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Electrokinetic Study of Adsorption Layers on Different Surfaces*

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In the first part the results of investigating interdependences of parameters of the electroosmotic displacement process (ED) and properties of adsorption layers of SAS have been considered. ED-process, connected with flow of two immiscible liquids (»water« and »oil«), filling the pores of diaphragm. Direct current is superimposed on the water-containing part. The complex investigations have been undertaken of the influence of SAS nature and concentration on the displacement kinetics, oil output, wetting angle, surface tension and the filtration velocity of oil with added SAS through the diaphragms, the SAS-layers in which were formed beforehand. In accordance with the results it follow that the character of dependences, in which the effectivenes is reflected, is very sensible to the change of adsorption layer properties, to their stability, the velocity of destruction, the values of the primary and residual wetting degree. In the second part the modifying action of ionic surfactants using 2 types of interfaces: oxides (quartz) and ion-exchange membrane have been considered and compared. The adsorpiton of positive (CTA^+) and negative (DS^-) surfactant ions at the quartz-aqueous solution interface, non-Longmuir type adsorption isotherm, a change in the sign of ζ-potential of quartz in CTAB solutions and surface conductivity in IEP are observed. Ion-exchange membranes in surfactant solutions exhibit a selective adsorption (only surfactant counterions are sorbed), sorption isotherms are of the Langmuir-type and as result of sorption of surfactant counterions the ion-exchange membranes lose its high permselectivity.

INTRODUCTION

In the first part of this paper results of the investigation of the relationship between parameters of the electroosmotic displacement process (further the abbreviation —ED) and properties of adsorption layers of SAS have been presented.

The rough scheme of the ED is given in Figure 1. There is a diaphragm, the pores of which are filled with two immiscible liquids. One is an aqueous solution of electrolyte (»water«), the other is any liquid insoluble in water (»oil«). Every liquid fills a separate diaphragm part. Direct current is superimposed on the water-containig part — »active part« (the thickness is marked

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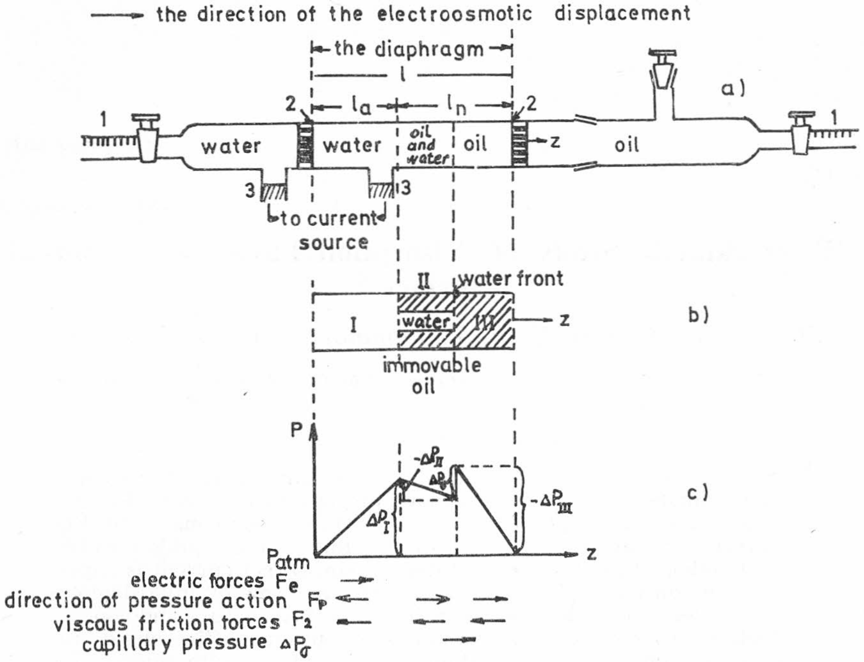


Figure 1. Schemes of the electroosmotic displacement: a) device: 1 ~ measuring capillaries; 2 ~ big pore disks; 3 ~ electrolytic bridge; b) scheme of displacement; c) sheme of internal pressure (Δ p) development and of the directions of general forces acting in diaphragms.

l_a). The electroosmotic stream arising in the active part is directed into other diaphragm part (»passive part«) being outside the electric field and containing oil at the beginning of the experiment.

