane as solvents has been studied in a constant interfacial cell area as a function of the stirring speed, concentrations of the transfer species, and the influence of chemical adsorption layers. For a detailed understanding of the mass transfer process, the electrophoretic mobility of oil-in-water droplets for these systems has been measured with a ZETA-sizer. The electrophoretic mobilities of the different reactive extraction systems were investigated for their dependence on the pH value and with different interfacial charges. It was proved that a change of the interfacial charge influences the zeta potential of the dispersed oil drops in the same way as the reaction kinetics, and thus the mass transfer of acetic acid.

Key Words. Mass transfer; Interfacial charge; Zeta potential; Electrophoretic mobility; Surfactants; Adsorption layers

INTRODUCTION

For the design of the technical extraction apparatuses (mixer-settler cascades or extraction columns) it is necessary to know two primary facts: the thermodynamic equilibrium behavior and the time-dependent mass transfer behavior. In the case of acetic acid a lot of information is available on the extraction equilibria in amine systems (1, 2). Very little information is available

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on the kinetics of acetic acid extraction, on the reaction mechanism, and on the rate-determining steps of the extraction process. Two different reaction mechanisms are proposed in the literature for the reaction kinetics: Siebenhofer (3) described a protonation of tri-*n*-octylamine before the anion can be extracted, and Tamada (4) proposed a direct reaction of tri-*n*-octylamine with the undissociated acid. When the interface is defined as the place of reaction the overall mass transfer can be generally limited by diffusion or by reactions (5).

However, when considering the extraction of ions at the liquid–liquid interface, the separation of the charges becomes important. Except at the isoelectrical point, the charge of the interfacial zone is either positive or negative. As a result, in addition to the chemical potentials there exists an electrical potential difference. When comparing different charged adsorption media, it is evident that electrostatic interactions have to be taken into account because the charge at the interface has a strong influence on the reaction kinetics as far as adsorption layers are involved (6–8). The influence of different ionic surfactants on the liquid–liquid equilibria has also been examined. By comparison between these and surfactant-free systems (1, 2), it was seen that there is no influence of surfactants on the extraction equilibria (7). Thus, the presence of a loaded organic/aqueous interface changes the interfacial concentration of the extractable aqueous species and therefore the flux of the acetic acid. Osseo-Asare and Lin described similar effects for other chemical systems (9): reaction inhibition is to be expected in the presence of an interface with the same charge as the extractable species (e.g., the Ni^{2+}/HDNNS–Aliquat 336 system). In opposition to this, an increase in extraction rate is to be expected when the aqueous transfer species and the interface are oppositely charged (e.g., the Ni^{2+}/HDNNS-LIX 63 system).

The present work is a systematic investigation on the mechanism of organic acid extraction, with acetic acid as the example. The enhancement and reduction of the initial mass transfer rate when using neutral, cationic, and anionic surfactants have been investigated in detail by considering the interfacial charge, especially the zeta potential.

EXPERIMENTAL

Chemicals

Acetic acid (Rotipuran, >99.7%), sodium hydroxide (Riedel de Haen, 1 M), sodium acetate (Riedel de Haen, >99%), and distilled water made up the aqueous phase. Tri-*n*-octylamine (Alamine 300, Henkel, >95%) in toluene (Riedel de Haen, >99.7%) or isododecane (Bayer, 2,2,4,6,6-pentamethylheptane, >98%) constituted the organic phase. The adsorption layers were generated with sodium dodecyl sulfate (Merck, >99%, anionic surfactant), 1-dodecanesulfonic acid sodium salt (Fluka, >98%, anionic surfactant), dodecyltrimethylammonium chloride (Fluka, >98%, cationic surfactant) and Triton X-100 (Boehringer Mannheim, octylpoly(ethylene glycol ether)$_{10}$, >99%, nonionic surfactant). Tri-*n*-octylamine was washed with 5 mM NaOH as a purification step. All other reagents were standard chemicals of analytical grade and were used without any further purification.

Analytics

The aqueous and organic samples were weighted with a Sartorius R300S and the acetic acid concentration was determined using an autotitrator (Orion 940/960) with NaOH. The interfacial tension γ of the presaturated phases was observed by the volume drop method with a tensiometer (TVT1, Lauda).

Kinetic Experiments

The kinetics experiments were carried out in a modified stirred Lewis-cell after Nitsch at 25°C (10, 11). The volume ratio of the aqueous to the organic phase was 1:1, with a total volume of 796 mL and an interface area of 44.2 cm^2. The experiments were started with the injection of the initial acetic acid concentration in the aqueous phase by a dosimate (Methrom 725) and a simultaneous start of the impellers (both at the same Reynolds number in each phase). The concentration–time curves were measured by taking samples of the organic phase with syringes. Samples were taken from the aqueous and the organic phases at the end of every experiment to control the mass balances.

Electrophoretic Mobility EM and Zeta Potential ζ

The organic phase (100 μL) was added to a 200-mL electrolyte solution (4 mM sodium acetate), and the pH value was adjusted with concentrated acetic acid or sodium hydroxide. After 5 minutes of homogenizing with an Ultra-Turrax-stirrer T50 (Janke & Kunkel Co.) at 7000 rpm, the electrophoretic mobility of oil-in-water droplets was measured with a ZETA-sizer (ELS-800, Otsuka Electronics Co.). The homogeneity of the emulsions and the particle size distribution were controlled by an Ultrafine Particle Analyzer (UPA).

