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Electrochemical Behavior of Benzene-Polycarmoxylic Acide with Different Number and Location of Containing Carboxylic Groups in Various Solvents

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Abstract: The purpose of the work is investigation of the influence of solvents – water, ethanol and ethylene glycol – nature on electrochemical behavior of nanoparticles of benzene-polycarboxylic acids with the different number and location of containing carboxyl groups: trimellitic acid (benzene-1,2,4-tricarboxylic acid), trimezintic acid (benzene-1,3,5-tricarboxylic acid), pyromelitic acid (benzene-1,2,4,5-tetracarboxylic acid), and mellitic acid with exactly six carboxylic groups (benzene-1,2,3,4,5,6-hexacarboxylic acid). The degree of adsorption of these acids was studied by the method of measuring the differential capacity of the double electrical layer depending on electrode potential. The determination of such as quality and as quantity of these acids in various solutions was carried out by polarographic method. On the base of experimental data were calculated the basic parameters characterizing behavior of nanoparticles at the mercury/solution interfaces. It was established that all the above mention acids have adsorption ability in double electrical layer, but in different degree. Namely, the values of parameters characterizing double electrical layer in the presence of benzene-polycarboxylic acids in various solutions are shown that mellitic acid has a greater adsorption capacity in ethanol solutions in comparison with aqueous solutions. It should be note that no acid shows adsorption ability in ethylene glycol solutions.

Keywords: differential capacity, adsorption, benzene-polycarboxylic acids, polarographic method.

1 Introduction

The different structures of benzene-polycarboxylic acids are widely applied in medicine and pharmacology in various directions. They are used in synthesis of the medicines containing blood maintenance substances, orthopedics, dentistry, also during creation of bio-elements and composite materials. Those acids are characterized by anti-carcinogenic activity as topical antiseptic inhalant decongestants. The benzene-polycarboxylic acids are used as part of a composite substance with a metallic cation in cosmetic and nutraceutical compositions [1]. The benzene polycarboxylic acids occur naturally in many plants and have a long history of use in folk medicine [2,3]. Those acids are used for the fungal skin diseases such as tuna [4], ringworm etc. It is clear that their studying has not only scientific, but also a practical importance.

It is well known, that in the living organism cell or plasma membranes separate the internal contents of any living cell from the environment. The composition of the solutions inside and outside the cells is different and the membranes themselves have selective permeability. The basis of the transport of substances through the membranes is the electrochemical regularities [5]. There are different theories about the ongoing processes in a living cell during metabolism through the plasma membrane [6]. However, all authors agree that simultaneously with other factors an important role is played the adsorption ability of substances at the interface of the plasma membrane and liquid. Therefore, the study of the electrochemical behavior of these acids, namely, the adsorption ability of nanoparticles at the interface of electrode and liquid is important.

The process that takes place at the membrane–solvent interface can be modeled on the basis of some electrochemical systems. Knowledge of the structure of the electrode–solution interface is of great importance. For this reason, we have studied the differential capacity of the

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double electrical layer in the presence of these acids. The information about behavior of substances, which using in medicine in the double electrical layer is useful for pharmaceutical science from the point of view that their adsorption ability facilitates the penetrating through the membrane into a living organism.

We study the differential capacity of double electrical layer in the presence of trimellitic, trimezinic, pyromelitic, and mellitic acids in various solvents. The number and location of functional groups can change the adsorbed/solvent interaction as well as the aromaticity of the benzene ring. The establishment of the adsorption ability of these acids in double electrical layer depending on the location and amounts of carboxylic groups is of great interest for electrochemistry and, especially, medical and pharmaceutical chemistry. The chemical properties of these acids are thoroughly studied [7]. As for the electrochemical properties, in the literature there is very limited information. There are works devoted to the adsorption capacity of individual acids on electrodes from different metals in both pure and mixed solutions. In the work [8], were studied four benzene-polycarboxylic acids at the solid electrodes (Pt, Cu, Ta, and Cu–Hg) in pure aqueous and mixed media (water–ethanol and water–pyridine mixtures) by the voltammetry method. A voltammetry and spectroscopic study of the adsorption and oxidation of benzoic acid on platinum and gold electrodes has been carried out in the aqueous acid medium [9–11]. The work [12] contains data of chemical synthesis of polypyrole film and adsorbing ability of aromatic polycarboxylic acids on this film. The purpose of this paper is to establish a comparative picture, depending on the functional groups in the various solvents.