The study of ED is interesting for problems dealing with flow phenomena of several different liquids in finely-porous diaphragms. During the ED some part of oil is as a rule retained behind the front of advancing water (»the front«), moving here very slowly or being immobile. Therefore the efficiency of ED is defined by two parameters: the volume velocity —q_{ed} and the oil output (related to the oil quantity at the beginning of the experiment) — β. The reasons of incomplete displacement are various. In particular, it may be caused by SAS adsorption layers (»ad-layers«). One of the main problems of the ED research is to establish the relationship of process parameters and properties of ad-layers.

The scheme of force distribution in a capillary system in some moment of process is represented in Figure 1 (it has been assumed that the surface of the solid has a hydrophilic character)[1].

The retention of the oil part behind the front means that $\beta < 1$ and that q_{ed} alters with time. The direction of the alteration, parallel with β, depends on the relation of liquids viscosities and on the direction of forces at the compared moments of time[1]. With equal viscosities, the front is inside the diaphragms (»unaqueous period of displacement« ~ scheme in Figure 1) and with

$\beta \neq 1$ the velocity must decrease with time — the more intensively, as β decreases (see position *1* in Figure 2, where the illustrative scheme of different meniscus positions can be seen. For visual demonstration it is assumed, that the region of the drop of water saturation is infinitesimal).

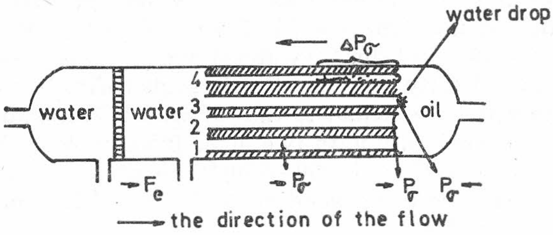


Figure 2. Illustration of the relative role of the capillary pressure and of the end effects for the diaphragm model.

The aforesaid character of velocity alteration with time occures until the front reaches the diaphragm outlet. When the front arrives at the capillary end, the following may be expected. First, (Figure 2, position *2*) the movement of the front decreases, due to the tendency of water to remain inside the hydrophylic capillary. An additional effort is needed to force it out. From this moment the meniscus changes its curvature, p_σ decreases and then possibly an inversion of its direction takes place (Figure 2, position *3*). The water drops appearing on the capillary end spread out and finally yield a continuous water phase. From this moment water can easily flow out (»the water-break«) with an increasing velocity. This is »an aqueous period of the process«.

The scheme in question is certainly more intricate in the case of real systems having a complicated porous-space (like soil). In the latter the front is diffused, where polar and nonpolar liquids form moving drops (Figure 2, position *4*) and where local capillary effects proceed[2]. The »end-effect« considered will have a greater influence when p_σ and F_e are mutually commeasurable.

In this case the observed value q_{ed} could pass through a minimum at some moment during the experiment. The depth of this minimum was related to the ad-layer, promoting an alternation of the wetting an structure parameters and their change in the process (e. g. destruction) — all these reasons affect essentially the distribution of forces and according to it, the main parameters of displacement.

In this paper the adsorption layers formed at the boundary of solid-oil (oilsoluble SAS) are considered.

Complex investigations have been undertaken regarding the influence of SAS nature and concentration on the displacement kinetics ($q_{ed} = f(t)$), the oil output (β), the wetting angle (Θ), the value of surface tension (σ) and the filtration velocity of oil with added SAS (q_f) through the diaphragms the SAS-layers in which were formed beforehand (corresponding to the filtered solution). The experiments were made on diaphragms of quartz powder with an equivalent particle diameters of 8—16 microns. For the statical wetting angle measurement a quartz ground end was sunk in oil and a water drop was placed on its surface. The experimental errors were: in the case of q_{ed} and q_f $\pm 2\%$, in Θ ±1°, in β — the precision was dependent on the measured value, for $\beta > 0.15$ it was more than 2%, while for smaller β values the error increased. The rough scheme of the device applied for electroosmose research is represented in Figure 1a[3]. The oil output was determined by water extraction on the passive part after the experiment was over. When measuring the velocity of filtration the whole diaphragm was filled with oil. In the experiment a water solution of KCl, $c = 10^{-3}$ mol dm^{-3} (»water«) and a mixture of nonpolar hydrocarbons with dissolved fatty and naphthenic acids (SAS) (»oil«) were used. The viscosity of the mixture was equal to that of water. The results represented in the paper were connected only with two acids from naphtha *N1* and *N2* the T_{BP} of which were (260—290) °C and (290—330) °C respectively. The values of Θ, σ and the minimal pressure corresponding to the oil filtration with broken ad--layers -p_0 are given in Table I.