In all measurements a standard ELS quartz cell (17 × 10 × 2 mm^3) was used. The principles of these measurements were based on the heterodyne method because of the extremely small Doppler shift of the frequency of the scattered light in comparison to the frequency of the incident laser light. The scattered light was mixed with a portion of the modulated incident laser light for a better determination on the photomultiplier (12–15). The ELS-800 measurement was made at five locations in the closed rectangular cell of the apparatus. The profile in Fig. 1 shows a parabolic distribution of velocity with the five measured values and their power spectra after the fast Fourier transformation for each heterodyne autocorrelation function. The parabola was cal-

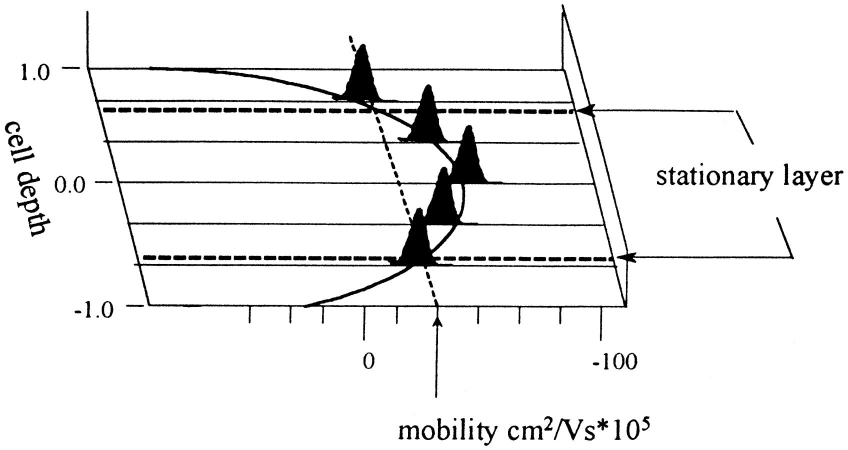

FIG. 1 Mobility profile measured in the closed ELS standard cell.

culated by the equation of Mori and Okamoto (16). The arrows mark the stationary layer in the rectangular cell. The intersection point of the parabola and the stationary layers gives the true electrophoretic mobility, and the quality of the measurements is good if the parabola is symmetric to the cell center.

RESULTS AND DISCUSSION

Adsorption Layers

The first part of the experimental results concerns the interfacial tension and the covering of amphiphiles at the interface. The second part resumes the zeta potential measurements. The investigated adsorption layers are realized by the hygroscopic–nonhygroscopic orientated adsorption of surfactants at the interface. The covering, and therefore the amount of surfactants per unit area adsorbed at the interface at a given temperature, was calculated on the basis of the Gibbs equation of adsorption:

$$\Gamma = -\frac{1}{m\Re T}\frac{d\gamma}{d\ln c} \tag{1}$$

Here c is the bulk aqueous concentration of the surfactants, γ is the interfacial tension, and m is the surfactant specific parameter which is 1 for neutral adsorbates and 2 for salt-free solutions of ionic surfactants. Figure 2 shows the interfacial covering Γ obtained for the system H_2O//10 wt% TOA/TOL versus log c curves. The nonionic surfactant aggregates to micelles at a lower bulk concentration (one decimal power) than the ionic surfactants. The interaction of the ionic headgroups, which counteracts a direct association of the surfactants, is a cause of their mutual repulsion. According to this mech-

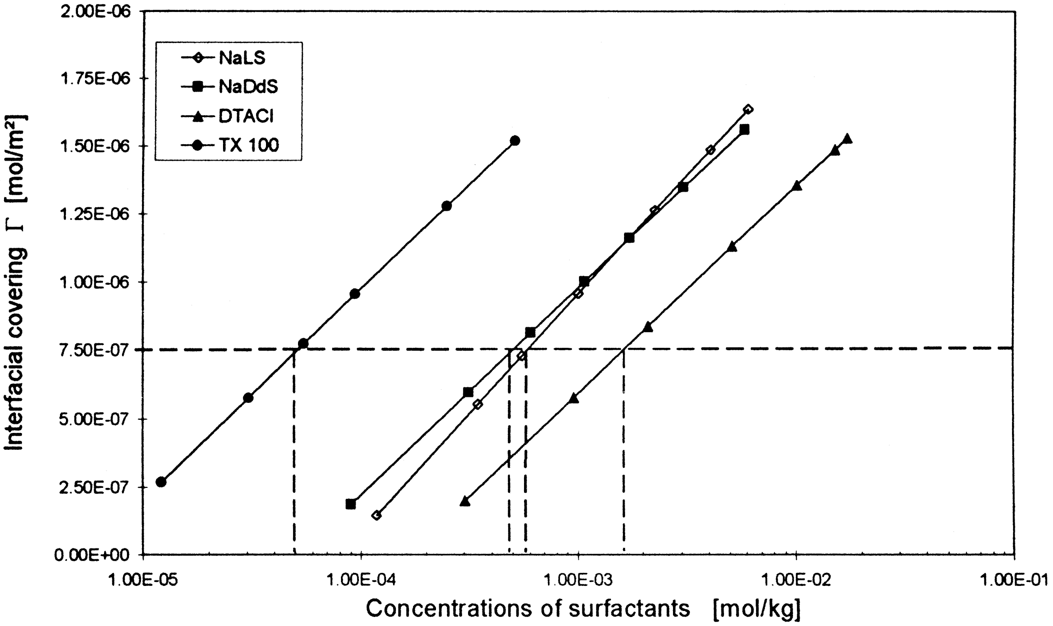

FIG. 2 Covering of surfactants per unit area calculated using the Gibbs equation, H_2O//10 wt% TOA/TOL.

anism, different amphiphilic concentrations have to be considered in order to get the same covering of surfactants at the interface, and therefore the same density of charge. The same is valid for the system H_2O//10 wt% TOA/ISO. Table 1 shows the chosen interfacial covering $\Gamma = 7.5 \times 10^{-7}$ mol/m^2 of the surfactants in dependence on their molalities in the aqueous bulk phase for both systems.

