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The effect of surfactants on interfacial mass transfer in binary liquid–liquid systems

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Abstract

The effect of surfactants on interfacial mass transfer in binary liquid–liquid systems has been investigated experimentally using the Schlieren and the Mach–Zehnder optical techniques. The Schlieren optical apparatus enabled the observation of the stability of the interfaces during the mass transfer process in the organic-aqueous systems selected, when the aqueous phase was ‘clean’ or ‘contaminated’ by selected surfactants. The Mach-Zehnder interferometer was used to measure molar fluxes in the same liquid–liquid systems. Results have shown that the presence of an ionic surfactant in the aqueous phase could either initiate or increase Marangoni convection and enhance (up to seven times) the measured molar fluxes. Slight decreases in molar fluxes were measured for the cases where Marangoni convection was observed to have been dampened by the addition of a non-ionic surfactant. The possibility is thus raised for the manipulation of interfacial stability, through the addition of a surfactant to induce or inhibit Marangoni convection, for the improved design and understanding of interfacial processes. © 2000 Published by Elsevier Science Ltd. All rights reserved.

Keywords: Interfacial convection; Marangoni convection; Surfactant effects; Schlieren/Mach–Zehnder optical techniques

1. Introduction

The mass transfer process in quiescent binary liquid–liquid systems occurs by diffusion, but it may sometimes be assisted by the occurrence of Marangoni convection at the interface. During the mass transfer process, interfacial tension gradients (originated from temperature gradients produced by the heats of solution) may be created at the interface and depending on the physical properties of the system, these may cause the onset of Marangoni convection which then lead to increased mass transfer rates.

Since Thompson [1] first reported his findings, Marangoni convection has been observed at many different liquid–liquid interfaces [2–4]. Its application to solvent extraction systems became apparent when Lewis and Pratt [5] observed rippling and erratic pulsations at the interface of drops of organic solvents dispersed in various solvents. Some of these pulsations were associated with shock waves, which were strong enough to cause a noticeable amount of mass transfer. Several workers, including Sherwood and Wei [6], Maroudas and Sawistowski [7] and Bakker and coworkers [8,9] later reported unexpected large increases in mass transfer rates, which were accompanied by the presence of Marangoni convection. In all these reports Marangoni convection occurred during the transfer of a solute across a liquid-liquid interface, i.e. ternary systems. Ying and Sawistowski [10], Perez de Ortiz and

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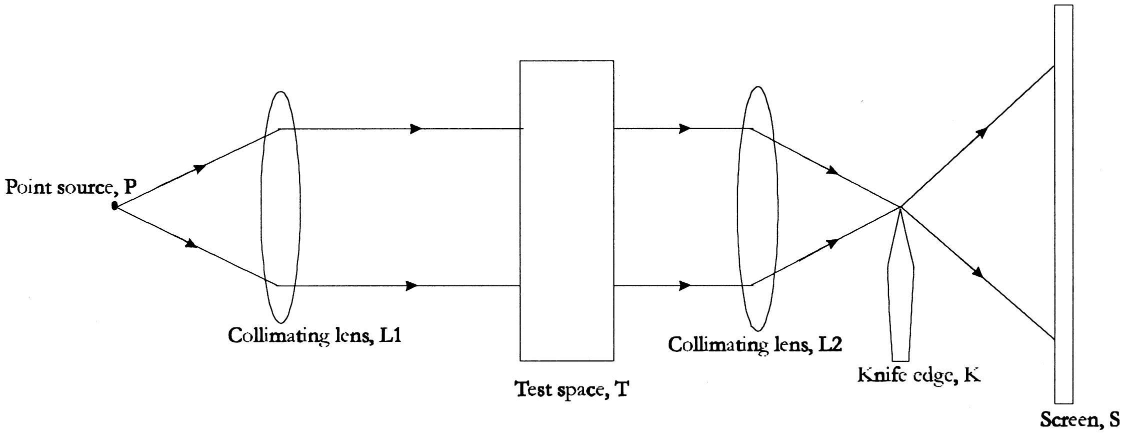

Fig. 1. Schematic of a Schlieren system.

Sawistowski [11], Davies and Thornton [12] and later Mendes-Tatsis and Perez de Ortiz [13] have also reported the presence of Marangoni convection in binary liquid–liquid systems.