TABLE I

NN of systems	Oil and SAS	c_{SAS} / % weight	σ / erg/cm^2	Θ	p_0 / cm H_2O
1	non-polar	0	51	29°	0
2	non-p+acid 1	0.1	43	38°	0
3	non-p+acid 1	0.5	34	48°	2.2
4	non-p+acid 2	0.5	32	39°	0.8
5	non-p+acid 1	1.0	29.5	42°	2.5
6	non-p+acid 2	1.0	29.5	41°	1.1

Results of the dependence: $\beta = f(I)$ are represented in Figure 3; $q_{ed\ rel} = f(t)$ ($q_{ed\ rel}$ is the relative displacement velocity at a given moment plotted against the velocity at some initial arbitrary moment). Any curve corresponds to some definite current. For the sake of convenient results comparison, each given system is marked with the same *N* in all the Figures and Table I.

Results obtained when using non-polar oil (*N1*) showed the folowing: a) the dependence of q_f on p conformed to the Darsi law ($p_0 = 0, \frac{dq_f}{dp} = \text{const}$); b) the oil output was big and only slightly depended on *I*; c) q_{ed} passed the clearly expressed minimum under the correspoding choice of *I* value.

All results obtained indicate the absence of spread boundary-layers (in accordance with literature[4] and can be explained on the basis of the aforesaid suggestion of the electroosmotic displacement mechanism, of the role of wetting and of the electric and capillar forces correlation.

Let us now consider the results obtained in the presence of SAS. The presence of SAS promoted in nearly all cases a decrease of Θ and σ and in many cases the distinction of p_0 from 0. Let us take notice of complicated dependence of Θ not only upon the nature, but also upon the SAS concentration[5].

The inequality of p_0 and 0 shows the existence of phases with resilient-plastic properties. In our case these phases could be adsorption layers only. A rise of the SAS concentration demonstrated the marked role of these properties in the overall balance of mechanical behaviour of the liquid in pores.

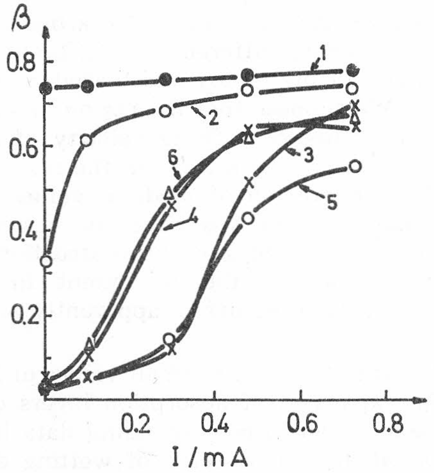
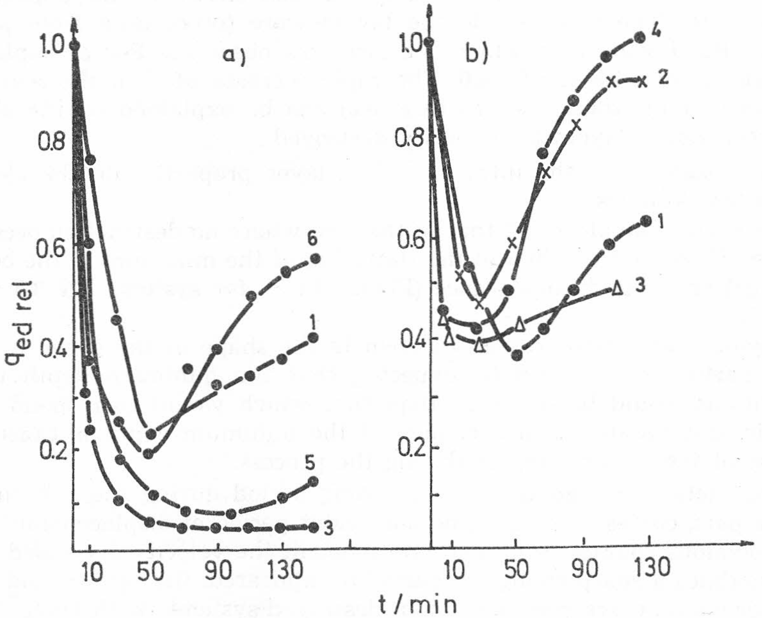


Figure 3. The dependence of non-polar oil to the SAS output (β) on the current (*I*). The numeration of curves corresponds to the system numeration in Table I.



a) I = 0.3 mA

b) curves 1,2,4 ~I = 0.5 mA
3 ~I = 0.75 mA

Figure 4. Change of the relative electroosmotic displacement velocity with time.

