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Study of the interaction phenomena of cetyl-trimethyl-ammonium bromide, cetylpyridinium chloride and benzethonium chloride with C. I. Acid Orange 52 and picric acid by two spectral methods

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Abstract

A study of solutions containing C. I. Acid Orange 52 (AO52) or picric acid (PA) and cationic surfactants in various molar ratios by UV–vis spectrophotometry and ion pair extraction spectrophotometry has been made. Cetyl-trimethyl-ammonium bromide (CTAB), cetylpyridinium chloride (CPC) and benzethonium chloride (BEC) have been chosen as cationic surfactants. AO52 and PA have been used as substances with coloured organic anion. The study focuses on investigation of solutions containing AO52 or PA in molar excess over the surfactants. The results show significant changes of absorption maxima in solutions of CTAB or CPC and AO52. The changes of absorption maxima in the case of AO52 excess are ascribed to the formation of associates soluble in water but insoluble in non-polar solvents. This process influences negatively the equilibrium of the ion pair formation. Consequently the amount of the ion pairs extracted during the ion pair extraction spectrophotometry is lowered and a hypochromic shift is recorded as the excess of the dye is increased. Therefore it may be concluded that the results of the ion pair extraction spectrophotometry and the direct UV–vis measurement correspond to each other as far as the systems CTAB + AO52 and CPC + AO52 are concerned. The PA excess does not cause any spectral shifts. Thus, this is in the agreement with the results of the ion pair extraction spectrophotometry, since the excess of PA does not influence the absorbance of the extracted ion pairs.

Keywords: Interaction; Associates; Spectrophotometry; Acid Orange 52; Picric acid; Surfactant

1. Introduction

Many methods have been proposed to determine the behaviour of cationic surfactants. Some papers [1–4] describe the utilization of the ion pair extraction spectrophotometry in submicellar region. If the extraction spectrophotometry is performed by a procedure developed by Gasparic et al. [5], anomalous phenomena are observed in comparison with cases when non-surfactants are determined in the same way. The procedure is also described in more available work [6]. Using the Gasparic procedure (simply: one step extraction), one can find the hypochromic shift of the extract, when molar excess of a counter ion is increased. This phenomenon has been observed in our laboratory when AO52 anion had been used as the counter ion. Similar behaviour in some other solutions has previously been reported by Sakai et al. [2] and Sedmikova and Gasparic [3]. However, neither of the authors has discussed their observations. Contrary to the described behaviour, if the

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picrate anion is used in the experiments instead of AO52 anion, no problems appear and the absorbance of the extract is independent on the molar excess of PA.

As far as the ion pair extraction spectrophotometry is concerned, a formation of ion pairs in molar ratio 1:1 between a univalent analyte and a univalent counter ion is presumed. A molar excess of the counter ion is used to shift equilibrium towards the ion pairs. The reasons for the hypochromic shift with increased molar ratio dye/surfactant are worthy of investigation. Besides analytical chemistry the studied phenomena would be important in processes of dyeing.

A possible reason for differences in the behaviour of the AO52 solutions and the PA solutions is that interaction between PA and the cationic surfactants in the solutions differ from the interaction between AO52 and the cationic surfactants. Buwalda et al. have recently published a spectrophotometric study of the systems containing AO52 and cationic surfactants [7]. Buwalda et al. observed shifts of absorption maxima in solutions of AO52 and some cationic surfactants, under the condition that the concentration of AO52 was kept constant and concentrations of the cationic surfactants varied. The molar concentrations of the cationic surfactants in the studied solutions were higher than the concentration of AO52, i.e. the surfactants were used in excess. Buwalda et al. have explained the origin of the spectral shifts by a formation of associates (not just ion pairs but generally some associates) containing both surfactant species and AO52 ones. Similar phenomena were investigated even by the other authors [8–10]. The authors of these references provide results in systems containing relatively high concentrations of the surfactants and low concentrations of the dyes. Dakiky et al. have utilized UV–vis spectrophotometry to determine ionisation constants and thermodynamic parameters of acid alizarin violet in micellar solutions [11]. Although many authors have studied dye–surfactant interactions, we have not found any article comparing interactions in PA + surfactant systems and in AO52 + surfactant systems.