In this work we also investigated the kinetics of the electrochemical discharge of these acids by the polarographic method with dropping mercury electrode in pure various solvents. For the study of the influence of nature of solvents on the electrochemical behavior of indicated acids were chosen following solvents – water, monoatomic alcohol – ethanol and diatomic alcohol – ethylene glycol, which resembles water in such properties as a high effect of solution, electric conductivity, wide electrode polarization, working temperature ranges and so on [13]. At the same time, they have different physical properties such as viscosity η (at temperature of 20°C water – 0.89, ethanol – 1.07, and ethylene glycol – 19.17mPa) and dielectric constant ε (water – 81, ethanol – 24, and ethylene glycol – 34.5) [14]. It is clear that those properties of the solvents have a significant effect on the behavior of nanoparticles in solution, especially, the diatomic alcohol – ethylene glycol is about 20 times more viscous liquid than water.

2 Experimental

The adsorption ability of above mentioned acids was studied at the mercury electrode in 0.5M NaClO4 or 0.2M Na2SO4 in different solvents by measuring the differential capacity C as a function of electrode potential E by means AC bridge (C,E-curves).The method is that we compare the double electrical layer capacity and the solution resistance with consecutively connected in series box of resistance and capacitance. When establishing equilibrium on the bridge by selecting the values of resistance and capacitance on the screen, the zero of the devices of the sinusoid turns into a horizontal line. We measure the differential capacitance at frequency of 400Hz. Hanging mercury drop electrode was used for the study, which was updated by small moving glass shoved located in the electrochemical cell. As an auxiliary electrode, a spherical form of mercury was used at the bottom of the cell.

Investigation of kinetic process of those acids qualitatively and quantitatively can be performed by the polarographic method with a dropping mercury electrode (t = 3.5s and m = 2.6mg/s). The polarographic method for a non-immiscible solution, in which diffusion is the predominant type of mass transfer, is one of the most useful and accessible methods for studying processes involving organic compounds. Anode was a platinum wire.

In both experimental methods of investigation, the value of potential was taken towards the saturated calomel electrode potential. All the solvents were used in twice-diluted form and experiments performed in the thermostatic three-electrode cell (T = 25ºC). Trimellitic, trimesic, pyromelitic, and mellitic acids were obtained from Aldrich and Fluka and used without further purification. For the determination of a degree of reproducibility, the experiments were repeated several times under the same conditions; the deviation made up to ±1.5%. The polarographic curves are recorded in potential range from 0 to –2.2V.

3 Results and Discussion

Experimental results of adsorption ability for acids are presented in **Figures 1–3**. In order to thoroughly study the electrochemical behavior of nanoparticles of these acids, on the base of experimental data by the theory of Frumkin–Damaskin for adsorption of organic substances on the electrode [15] we calculated the following significant parameters characterizing the processes taking place in the double electrical layer in various solvents: attraction constant a, the value of maximum adsorption Γ_m [mol·cm^{-2}], adsorption equilibrium constant B [L·mol^{-1}], standard adsorption energy ΔG_A [kJ·mol^{-1}], and charge on the electrode surface σ [μC·cm^{-2}]. In this article, were calculated above listed adsorption parameters for four acids on the basis of experimental data, but detailed calculation is given only for trimellitic acid in aqueous solution as an example. The calculated values of the adsorption parameters for all acids are shown in the **Table 1**.

