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The Effect of Different Factors on the Adsorption of Cucurbituril Complexes at Electrode/Solution Interfaces[1]

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Abstract—Results of the studies in the adsorption properties of CB6, CB7, CB8 cucurbituril complexes with inorganic cations (Na^+, K^+, Cs^+), cation of 3,3'-diethyltiocarbocyanine iodide organic dye and neutral organic compounds: adamantanol-1 and ferrocene at the electrode/solution interfaces are summarized. Effects of different factors on the adsorption behavior of the studied supramolecular complexes are analyzed.

Keywords: cucurbituril complexes CB6, CB7, CB8, inorganic cations, adamantanol-1, ferrocene, 3,3'-diethyltiocarbocyanine iodide dye, adsorption properties, electrode/solution interface

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INTRODUCTION

In recent years, numerous studies appeared in supramolecular chemistry, i.e., the chemistry of compounds formed by non-covalent interactions; they were inspired by pioneer research of Lehn [1], a Nobel Prize winner. These studies mainly deal with the preparation and studying of molecular cavitands, that is, bulky molecules with rigid structure, able of forming inclusive complexes of different type. Among these compounds, a special place is hold by relatively new group of molecular containers that belong to the cucurbituril family and resemble in shape a pumpkin (family *cucurbitaceae*). The very first member of the group was synthesized as long ago as 1905 [2]; however, the structure of the molecular containers was elucidated no sooner than 1981 [3]. Their molecules (CBn_s) are formed by n glycoluril fragments. To date, the molecules with n equal to 5–8, and 10 have been synthesized. Their unique feature is the presence of a hydrophobic intramolecular cavity with two portals framed with carbonyl groups. Thereby, the cucurbiturils are able of simultaneously forming inclusive complexes with organic compounds through their placing into the molecular container's cavity, as well as with inorganic cations via their interaction with the portals' carbonyl groups. These unique properties underlie the ever growing interest to the studying of this group of supramolecules, as well as the potentialities of their applications in supramolecular chemistry, pharmacy, biochemistry, and nanotechnologies. Recent results of the intense studies on the homological series of the cavitands are summarized in some review-articles [4–6].

Until quite recently, systematic studies of behavior of the discussed compounds at interfaces were practically absent; the same is true for supramolecular compounds of any type. In previous work, we studied adsorption properties of cationic cryptate complexes at electrode/solution interfaces [7]. Recently, we investigated CB5–CB8 cucurbituril complexes with cations and neutral organic compounds [8–11]. In this work, we aimed at a generalizing of the recently obtained data and analyzing of effects of different factors on the adsorption behavior of the studied supramolecular complexes at electrode/solution interfaces.

EXPERIMENTAL

We carried out our studies at a steady-state Hg-drop-electrode by the measuring of the potential dependence of its differential capacitance C, using an Autolab potentiostat/galvanostat equipped with a FRA-software (Eco Chemie, Netherlands). The measurements were carried out at a frequency $f = 370$ Hz and ac voltage amplitude of 1 mV. The electrode potential E was measured in increments of 20 mV. The mercury drop had a surface area of 0.016 cm^2, which was determined by weighting few drops. The drop was mounted at an end of cone-shaped capillary (inner diameter: 80 μm) using a special setup from complete set of a polarographic analyzer PA-3 (Czechoslovakia). The working electrode potential was measured

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(and presented in this work) against saturated calomel electrode (SCE). The differential capacitance was measured after the working electrode exposure for t = 0–35 s to each potential E value after its setting up. The cucurbit [7]uril (Aldrich), adamantanol-1 (Merk), ferrocene (Fluka), 3,3'-diethyltiocarbocyanine iodide (Sigma-Aldrich) were used without additional purification, Na_2SO_4, K_2SO_4, Cs_2SO_4 were crystallized twice and calcinated` at 500°C; water was purified with a Millipore setup; solutions were deaerated using high-purity argon.

RESULTS AND DISCUSSION

The studies of adsorption phenomena at the electrode/solution interface were carried out in systems containing cucurbituril complexes with inorganic cations (Na^+, K^+, Cs^+), organic compounds: adamantanol-1, ferrocene, and organic dye 3,3'-diethyltiocarbocyanine iodide. Among numerous organic compounds forming inclusive complexes with the CBn_s, the adamantane and ferrocene derivatives are distinguished by the forming of exceptionally strong complexes with CB7. For instance, the constants (K_s) of complex formation between CB7 and adamantanol-1 and hydroxymethylferrocene are 2.8×10^{10} M^{-1} [12] and 3.0×10^9 M^{-1} [13] respectively (the exact value of K_s for the CB7–ferrocene complex is unknown; however, presumably it is rather high). For some other adamantane and ferrocene derivatives, the constants of complex formation with CB7 may be as high as 10^{14}–10^{15} M^{-1}. Evidently, in the systems under study, where the ligand and the organic substance enter the complex in equimolar amounts, practically no free ligand molecules are present in the solution bulk at such values of K_s. It was thought that in the complex formation due to non-covalent interactions such high values of K_s are within reach only for high-molecular (protein) substances. One of the best examples of this kind is the avidin-biotin complex, a strongest bimolecular system whose value of K_s is extraordinary high ($\approx 10^{15}$ M^{-1}) [12]. For the CB7 complex with the 3,3'-diethyltiocarbocyanine$^+$ dye cation, $K_s = 2.8 \times 10^4$ M^{-1} [14]. Nowadays, the K_s values are known for all CB5–CB8 cavitand complexes only with Na^+ cation [4, 15]. The strongest of the complexes is the one with CB6 ($\log K_s = 3.49$); the less strong, with CB5 ($\log K_s = 1.85$); for CB7 and CB8, the K_s values are 2.89 and 2.6, respectively. Evidently, it is the complex strength that determines the free ligand CBn concentration in the studied solutions. For the less strong complexes, the presence of the cavitand molecules allows their participation in the adsorption layer formation (under some special conditions).