The interactions in solutions would be in the relation with structures of PA and AO52. The structures are given in Fig. 1. The molecule of PA is able to form intramolecular hydrogen bond between the hydroxyl group and the nitro group. Its tendency to hydrophobic interactions is lowered due to a high electron density on the nitro groups. AO52 is of different molecular structure. Two benzene rings are connected by azo group and substituted in the *para* positions. One substituent belongs to sulpho group, and the other substituent to dimethylamino group. Consequently the molecule of AO52 contains both a hydrophilic part and a hydrophobic part. Therefore the molecule can exhibit both ionic and hydrophobic interactions.

Structures of the used surfactants are given in Fig. 1. CTAB and CPC have hydrophobic tails of the same length (16 carbon atoms) and differ not only in the hydrocarbon structures surrounding their nitrogen atoms but also in the anions. BEC has hydrophobic tail of more complicated structure, which contains two oxygen atoms. Total number of atoms in the BEC tail is 20 (carbon and oxygen atoms) and the chain is not linear.

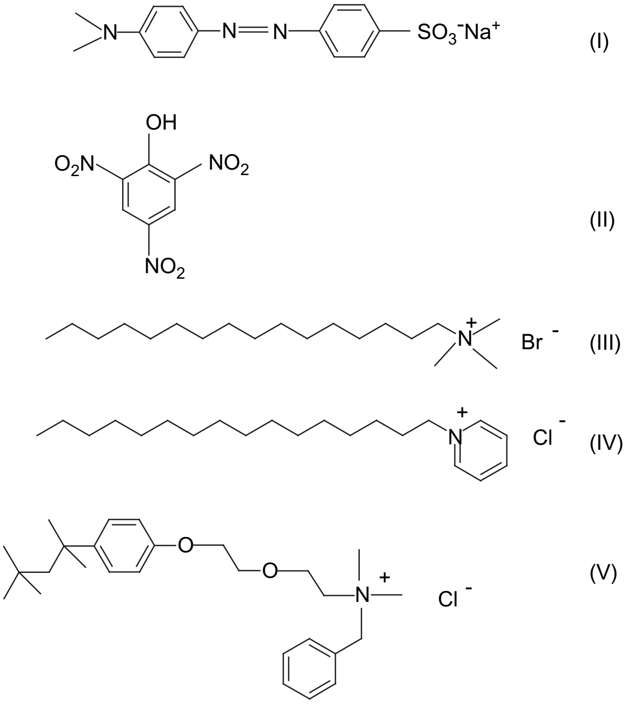

Fig. 1. Chemical structures of the studied substances: AO52 (I), PA (II), CTAB (III), CPC (IV), and BEC (V).

Our work deals with further development of Buwalda method and studies the interaction in both the surfactant + AO52 system and the surfactant + PA system. The experiments are performed at various molar ratios of AO52 or PA and surfactants, but the molar excess of AO52 or PA has attracted the main attention. The molar excess of AO52 and PA is studied by the ion pair extraction spectrophotometry, too.

2. Experimental

2.1. Reagents

Picric acid p. a. (Fluka), C. I. Acid Orange 52 (Merck), boric acid (Lachema Brno), acetic acid (Lachema Brno), phosphoric acid (Lachema Brno), sodium hydroxide (Lachema Brno), chloroform (Lachema Brno), CTAB (Fluka), CPC (Sigma), BEC (Sigma). Picric acid and Acid Orange 52 were recrystallized from ethanol. Chloroform was twice redistilled before every use. Surfactants were used without purification.