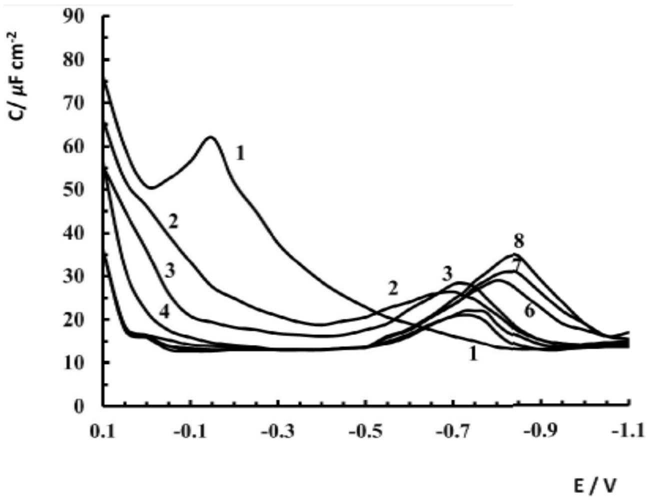


Fig. 1: Dependence of differential capacitance on potential of mercury electrode in 0.2M Na2SO4 aqueous solutions with addition of trimellitic acid in following concentrations: 1 – 0, 2 – 4.7•10–4, 3 – 9.4•10–4, 4 – 1.4•10–3, 5 – 1.8•10–3, 6 – 2.2•10–3, 7 – 2.7•10–3, and 8 – 3.2•10–3M.

parameters, these acids have been given quantitatively somewhat different pictures.

As can be seen from Figure 1, the maximum decrease of capacity (13μF•cm–2) occurs in solution with acid concentration of c = 1.4•10–3M and with further increase in the concentration, there is observed insignificant increase in the capacity (0.6μF•cm–2). The value of capacity becomes constant in certain concentration region of 1.8•10-3–2.7•10-3M (Curves 5–7). The capacitive minimum is bounded by the cathode maximums, the height and potential of which varies depending on the over shifts to the cathode side, which indicates their adsorption–desorption character. At a high negative value of potential, adsorbed nanoparticles are desorbed from the electrode surface.
According to the work [8], at the concentration of trimellitic acid in millimolar solutions simultaneously there are both large-sized anions and non-dissociated molecules. Probably, during the adsorption process, several effects are superimposed on each other: expulsion of adsorbed molecules from bulk solution, the effects of the π-electron interaction with the positively charged electrode surface, etc. In diluted solutions, these effects are added with stereo and dipole factors, which in dilute solutions are causing the maximum decrease in the capacity. With further increasing of concentration, the attraction of anionic adsorbed particles by the positively charged electrode became stronger. As a result, the thickness of the adsorption layer is decreased and the capacity increase. The obtained data were processed according to the Frumkin–Damaskin theory for adsorption of organic substances [15]. By integrating the C,E-curves (Figure 1), the charge on the electrode surface σ was calculated and then σ,E-curves were plotted for the further integration from which, we calculated the interfacial tension and belt γ,E-curves. For clarity, the γ,E-curves are given for trimezinic acid in ethanol solution (Figure 2).

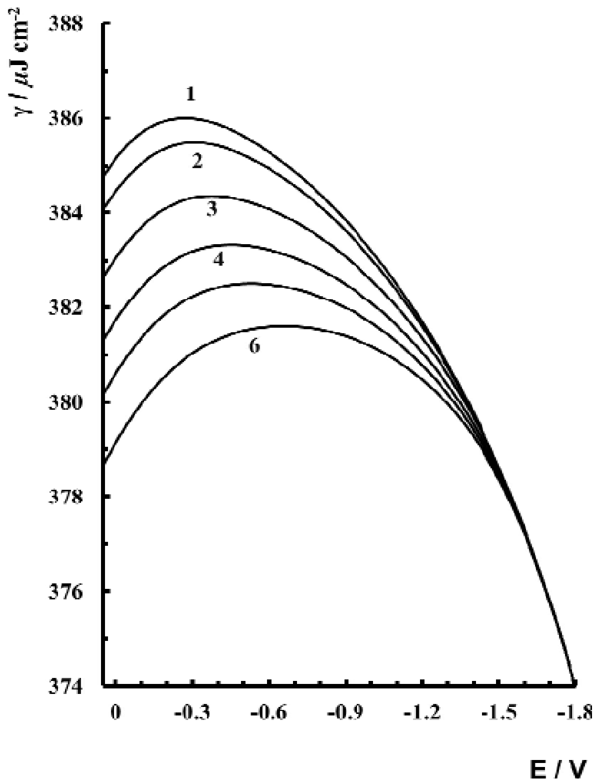


Fig. 2: Calculated electrocapillary curves on mercury electrode in 0.5M NaClO4 ethanol solutions with addition of trimezinic acid in following concentrations: 1 – 0, 2 – 1•10–4, 3 – 4•10–4, 4 – 8•10–4, 5 – 1.4•10–3, and 6 – 3.8•10–3M.