Reports by, for instance, Blokker [14], Davies and Wiggill [15] and Mudge and Heideger [16], about the effect on mass transfer of the presence of surfactants in ternary systems indicated that they decreased mass transfer. Reductions in the mass transfer rates were attributed either to a mechanical resistance (barrier effect) or the dampening of Marangoni convection at the interface, caused by the formation of a rigid interfacial film. The presence of surfactants was therefore considered disadvantageous in most mass transfer studies and solvent extraction processes. Renewed interest in Marangoni convection has arisen, in the last decade, due to reports that the presence of surfactants in some systems caused the appearance of Marangoni convection [17–21]. Surfactants are typically used in considerably smaller concentrations (~0.05% v/v) than are other solutes that have been associated before with Marangoni convection. If surfactants are not only able to dampen interfacial convection, but are also capable of inducing Marangoni convection, then such developments may offer the process designer and operator the possibility of manipulating the interfacial region to suit a desired application, with obvious implications on solvent extraction and other related processes. In solvent extraction, high rates of mass transfer are required and therefore ways of improving the mass transfer process are always desirable.

Marangoni convection has also found applications, which extend beyond the confines of solvent extraction. For instance, Sharma and Ruckenstein [22] suggested that wetting and cushioning solutions for contact lenses may help avoid conditions that lead to sore/dry eyes if they include substances that promote Marangoni convection at the cornea tear film, since Marangoni convection inhibits the break-up of the cornea tear-film by continually replenishing tear-film fluid. Also, Leenars et al. [23] note that in the manufacture of silicon wafers for liquid crystal displays induced Marangoni convection aids the (clean) drying of silicon wafers, where conventional drying methods are unsatisfactory.

This paper reports the findings from an experimental programme, which has been carried out with the following objectives:

1. to examine systematically the effect of a selected range of surfactants on the interfacial stability of different binary liquid–liquid quiescent systems and
2. to examine the effect on interfacial mass transfer rates of the presence of interfacial convection in those systems.

2. Experimental

Two optical techniques have been employed to study the mass transfer process at binary liquid–liquid interfaces, where mass transfer is expected to occur by diffusion only. The first, the Schlieren technique [24,25], was used to observe the effect of the presence of various surfactants on the stability of liquid–liquid interfaces; the second, the Mach–Zehnder technique, was used to measure the liquid–liquid interfacial mass transfer rates both in the presence and in the absence of the surfactants.

In the Schlieren technique (Fig. 1) light from a point

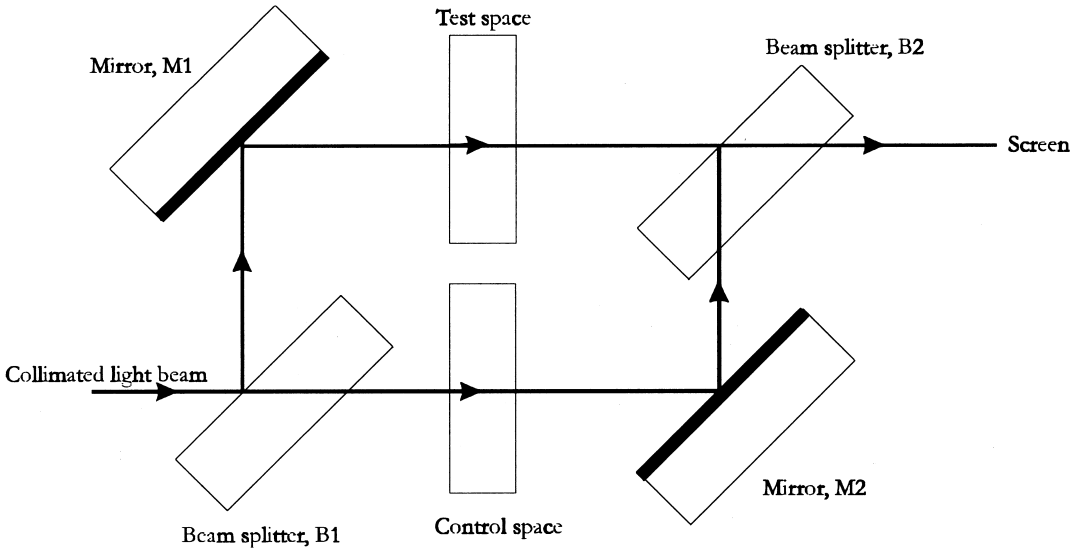

Fig. 2. Schematic of a Mach–Zehnder interferometer.