2.2. Ion-pair extraction spectrophotometry procedure

The general procedure is described elsewhere [5,6]. Our experiments were carried out as follows: 1.0 ml of AO52 or PA solution of an appropriate concentration, 1.0 ml of surfactant solution (0.1000 mmol/l), 3.0 ml of Britton–Robinson buffer (pH = 7.96) and 5.0 ml of chloroform were pipetted into a test tube. The test tubes containing two liquid layers were sealed with parafilm, shaken for 20 min with a laboratory shaker and remained untouched for 10 min in order to allow both liquid phases to separate completely. The absorbance of the chloroform layer was then directly measured against a blank using a Spekol spectrophotometer (Carl Zeiss, Jena), equipped with a special accessory allowing to use test tubes instead of cuvettes. The blanks were prepared the same way as the mixtures; nevertheless, instead of 1.0 ml of surfactant solution 1.0 ml of distilled water was used.

2.3. UV–vis spectrophotometry procedure

Stock solutions of AO52, PA and surfactants and Britton–Robinson buffer solution (pH = 7.96) were used to prepare solutions containing either AO52 or PA and a surfactant in accurate molar ratios. Actual concentrations are provided in appropriate figures. Then spectra of these solutions were measured in quartz cuvettes by UV–vis spectrophotometer HP 8453 (Hewlett-Packard) against blanks containing no surfactant but containing the relevant excess amount of the dye, eventually.

All experiments were carried out at 25 °C.

3. Results and discussion

The dependence of the absorbance of chloroform extract on the molar excess of AO52 determined by the ion pair extraction spectrophotometry is described in Fig. 2. The surfactant concentration in the water layer was kept constant (0.0200 mmol/l). Hypochromic shifts are shown with increasing dye excess. The curve of BEC exhibits somewhat different shape when compared with the other two. The same dependence, however, with PA used instead of AO52, is given in Fig. 3, in which it may be seen that the absorbance values are not influenced by excess of PA. In both figures, the excess is given as the molar ratio (E) of AO52 or PA and surfactant. In the studied species only the ion pairs and free surfactant particles are supposed to be soluble in the organic layer (chloroform) and the absorbance of this layer in the visible spectral region is therefore ascribed to the ion pairs. A possible explanation of the hypochromic shifts is following: the higher concentration of AO52 in the water layer causes a formation of water soluble as well as chloroform insoluble species. This process shifts the equilibrium of the ion pair formation towards the reactants (i.e. surfactant cations and AO52 anions). The ion pair formation is thus suppressed and a lower amount of the ion pairs is extracted to chloroform. Since the hypochromic shifts are not observed in the systems with PA, the water-soluble species formation is probably strongly supported if significant hydrophobic interactions between the surfactant and the counter ion may be expected. Another explanation of the hypochromic shifts may be the existence of higher concentrations of AO52. The extractability of the ion pairs is negatively affected by changes in the interface between water and chloroform. A question may appear, whether 20 min of shaking is sufficient to reach equilibrium. This was tested by performing the extraction for longer period. Even after 210 min the results remained the same (data not given).

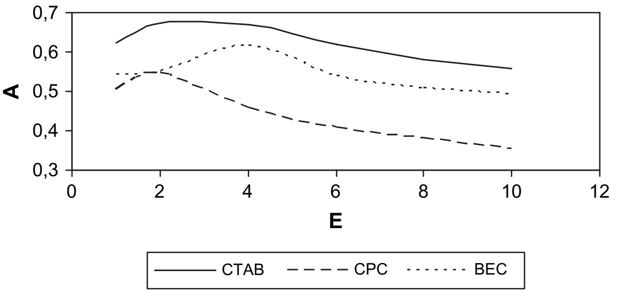

Fig. 2. The hypochromic shifts in the systems of the surfactants and AO52. Surfactant concentration $c = 0.0200$ mmol/l, pH = 7.96, $\lambda = 420$ nm after 20 min extraction.

