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FEATURES OF ADSORPTION OF BINARY MIXTURES OF CATIONIC AND NON-IONIC SURFACTANTS BY NON-POROUS CARBON SORBENT

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The adsorption of cationic hexadecylpyridinium bromide and non-ionic oxyethylated octylphenols (TX-45, TX-100, TX-305) surfactants from their mixed solutions at non-porous hydrophobic carbon sorbent such as thermal graphitized carbon black has been studied. The molar fraction of oxyethylated octylphenols in the mixtures was 0.2, 0.4, 0.6, and 0.8. The results have shown that the adsorption values of hexadecylpyridinium bromide depend on the total concentration of the surfactant and the composition of mixtures.

It has been found that the molar fraction of nonionic surfactants in the mixed adsorption layers at carbon black surface decreases in the series: TX-45 > TX-100 > TX-305. The regularities of the change in the surface activity of the surfactant mixtures at the solution-solid sorbent interface with an increase in the total surfactant concentration in the solution correlate with those for the solution-air system, but are more pronounced. The areas per hexadecylpyridinium bromide and the oxyethylated octylphenols molecules in the adsorption layer while adsorbing from their binary solutions are lesser than ones for the surfactants adsorption from their individual solutions, and smaller than the corresponding areas at the solution-air interface. These findings indicate the consolidation of the adsorption layer at the solid surface and confirm that the surfactants adsorb at carbon black surface as associates.

Keywords: adsorption, surfactants, binary mixtures, adsorption layer, graphitized carbon black

INTRODUCTION

Adsorption of surfactants at a solid-liquid interface has an important role in many processes such as ore flotation, oil extraction, household and personal care applications, in textile, cosmetic, food industries, biotechnology and water treatment [1, 2]. In many cases, the employing of the surfactants mixtures is more beneficial than using of individual surfactants [2, 3]. Therefore, understanding the main features of mixed adsorption of surfactants at solid surfaces is of vital importance for prediction the properties of the surfactant mixtures and their applications.

Nowadays, adsorption of the surfactants at carbon sorbents is a subject of numerous studies, but majority of the works in this area is devoted to investigation of the surfactant adsorption from single component solutions [4–6]. Currently the main research topics related to the surfactants mixtures are the development of theoretical approaches to describe the colloid-chemical interactions between the surfactants in the binary solutions. The processes of micelle formation in the bulk solution as well as adsorption at an air-

solution interface attract great research attention [7–9]. As for a solid sorbent-solution interface, the studies were conducted mainly with hydrophilic sorbents [10–12].

With regards to non-polar hydrophobic sorbents, the adsorption of a mixture of non-ionic and cationic surfactants on the surface of hydrophobic glass was investigated [13, 14]. The authors of paper [13] studied the mutual influence of Triton X-305 and tetradecyltrimethylammonium bromide surfactants on their adsorption on the hydrophobic glass and the interfacial tension values at the solid-liquid interface. In the concentration region of 10^{-4} – 10^{-3} M it has been found a synergistic effect when the adsorption value from the surfactants mixtures exceeds the adsorption values from the individual surfactants solutions. The authors of study [14] investigated the effect of dodecylpyridinium bromide and Triton X-100 concentrations on the interaction energy of SiO₂ particles. The synergetic effect is found in a wide range of concentrations at both the surfactant solution-air and the surfactant solution-hydrophobic solid interfaces. As for activated carbon, it was shown synergetic

adsorption of surfactants due to electrostatic interaction of oppositely charged ions of anionic and cationic surfactants [15].

Ridaoui *et al.* [16] investigated the change in the charge of carbon black (CB) particles in mixed aqueous dispersions of polystyrene-polyethylene oxide copolymers and cetyltrimethylammonium chloride. It has been shown that the adsorption value depends on the surfactant type. Adsorption of the surfactant or polystyrene copolymer on CB surface leads to electrostatic or steric repulsion of CB particles [16].

Ma and Xia [17] evaluated the stability of CB dispersions in mixed solutions of sodium dodecyl sulfate and ethoxylated nonylphenols. The authors showed the adsorption of the ionic surfactant to decrease in the presence of the nonionic surfactant.