source is collimated by a lens, L1, and passes through a test object space, T. This test space is, in this work, the flow field around drops. The beam is subsequently focused onto a knife-edge, K, by a second collimating lens, L2, and then projected onto a viewing screen S, which is then photographed or filmed. Schlieren images

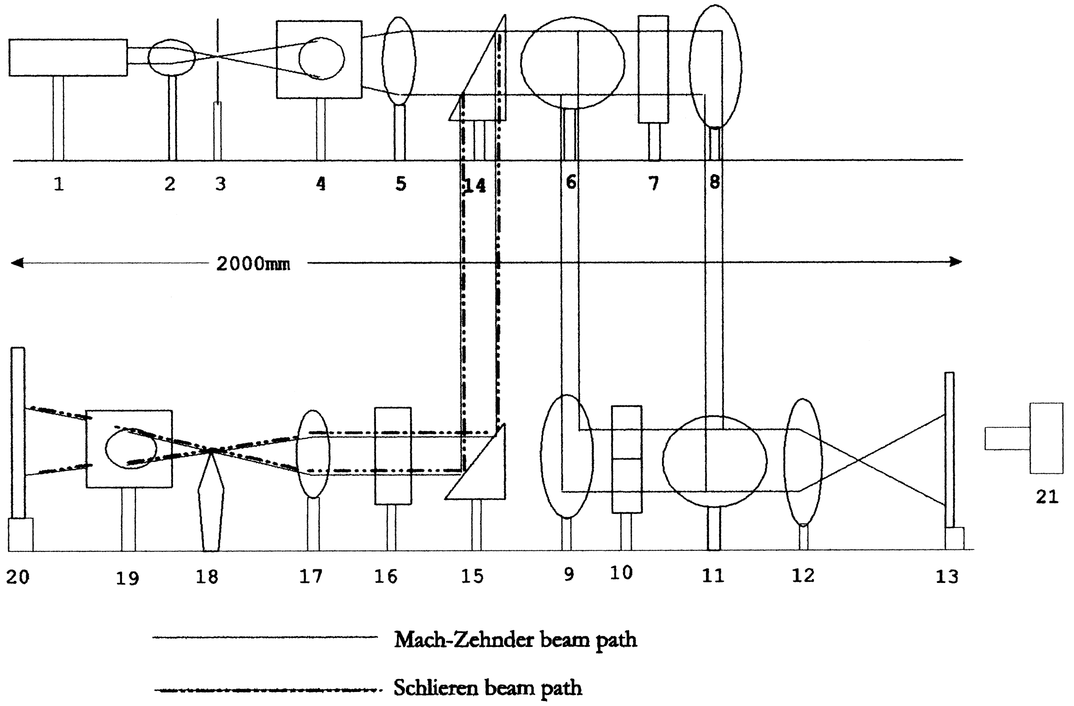

Fig. 3. Integrated Schlieren/Mach–Zehnder optical apparatus. Labels: (1) He–Ne laser; (2) microscope objective; (3) 25μ pinhole; (4, 19) circular diaphragms; (5) f500 mm plano-convex lens; (6, 11) semi-reflecting mirrors; (7) interferometer reference cell; (8, 9, 14, 15) fully reflecting mirrors; (10) interferometer test cell; (12) f125 mm plano-convex lens; (13, 20) translucent screens; (16) Schlieren drop cell; (17) f75 mm bi-convex lens; (18) tapered knife edge; (21) photographic/video camera.

Table 1
Surfactant concentrations used in experiments and their respective values of critical micelle concentration (CMC) and relative molecular mass (RMM)

Surfactant	LDS	SDS	DTAB	Softanol 30	Softanol 120	ATLAS G1300
Concentration, g/100 ml	0.05	0.05	0.05	0.002	0.002	0.002
CMC, g/100 ml	0.243	0.236	0.493	2.107×10^{-3}	3.998×10^{-3}	2.735×10^{-3}
RMM, g/mol	273.3	288.3	308.3	330	726	~ 9500

of a homogenous test space consist of a black background, due to the absence of light blocked out by the knife-edge. If there are any inhomogeneities in the test space, some light will be deflected and will therefore no longer be focused on the knife-edge. This results in the formation of bright patches on the Schlieren image.