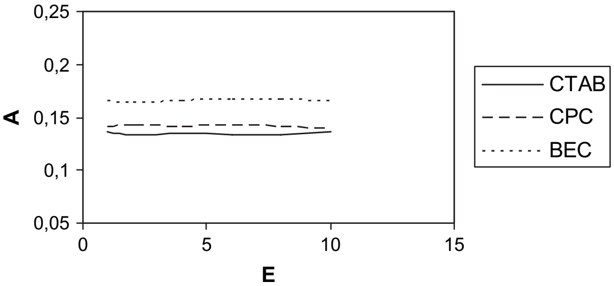

Fig. 3. The dependence of absorbance (A) of the chloroform extract on the molar excess (E) of PA at surfactant concentration $c = 0.0200$ mmol/l, pH = 7.96, $\lambda = 420$ nm after 20 min extraction.

Some other circumstances should be taken into account in case a discussion about the explanation of the hypochromic shift takes place. First problem occurs if the absorbance of the chloroform layer is influenced by free AO52 or PA dissolved in chloroform. If pH (7.96) is used, both AO52 and PA are completely dissociated and therefore insoluble in chloroform. This was verified by a comparison of the absorbances of blanks with the absorbance of pure chloroform at λ_{max}. The measured

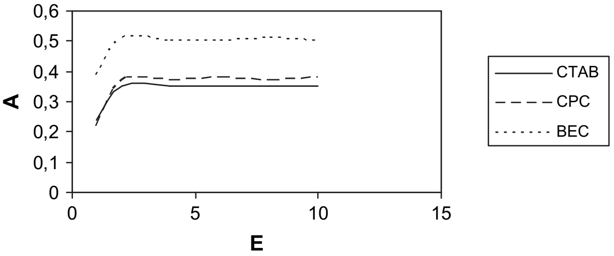

Fig. 4. The dependence of absorbance (A) of the chloroform extract on the molar excess (E) of AO52, if the surfactant was dissolved in chloroform before extraction. Surfactant concentration $c = 0.0200$ mmol/l, pH = 7.96, $\lambda = 420$ nm after 20 min extraction.

absorbance values were very low (near zero) and comparable to each other. They showed that neither AO52 nor PA is soluble in chloroform. The other problem is dimerization of AO52 in the water layer. This problem is discussed later. The surfactants (CTAB, CPC, BEC) are well soluble in both water and chloroform. Their solubilities in chloroform can raise an idea of formation either inverse micelles or some other associates in the chloroform layer. Since the bulk concentration of the surfactants in the chloroform + water system is low (0.0100 mmol/l), a formation of the inverse micelles in the organic layer is not expected and single surfactant molecules are presumed to be present in the chloroform layer. There is an interesting question: whether the single surfactant molecule in the chloroform layer can form premicellar associates by interaction with the ion pairs extracted from the water layer and contribute to the hypochromic shift this way? To check such hypothesis the following experiment has been carried out: 1.0 ml of AO52 solution of appropriate concentration, 3.0 ml of Britton–Robinson buffer (pH = 7.96), 1.0 ml of distilled water and 5.0 ml of surfactant solution in chloroform (0.0200 mmol/l) were mixed in a test tube and the extraction spectrophotometry was performed. Results are given in Fig. 4. The results are sharply different from those in Fig. 2, since no hypochromic shifts have been recorded. The absorbance of the chloroform layer is constant in the range of high excess of AO52, which ensures that the ion pair formation reaction occurs quantitatively. This confirms that the hypochromic shift is observed due to the interactions in the water layer. Our experiments have brought no evidence of association in the chloroform layer. The different results in Figs. 2 and 4 may be ascribed to different ways of ion pair formation. If AO52 is dissolved in the water layer and the surfactant in the chloroform layer (before the extraction), the ion pairs are formed in the interface of both layers and then immediately extracted to chloroform, where probably no further interactions occur. If both AO52 and the surfactant are dissolved (before the extraction) in the water layer, complex equilibria among species in the water solution are set due to both electrostatic and hydrophobic interactions. Consequently lower amount of the ion pairs is extracted into the chloroform layer. If the last experiment is performed with PA, the results are identical with those in Fig. 3. In the case of AO52 the existence of the complex equilibria in the water layer is derived from the experience of some authors, e.g. [1], who performed their experiments by extraction in several steps and observed no hypochromic shifts.