Bossoleli *et al.* [18] studied adsorption of sodium polystyrene sulfonates and ethoxylated nonionic surfactants (NP-12, NP-40, NP-100) on the CB surface. For all surfactants used, adsorption isotherms of the Langmuir type and the negative values of adsorption energy were obtained. It was assumed that the polyethylene oxide chains of adsorbed non-ionic compounds had coil conformation on CB surface [18].

The purpose of this work is to study the features of adsorption of binary mixtures of cationic and non-ionic surfactants on a non-porous hydrophobic carbon sorbent such as graphitized CB.

MATERIALS AND METHODS

Cationic and non-ionic surfactants were used in the study. Non-ionic surfactants such as oxyethylated octylphenols of the molecular formula $C_8H_{17}C_6H_4O(CH_2CH_2O)_nH$ with the degree of oxyethylation $n = 5, 9, 10$ and 30 (Triton X-45, X-100 and X-305 (TX-45, TX-100, TX-305), and a cationic surfactant such as hexadecylpyridinium bromide ($C_{16}H_{33}NC_5H_5Br$) (HDPB) were obtained from Sigma-Aldrich (USA) and used as received.

Thermally graphitized CB with the specific surface area of $105 \text{ m}^2/\text{g}$, as was evaluated by BET method with argon adsorption, was used as a non-porous carbon sorbent. For removal of inorganic ash components, CB was treated with 2M solutions of HNO_3 and with concentrated HF by the method, described previously [19]. The samples were washed out with distilled water

until no traces of NO_3^- and F^- ions in washing waters were detected, and dried at 105°C .

Determination of the critical micelle concentration (CMC) values in the surfactants solutions was carried out by using a tenziometric method, which is based on plotting of the dependence of the surface tension (σ) versus the equilibrium surfactant concentration (C). The CMC value of the surfactant is defined as the concentration which corresponds to the break point on the $\sigma(\ln C)$ plot.

Measurement of the surface tension was carried out by Wilhelmy method by balancing the platinum plate. The cleanliness of the plate was controlled by the surface tension of the bidistilled water ($\sigma = 72.5 \text{ mJ/m}^2$). The accuracy of the evaluation, which was based on the average data for 3–5 measurements, was $\pm 0.5 \text{ mJ/m}^2$.

To obtain adsorption isotherms, the solutions with different initial concentrations of the surfactants but at a constant sorbent loading were shaken at 6000 rpm. The solution volume was 0.025 dm^3 . After reaching the adsorption equilibrium, the solutions were separated from the adsorbent by centrifugation. The equilibrium concentrations of Triton X surfactants in the solutions were determined by spectrophotometric method at $\lambda = 274 \text{ nm}$, while the concentrations of HDPB in the solutions were evaluated by two-phase titration method [20]. The measurement errors did not exceed 1 and 3 %, respectively. Investigations of the adsorption kinetics have shown that the adsorption equilibrium in the aqueous solution of surfactant is achieved in 8 h.

The specific adsorption value was calculated from the ratio:

$$A = \frac{C_o - C_e}{m} V \quad (1)$$

where A is adsorption value (mol/g), V is the volume of the solution (dm^3), m is CB loading in the solution (g), C_o and C_e are the initial and equilibrium surfactant concentrations (mol/dm^3).

The colloidal-chemical characteristics of the used surfactants were determined as described previously [21], and are presented in Table 1.

RESULTS AND DISCUSSION

Fig. 1 shows the adsorption isotherms of HDPB, TX-45, TX-100 and TX-305 surfactants from individual aqueous solutions on CB

surface. Adsorption of HDPB and Triton X surfactants is due to hydrophobic interactions between alkyl radicals in the surfactant molecules and CB surface. These interactions are recognized as the main driving force that cause the surfactant adsorption at hydrophobic surfaces [1, 10, 22–24].