A Schlieren image of a non-homogenous test space is therefore characterised by the presence of light and dark patches, and non-symmetric movements of these light patches indicate the presence of Marangoni convection.

In Fig. 2 a schematic of the Mach–Zehnder interferometer is shown. A coherent parallel beam of light from the He–Ne laser is incident on B1, a beamsplitter (semi-reflecting mirror), where it is split into two branches, the test beam and the control beam, which travel different paths before being recombined at B2, the second semi-reflecting mirror. The test beam is initially incident on M1, a fully reflecting mirror, after which it traverses a test space in which its optical path is modified. It is finally incident on B2. The control beam traverses a control space (usually a reference cell of identical dimensions to the test cell), is then incident on M2, a fully reflecting mirror, and finally rejoins the test beam at B2. An interference fringe pattern/interferogram is formed when the two beams recombine and can be seen on a screen. The mirrors are assumed to be flat, vertical and parallel to each other, with their centres forming the corners of a parallelogram, and all beams are horizontal and incident on all mirrors at an angle of 45°.

Since both these techniques share some common features, an integrated apparatus was assembled (Fig. 3), which permitted Schlieren or Mach–Zehnder studies to be carried out, depending on the optical path chosen (as indicated in Fig. 3). For the interferometric studies the split light beams crossed the reference cell and the test cell (components 7 and 10, respectively) and were recombined at a semi-reflecting mirror (component 12) to form an image on the translucent screen (component 13). However, when the apparatus was used for Schlieren studies, the fully-reflecting mirror (component 14) was introduced to change the optical path and the Schlieren drop cell (component 16) was utilised to obtain images on the translucent screen (component 20).

The Mach-Zehnder technique has been used extensively [26] to measure the concentration of a transferring solute in an aqueous or in a solvent medium. It involves the formation of an interference pattern (interferogram) of the medium under study, and from the deflection of fringes on the interferogram it is possible to determine the concentration (related to the refractive index) at any location in the medium under study, at various times.

2.1. Selection of systems

Four different binary liquid–liquid systems were selected: aniline/water, 1-hexanol/water, isobutanol/water and ethyl acetate/water. The solvents were all of HPLC quality and obtained from Aldrich Chemical Co. and nano-pure water was used for the aqueous phases. Six different surfactants, three ionic and three non-ionic were selected as 'contaminants' for the aqueous phase. The ionic surfactants were sodium dodecyl sulphate (SDS), lithium dodecyl sulphate (LDS) and dodecyl trimethyl ammonium bromide (DTAB), all supplied by Sigma Chemicals. The non-ionic surfactants were Softanol 30 and Softanol 120, supplied by Honeywill and Stein Ltd., and ATLAS G1300, supplied by BP Chemicals. The surfactant concentrations used in the aqueous solutions were always below the critical micelle concentration (CMC) and are included in Table 1. The CMC of each surfactant in aqueous solution was determined according to the method suggested by Porter [27], and surface/interfacial tension measurements were carried out on a Kruss K10 Tensiometer [28]. Values of the CMC and the relative molecular mass (RMM) for the selected surfactants are also given in Table 1. Values for partition coefficients have either been measured or estimated and are reported elsewhere [28].

2.2. Schlieren experiments

The experiments to investigate the interfacial stability of the various liquid–liquid systems involved examination of the interface of a drop of the organic