TABLE 1
Surfactant Concentration in the Aqueous Bulk Phase for the Realization of an Interfacial Covering $\Gamma = 7.5 \times 10^{-7}$ mol/m^2 for Both Systems

System H_2O//	Surfactant	Molality m_{Tensid} (mol/kg)
10 wt% TOA/ISO	DTACl	3.354E-03
	NaDdS	5.715E-04
	NaLS	4.804E-04
	TX-100	1.134E-05
10 wt% TOA/TOL	DTACl	1.608E-03
	NaDdS	4.974E-04
	NaLS	5.802E-04
	TX-100	5.112E-02

This interfacial covering (see Fig. 2) was used as a reference to compare the influence of charged interfaces on the reaction kinetics as well as on the respective electrophoretic mobility, the zeta potential, for the different amphiphiles. However, the resulting bulk concentrations for the different surfactants are situated in the lower range of sensitivity of the adsorption isotherms and remote from the critical micelle concentrations (cmc). Consequently, the influence of amphiphiles on the mass transfer and on the zeta potential is caused only by the charge density at the interface. For a relative low acetic acid concentration, a transport limitation due to a heterogeneous reaction at the interface could be found for an aqueous impeller speed range of 120 to 180 rpm. Above 180 rpm the liquid–liquid interface becomes unstable and organic droplets move into the aqueous phase. Below 120 rpm the transfer rate is dominated by diffusion or even a mixed regime, which is also recognized by the flux at higher acetic acid concentrations. The transfer rates are calculated by correlating the organic phase concentrations obtained with a function developed by Kamenski and Dimitrov (17):

$$f(t) = C_1 \frac{t + C_2}{t + C_3} \tag{2}$$

C_1, C_2, and C_3 are correlated parameters and t is the experimental time. The initial transfer rate R_0 is then

$$R_0 = \left.\frac{\partial f(t)}{\partial t}\right|_{t=0} \frac{w_s}{A} \tag{3}$$

where A denotes the interfacial area and w_s is the weight of the organic solvent. Figure 3 shows the significant plateau regions for the calculated initial mass transfer of the surfactant-free system in comparison to the amphiphiles used. The induced charge at the interface, which is caused by a hygroscopic–nonhygroscopic orientated covering of surfactants at the interface, has a strong influence on the flux. The anionic active layers decrease the flux while it is increased by cationic surfactants and independent of nonionic surfactants at an equal covering and charge density. Furthermore, there is no difference between the two anionic surfactants NaDdS and NaLS, which means that the change in flux is determined by the induced charge and not by the adsorbed substance.

This is a very important fact because the adsorbate matrix, and therefore also the charge at the interface, has a markedly effect on the kinetics at the interface. Figure 4(c) represents the case of a cationic-charged interface where the anionic extractable aqueous species (acetate anion) is preferably accumulated in the resulting double layer due to electrostatical interactions. Therefore, the corresponding hydrogen protons have free access to enhanced protonation of the interfacially adsorbed TOA molecule. This and the high acetate

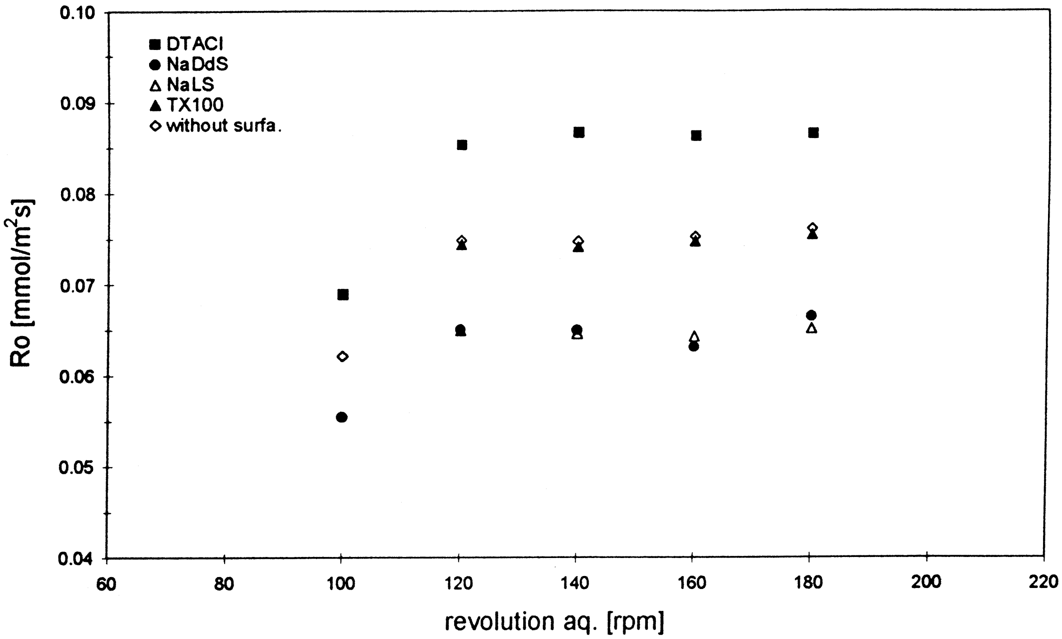

FIG. 3 Influence of surfactants on the reaction limited regime, $\Gamma = 7.5 \times 10^{-7}$ mol/m^2, 0.2 wt% HA/H$_2$O//10 wt% TOA/TOL.