As seen in Fig. 1, the adsorption isotherms of the surfactants in the studied concentration range are of the Langmuir type with a clearly defined plateau. Adsorption saturation for HDPB was achieved at a higher equilibrium surfactant concentration (6.5 mmol/l) than for Triton X (about 0.4 mmol/l). The maximum adsorption

value for Triton X surfactants increases with a decrease in the length of the polyoxyethyl chain of the surfactant molecules: $A_{TX-45} > A_{TX-100} > A_{TX-305}$, which corresponds to the previously published data [25, 26]. Earlier it was reported that the main parameter, which restricts the Triton X adsorption, is the polyoxyethylene chain protruded in the bulk solution [17]. The long polyoxyethylene chains could take a coil conformation in the solution [25, 27] and therefore restrict the packing density of the molecules in the adsorption layer, thus reducing the adsorption value.

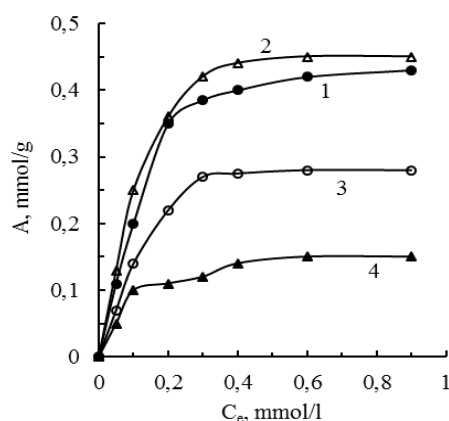


Fig. 1. Adsorption isotherms of HDPB (1), TX-45 (2), TX-100 (3) and TX-305 (4) on CB surface from individual surfactant solutions

Fig. 2 depicts the adsorption isotherms of HDPB at CB surface from binary mixtures with TX-45 (a), TX-100 (b) and TX-305 (c) surfactants at different mole fractions of Triton X in the mixtures: $\alpha_{TX} = 0.2, 0.4, 0.6$ and 0.8 . The results have shown that the adsorption values of HDPB from these mixtures depend on the total concentration of the surfactant and the molar fraction of Triton X in the mixture.

As seen in Fig. 2 a, b, at a low HDPB content in the mixture ($\alpha_{TX} = 0.8$), adsorption of HDPB from the binary solutions on CB surface is low. With increasing of α_{HDPB} , the maximum adsorption value of HDPB from the mixture is higher compared to the adsorption from the individual HDPB solution. This means that adsorption of cationic surfactant is increased in the presence of the molecules of the non-ionic surfactant. A similar effect was observed for the cetyltrimethylammonium bromide-Triton X-100 system at the mixture concentration, which corresponds to an unsaturated monolayer [28].

This effect is enhanced with increasing of the mole fraction of HDPB in the mixture from 0.2 to 0.6. The largest increase in the surfactant adsorption is observed at $\alpha_{HDPB} = 0.6$, when its value reaches 0.50–0.63 mol/kg in the studied concentration range and there is a tendency for its further increase.

Such results can be explained by the fact that the embedding of the molecules of the non-ionic surfactant in the mixed adsorption layer reduces the electrochemical repulsion between positively charged ions of HBPB. This leads to higher adsorption of the cationic surfactant from its mixture with the non-ionic surfactants than the one from the individual HDPB solution. The maximal effective balance of the acting driving forces of adsorption is realized at $\alpha_{TX} = 0.4$, probably due to adsorption of both individual HDPB ions and mixed TX-HDPB aggregates.

To calculate the composition of mixed adsorption layers of the surfactants and parameters of intermolecular interaction between

the surfactants, a phase separation model (the Rubin-Rosen approach) was used [2, 29]. According to this model, the coefficient of intermolecular interaction in the adsorption layer β^s was estimated by using the equation:

$$\beta^s = \frac{\ln(\alpha_1 C_{12} / C_1 \chi_1)}{(1 - \chi_1)^2} \quad (2)$$

where α_1 is the molar fraction of surfactant 1 in the solution, χ_1 is the molar fraction of surfactant

1 in the mixed adsorption layer, C_1^0 and C_{12} are the molar concentrations of the surfactant 1 solution and the binary mixture at the same adsorption value.