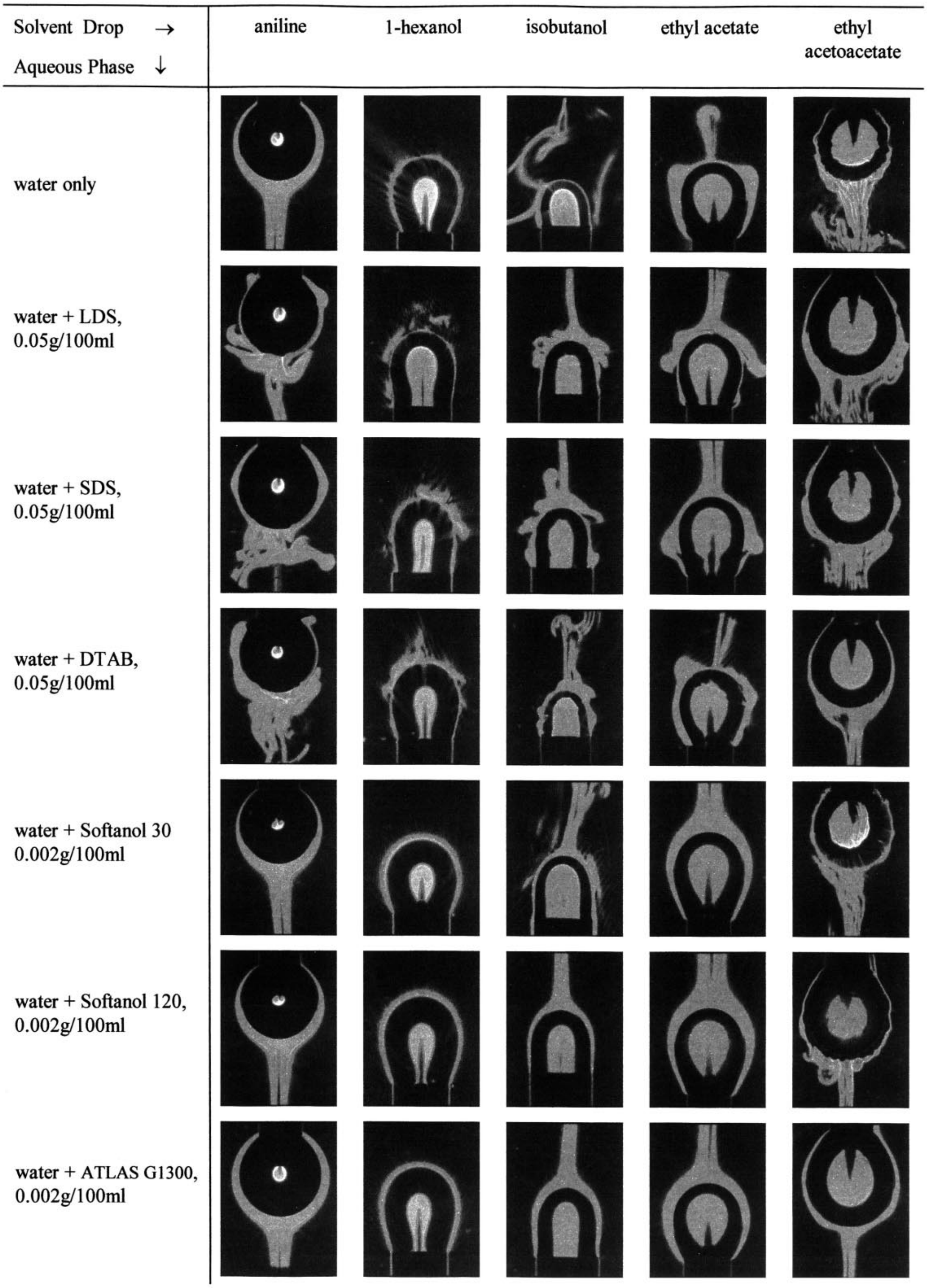

Fig. 4. Schlieren photographs of organic phase drops immersed in 'clean' or 'contaminated' aqueous phases during mass transfer from the drop to the outer aqueous phase.