As it can be seen from the results of dependences $\beta = f(I)$ (Figure 3.) the latter were substantially more complicated in the presence of SAS than in their absence, the observed character of dependence being sufficiently various. Here parts of slight dependence β on I and parts with considerable alternation of β can be observed.

In some cases (~ curves 2, 3, 5 in Figure 3) a big rise of β was observed within narrow limits of I, being different for different cases; in some cases in wide limits of I values, an invariably substantial β rise was observed (see Figure 3 — curves 4, 6). We suppose that the region of substantial β alteration corresponded to the appearance of a finite velocity of adsorption layers destruction and its gradual increase with time. In the case of systems with sharp change of β observed in the region of medium values of I (curves 3, 5) — when I values were smaller, there was either no destruction at all or very slight destruction; when I's were bigger, the destruction occurred as soon as the field was superimposed and to the full extent. In the cases of systems *2, 4, 6,* a considerable destruction occurred, apparently already at small values of I.

By comparison of results obtained at small values of I ($I \rightarrow 0$) the influence of the wetting and the expansion of adsorption layers on the oil output can be estimated. It can be seen, when corresponding data in Table I and results in Figure 3 are compared, that a decrease of wetting due to the adsorption layer development, always resulted in oil output decrease, being increasing as a rule with the wetting degree decrease and with the expansion of the adsorption layers (p_0) (compare, for example, systems *1—2, 2—3*).

The increase of layer relative stability was also shown by the appearance of curve $\beta = f(I)$. The more stable the layers were (proceeding from p_0 the bigger were the I values, at which the rise was observed. For example one can compare systems 2—3; 3,5—4,6. The rapid increase of β in the region of small values of I for some systems (e. g. *N2*) can be explained by the ability of the corresponding layers to be easily destroyed.

Let us consider now the influence of ad-layer properties on the electroosmotic process kinetics.

It folows from the aforesaid that in the case where no destruction occurred the decrease of wetting resulted in the flattering of the minimum in the curves of $q_{\text{ed rel}} = f(t)$ or in its disappearance (Figure 4a ~ for systems *NN* 3,5 when $I = 0.3$ mA).

The layers destruction was also shown in the shape of the curve $q_{\text{ed rel}} = f(t)$. In particular, it could be expected that the minimum depth under these conditions would be different than that which would correspond to Θ measured in static state. Such a change of the minimum depth is caused by the increase of the wetting degree during the process.

The destruction was going on over a long period during the experiment. The curve's part, corresponding to the non-water period of displacement, could gain an anomalous course- from a certain moment the velocity decreased more slowly, sometimes a sharp change in curvature appeared, the curve could cross with another curve, corresponding to non-destroyed systems. With layers being destroyed sufficently slowly, the minimum could turn out to be very pronounced; sufficiently complete destruction during the experiment caused a considerable increase of relative velocities in the final stages of the experiment.

The aforesaid facts were confirmed in most cases (Figure 4a ~ to systems *6* when $I = 0.3$ mA and in Figure 4b to systems *NN* 2,4 ~ $I = 0.5$ mA, the influence of the layer destruction on the minimum display ~ curves 3 in Figure 4a and 4b).

The last fact to which attention should be payed in this part of our paper is the considerable increase of the displacement efficiency of the oil containing SAS when the electric field was superimposed.

In accordance with results represented here it follows that the presence of SAS has a very strong influence on the displacement efficiency — the oil output, the process velocity and its kinetics. The character of dependences reflecting the efficiency is very sensible to the change of adsorption layer properties, to their stability, the velocity of destruction and values of the primary and residual wetting degrees. We think that these results are interesting not only because certain information regarding the influence of SAS on the electroosmotic displacement efficiency can be obtained, but also because by this method extensive investigation of adsorption layer properties can be performed. By this method more information about the adsorption layer properties and their behaviour in different conditions can be obtained than by filtration.

In the second part of our paper we will discuss the results of comparative investigations of the modifying action of ionic surfactants using two types of interfaces: oxides (quartz as a typical oxide) and ion-exchange membranes. The quartz samples were disk (made from crystalline and melted quartz), diaphragms from fibres (100 μm in diameter) and powder (particle diameters of 5 to 8 μm). The ion-exchange membranes used were a strongly acidic cation — exchanger, MK-40, and a strongly basic anion-exchanger, MA-40, (USSR). Sodium dodecyl sulphate (NaDS) and cetyltrimethylammonium bromide (CTAB) were used as ionic surfactants.