The composition of the adsorption layer was calculated by the ratio [2, 29]:

$$(\chi_1)^2 \ln\left(\frac{\alpha_1 C_{12}}{C_1 \chi_1}\right) = (1 - \chi_1)^2 \ln\left(\frac{(1 - \alpha_1) C_{12}}{C_2 (1 - \chi_1)}\right). \quad (3)$$

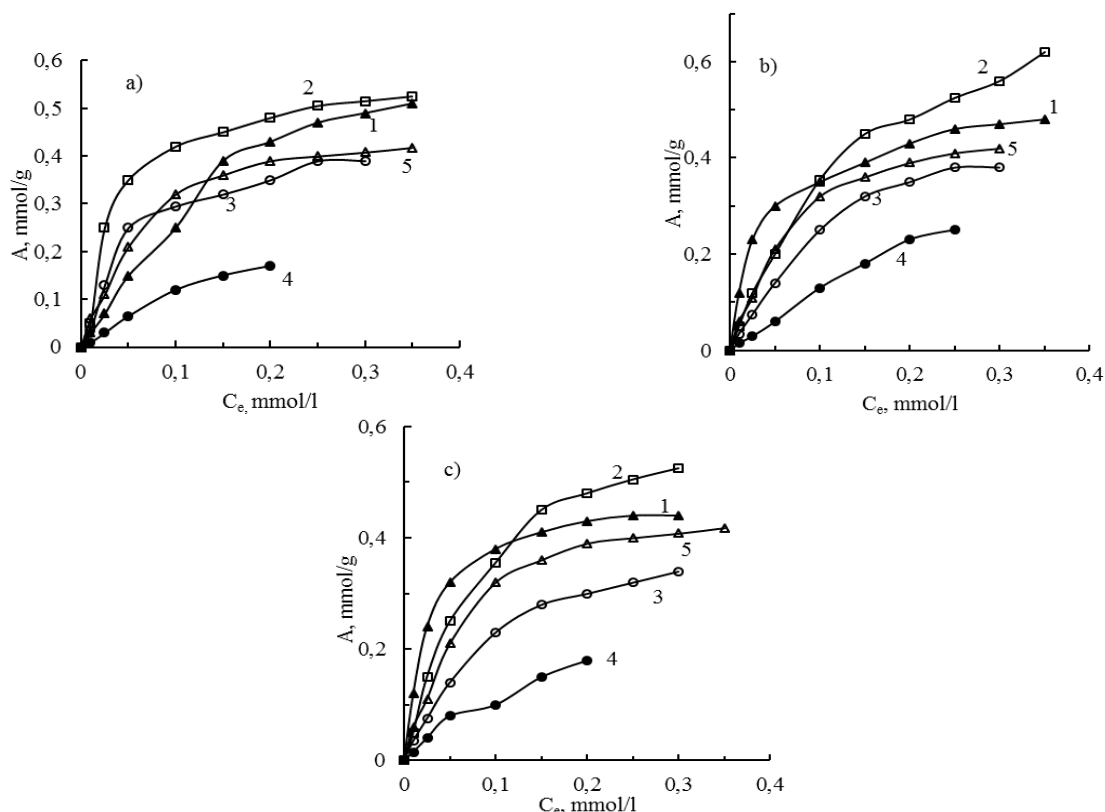


Fig. 2. Adsorption isotherms of HDPB at CB from single solution (5) and mixed solutions with TX-45 (a), TX-100 (b) and TX-305 (c) at different mole fraction of Triton X (α_{TX}) in the mixtures: $\alpha_{TX} = 0.2$ (1), $\alpha_{TX} = 0.4$ (2), $\alpha_{TX} = 0.6$ (3), $\alpha_{TX} = 0.8$ (4)

Thus, having determined from the experimental data the concentrations of the surfactant mixtures and the individual solutions, at which a given adsorption value is reached, the composition of the mixed adsorption layer at the specific adsorption value was calculated. The calculation results show (Table 1) that the composition of the adsorption layer on the CB surface is notably different from the surfactants ratio in the initial solution.