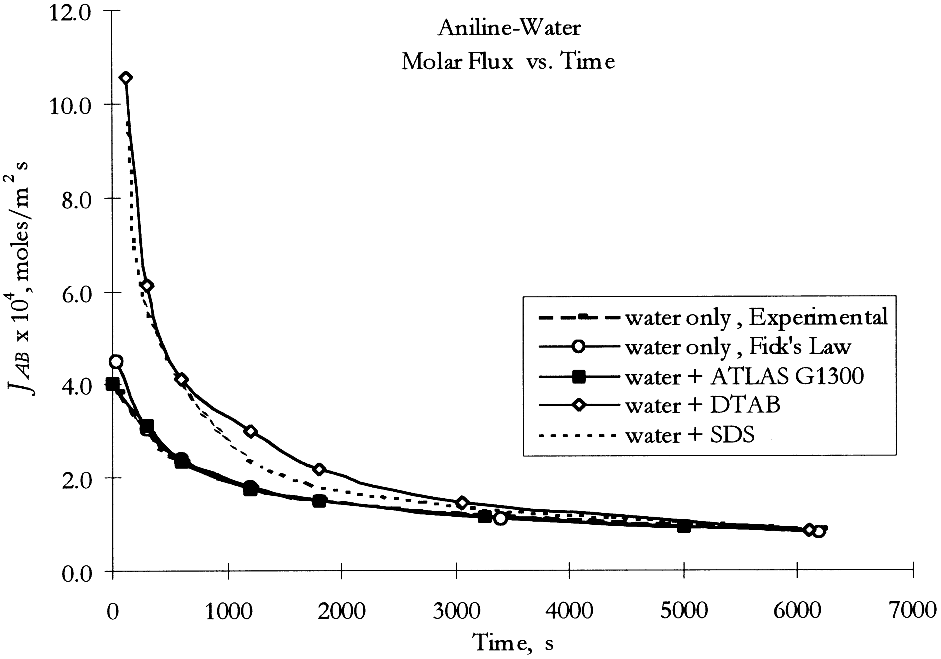

Fig. 5. Molar flux vs. time for the transfer of aniline into different aqueous phases.

phase (saturated with water) which was immersed in an aqueous phase of pure water or of water with a 'contaminant' surfactant, so that mass transfer occurred out of the drop. The drops were formed at the end of a 2.11 mm (OD) capillary needle immersed in a cell, which was constructed from quartz glass and PTFE. Images of the liquid–liquid interface were obtained with the Schlieren optical technique for later analysis.

Results from this Schlieren study are presented in Fig. 4, and consist of photographs of drops of each of the organic phases investigated, immersed in either 'clean' water or in an aqueous phase 'contaminated' with a surfactant. These photos were taken 10 s after the formation of the drop. At the aniline/water interface no instabilities are observed while at the 1-hexanol/water interface an indication of (slight) motions is consistent with observations by Ying and Sawistowski [10]. The presence of instabilities at the isobutanol/water, ethyl acetate/water and ethylacetoacetate/water interfaces is clear and is in agreement with reported observations by Aguirre et al. [29], Ying and Sawistowski [10] and Mendes-Tatsis and Perez de Ortiz [13], respectively.

The presence of LDS, SDS and DTAB as 'contaminants' in the aqueous phase is seen to induce Marangoni convection in the case of the aniline/water system. In the 'clean' systems where convection was observed to exist the presence of these surfactants is observed to increase further the intensity of the Marangoni convection. For example, in Fig. 4, the intensity of interfacial convection in the image corresponding to the ethyl acetate/water + DTAB system is greater than that corresponding to ethyl acetate/water. This conclusion is illustrated more clearly in the video films from which these photos have been extracted.

On the other hand, the surfactants Softanol 30, Softanol 120 and ATLAS G1300 tend to have either no effect on the interfacial stability or dampen any convection that was present in the 'clean' system. For instance, the surfactant ATLAS has little or no effect on the stable interface of aniline/water shown; however, it is seen to dampen the instabilities that were present at the isobutanol/water interface.

2.3. Mach–Zehnder experiments

In this work, the mass transfer studies were carried out in a diaphragm cell similar to that used by Mudge and Heideger [16]. The cell was constructed from quartz glass and PTFE, held together by an outer aluminium frame. The organic solvent was always presaturated with water so that only transfer of the organic solvent into the aqueous phase took place. Two initially separate liquid phases were contacted by removal of a central partition and an image of the area close to the interface appeared on the screen. The resultant interferogram of the planar interface was photographed/filmed at regular intervals. The software Optimas v5.2 was used to analyse the interferograms