concentration in the near vicinity are the reasons for an increase of the extraction rate. This corresponds with the upper values in Fig. 3. The opposite effect is shown in Fig. 4(a) where the protons are bound in the double layer and make the protonation of the adsorbed TOA molecule more difficult. Additionally,

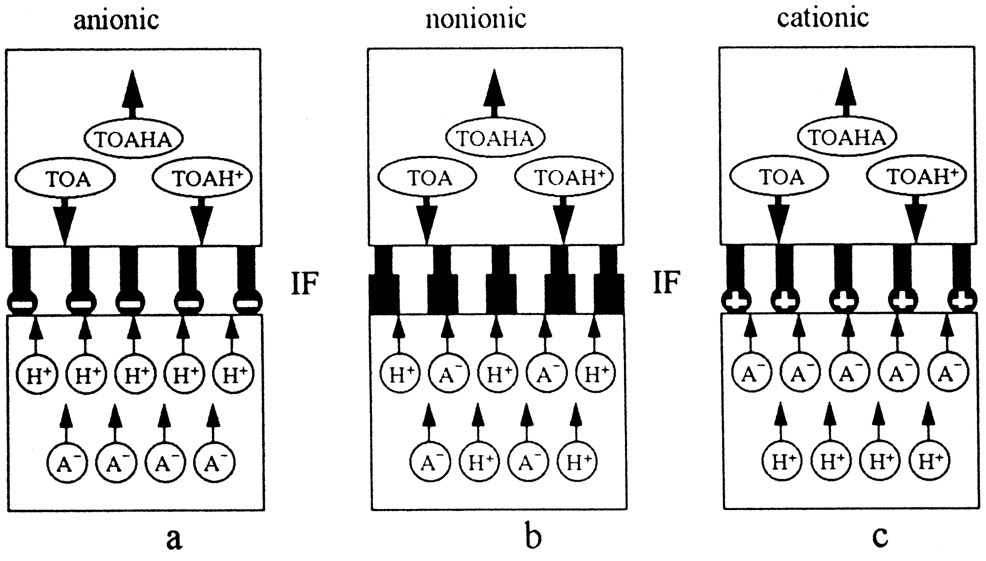

FIG. 4 Chemical adsorption layers as a matrix for the interfacial reaction (IF = interface).

the negative charge repulses the acetate anion, which overall leads to a decrease of the extraction rate as depicted in the lower values in Fig. 3. The neutral case is shown in Fig. 4(b); the equal distribution of hydrogen protons and acetate anions at the interface leads to a balanced extraction rate which is comparable to the surfactant-free system (see the middle values in Fig. 3). So the presence of a loaded organic/aqueous interface changes the interfacial concentration of the extractable aqueous species and thus the extraction rate.

The influence of the interfacial charge of the anionic surfactant NaDdS and the cationic surfactant DTACl on the transfer rate is shown in Fig. 5. The upper limit in these experiments is given by the cmc of the components. In the range of lower interfacial charge the systems behave as in Fig. 3; an increase of the surfactant concentrations leads in the same way to a decrease of the initial flux for NaDdS as to an increase for DTACl. But at higher concentrations the mass transfer for the cationic surfactant increases non-proportional with the interfacial charge, and in a similar manner is even an increase instead of a stronger decrease in the anionic case. This can be explained by the constitution of the surfactant film at the interface, because an increase of the surfactant concentration leads to a transformation of the gas-analogous film into a condensed and strictly ordered surfactant film, which makes penetration and mass transfer easier (18). This is in accordance with other systems, e.g., water/acetone/toluene in the presence of NaDdS (19).

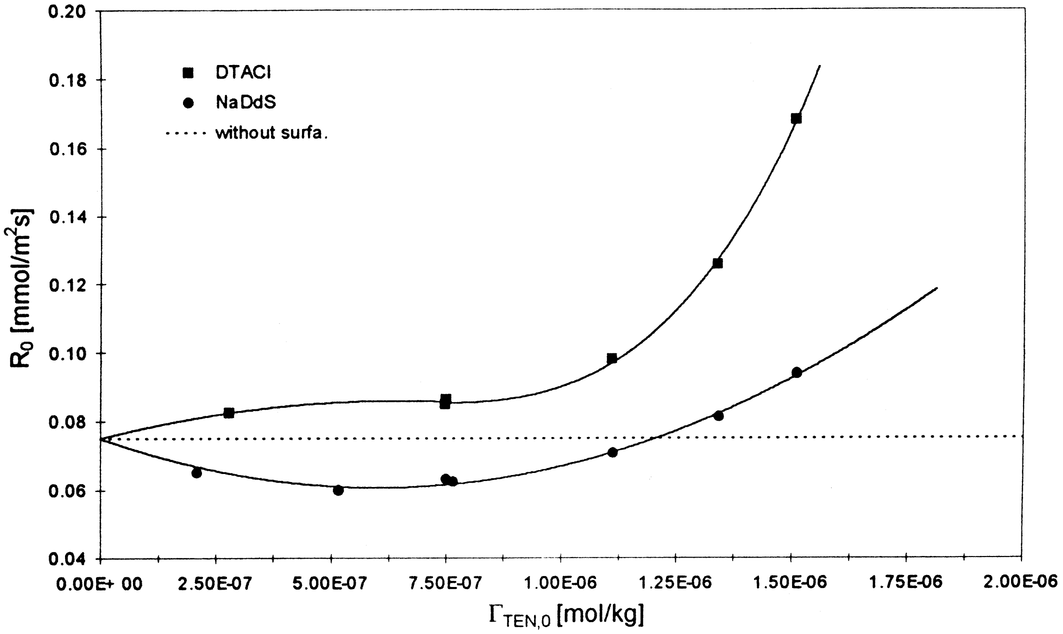

FIG. 5 Influence of surfactants on the reaction limited regime, Variation of Γ, 0.2 wt% HA/H_2O//10 wt% TOA/TOL, n_{aq} = 160 rpm.