Electrokinetic properties (the ζ-potencial and surface conductivity) for quartz diaphragm and electrochemical characteristics (transport numbers of ions and electric conductivity) for membranes in solutions of SAS as well as surfactant sorption on these samples were investigated.

RESULTS AND DISCUSSION

First we will consider quartz. According to modern concepts, the surface charge of oxides, σ, appears as a result of adsorption Γ of potential-determining H^+ and OH^- ions:

$$SiO^- + 2H^+ \rightleftharpoons SiOH_2^+$$

$$\sigma = F\,(\Gamma_{OH^-} - \Gamma_{H^+})$$

In the neutral pH range the quartz surface has a negative charge owing to the preferential adsorption of OH^- (the isoelectric point, IEP, of our quartz samples was 2.0). One could expect therefore that the sorption behaviour of CTA^+ and DS^- ions on quartz would be different. The adsorption isotherms for NaDS and CTAB on quartz powder are shown in Figures 5 and 6[6].

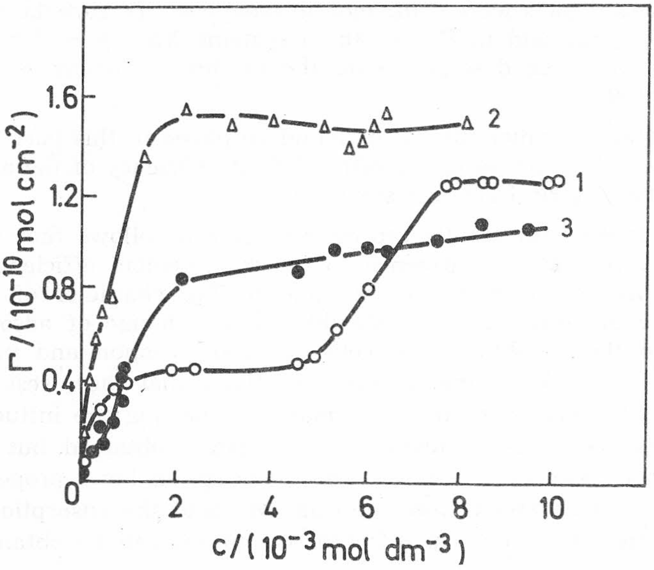


Figure 5. Adsorption isotherms for NaDS on quartz powder; (1) — pH = 6, t = 20 °C; (2) — pH = 3, t = 20 °C; (3) — pH = 6, t = 50 °C.

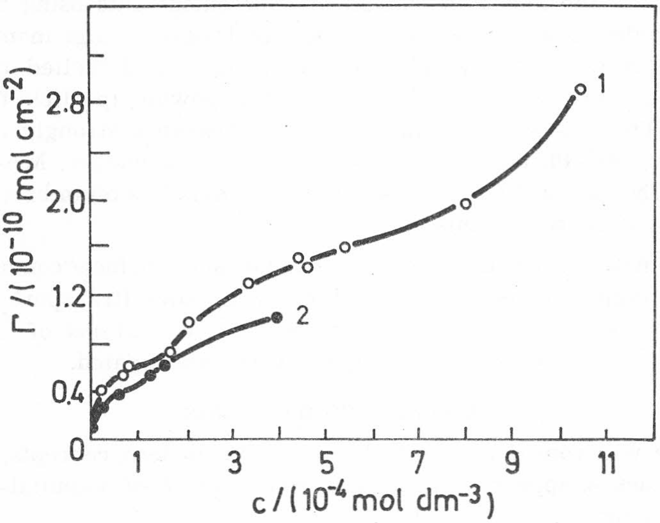


Figure 6. Adsorption isotherms for CTAB on quartz powder, at pH = 6, (1) t = 20 °C; (2) t = 50 °C.

The isotherms consist of two regions; in the neutral pH range the adsorption of CTAB greatly exceeds that of NaDS. This complex character of isotherms can be explained by the formation of the first ion layer on the solid-liquid interface as a result of electrostatic attraction and by the formation of the second layer (or hemimicelles) owing to the van der Waal's and hydrophobic interactions. This adsorption mechanism is also confirmed by the character of changes of contact angles on the quartz surface in solutions of these surfactants: an increase in hydrophobic properties in the first plateau region and their decrease in the second plateau region. Probably, negative SiO^- surface groups are adsorption centres for CTA^+ whereas positive $SiOH_2^+$ groups are the centres for DS^-. The presence of negative SiO^- groups prevents adsorption of DS^-, consequently when the negative surface charge decreases, the sorption of DS^- should increse. In fact, the adsorption of NaDS at $pH = 2$ (this corresponds to IEP for quartz) is much higher than that at $pH = 6$. The temperature increase has also an effect (Figures 5 and 6).