Nowadays, we do not know whether the CB7 complexes with adamantanol-1 and ferrocene can simultaneously form complexes with inorganic cations. Such a process was observed for CB6 complexes with some organic dyes and small organic molecules [16, 17]. In principle, the conclusion on the CB7 simultaneous complex formation with organic molecules and inorganic cations can be drawn basing on the impedance measurements' data. First of all, the conclusion can be based on the position of the adsorption potential region that depends on the charge of the adsorbing particles.

C,E-dependences were obtained in 0.1 M solutions of sodium, potassium, and cesium sulfates in the presence of CB6, CB7, and CB8 of different concentrations, as well as in 0.1 M Na_2SO_4 solution with different equimolar CB7 concentrations with adamantanol-1, ferrocene, or 3,3'-diethyltiocarbocyanine iodide. The measurements were carried out using both directions of the potential sweeping, at a different time of the electrode exposure to each potential value upon its setting. In Fig. 1 we give typical data for the studied systems in 0.1 M Na_2SO_4 + 10^{-5} M CB8 solution, obtained for different electrode exposures to each potential and using the opposite directions of the potential scanning. As for other members of the homological series, the CB8 shows unusually broad region of the adsorption potentials (~2 V), where the C values appeared being lower than in additive-free indifferent electrolyte solutions. It is well known that the adsorption potential region for neutral organic compounds rarely exceeds 1 V at some electrodes. For CB8, as for cationic complexes of the other homologues, the limiting electrode surface coverage with the organic molecules has been reached at extremely low additive concentration. A distinctive feature of the data shown in Fig. 1, which was observed for the complexes with inorganic cations with other cucurbiturils as well, is the presence of two segments in the C,E-dependences, which may evidence the formation of adsorption layers with different structures. The transition region between these segments is located near the electrode zero-charge potential in the indifferent electrolyte solution (–0.44 V). Moreover, this allows suggesting that the potential change in this region is accompanied with some change in the charge of the adsorbed particles. The broadest part of the adsorption region (≈–1.85...–0.44 V) corresponds to negative charge of the electrode surface, which leads to the conclusion that there are the cucurbituril cationic complexes that adsorb over this potential region.

The complexes' positive charge must prevent their adsorption at positive surface charges; however, in some cases the positive boundary of the adsorption region is shifted with respect to the zero-charge potential by ca. 0.7 V toward positive potentials. This may be due to the possible participation of the cucurbituril molecules in the adsorbate layer formation in this potential region.

The adsorption layer formation for the systems containing some cationic complexes of CBn_s strongly