The water phase was studied further by means of UV–vis spectrophotometry. In the first step a former experiment described by Buwalda et al. [7] was performed. Studied water solutions consisted of CTAB and Britton–Robinson buffer (pH = 7.96) with either AO52 or PA. Spectral curves in the system of CTAB + AO52 were measured at concentration of AO52 0.0250 mmol/l and at various concentrations of CTAB from 0.0125 mmol/l to 2.5000 mmol/l at pH = 7.96. The obtained spectra are given in Fig. 5. Buwalda et al. [7] measured the spectral curves at the same concentrations of CTAB and AO52, but at pH = 9.40 and the spectral shifts were explained as a result of the interaction between CTAB and AO52. Our measurement at pH = 7.96 was carried out to verify the spectral shifts at the pH, which corresponds to the other results provided by this work. Comparing Fig. 5 (results at pH = 7.96) and Ref. [7] (results at pH = 9.40) the shifts of the absorption maxima are found in the same range of concentrations of CTAB from 0.0320 mmol/l to 0.6300 mmol/l and consequently the explanation of the shifts should be the same as in Ref. [7], i.e. the interaction between CTAB and AO52. The wavelengths of the absorption maxima in Fig. 5 begin to return to the higher values at CMC of CTAB. Note that the CMC of a surfactant (actually CTAB) decreased in a presence of strong electrolytes (e.g. components of buffer solutions). Our measurements [12] give CMC value 0.23 mmol/l (log CMC = −3.64) for CTAB in the solution of Britton–Robinson buffer pH = 7.96.

At the same concentrations and pH, the spectral curves were measured in the CTAB + PA system. The results are shown in Fig. 6. Some spectral shifts are observable in the concentration range from 0.0500 mmol/l to 0.2000 mmol/l. While the system CTAB + AO52 exhibits blue shifts, in the system of

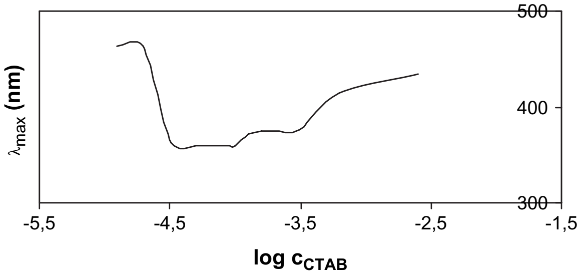

Fig. 5. The spectral shifts in the buffered water solutions containing AO52 (0.0250 mmol/l) and CTAB at pH = 7.96.

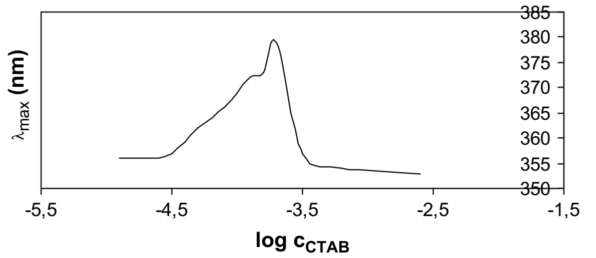

Fig. 6. The spectral shifts in the buffered water solutions containing PA (0.0250 mmol/l) and CTAB at pH = 7.96.

CTAB + PA red shifts are found. This indicates that the interaction in the former system differs from that in the latter. Consequently the formed species in both systems are different. The blue shifts in the CTAB + AO52 system were ascribed to the formation of some associates in the solutions. The explanation of the red shifts in the CTAB + PA system is dissimilar. From Fig. 6 it could be seen that λ_{max} increases within the concentration range of CTAB from 0.0500 mmol/l to 0.2000 mmol/l. This can be explained as a result of increasing perturbations of ionic atmosphere and solvation sheath of the ion pairs by free surfactant ions, especially by their hydrophobic tails causing the compactness of both shells to be disturbed. The perturbations lead to easier excitations of π-electrons by lower energy and consequently to absorption of light at higher wavelengths. These perturbations are cancelled when a self-association (micellization) of the surfactant takes place. A sharp decrease of λ_{max} in the concentration range from 0.2000 mmol/l to 0.3160 mmol/l is therefore observed. The CMC value (0.23 mmol/l) corresponds with this result. Although the origin of the spectral shifts in both studied mixtures is not the same, a hydrophobic character of alkyl chains is always an important factor.