We were plotted Δγ,logc-curves at the value of potential of E = –0.4V (Figure 3a). On the basis of the dependence of Δγ from logc and the equation

$$\Gamma = -c/RT \qquad (1)$$

we calculated the value of Γ. From Γ,*c*-curve, the maximum adsorption $\Gamma_m = 1.52 \cdot 10^{-11} mol \cdot cm^{-2}$ was determined. For various concentrations of trimellitic acid with the help of the equation $\Gamma / \Gamma_m = \theta$ it was calculated values of θ and constructed θ,*c*-curve of the adsorption isotherm. The adsorption isotherm has the shape of *S*, which it is characteristic of those substances, adsorbed molecules of which are attracted to each other. For calculation of this interaction (the attraction constant *a*) we used the Frumkin isotherm equation:

$$B\,c = (\theta / (1 - \theta)) \exp(-2\,a)\,. \qquad (2)$$

From this equation, when $\theta = 0.5$, we get:

$$a \approx 2.3 / (1 - 2\theta) \log ((y\,(1 - \theta)) / \theta)\,. \qquad (3)$$

From above equation, the attraction constant is of a = 1.6. Sane value we were calculated by another method as well –

in logarithmic coordinates:

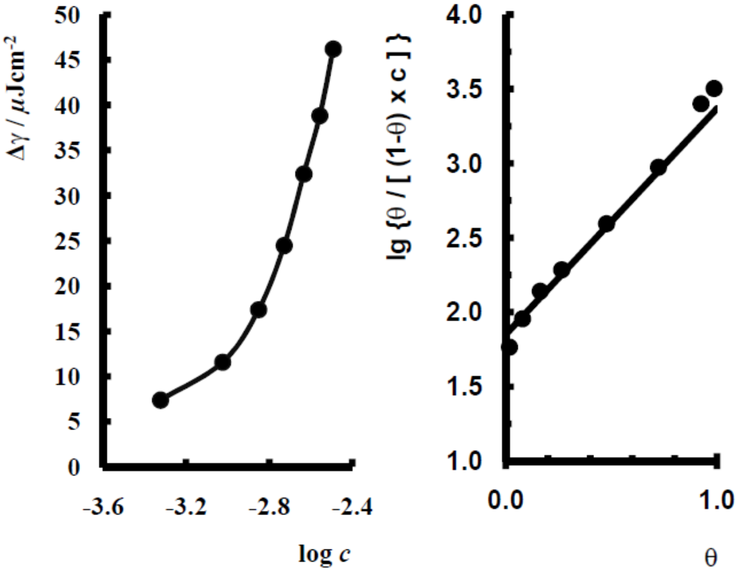
$$\log(\theta/((1-\theta)\,c)) \approx \log B + 2\,a\,\theta/2.3 - \log 55.5\,, \qquad (4)$$

where 55.5 is the quantity of moles in 1L of water, B is the adsorption equilibrium constant. For molecules of trimellitic acid in **Figure 3b**, the dependence of log ($\theta/((1-\theta)\,c)$) on θ is given, which represents a straight line and value of attraction constant by this method $a = 1.7$. The values of attraction constant calculated by different methods are in good agreement. In **Figure 3b**, the point of intersection of the straight line with axis of ordinates gives value $\log B \approx \log 55.5$, from which calculated value of the adsorption equilibrium constant is equal to 3926L·mol^{-1}. The value of standard free energy of adsorption ($-\Delta G_A$ = 20.15μJ·mol^{-1}) has been calculated from equation:

$$-\Delta G_A = R\,T\ln B. \qquad (5)$$

Analysis of experimental data within the framework of the Frumkin–Damaskin theory shows, that the adsorption of trimellitic acid conforms well to the Frumkin isotherm for particle–particle interaction, which corresponds the attractive interaction of adsorbed particles. It should be noted that during the anodic polarization of the electrode, the adsorption of particles is caused by strong π-electronic interaction of aromatic ring with electrode surface. The calculated parameters according to the above procedure for these acids in ethanol are given in Table 1.