Zeta Potential

Electrophoresis measures the migration of charged particles under the influence of an external electrical field (20, 21). The zeta potential (ζ) is the designation of the Galvani potential in the diffusive part of the electrochemical double layer at the interface of two nonmixable liquids. It is the outer directed potential and is responsible for the electrokinetic appearance of a charged particle. The zeta potential is the potential at the plane of shear which separates the stationary and mobile phases in a tangential flow (9). It is possible to convert the electrophoretic mobility into the zeta potential with the Henry equation which describes the general case:

$$u = \frac{2\varepsilon\zeta}{3\eta} f(\chi, a) \tag{4}$$

where ε and η are the dielectric constant and the viscosity of the continuous phase, respectively; a is the particle radius; and χ is the Debye–Hückel parameter:

$$\chi = \left(\frac{e^2 N_A \sum c_i z_i^2}{\varepsilon k T} \right)^{0.5} \tag{5}$$

e is the elemental charge; N_A is Avogadro's constant; c_i and z_i are the concentration and valence of the ion i, respectively; k is the Boltzmann constant; and T represents the absolute temperature. In this case, where the particle radius is much larger than the compared double layer thickness χ^{-1}, the zeta potential can be described by the Smoluchowski equation:

$$u = \varepsilon\zeta/\eta \tag{6}$$

The influences of diluents and TOA concentrations in the amphiphile-free system are given in Figs. 6 and 7. An increase of the pH value from acidic to alkaline leads to an decrease of the zeta potential from positive to negative values (9). Furthermore, it can be seen that the tertiary amine TOA does not essentially influence the potential of the oil-in-water emulsions. However, an increase of the TOA concentration leads to a shift of the isoelectrical point to higher pH values. These results were expected for an extractant with a basic active component. By comparing Figs. 6 and 7 it can be recognized that the isoelectrical point is shifted about two pH units to higher pH values in the isododecane system at the same TOA concentrations as in the toluene system. In general, higher zeta potentials were reached. This can be explained by the larger number of free adsorption places for TOA in the isododecane system, which could be proved by dynamic surface tension measurements (18).

The presence of surfactants in the aqueous phase markedly affects the zeta potential of oil drops through interfacial adsorption of the surfactant

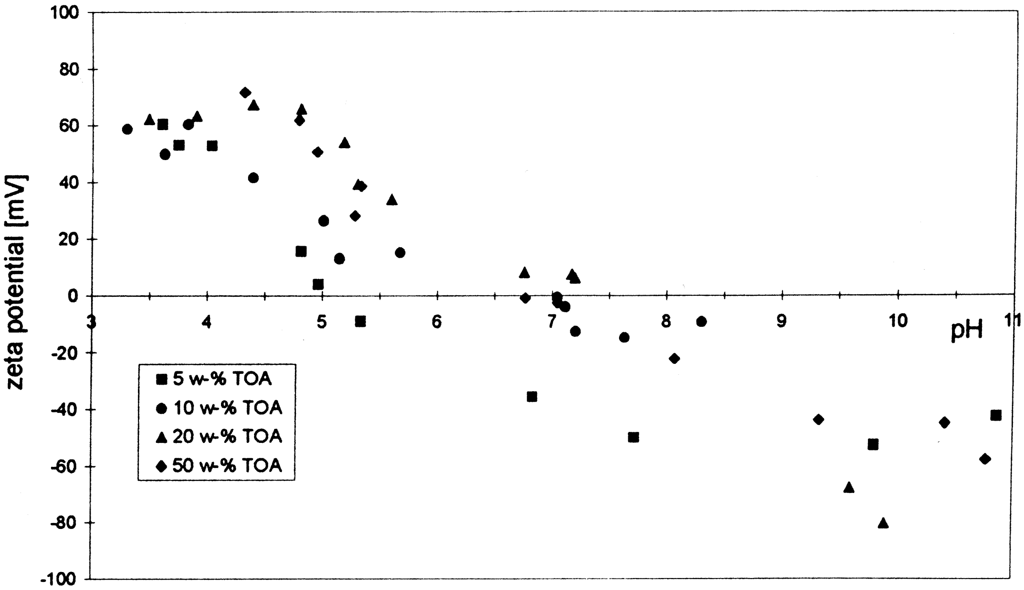

FIG. 6 Zeta potential–pH curves for different wt% TOA/TOL//H_2O emulsions.

molecules at the interface. Figures 8 and 9 show the influence of the surfactants on the zeta potential of oil drops for the toluene and the isododecane systems at the same charge density as in the mass transfer experiments.