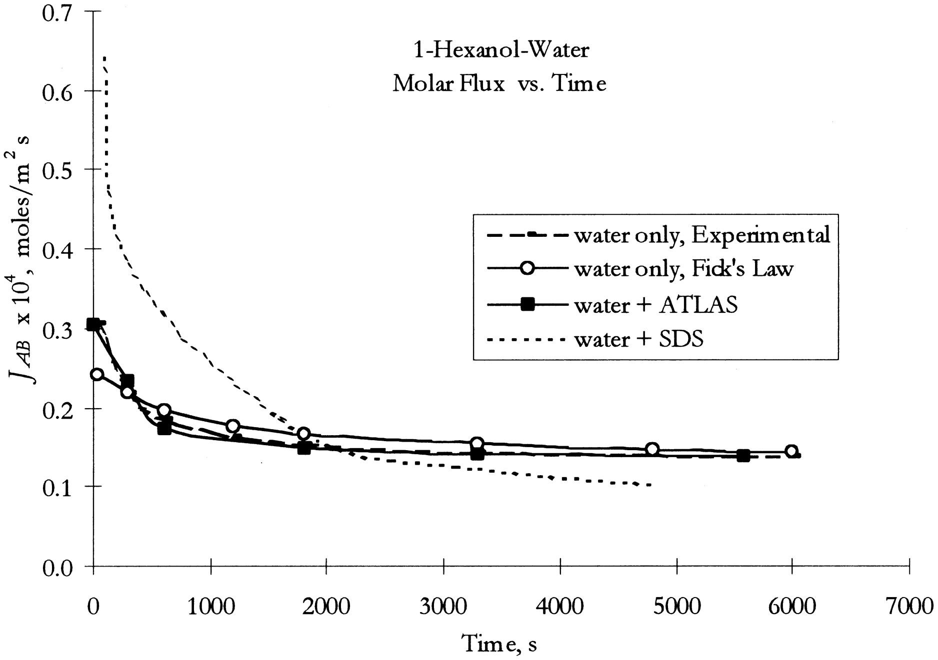

Fig. 6. Molar flux vs. time for the transfer of 1-hexanol into different aqueous phases.

and full details of the method used is included in Agble's work [28]. Prior measurements of the refractive index of the aqueous solutions containing different concentrations of the solutes under study, enabled the calculation of the values of concentrations at different locations from the interface and at different times, from which concentration profiles were plotted. However, mass transfer results are usually expressed as

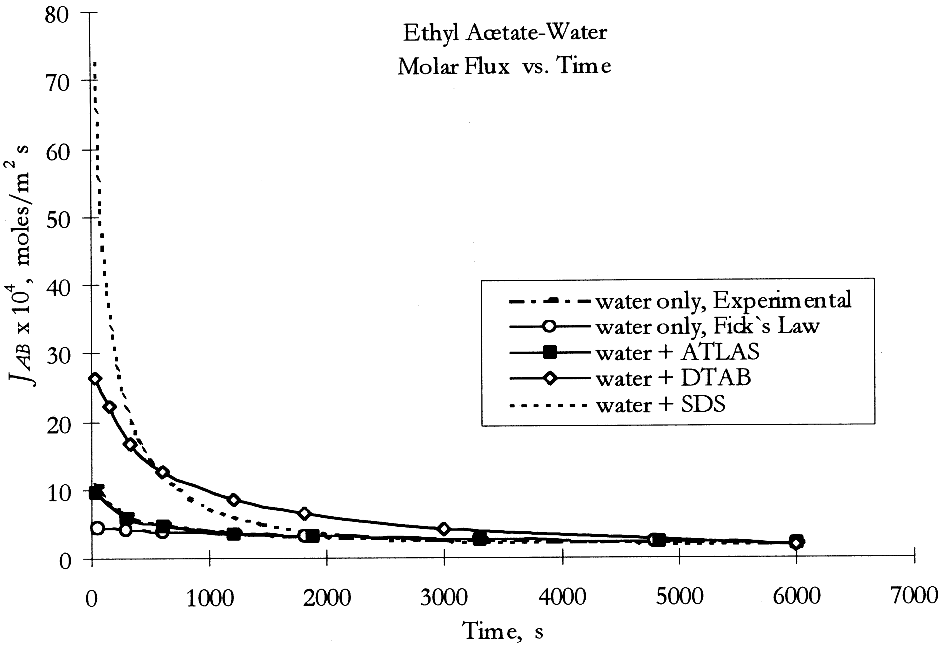

Fig. 7. Molar flux vs. time for the transfer of ethyl acetate into different aqueous phases.

the molar flux (J_{AB}) into a phase plotted against time. The procedure required to convert the concentration vs. distance plots into molar flux vs. time plots is straightforward and is summarised below.