The χ values indicate that the mixed adsorption layers on CB surface are enriched with molecules of the non-ionic surfactants, even

at their low content in the solution ($\alpha_{TX} = 0.2$). With increasing of the Triton X molar fraction in the binary solution from 0.2 to 0.8 its share in the adsorption layer increases by approximately 2.5 times.

Fig. 3 shows the ratio of the molar fraction of HDBP in the bulk solution and in the mixed adsorption layer on the CB surface. If the HDBP content at CB surface were the same as in the solution, the experimental data would fall in the dashed line in Fig. 3. However, as seen in Fig. 3, the non-ionic surfactants are present in excess on CB surface, and their mole fraction in the

adsorption layer decreases in the following order: TX-45 > TX-100 > TX-305. These data indicate that more hydrophobic non-ionic surfactants are predominantly adsorbed on CB surface from mixed solutions with HBPB.

Negative values of β^s interaction parameter (Table 1) indicate notable attraction of the surfactant molecules and ions in the mixed adsorption layers. An increase in the absolute value of β^s parameters with increasing α_{TX} in the solution characterize the strengthening of the interactions between the components in the adsorption layer. Fig. 4 shows the dependence of

the coefficients of intermolecular interaction β^s versus the mole fraction of TX-45, TX-100, and TX-305 surfactants in the mixtures. As seen in Fig. 4, an increase of α_{TX} value in the initial solution to 0.6 is accompanied by an increase in the absolute value of the coefficients β^s at a fixed adsorption value. The strengthening of intermolecular interactions in the surface layer can be due to the formation of hydrogen bonds, as well as due to dispersion interactions between π -electrons of phenyl rings of the surfactant molecules and π -electrons of the graphite lattice of CB.

Table 1. Parameters β^s and molar fractions of Triton X in the mixed adsorption layers on the CB surface (χ_{TX}): $A = 1.8 \times 10^{-4} \text{ mol/g}$

α_{TX}	0.2	0.4	0.6	0.8
HDPB/TX-45				
β^s	-6.47	-6.75	-8.28	-5.43
χ_{TX-45}	0.40	0.73	0.85	0.90
HDPB/TX-100				
β^s	-5.61	-5.22	-7.53	-4.80
χ_{TX-100}	0.30	0.55	0.73	0.76
HDPB/TX-305				
β^s	-4.87	-5.02	-5.66	-3.58
χ_{TX-305}	0.52	0.24	0.68	0.70

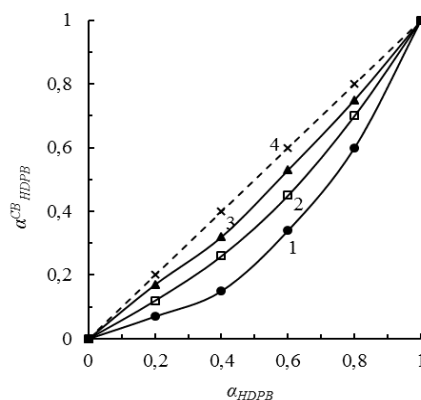


Fig. 3. Molar fraction of HDBP in the bulk solution (α_{HDBP}) and in the mixed adsorption layer on the CB surface (α^{CB}_{HDBP})

Additional information related to the surfactant adsorption from binary solutions on the sorbent can be obtained by comparison with adsorption at an interface between a binary solution of the surfactant and air. Using the isotherms of the surface tension of the HDBB, TX-45, TX-100, TX-305 surfactants and their mixtures, which were previously obtained [20], the maximum adsorption values (Γ_∞) at the interface surfactant solution-air and the values of the area per the adsorbed surfactant molecule in

mixed adsorption layer (S_{min}) were evaluated (Table 2).

By using the adsorption isotherms of the surfactants on CB (Figs. 1, 2), the values of the maximal surfactants adsorption in the plateau region (A_{max}) were found. The area per molecule in the mixed adsorption layer at the CB surface was calculated by using the equation [30]:

$$\omega = \frac{S}{A_{max} N_a}, \quad (4)$$

where S is the surface area of CB, ω is a minimal area per molecule in the adsorption layer at CB surface, N_a is the Avogadro number and A_{max} is the adsorption value at a concentration, which is close to saturation.