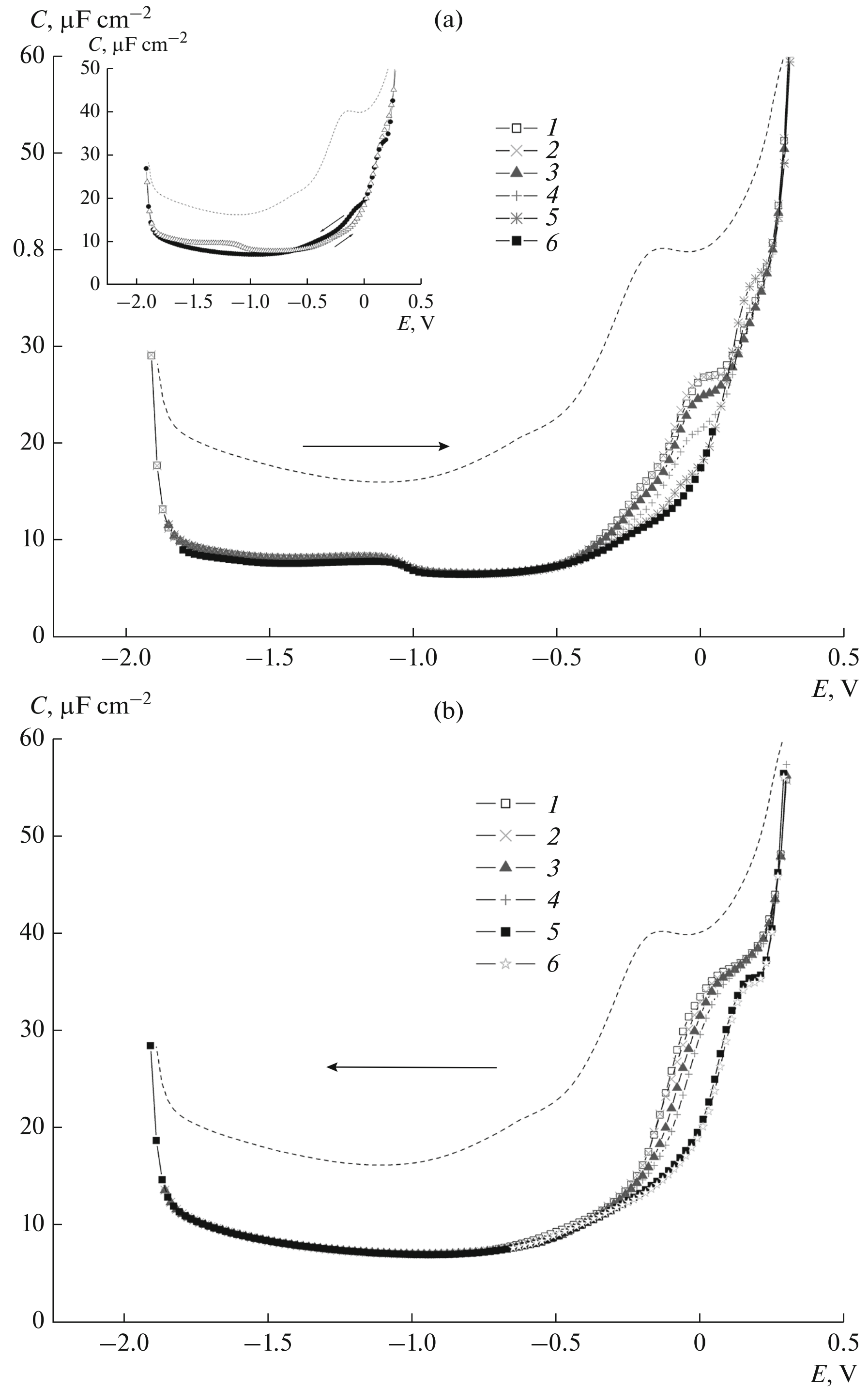

Fig. 1. *C,E*-dependences in 0.1 M Na_2SO_4 solution containing 5×10^{-6} M CB8, taken during the potential scanning from negative to positive values (a) and in the reverse direction (b), with the exposing of the working electrode at each *E* value for the time *t*, s: (*1*) 0, (*2*) 1, (*3*) 3, (*4*) 10, (*5*) 15, (*6*) 35 (a) and (*1*) 0, (*2*) 1, (*3*) 3, (*4*) 10, (*5*) 35, (*6*) 50 (b). Inset: *C*,*E*-dependences in 0.1 M Na_2SO_4 solution containing 10^{-5} M CB8, taken during the potential scanning in opposite directions, *t* = 35 s. Dashed line—*C*,*E*-dependence in 0.1 M Na_2SO_4 solution.

differ in the potential regions corresponding to different segments of the *C*,*E*-dependences. Thus, on the cucurbituril cationic complexes adsorption in the potential region corresponding to the electrode surface negative charge, the capacitance remains practically independent of the complex concentration, the time of electrode exposure at each potential, and the potential scanning direction. Yet, these dependences were observed in the potential region corresponding to the electrode surface positive charge, and this leads to the appearance of hysteresis (Fig. 1a, inset). In this potential region we observed the slowed down adsorption layer formation, which manifested itself in the C value gradual decrease with the increasing of t; the constant values of C to be achieved used at the t values of 20–30 s by order of magnitude. The slowing down can be explained by the inhibiting during the adsorption of positively charged particles at the likely charged electrode and low concentration of the cavitand molecules that do not participate in the complex formation; their adsorption in this region is preferable, thanks to the presence of electron-donor carbonyl groups at the portals. When the potential is swept toward negative potentials, we observed step formation in the C,E-dependences, which points out to the presence of some structures in the adsorption layer, which are relatively stable over narrow potential ranges. During the reverse potential scanning, similar structures are formed at low t only. On the whole, the differences in the properties and formation of the cucurbituril complexes' adsorption layers at the opposite potential scanning directions can be explained by the fact that during the scanning toward positive potentials there occurs restructuring of the previously formed adsorption layer, concurrently with the desorption. During the reverse scanning direction, the adsorbate layer forms simultaneously with the diffusion delivery of the adsorbable particles from the solution bulk. The effect of formation of adsorption layers with different properties in different potential regions, observed for the CBn_s + 0.1 M Me_2SO_4 systems (Me: Na^+, K^+, Cs^+), leads to the reasonable conclusion that the entire array of experimental data cannot be described by a unique set of the adsorption parameters. On this reason, we found the adsorption parameters for the systems, by using the regression analysis method [18], separately for each above-discussed segment in the adsorption-potential region.

In the calculations, we used the following independent adsorption parameters: φ_m is the maximal adsorption potential expressed in the rational scale; $\ln B_m$ is the logarithm of the adsorption equilibrium constant at the maximal adsorption potential; a_m is the parameter of intermolecular interaction in the adsorption layer at $\theta = \theta_m$; C_m is the differential capacitance at the limiting surface coverage with the adsorbate and $\theta = \theta_m$; $A = RT\Gamma_m$, where Γ_m is the adsorbate limiting surface concentration at $\theta = 1$; R and T is the universal gas constant and absolute temperature, respectively. Some coefficients characterizing potential dependences of the parameters a and C_m were also used. Thus obtained adsorption parameters are given in some publications [8–11]. Their analysis allowed concluding on a significant difference of these quantities in the two segments of the adsorption region. For example, the ln B_m values for the negative potential segment exceeds those for the positive potential segment by a factor of 1.5–2. This can be explained by a significant contribution from electrostatic interaction. The attraction interaction parameters also differ in the two segments. In the "negative" segment the parameter a is positive, which evidenced an attractive particles' interaction in the adsorption layer; by contrast, in the "positive" segment, the parameter a approached zero or is negative. The limiting adsorption Γ_m differs nearly by a factor of two for the adsorbate layers in the two different adsorption segments. In the "positive" segment, this quantity corresponds to nearly full surface coverage by the CBn molecules oriented with the portals' carbonyl groups toward the electrode surface. This orientation was observed in the CBn_s and their complexes adsorption at uncharged surfaces of some metals [19]. In the "negative" segment, the Γ_m value is approximately twice as large, which can be explained by the formation in this segment of an adsorption bilayer. Such a bilayer can be formed due to simultaneous binding of two cavitands CBn by a sodium cation. Of the two cavitands, one is located at the electrode surface; the other is oriented toward the solution. This is the bilayer structure was observed at gold in the CB6 solutions in the presence of sodium cations [19].