Electrokinetic measurements in solutions of SAS substantiate these concepts of mechanism of adsorption of CTAB and NaDS on a quartz surface. The streaming potentials, E_s, were measured with diaphragms of quartz fibers or with rotating disc on which a film of the SAS solution was spread. In the latter case E_s was calculated by the equation

$$\zeta = \frac{8 \pi \varkappa \nu E_s}{\varepsilon \omega^2 (r_2^2 - r_1^2)}$$

where $\varkappa$ and ε are the specific conductivity and dielectric permeability of solution, ν is its kinetic viscosity, ω is the angular velocity of disc rotation and r is the distance from the centre of disc to two ring electrodes. The curves of ζ vs. lg c are shown in Figure 7.[7] For CTAB a typical S — shaped curve of specifically adsorbed counter-ions, exhibiting a change in the sign of charge at $c = 4.0 \times 10^{-5}$ mol/dm^3 IEP was observed. The run of curve 2 for the NaDS solutions differs from that observed for an indifferent KCl electrolyte.

In contrast to KCl for which $|\zeta|$ decreases with increasing c, in NaDS solutions (owing to DS^- adsorption) the negative charge of the surface increases

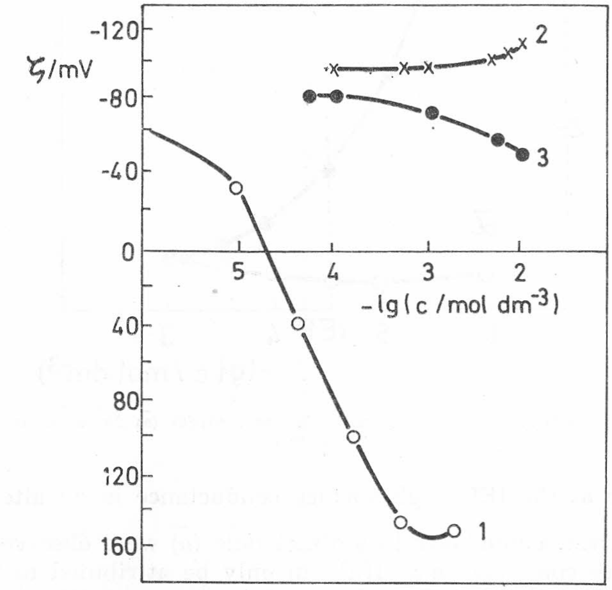


Figure 7. Variation of the electrokinetic potential of the quartz disc as a function of concentration, 1 — CTAB, 2 — NaDS, 3 — KCl.

with increasing c and compensates the compression of the diffuse part of the electrical double layer. As a result, practically no change in the value of ζ occurres.

The results of investigations of ζ vs. pH in mixed systems NaDS + HCl ($c_{\mathrm{NaDS}} = 10^{-2}$ mol dm^{-3}) provide another evidence for NaDS adsorption on quartz. It was found that the IEP of quartz shifts by I towards lower pH, which indicates that the anion is specifically adsorbed. The values of adsorption potentials Φ_i of CTA$^+$ and DS$^-$ ions were calculated for different adsorption models from IEP's using the Langmuir-Stern equation for sorption of several ions and the expression for the equilibrium constant K_i at $\Psi_i = 0$ $K_i = e^{-\Phi/TR}$. The value of $\Phi_{\mathrm{CTA^+}}$ is of the order of magnitude of 10—14 kcal/mol while $\Phi_{\mathrm{DS^-}}$ amounts to 5 kcal/mol.

Now we shall consider the results of the surface conductivity ($\varkappa_s$) measurements, in a solution of SAS, made for quartz fibre diaphragms. It was found that the values of the specific surface conductance K_s in surfactant solutions exceeded the corresponding K_s values in solution of simple ions by almost an order of magnitude. These results are due to the high adsorption of surfactant ions on the quartz surface.