It is seen from the data presented in Table 2 that the surfactants adsorption values from their binary mixtures at the solution-CB interface significantly exceed the adsorption values at the solution-air interface. This is due to the formation of mixed HPDP-TX aggregates at CB surface, as well as due to hydrophobic

interactions of the alkyl chains of the surfactant molecules with the nonpolar CB surface.

The areas per HDPB and the Triton X molecules in the adsorption layer while adsorbing from their binary solutions are lesser than those for the surfactants adsorption from their individual solutions, and smaller than the corresponding areas at the solution-air interface (Table 2). These findings indicate the consolidation of the adsorption layer at the solid surface and confirm that the surfactants adsorb on CB surface as associates.

Table 2. Comparison of the main adsorption parameters of the surfactants mixtures at the solution-air and the solution-CB interfaces

		HDPB/TX-45					
α_{TX-45}		0	0.2	0.4	0.6	0.8	1.0
$\Gamma_{\infty} \times 10^6$, mol/m ²	2.76	3.64	5.01	6.72	4.13	2.66	
S_{min} , nm ²	0.60	0.46	0.33	0.22	0.40	0.62	
$A_{max} \times 10^6$, mol/m ²	3.81	6.35	8.26	9.74	7.18	4.29	
ω , nm ²	0.43	0.26	0.20	0.17	0.23	0.39	
		HDPB/TX-100					
α_{TX-100}		0	0.2	0.4	0.6	0.8	1.0
$\Gamma_{\infty} \times 10^6$, mol/m ²	2.76	3.38	4.80	5.71	3.90	2.51	
S_{min} , nm ²	0.60	0.50	0.35	0.29	0.42	0.66	
$A_{max} \times 10^6$, mol/m ²	3.81	5.49	7.30	8.56	6.95	2.67	
ω , nm ²	0.43	0.30	0.23	0.19	0.24	0.62	
		HDPB/TX-305					
α_{TX-305}		0	0.2	0.4	0.6	0.8	1.0
$\Gamma_{\infty} \times 10^6$, mol/m ²	2.76	2.63	3.05	4.27	2.78	1.47	
S_{min} , nm ²	0.60	0.63	0.54	0.39	0.60	1.13	
$A_{max} \times 10^6$, mol/m ²	3.81	4.32	4.91	5.50	3.96	1.55	
ω , nm ²	0.43	0.39	0.34	0.30	0.42	1.07	

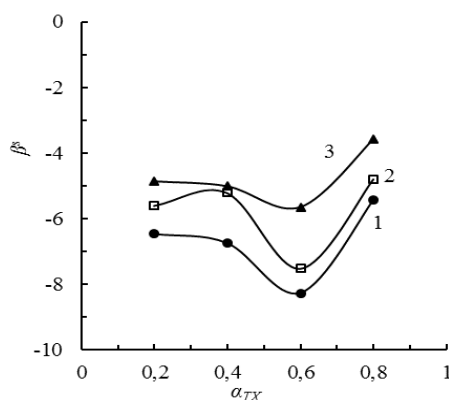


Fig. 4. Dependence of the coefficients of intermolecular interaction β^s versus the mole fraction of TX-45 (1), TX-100 (2) and TX-305 (3) surfactants in the mixtures (α_{TX}); $A=1.8 \times 10^{-4}$ mol/g