Of interest is the comparing of experimental data obtained for different CBn_s with the same cation and for the same CBn with different cations. In Fig. 2 we compare C,E-dependences for the CB6, CB7, and CB8 complexes with the Na^+ cation. Evidently, at negative surface charge these dependences approach each other, serious discrepancies are observed in the positive branches of the C,E-curves, and noticeable difference between them begins from the zero-charge potential. We see that the adsorption region of the CB6 $\subset$ Na^+ complex is narrower than that of other complexes. This can be explained by the above-mentioned much larger strength of the complex. Moreover, this stipulated much lower concentration of the free ligand in the solution, hence, practical impossibility of a mixed adsorption layer formation. Thus, we may suggest the stepwise jumps in the C,E-dependence (Fig. 2a) in the solution containing the CB6 $\subset$ Na^+ complex to be caused by desorption of this complex. It should be noted that the like steps in the C,E-dependences at the adsorption–desorption potentials are characteristic of compounds forming two-dimensional condensed layers at electrodes [20]. Accordingly, in the reverse run of the curve the capacitance decrease in this solution (Fig. 2b) is due to the loading of this complex onto the electrode surface as well. It should be noted that the value of C corresponding to the step in the C,E-dependence for the CB6 $\subset$ Na^+ complex is twice as high as that at more negative potentials, which can be explained by a twofold decrease in the adsorbate layer thickness. The CB7