When comparing the calculated parameters given in the Table 1, it is evident that the adsorption ability of trimellitic acid is more significant than that of trimesinic. The reason for this is the arrangement of the carboxylic groups in case of trimellitic acid (1,2,3). It is known that the effect of the carboxyl groups on each other is stronger the closer they are. Trimellitic acid is stronger than trimezinic acid and in the summed adsorption pattern, the role of trimellitic acid is aggravated by anodic polarization due to electrostatic attraction of the anions located closely.



(a) (b)

Fig. 3: Dependence of change of boundary tension on logarithm of concentration of trimellitic acid (a) and linear relationship between log ($\theta/(1-\theta)\,c)$) and θ (b).

Table 1. Adsorption parameters for benzene-polycarboxylic acids.

Benzene-polycarboxylic acid	**Solution**	**Attraction constant *a***	**Standard adsorption energy $-\Delta G_A$, μJ·mol^{-1}**	**At half value of coverage $c_{\theta=0.5}$, mol**	**Adsorption equilibrium constant *B*, L·mol^{-1}**
Trimellitic	Aqueous	1.36	20.15	$2.4\cdot10^{-3}$	3926.4
Trimezinic	Aqueous	1.47	22.22	$1.5\cdot10^{-3}$	9204.5
	Ethanol	1.68	21.16	$3.0\cdot10^{-4}$	5956.6
Pyromelitic	Aqueous	1.80	19.13	$3.8\cdot10^{-3}$	2580.5
Mellitic	Aqueous	1.57	18.79	$4.4\cdot10^{-3}$	2243.9
	Ethanol	1.61	23.52	$2.1\cdot10^{-4}$	15659.0

The difference in the adsorptive ability of these acids from the ethanol and aqueous solutions also has to be caused by various orientations of these solutions to electrode surface. According to available data [8], in the both solvents are big sizes acids anions and some quantities of non-diagnosed molecules. It is possible to assume some simultaneous effects: expulsion of adsorbed molecules from bulk solution on electrode surface, effect of the π-electronic interaction on the positively charged surface of the electrode, effect of electrostatic interaction of carboxyl group to surface, etc.

Lower value of a depression of capacity in ethanol than in aqueous solutions are explained in the most part by that component, in the size of adsorption which corresponds to orientation of carboxyl group in ethanol solutions to the electrode surface. A decrease in the thickness of the adsorption layer leads to an increase in the capacity. Following adsorptive parameters are calculated from Frumkin–Damaskin theory: minimum value of capacity C', concentration corresponding to half of degree of fullness ($y = c_{\theta=0.5}$), constant of interaction of the adsorbed molecules a, the adsorptive constant released at adsorption standard energy $-\Delta G_A$. Released standard energy at adsorption of benzene-carboxylic acids has less value in water solutions than in the ethanol solutions that indicates more adsorptive ability of benzene-carboxylic acids in the ethanol solutions in comparison with aqueous solutions.

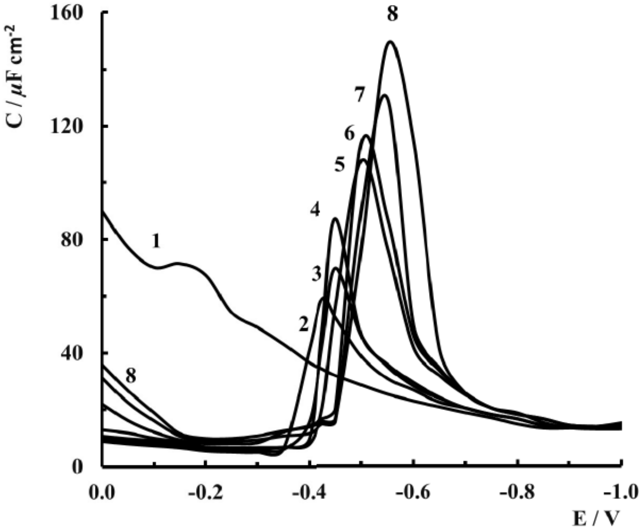


Fig. 4: Dependence of differential capacitance on potential of mercury electrode in 0.2M Na_2SO_4 aqueous solutions with addition of mellitic acid in following concentrations: 1 – 0, 2 – $8.7\cdot10^{-4}$, 3 – $1.7\cdot10^{-3}$, 4 – $2.6\cdot10^{-3}$, 5 – $3.4\cdot10^{-3}$, 6 – $4.3\cdot10^{-3}$, 7 – $5.1\cdot10^{-3}$, and 8 – $6.0\cdot10^{-3}$M.