CONCLUSIONS

Adsorption of HBPB from its binary mixtures of different composition with TX-45, TX-100 and TX-305 surfactants at graphitized CB was studied. It has been found that at low surfactants concentrations, adsorption of HBPB from the surfactants mixtures is higher in the presence of the non-ionic surfactant than HBPB adsorption from its individual solutions. This can be explained by the fact that the inclusion of the molecules of the non-ionic surfactant in the mixed adsorption layer reduces electrostatic repulsion between charged hydrophilic groups of the cationic surfactants and thus promotes HBPB adsorption. It has been found that the mixed adsorption layers are enriched with molecules of

the non-ionic surfactant. The molar fraction of the non-ionic surfactants in the adsorption layer decreases in the series: TX-45>TX-100>TX-305. The results obtained show that the more hydrophobic non-ionic surfactant is better adsorbed at CB surface from mixed solutions with cationic surfactant. The regularities of the change in the surface activity of the surfactants mixtures at the solution-CB interface with an increase in the total surfactant concentration in the solution correlate with ones at the solution-air interface, but are more pronounced. These findings indicate the consolidation of the adsorption layer at the solid surface and confirm the fact of the surfactants adsorb at CB surface as associates.

Особливості адсорбції бінарних сумішей катіонних і нейонних поверхнево-активних речовин непористим вуглецевим сорбентом

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Досліджено адсорбцію катіонного гексадецилпіридиній броміду та нейонних оксиетильованих октилфенолів (TX-45, TX-100, TX-305) із змішаних розчинів на непористому гідрофобному вуглецевому сорбенті – термічній графітованій сажі. Мольна частка гексадецилпіридиній броміду в сумішах становила 0.2, 0.4, 0.6 і 0.8. Результати показали, що величини адсорбції гексадецилпіридиній броміду залежать від загальної концентрації поверхнево-активних речовин (ПАР) і складу сумішей.

Встановлено, що мольна частка нейонних ПАР в змішаних адсорбційних шарах на поверхні графітованої сажі зменшується в ряду: TX-45 > TX-100 > TX-305. Закономірності зміни поверхневої активності сумішей ПАР на межі поділу рідина-твердий сорбент зі зростанням загальної концентрації ПАР в розчині корелюють з такими для системи рідина-повітря, але виражені сильніше. При адсорбції ПАР на поверхні графітованої сажі з бінарних розчинів значення площі, що займають молекули гексадецилпіридиній броміду і оксиетильованих октилфенолів в адсорбційному шарі, менші, ніж при адсорбції з індивідуальних розчинів, і менші відповідних площ на межі розділу фаз розчин – повітря. Це свідчить про ущільнення адсорбційного шару на твердій поверхні і підтверджує факт адсорбції ПАР на поверхні графітованої сажі у вигляді асоціатів.

Ключові слова: адсорбція, поверхнево-активні речовини, бінарні суміші, адсорбційний шар, графітована сажка

Особенности адсорбции бинарных смесей катионных и неионных поверхностно-активных веществ непористым углеродным сорбентом

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Исследована адсорбция гексадецилпиридиний бромида и оксиэтилированных октилфенолов (TX-45, TX-100, TX-305) из смешанных растворов на непористом гидрофобном углеродном сорбенте - термической графитированной саже. Мольная доля гексадецилпиридиний бромида в смесях составила 0,2, 0,4, 0,6 и 0,8. Результаты показали, что величины адсорбции гексадецилпиридиний бромида зависят от общей концентрации поверхностно-активных веществ (ПАВ) и состава смесей.

Установлено, что мольная доля неионных ПАВ в смешанных адсорбционных слоях на поверхности графитированной сажи уменьшается в ряду: TX-45 > TX-100 > TX-305. Закономерности изменения поверхностной активности смесей ПАВ на границе раздела жидкость-твердый сорбент с ростом общей концентрации ПАВ в растворе коррелируют с таковыми для системы жидкость-воздух, но выражены сильнее. При адсорбции ПАВ на поверхности графитированной сажи из бинарных растворов величина площади, занимаемой молекулами гексадецилпиридиний бромида и оксиэтилированных октилфенолов в адсорбционном слое, меньше, чем при адсорбции из индивидуальных растворов, и меньше соответствующих площадей на границе раздела фаз раствор - воздух. Это свидетельствует об уплотнении адсорбционного слоя на твердой поверхности и подтверждает факт адсорбции ПАВ на поверхности графитированной сажи в виде ассоциатов.

Ключевые слова: адсорбция, поверхностно-активные вещества, бинарные смеси, адсорбционный слой, графитированная сажа

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