For DTACl, the zeta potential is positive over the whole pH range. In the case of anionic NaDdS and NaLS, the zeta potential becomes negative in

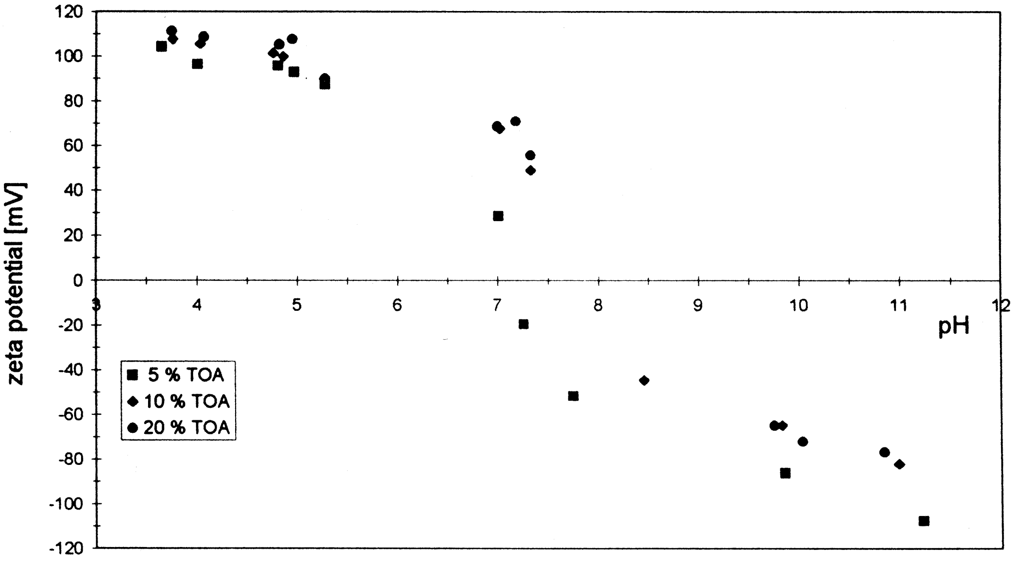

FIG. 7 Zeta potential–pH curves for different wt% TOA/ISO//H_2O emulsions.

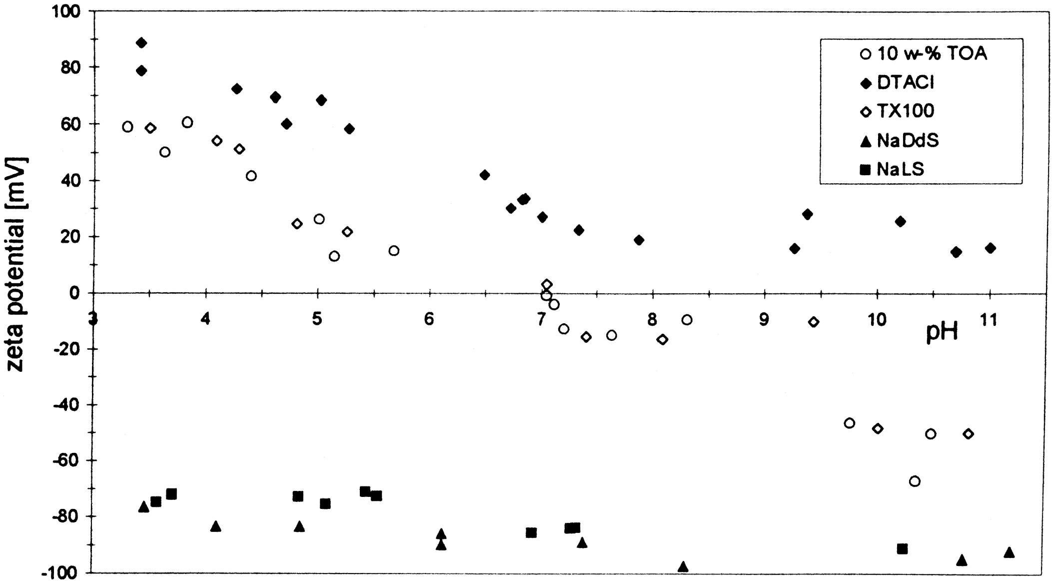

FIG. 8 Zeta potential–pH curves for different wt% TOA/TOL//H_2O emulsions in the presence of surfactants at an interfacial covering $\Gamma = 7.5 \times 10^{-7}$ mol/m^2.

about the same way. The nonionic surfactant TX100 does not affect the zeta potential over the pH range and is in line with the values obtained for the surfactant-free system. These results are in a very good agreement with the measured reaction kinetics in the stirred cell (Fig. 3). In other words, reaction in-