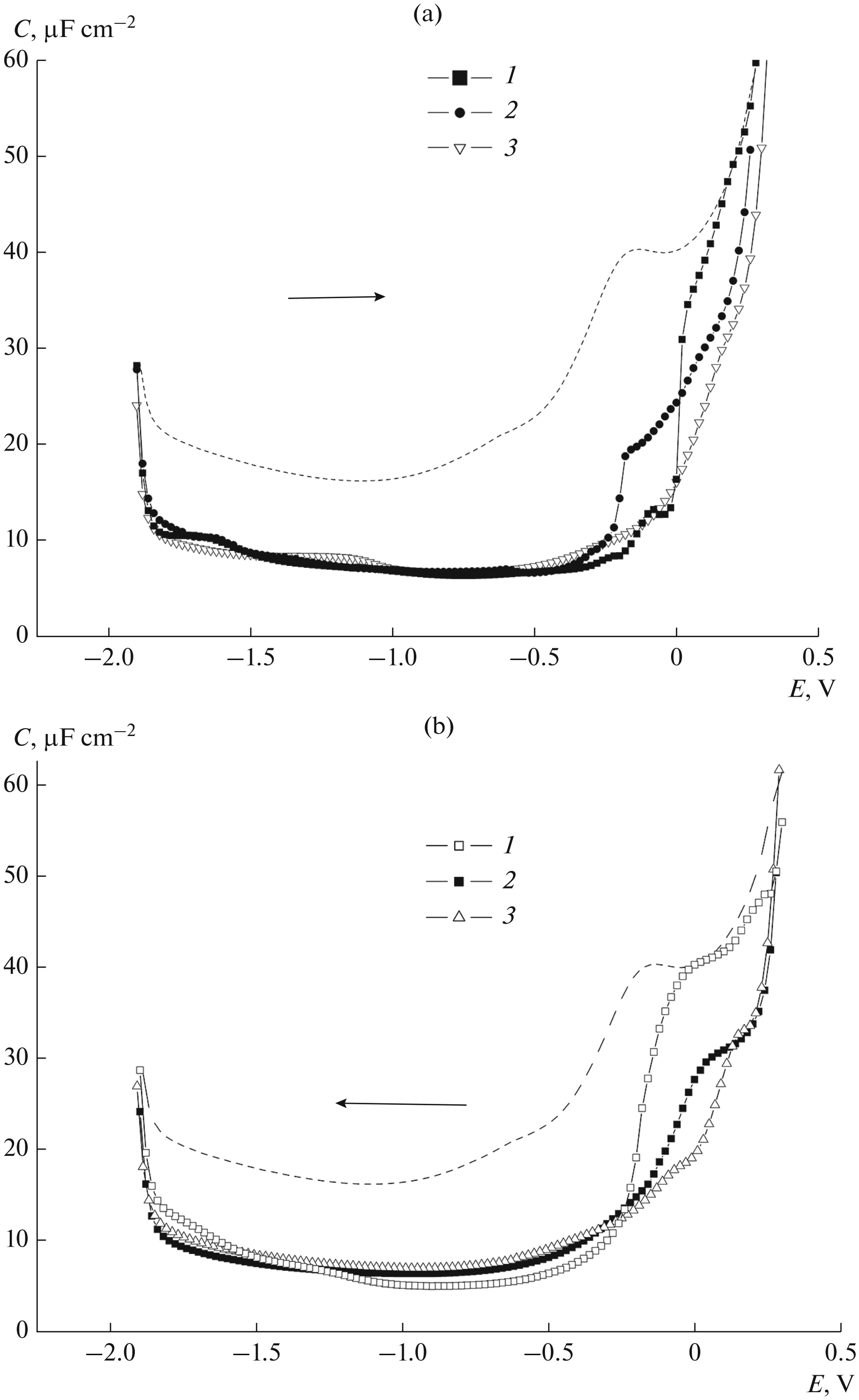

Fig. 2. *C,E*-dependences in 0.1 M Na_2SO_4 solution containing 5×10^{-6} M CB*n*, taken during the potential scanning from negative to positive values (a) and in the reverse direction (b), with the exposing of the working electrode at each *E* value for the time $t = 35$ s: (*1*) CB6, (*2*) CB7, (*3*) CB8. Dashed line—*C*,*E*-dependence in 0.1 M Na_2SO_4 solution.

and CB8 complexes' adsorption-potential regions are broader thanks to the shift of their positive boundaries. At positively charged electrode surfaces, these complexes demonstrated steps in the *C*,*E*-dependences, which evidences the formation of some adsorption layers that differ in their structure from those occurring at negatively charged surface. As mentioned above, most probable seems the suggestion on the par-

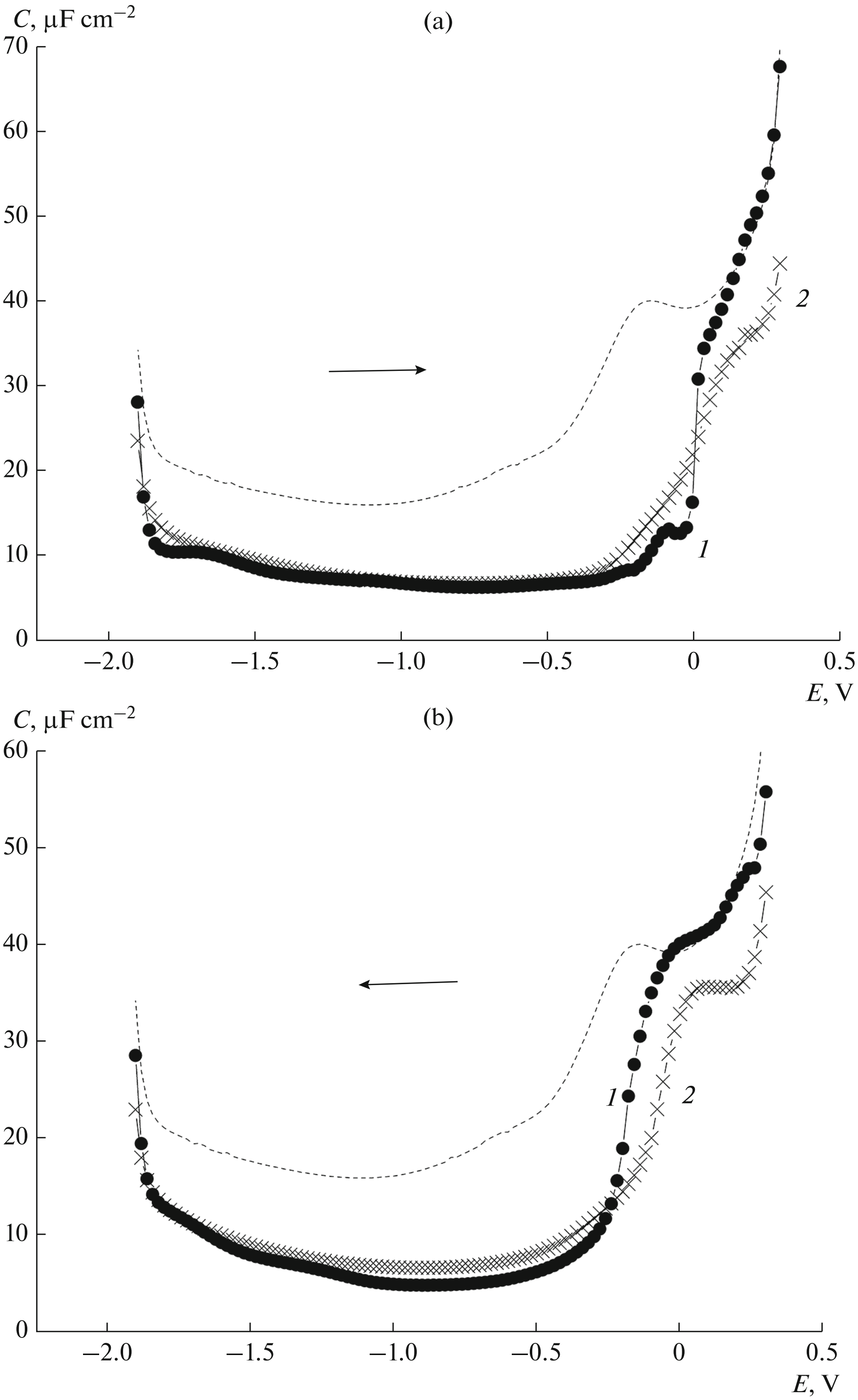

Fig. 3. C,E-dependences in 10^{-4} M CB6 solution containing 0.1 M Na_2SO_4 (*1*) and 0.1 M K_2SO_4 (*2*), taken during the potential scanning from negative to positive values (a) and in the reverse direction (b), with the exposing of the working electrode at each E value for the time $t = 30$ s. Dashed line—C,E-dependence in 0.1 M Na_2SO_4 solution.

ticipation of the free ligand-molecules in the formation of the mixed adsorption layers at so high positive surface charges.