The area under each one of the plots of concentration vs. distance, obtained at different times, is equivalent to the integral/total mass transfer (M_{AB}) into the receiving phase, at the corresponding time. Hence, plots of M_{AB} vs. time may be produced. The molar flux (J_{AB}) or mass transfer rate, at a particular time, is given by the gradient of the plot of M_{AB} against time. This procedure for the manipulation of the data was performed using the software packages Microsoft Excel and Mathematica v2.3. The procedure described has been applied to the systems examined in the Schlieren study and selected mass transfer results are presented below.

The experimental values of J_{AB} for the transfer of aniline into water and into various 'contaminated' aqueous phases are shown in Fig. 5. In Figs. 6 and 7 the same is shown for 1-hexanol and ethyl acetate as transferring solutes. Also shown in the same figures are the theoretically predicted molar fluxes, based on Fickian diffusion of the pure solvent into an aqueous solution. As seen in Fig. 5, the measured molar flux of aniline transferring into pure water closely follows the theoretically predicted molar flux. This is expected since no interfacial activity was observed at the aniline/water interface in Fig. 4. The molar flux also remains unchanged when the surfactant ATLAS G1300 is added to the aqueous phase, and this is once again consistent with the Schlieren images shown in Fig. 4. However, when the surfactants SDS or DTAB are present in the aqueous phase, a large increase in the initial molar flux of aniline into the aqueous phase is observed in Fig. 5. This result can be associated with the observed interfacial convection in their respective Schlieren images in Fig. 4.

In Fig. 6, a similar pattern of behaviour is observed for the transfer of 1-hexanol into water only and water 'contaminated' with surfactants. In Fig. 7, the measured molar flux of ethyl acetate into water is higher than that predicted by the Fick's diffusion equation. The cause for this increase is the interfacial convection (caused by Marangoni and gravitational effects as reported by Austin et al. [30] and Agble and Mendes-Tatsis [31]), which exists at the ethyl acetate/water interface and which is seen in the Schlieren study shown in Fig. 4.

Addition of the surfactants SDS and DTAB to the aqueous phase increases further the initial molar flux of ethyl acetate into water, consistent with the intensified interfacial convection observed in the corresponding Schlieren images in Fig. 4. The addition of ATLAS G1300 to the aqueous phase is observed to cause a slight decrease in the initial molar flux of ethyl acetate into water, perhaps due to the slight dampening of interfacial convection that was noted on the corresponding Schlieren image in Fig. 4.

Values for J_{AB} were determined from data, which was averaged from at least five runs in the case of experiments involving 'clean' systems, and from at least two runs for experiments involving 'contaminated' systems. The total experimental error associated with determining J_{AB} for the transfer of aniline, 1-hexanol and ethyl acetate into their various aqueous phases was 7.28, 17.82 and 10.60%, respectively. This total error is comprised of the error associated with reproducing an individual mass transfer experiment and the error associated with the procedure for obtaining J_{AB} from concentration vs. distance profiles, which was calculated to be 3.5% [28]. Differences in the error associated with reproducing an experiment arise due to differences in the total number of fringes that are present in the system, which affect the accuracy of measurements of the fringe shift, and the differences in the interfacial stability of the system, which affect the swing of the actual fringe shifts measured.

The experimental results presented have exposed the need for further experiments to be carried out with other liquid–liquid systems and with other surfactants. Work is now under way to obtain a physical understanding of the effects observed, particularly with the ionic surfactants, and to find criteria to predict them. Stability criteria has been established before for the case of ternary [32] and binary systems [33]; for the cases when there is a surfactant present in the system Hennenberg and et al. [34,35] and Nakache et al. [17,36] have proposed stability criteria. A full discussion on the applicability of these criteria to the systems investigated in this paper, including assumptions that had to be made, is included elsewhere [28] where it is concluded that the one proposed by Nakache and co-workers predicts better the experimental observations discussed in this paper.

3. Conclusions

The Schlieren and Mach–Zehnder experimental results presented clearly show that the presence of the selected ionic surfactants in the liquid–liquid systems investigated may either initiate or increase Marangoni convection with a consequent increase in molar fluxes. This conclusion is of great importance because it contrasts with the accepted notion that surfactants always decrease mass transfer rates. In this study this was only found to happen for the cases of non-ionic surfactants which had either a neutral effect or dampened interfacial convection. Further work is necessary to be able to understand and predict these effects as well as to extend this study to multicomponent mass transfer systems and to other surfactants.

Acknowledgements

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