Our experiments were then directed to study the molar excess of AO52 or PA in buffered water solutions of surfactants. The experiments were carried out at the surfactant concentration 0.0200 mmol/l and at various concentrations of the dyes. The concentration 0.0200 mmol/l is the bulk concentration of the surfactants in the water layer in the ion pair extraction spectrophotometry. If the dye concentration exceeds the concentration of the surfactant, a broad band of the free dye appears in the spectrum. The dye broad band overlaps the other bands of the interest; hence these measurements require a compensation of the dye absorption band in the spectra. The compensation was performed by addition of the excess amount of the dye into the blanks. Since the ion pair formation was expected at the molar ratio 1:1, the excess concentration of the dye was calculated as a part of the dye concentration exceeding the concentration of the surfactant in the appropriate sample. Elimination of the absorption of free dye was thus successfully reached. A drawback of this method is that the absorbance values lose their information ability in the cases when the formation of species different from the ion pairs occurs. The spectra obtained with solutions of various concentrations of AO52 at the constant concentration of CTAB, CPC and BEC are given in Figs. 7, 8 and 9, respectively. In Figs. 7 and 8 large blue shifts are recorded if the molar concentration of AO52 exceeds the molar concentration of the surfactants. A "transition" molar ratio of AO52 and the surfactant is 2. At this ratio absorption band at 363 nm (resp. 357 nm) begins to rise at the expense of absorption band at 464 nm. At the molar ratios higher than 2 the absorption band at 363 nm (resp. 357 nm) is dominant. Intersections of the spectral curves at approximately 400 nm and 541 nm are too scattered to consider them to be isosbestic points. The band at 464 nm is the main absorption band of pure AO52 in the visible spectral region. The absorption maximum at 464 nm is observed in Figs. 7 and 8 if the molar ratio of AO52 and the surfactant is 1:1, i.e. if the probability of the ion pair formation is very high. Hence we conclude that the absorption maximum of the ion pairs in the studied systems is the same as the absorption maximum of pure AO52 and large blue shifts in Figs. 7 and 8 are not caused by ion pairs in the solutions.

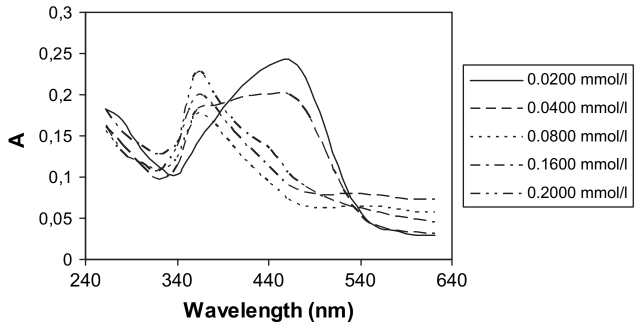

Fig. 7. The spectra of the buffered water solutions containing CTAB (0.0200 mmol/l) and AO52 at pH = 7.96. AO52 concentrations are given in the legend.

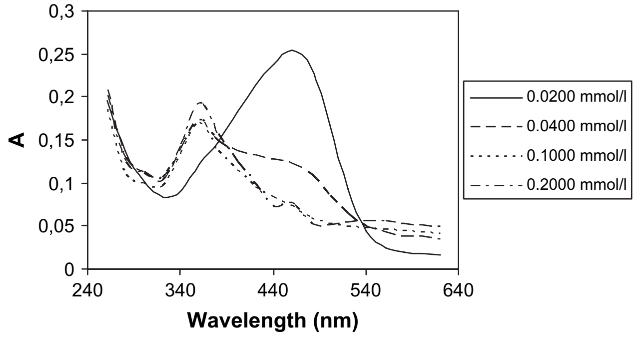

Fig. 8. The spectra of the buffered water solutions containing CPC (0.0200 mmol/l) and AO52 at pH = 7.96. AO52 concentrations are given in the legend.