The comparing of data for the $CB6 \subset Na^+$ and $CB6 \subset K^+$ complexes (Fig. 3) shows that the adsorption-potential regions for these complexes are different. Moreover, for the one with broader adsorption region we observed somewhat different form of C,E-dependence in the region of positive potentials: the characteristic steps are formed at relatively high values of C. As mentioned above, the form of C,E-depen-

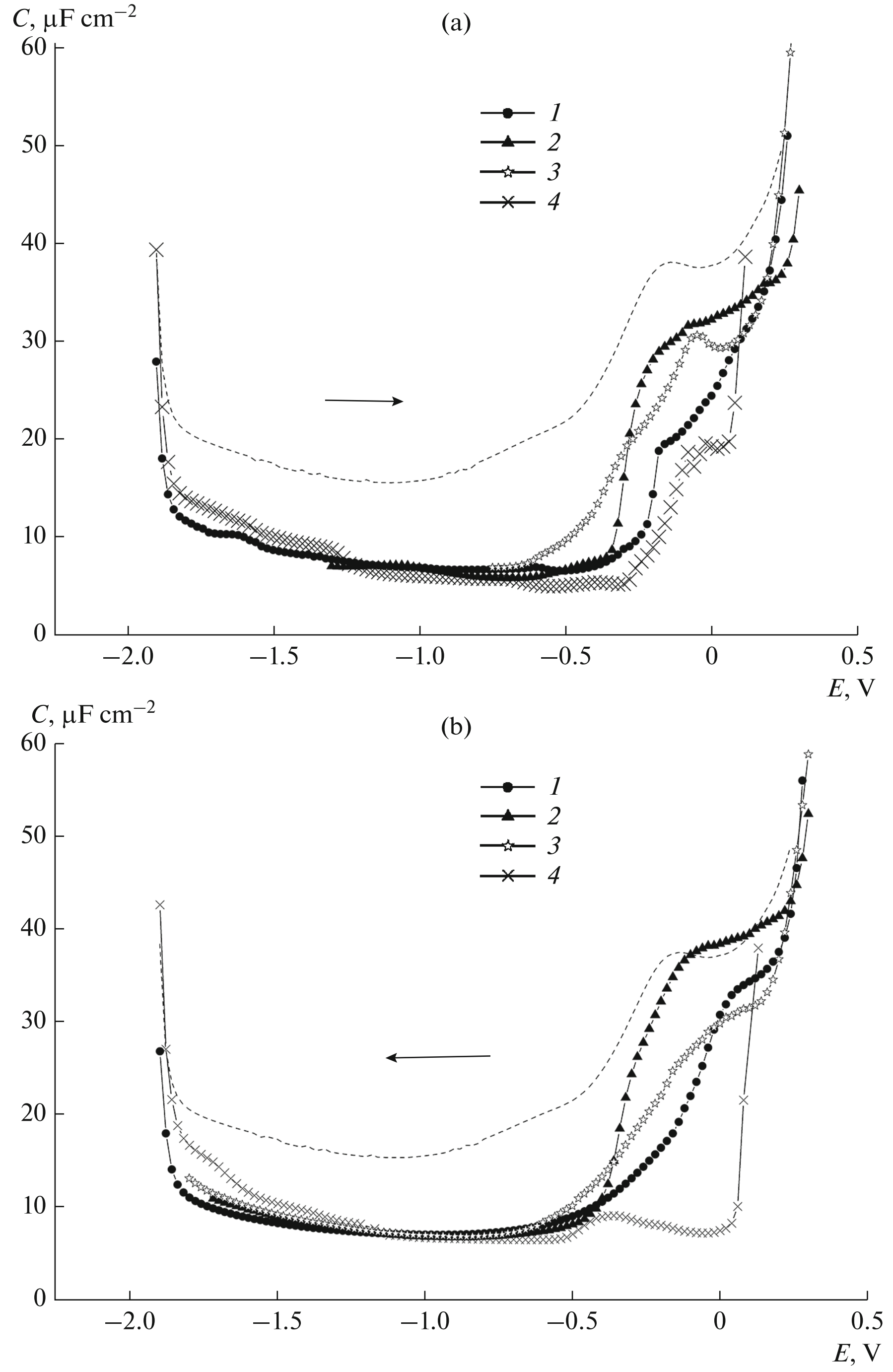

Fig. 4. C,E-dependences in 10^{-5} M CB7 solution containing 0.1 M Na_2SO_4 (*1*), 0.1 M K_2SO_4 (*2*), 0.1 M Cs_2SO_4 (*3*) and in 5 × 10^{-6} M CB7 solution containing 0.1 M Na_2SO_4 + 5 × 10^{-6} M 3,3'-diethyltiocarbocyanine$^+$ (*4*), taken during the potential scanning from negative to positive values (a) and in the reverse direction (b), with the exposing of the working electrode at each E value for the time t = 30 s. Dashed line—C,E-dependence in 0.1 M Na_2SO_4 solution.