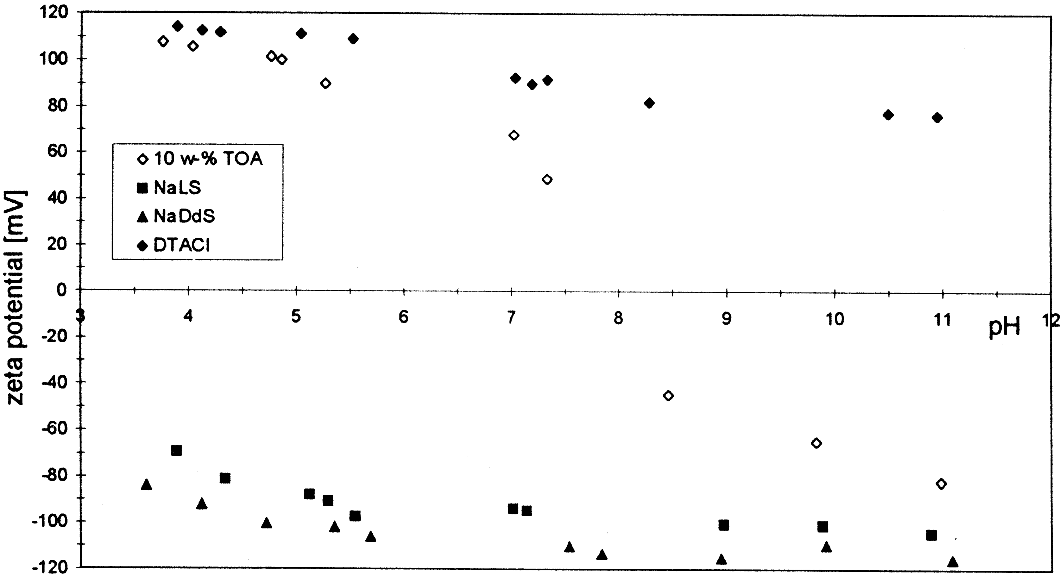

FIG. 9 Zeta potential–pH curves for different wt% TOA/ISO/H_2O emulsions in the presence of surfactants at an interfacial covering $\Gamma = 7.5 * 10^{-7}$ mol/m^2.

hibition is to be expected in the presence of an interface with the same charge as the extractable species. In opposition to this, an increase in extraction rate is to be expected when the aqueous transfer species and the interface are oppositely charged (see also Fig. 4).

Figure 10 shows the variation of the interfacial charge for different amounts of the anionic surfactant NaDdS. As expected, the values of the measured zeta potentials generally decrease with an increase of the amphiphile concentration at higher interfacial coverings. However, in the measurement series at $\Gamma = 1.9 \times 10^{-7}$ mol/m^2, changes of the zeta potential values in the acid area were not expected. At that concentration the induced negative charge is too low for the sensitivity of the ZETA-sizer. So the protons and therefore the accumulation of acetate ions determine the electrophoretic mobility in the acid area at this concentration of anionic surfactants, and the values show the same progress up to the isoelectrical point as in the amphiphile-free system. In the neutral pH area the surfactant has an effect through the logarithmic decrease of hydrogen protons, and the surfactant predominates the potential curve in the same way as it does at higher amphiphile concentrations. This phenomenon has even been described for very low anionic amphiphile concentrations by Puls et al. who investigated the influences of different concentrations on large (100 nm) iron oxide particles (22).

Figure 11 shows the variation of the interfacial charge for the cationic surfactant DTACl. At different coverings of DTACl it was observed that the zeta potential shifts to higher values with an increase of the surfactant concentration at higher interfacial charges. Upon comparing the overall behavior of the

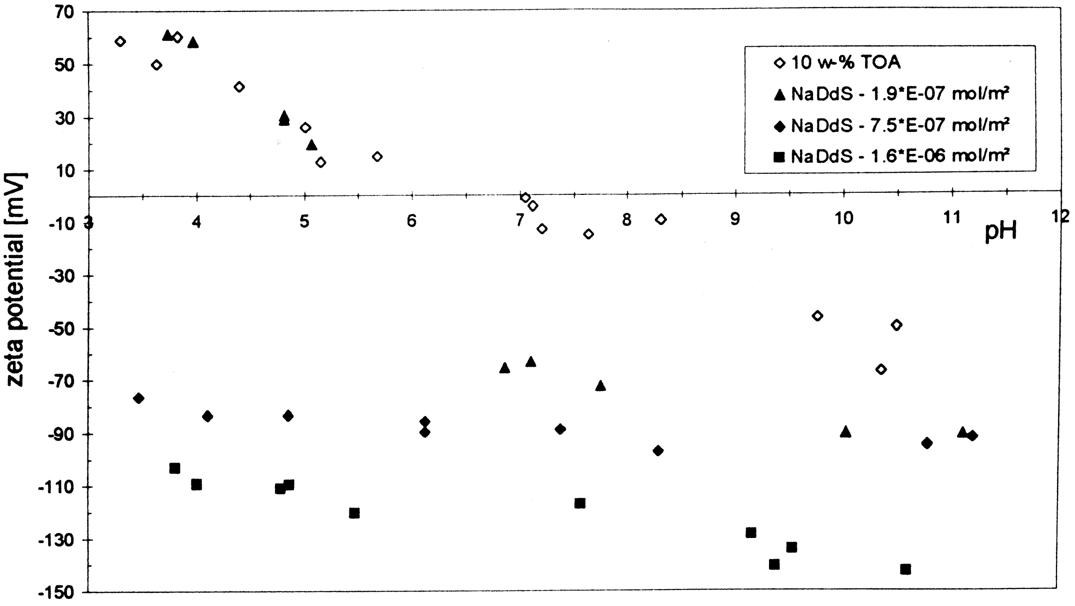

FIG. 10 Zeta potential–pH curves for different wt% TOA/TOL/H_2O emulsions in the presence of NaDdS at different interfacial coverings.