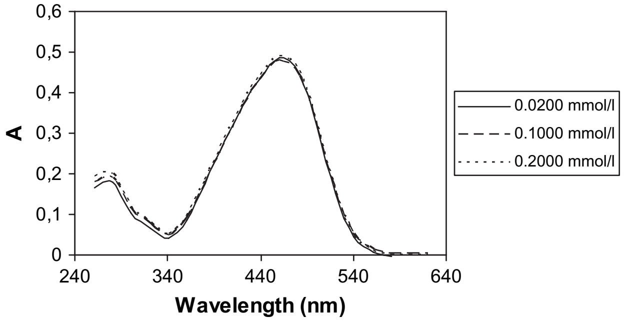

Fig. 9. The spectra of the buffered water solutions containing BEC (0.0200 mmol/l) and AO52 at pH = 7.96. AO52 concentrations are given in the legend.

Similar opinion has been published by Buwalda et al. [7]. At higher concentrations of AO52 the existence of its dimmers should be discussed. We cannot exclude a presence of the AO52 dimmers in the solutions; however, since the dimmer absorption band exhibits a blue shift of 4 nm from the band of AO52 monomer [13], the large shifts obtained in this work cannot be ascribed to the formation of AO52 dimmers. The shifts in the Figs. 7 and 8 are comparable with those in Fig. 5 and in publication [7]. Hence we can suppose a certain similarity of arrangement of the species causing the large spectral shifts in Figs. 5, 7 and 8. Such species (associates) are formed in solutions containing both AO52 and CTAB or CPC since the shifted absorption maxima are observed just in the mixtures of AO52 and CTAB or CPC (not in the solutions without the surfactants). Consequently the absorption bands at 363 nm (resp. 357 nm) can be explained by a formation of associates including both AO52 and the surfactant in their structures. The associates are expected not to be soluble in chloroform. This is derived from the hypochromic shifts themselves. But if we presumed the solubility of the associates in chloroform, our results would not be in agreement with the presumption. Buwalda et al. [7] have shown and we have verified (data not given) that in non-polar solvents the spectral shifts observable in water are not found. This is interpreted as an absence of any associates. Therefore associates soluble in chloroform should be decomposed after extraction to chloroform to form the ion pairs and free molecules (not ions) of either AO52 or the surfactant. Neither free molecules of AO52 nor free molecules of the surfactant would cause the hypochromic shifts after the equilibrium having been achieved. The presence of the free molecules of surfactant in the chloroform layer has been studied (see Fig. 4). The presence of the free molecules of AO52 in the chloroform layer would rather lead to hyperchromic shifts. Finally, the explanation of the observed hypochromic shifts is possible, if the associates are insoluble in chloroform.

The spectra in Fig. 9 are different from those in Figs. 7 and 8. All spectral curves have their absorption maxima at 464 nm and the AO52 excess has not caused any changes in the spectra. Therefore the hypochromic shift in Fig. 2 cannot be explained by the formation of the associates in the water layer. The hypochromic shift obtained by the extraction spectrophotometry with the BEC solutions can be connected with interfacial phenomena in the water + chloroform system but this hypothesis requires a confirmation, which is beyond the frame of this work.

The spectra of the solutions with PA instead of AO52 were taken but no spectral shifts were recorded. Absorption maxima of all spectral curves were found at 356 nm (i.e. at the absorption maximum of pure PA) and no additional maxima appeared there. In Fig. 10 the spectra of CPC + PA system are given as an example. If just the ion pairs are formed in the solutions of the surfactants and PA, spectral shifts are not caused by their formation. This confirms again the conclusion of Buwalda et al. [7] who have doubted that the spectral shifts could be raised by the formation of the ion pairs.