dence in the presence of CB6 $\subset$ Na^+ complex is characteristic of organic surfactants that form condensed adsorption layers. Thus, one may qualitatively judge on the cationic complex stability by the form of C,E-dependence and location of the adsorption region. Similar comparison for the CB7 cationic complexes is shown in Fig. 4. Along with the data for inorganic cation complexes, we give those for the CB7 inclusive complex with the cation of the organic dye, 3,3'-diethyltiocarbocyanine iodide. Like for the above-described systems (Figs. 2 and 3), the basic difference in the adsorption behavior of the complexes occurs at

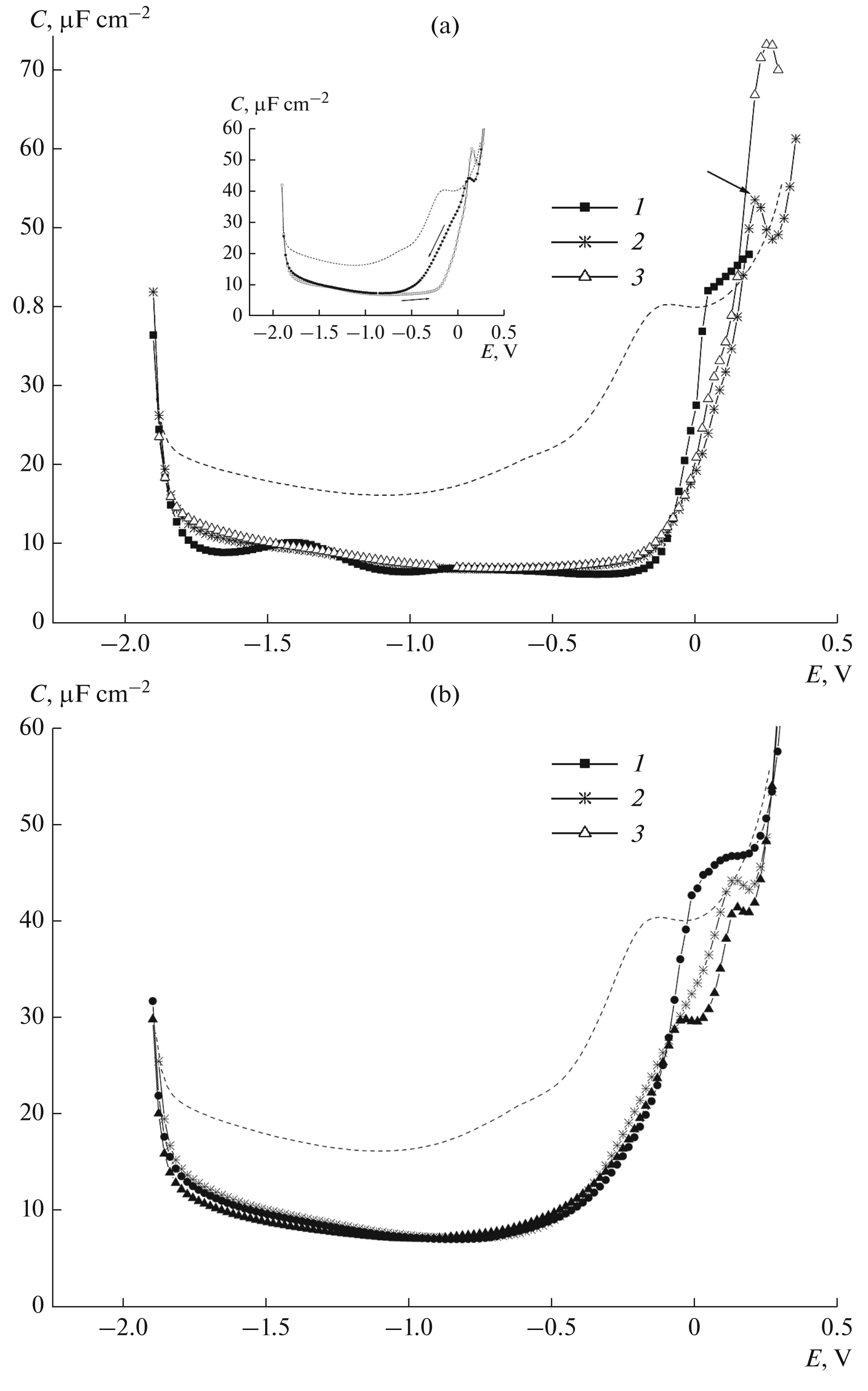

Fig. 5. *C*,*E*-dependences in 0.1 M Na_2SO_4 solution containing 10^{-5} M CB7 + 10^{-5} M ferrocene (*1*), 5 × 10^{-5} M CB7 + 5 × 10^{-5} M ferrocene (*2*), 10^{-4} M CB7 + 10^{-4} M ferrocene (*3*), taken during the potential scanning from negative to positive values (a) and in the reverse direction (b), with the exposing of the working electrode at each *E* value for the time $t = 30$ s. Inset: *C*,*E*-dependences in 0.1 M Na_2SO_4 solution containing 5 × 10^{-5} M CB7 + 5 × 10^{-5} M ferrocene ($t = 30$ s), taken during the potential scanning in opposite directions. Dashed line—*C*,*E*-dependence in 0.1 M Na_2SO_4 solution.