Most interesting results were obtained in CTAB solutions (Figure 8).[8]

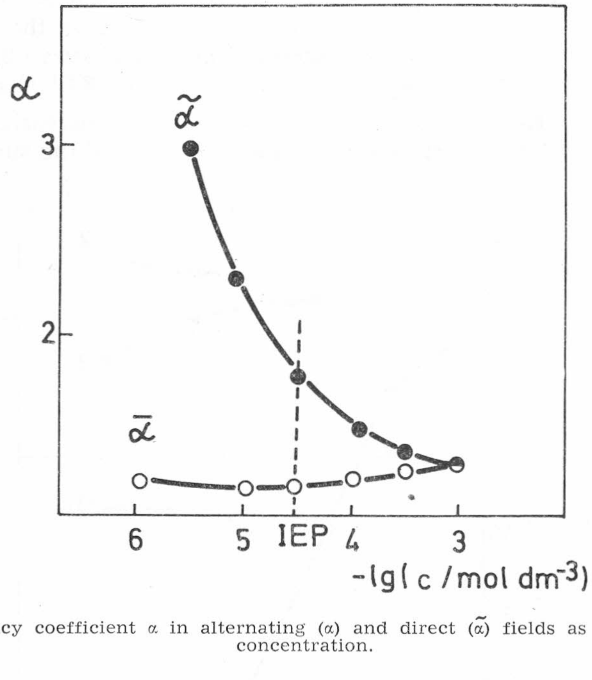


Figure 8. Efficiency coefficient α in alternating (α) and direct $(\tilde{\alpha})$ fields as function of CTAB concentration.

In this case at the IEP, high surface conductance in an alternating field $(\tilde{\alpha})$ and low surface conductance in a direct field $(\bar{\alpha})$ were observed. The appearance of surface conductance at IEP can only be attributed to the fact that CTA$^+$ ions are adsorbed on the external surface of quartz and cannot penetrate the »porous« layer permeable for ions (the »gel layer«). Hence at IEP, small counterions (mostly H^+ ions) also remain in the gel layer, compensating the negative charge of the matrix and exhibiting mobility in an alternating field (a). So in this case CTA^+ ions act as a kind of probe revaling the existence of fine surface structure.

By comparison of the values of K_s and Γ for CTAB the evaluation in the first approximation of the mobility of ions (λ_s) in the electric double layer from the equation $K_s \approx \Gamma' \lambda_s$ was possible. It was found that at low surface coverage the mobility of bromide ions λ_{Br^-} is equal to that in a free solution. The value of λ_{Br^-} decreases with increasing c_{CTAB} in solution (in accordance with the increasing surface coverage); probably this is due to increasing hindrances to ion motion with the increase of c_{CTA^+} on the surface.

Summing up the results of investigations of surfactant adsorption on quartz, it should be noted that surfactant ions bearing charges of both signs are adsorbed, that the adsorption isotherms for both ions consist of two regions and that in the case of the adsorption of surfactant counterion the surfaces is recharged.

Different situation is observed for ion exchange membranes in surfactant solutions. Studies of sorption (Figure 9) showed that these membranes exhibit a pronounced selective adsorption: only surfactant counterions are sorbed, while virtually no sorption of organic co-ions occurs. Moreover, sorption isotherms are of the Langmuir type and their plateau is near *c.m.c.* It appears that no formation of surfactant hemimicelles in membranes takes place; this is also indicated by similar values of surfactant sorption and exchange capacity of membranes. For DS^- on the MA-40 anionic membrane the value of sorption is somewhat higher than the exchange capacity. The result as well as the slow attainment of sorption equilibrium (over a month for CTAB) indicate that steric hindrances profoundly affect the sorption of organic counterions in membranes.