Comparing the studied systems with AO52 and with PA we can deduce that different interactions lead to the formation of different associates in the appropriate solutions and more or less complicated equilibria are set. Actually, in the solutions of AO52 and CTAB or CPC associates of AO52 and the surfactant are formed according to the results of UV–vis spectrophotometry. The ion pair extraction spectrophotometry shows the associates to be insoluble in chloroform; hence they have an ionic character (on the contrary to the ion pairs). The formation of the ionic associates competes with the formation of the ion pairs. At the high concentrations of AO52, the formation of the ionic associates can be thermodynamically more convenient due to the sharper drop of the Gibbs energy, caused by sharper decrease in the bulk contribution of the AO52 chemical potential. In addition, some ion pairs can be incorporated into the structure of the ionic associates (solubilization) [14]. Thus two processes (the formation of the water-soluble

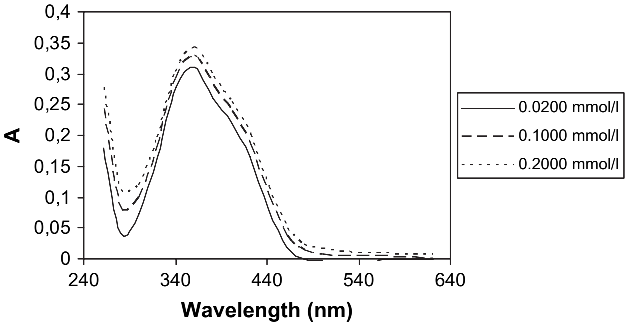

Fig. 10. The spectra of the buffered water solutions containing CPC (0.0200 mmol/l) and PA at pH = 7.96. PA concentrations are given in the legend.

associates and the solubilization of the ion pairs) can control the formation of water-soluble associates and can be responsible for the lowered amount of the ion pairs in the chloroform extract, when the concentration of AO52 is increased. On the contrary, in the case of solutions containing PA the only thermodynamically preferable process is the ion pair formation. The solubilization of the ion pairs in the surfactant + PA system is not thermodynamically convenient probably due to the significantly lower hydrophobic character of picrate anion compared to AO52 anion.

4. Conclusions

1. According to our observation, PA forms the ion pairs with CTAB, CPC and BEC in the water solutions and no more complex associates occur there, even if PA is used in the molar excess. Its behaviour is, therefore, different from that of AO52 and this is confirmed by the results of extraction spectrophotometry. In the solutions of AO52 and CTAB or CPC various associates are formed. A significant portion of them is soluble in water but insoluble in chloroform and the water-soluble associates are formed at the expense of the ion pairs.
2. Although the hypochromic shift of the chloroform layer has been observed with the solutions of CTAB, CPC and BEC, in the BEC solutions no associates (different from the ion pairs) have been confirmed by UV–vis spectrophotometry. Hence the hypochromic shift in the case of BEC is estimated as a matter of interfacial phenomena in the water + chloroform system. Different ways of the origin of the hypochromic shifts are probably connected with the different structures of the surfactants. The oxygen atoms in the BEC hydrocarbon chain significantly lower its hydrophobicity and the ability of BEC to form more complex associates with AO52.
3. The results obtained by both methods used in this work are in a very good agreement. The only exception is the system BEC + AO52, where the hypochromic shift in the chloroform layer is not explainable by the UV–vis spectrophotometry results. As indicated above some of our results support the conclusions of Buwalda et al. [7].
4. A connection of UV–vis spectra, ion pair extraction spectrophotometry results and the hydrophobicity of hydrocarbon chains in the structures of CTAB and CPC is shown. Thus a great significance of the hydrophobicity for the interactions of dyes and surfactants is confirmed again. Similarity of the results obtained with CTAB and CPC supports this conclusion since both surfactants are of the same hydrophobic tail; however, they differ in the anions and in the structures surrounding their nitrogen atoms.

Acknowledgement

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