positively charged electrode surfaces. The most surface-active in this region is the CB7 ⊂ 3,3'-diethyltiocarbocyanine$^+$ complex. This can be explained by the significantly weaker effect of the electrical field of electrode on the charge of the cation included to the cavitand cavity. The high strength of the complex, like

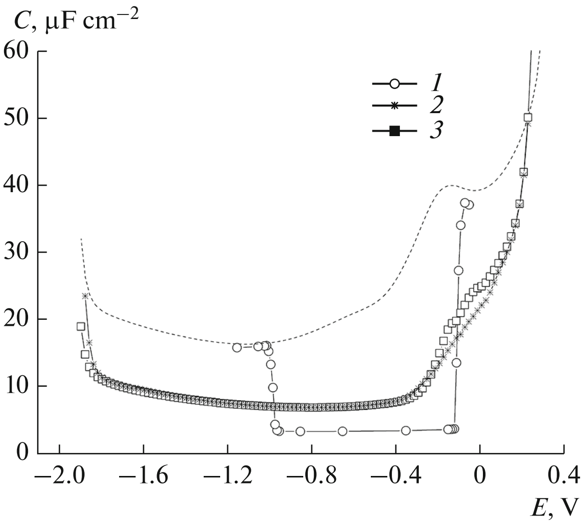

Fig. 6. C,E-dependences in 0.1 M Na_2SO_4 containing 2.5×10^{-4} M adamantanol-1 (*1*), 2.5×10^{-4} M CB7 (*2*), 2.5×10^{-4} M adamantanol-1 + 2.5×10^{-4} M CB7 (*3*); t = 35 s. Dashed line—C,E-dependence in 0.1 M Na_2SO_4 solution.

in the case of CB6 $\subset$ Na^+, leads to a stepwise change of the C values caused by the complex desorption. The CB7 $\subset$ Cs^+ complex seems being the less strong of all; the positive branch of its C,E-dependence is most expanded.

No studies of electrochemical behavior of systems in which the cucurbituril can simultaneously form complexes with neutral organic compound and cation has been carried out so far. We studied adsorption phenomena at electrode/solution interface in 0.1 M Na_2SO_4 solution containing CB7 complexes with ferrocene and adamantanol-1. As we mentioned above, these compounds form extremely strong complexes with CB7. In Figs. 5 and 6 we show C,E-dependences obtained in the 0.1 M Na_2SO_4 solution in the presence of equimolar amounts of CB7 and ferrocene (Fig. 5) and CB7 and adamantanol-1 (Fig. 6). Basic adsorption features obtained in the Na_2SO_4 + CB7 + ferrocene system are similar to those of the CBn_s complexes with the cation Na^+: the adsorption layer slow formation at positively charged electrode surfaces, formation of some intermediate structures at positive charges, and the hysteresis effects. Thus, it is believed that in the studied system the CB7 $\subset$ ferrocene complex formed through ferrocene inclusion to the CB7's hydrophobic cavity simultaneously forms another complex with the Na^+ cation as a result of the cation interaction with the portals' carbonyl groups (in the same way as it occurs in the absence of ferrocene in the system). Here, at negative and small positive surface charges, the adsorption layer is formed by the CB7 $\subset$ ferrocene $\subset$ Na^+ cationic complex; with the increasing of positive charge, neutral CB7 $\subset$ ferrocene complexes are involved in the adsorption layer formation.

The data given in Fig. 6 clearly demonstrate the effect of the CB7–adamantanol-1 complex formation. We see (Fig. 6, curve *1*) that a stable condensed layer of adamantanol-1 molecules is formed at the electrode in the absence of the cavitand. This is evidenced by some typical features of the C,E-dependence, first of all, the abrupt jumps in the capacitance at the adsorption region boundaries, which point to a phase transition in the adsorption layer. We see that the adding of the cavitand equimolar amount removed the effects of two-dimensional condensation completely. The reason is the adamantanol-1 molecule full involvement to the extremely strong complex thus formed. Figure 6 also allowed comparing the adsorption potential region typical of neutral organic compounds and that of the studied supramolecular complexes (which practically is twice as broad). Based on this data, one may generally conclude that the adsorption of cationic complexes at the electrode undoubtedly occurs from 0.1 M Na_2SO_4 solutions containing equimolar amounts of CB7 and ferrocene (or CB7 and adamantanol-1). This conclusion is proved, front of mind, by the practically coinciding potential regions in the 0.1 M Na_2SO_4 + CB7 solutions in the presence and in the absence of ferrocene or adamantanol-1. Thus, the inclusion of a neutral organic molecule to the cavitand cavity undoubtedly has no effect on the cavitand ability to form complexes with inorganic cations; we attribute this phenomenon to the cations' interaction with the portals' carbonyl groups.

CONCLUSIONS

The results of the present work evidence that supramolecular complexes formed in solutions containing alkali metal sulfates (0.1 M) and cavitands of some cucurbiturils are highly surface-active and adsorb at the electrode over extremely broad potential region. The adsorption layers formed at opposite electrode surface charges are shown to have different structure and properties. At negative potentials, the adsorption layers have bilayer structure. At positive surface charges, mixed adsorption layers are presumably formed. Because such layer formation involved the cavitand free molecules, the adsorption phenomena must be strongly affected by any factor governing the concentration of these molecules (the complex strength, the electrolyte concentration). The neutral organic molecule inclusion to the cavitand cavity does not prevent from simultaneous formation of complexes with inorganic cations. The encapsulating of organic cations evidently must change the very essence of the phenomenon radically.

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest

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