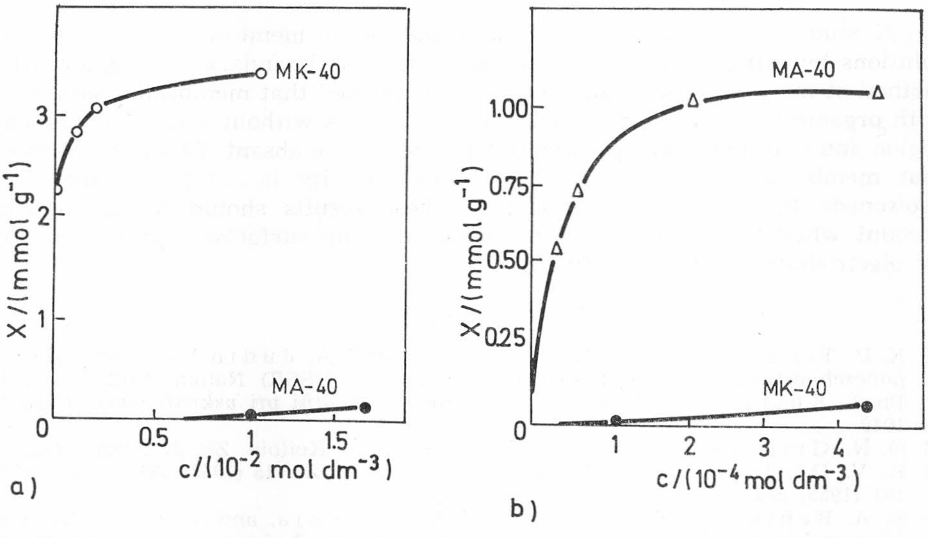


Figure 9. Sorption isotherms of NaDS (a) and CTAB (b) on ionic exchange membranes MK-40 and MA-40.[9]

It was possible to follow the changes in electrochemical properties of ion exchange membranes during the sorption of surfactant counterions, by measuring the counterion transport numbers t_i in membranes. The values of t_i were found to decrease during the surfactant sorption from $t_i \approx 1$ up to $t_i \approx t_i^0$ (where t_i^0 is the transport number in a free solution). This situation is observed for MA-40 saturated with NaDS. For quartz diaphragms the sorption of a surfactant counterion (CTA^+) led to a change in the sign of ζ, whereas in this case only complete screening of the matrix charge was observed.

A study of the temperature dependence of the electric conductivity of membrane showed that for these membranes the electric conductivity activation energy in surfactant solutions is 8—10 kcal/mol, in comparison with the value of 5—6 kcal/mol for these membranes in solution of simple ions.

An investigation of electric conductivity also showed that the electric resistance of membranes saturated with surfactant counterions, is by one or two orders of magnitude higher than that of membranes in solution of simple ions, whereas the mobility and diffusion coefficient of the former are lower by approximately an order of magnitude. These data are shown in Table II.

TABLE II

The system	$\frac{c}{10^{-3}\ \mathrm{mol\ dm^{-3}}}$	Water content	Coeff. of structure resistance β	Efficiency coeff. α	$\frac{\bar{\lambda}_i}{\Omega^{-1}\ \mathrm{cm^2\ mol^{-1}}}$	$\frac{\bar{D}}{10^{-7}\ \mathrm{cm^2/s}}$
MK-40 NaCl	1	0.37	3.9	15	0.40	1.0
MK-40 CTAB	0.4	0.35	4.0	< 1	0.02	0.06
MA-40 NaCl	10	0.40	9.2	7	1.90	4.8
MA-40 NaDS	9.8	0.22	20.4	1	0.07	0.2

A study of the concentration polarization of membranes in surfactant solutions by using $i - e$ curves and the method of boundary polarization (the method of membrane polarization potential) showed that membranes saturated with organic counterions exhibit linear i—e curves without a limiting current region and that membrane polarization potentials are absent. This also indicates that membranes lose their electrochemical activity ($t_i \approx t_i^0$), i. e. they are »poisoned« by surfactant counterions. These results should be taken into account when the treatment of sewage containing surfactants is carried out by electrodialysis and hyperfiltration.

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SAŽETAK

Elektrokinetička istraživanja adsorpcijskih slojeva na različitim površinama

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Na temelju elektroosmotskog pomaka istraživana su svojstva adsorpcijskih slojeva na faznoj granici krutine i otopine u kojoj je površinski aktivna tvar otopljena u otapalu koje se ne miješa s vodom. Dijafragme korištene kod mjerenja elektroosmotskog pomaka bile su od kvarcnog praška kojemu je promjer čestica iznosio od 8 do 16 μm. Vodena faza bila je otopina KCl, a kao faza koja se ne miješa sa vodom upotrijebljena je smjesa nepolarnih ugljikovodika u kojima su bile otopljene masne kiseline i kiseline iz nafte, kao površinski aktivne tvari. Ispitan je utjecaj prirode i koncentracije površinski aktivnih tvari na različite parametre koji se mogu odrediti na temelju mjerenja elektroosmotskog pomaka.

Također su izvršena usporedna istraživanja utjecaja ionskih površinski aktivnih tvari na različite površine kvarca, kao i na ionsko-izmjenjivačke membrane. Kao površinski aktivne tvari primijenjeni su natrij-dodecilsulfat i cetiltrimetilamonij-bromid. Za kvarcne dijafragme određen je elektrokinetički potencijal i površinska vodljivost, a za membrane u otopinama površinski aktivnih tvari određeni su prijenosni brojevi iona, vodljivost te adsorpcijski parametri.

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