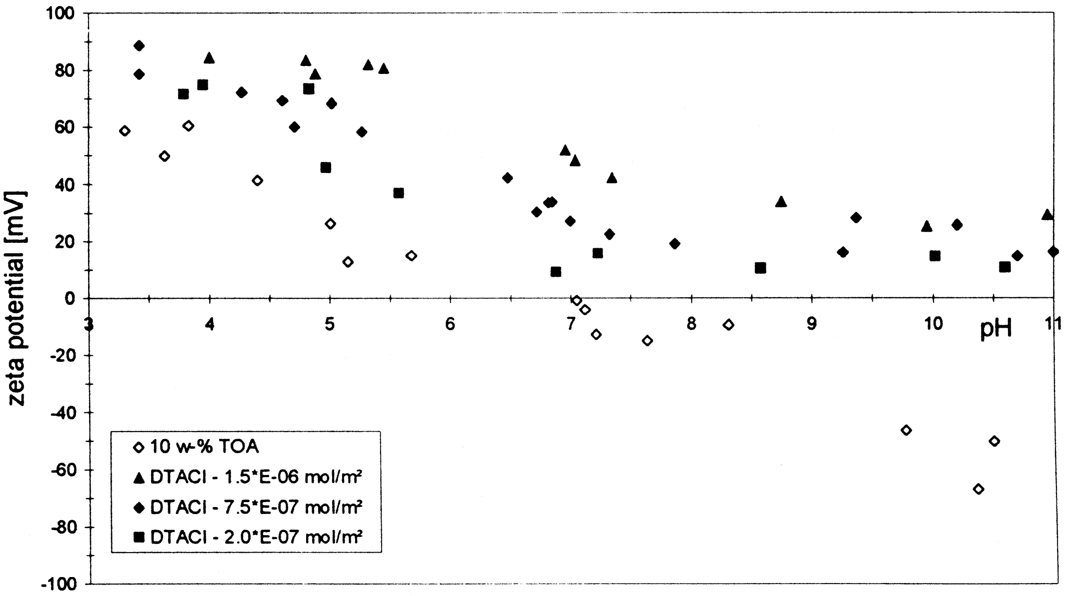

FIG. 11 Zeta potential–pH curves for different wt% TOA/TOL//H_2O emulsions in the presence of DTACl at different interfacial coverings.

anionic surfactant NaDdS and the cationic surfactant DTACl, it can be recognized that the data in Fig. 11 are closer together for a similar spread at interfacial coverings for both ionic surfactants. This effect can be explained because of the larger reduction of the double-layer thickness due to the larger DTACl concentrations for comparable interfacial charges. That is, the compensation for the charge occurs much closer to the surface because of the much higher concentration of counterions. Assuming that the surfactants are completely dissociated, then according to Eq. (5) the thickness of the double layer χ^{-1} is reciprocally proportional to the total ionic strength I of the aqueous solution: $\chi^{-1} \propto 1/\sqrt{I}$. Table 2 shows the calculated values for the chem-

TABLE 2
Interfacial Coverings Γ, the Associated Molalities m_i, and the Resulting Electrochemical Double Layer Thickness in Dependence of the Total Ionic Strength I for NaDdS and DTACL in the Toluene System

System H_2O//10 wt% TOA	Surfactant	Covering Γ (mol/m^2)	$m_{\text{Surfactant}}$ (mol/kg H_2O)	Layer thickness (nm)
TOL	DTACl	2.0×10^{-7}	3.032×10^{-4}	4.6
		7.5×10^{-7}	1.608×10^{-3}	4.1
		1.5×10^{-6}	1.710×10^{-2}	2.1
	NaDdS	1.9×10^{-7}	9.051×10^{-5}	4.7
		7.5×10^{-7}	4.974×10^{-4}	4.5
		1.6×10^{-6}	5.806×10^{-3}	3.1

ical double-layer thickness in dependence of the total ionic strength *I* which includes 4 mM sodium acetate as the background electrolyte and different concentrations of DTACl and NaDdS.

CONCLUSIONS

Different concentrations of surfactants in the aqueous bulk phase have to be considered to get an equally amphiphilic-covered interface. At an interfacial covering of $\Gamma = 7.5 \times 10^{-7}$ mol/m^2, the effect of amphiphiles on the mass transfer and on the zeta potential is caused only by the interfacial charge density. The amphiphile has a strong influence on the initial mass transfer rate in the reaction-dominated regime. Cationic layers increase the mass transfer of acetic acid while anionic layers give a decrease and nonionic surfactants have no effect. In general, those influences are not caused by a shifting of the corresponding extraction equilibria but by the charged interface which changes the amount of extractable aqueous species. So the adsorbate matrix and therefore also the charge at the interface has a marked effect on mass transfer kinetics. The measurement of the electrophoretic mobility is a suitable tool to evaluate these effects in the TOA–surfactant systems with either toluene or isododecane as diluent. The tertiary amine is primarily adsorbed as a neutral substance and does not affect the potential of oil-in-water emulsions. This is in contrast to surfactant systems which were used for comparison at the same interfacial covering as in mass transfer experiments. Here the zeta potential becomes positive over the whole pH range for cationic DTACl, negative for anionic NaDdS and NaLS, and equal to the values obtained for surfactant-free systems in the case of the nonionic TX100. An increase in the cationic or anionic surfactant concentrations leads to a proportional increase of the zeta potential in the positive or negative direction. However, at higher surfactant concentrations the charge at the interface is overlapped by the constitution of the interfacial surfactant film (transition from a gas-analogous film to a condensed film), which even in the anionic system leads to an increase of the flux instead of a stronger decrease. Furthermore, the variation of surfactant concentrations up to its cmc leads to a reduction of the double layer and therefore the increase of the zeta potential in relation to the interfacial charge is nonproportional (especially for DTACl).

NOMENCLATURE

Aqueous Phase

HA	acetic acid
NaOH	sodium hydroxide
NaA	sodium acetate

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Organic Phase

TOA	tri-*n*-octylamine
TOL	toluene
ISO	isododecane

Surfactants

NaDdS	sodium dodecyl sulfate, anionic
NaLS	1-dodecanesulfonic acid sodium salt, anionic
DTACl	dodecyltrimethylammonium chloride, cationic
TX100	octylpoly(ethylene glycol ether)$_{10}$, nonionic

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