The adsorption behavior of the pyromelitic acid was studied in the same condition that we have been researched the above mention acids. The form of curves and the calculated parameters of them indicated that the pyromelitic acid has the adsorption ability on the mercury/aqueous and mercury/ethanol solutions interface. The corresponding calculated parameters of the double electric layer in presence of pyromelitic acid are presented in the Table 1.

In the **Figure 4**, it is shown the dependence of the differential capacity on the potential of the mercury electrode in 0.2M Na_2SO_4 aqueous solution with the addition of mellitic acid. There are two areas of capacity reduction on C,E-curves. At anodic polarization π-electron interaction of adsorbed molecules with a positively charged surface occurs. The maximum decrease of capacity occurs in too diluted solutions ($8\cdot10^{-4}$M), where mellitic acid mainly are as a large aromatic ion because the degree of dissociation is great. Consequently, in the first section of adsorption (anodic side from $E = -0.3$V) overlap the effects of the π-electron and electrostatic interactions with electrode surface. When the cathode polarization of the electrode is more towards the cathode side from $E = -0.3$V, the adsorbed molecules ejected from the bulk solution (first plot). The height of the cathode peaks increases with increasing concentration and their potential shifts to the cathode side, which indicate that they are adsorption–desorption peaks. At high negative potential ($E = -0.95$V), the curves over the entire concentration range coincide with each other, which indicates that the acid molecules are completely desorbed from the electrode surface. A sharp decrease in capacitance at low acid concentration adds the stereo and dipole effects to the above factors. With increase in the concentration of acid, increases the pushing out of large molecules from the bulk solution and reduce the stereo effects. Therefore, increase charge density of negative ions in the adsorption plane causes increasing electrostatic attraction and, consequently, reduces layer thickness and it means increase in the capacity value.

The dependence of the differential capacitance on the potential of the mercury electrode in 0.5M Na_2SO_4 ethanol solutions changes with the addition of mellitic acid. Mellitic acid is adsorbed in the anode region of polarization $E = -0.1 - +0.2$V, which is caused by the π-electron interaction of aromatic ring with a positively charged electrode surface. Maximum capacity reduction ($C = 11.6\mu F\cdot cm^{-2}$) occurs at $E = +0.5$ V. Capacity depression is from 30 to $18\mu F\cdot cm^{-2}$, where $30\mu F\cdot cm^{-2}$ is the value of the differential capacitance of supporting electrolyte. As can be seen from **Figure 3**, in an aqueous solution, the maximum decrease of capacitance ($C' = 5.2\mu F\cdot cm^{-2}$) takes place in the region of potentials $E = -0.1 - +0.4$V and the depression of the capacitance is hesitant within 55–$35\mu F\cdot cm^{-2}$. In ethanol solutions, in the adsorbed state, mellitic acid in the double electric layer is located in a narrow potential interval and at $E = -0.5$V it is almost removed from the electrode surface. In the aqueous solution full desorption of mellitic acid occur at the potential $E = -1.0$V. The different adsorption behavior of mellitic acid in aqueous and ethanol solutions is apparently caused by the different orientation of these solutions towards the electrode surface. In both solvents according to [8], mellitic acid in solution is not only in the form of large anions but also in a certain number of not dissociated molecules (mellitic acid is dibasic acid).

Table 1 shows the values of the adsorption parameters for all four acids calculated on the basis of experimental data and the theory of Frumkin–Damaskin.

Taking into account the fact that organic acids studied by us are used in medicine, we carried out the determination of their concentration by a polarographic method, which is characterized by high sensitivity and comparative simplicity. The polarographic measurement in not mixed solution, in which diffusion appears the prevailing stage of a mass transfer, is one of the most useful and available research technique of electrode processes with participation of organic connections. Polarographic measurements are widely used in analytical chemistry for determination of substance concentration. When studying the degree of adsorption of above acids, it became clear that no one of them shows adsorption capacity in ethylene glycol. Consequently, the study of the kinetics of the process of discharge of acids in ethylene glycol allows us to avoid the influence of adsorption effect, which usually considerably complicates the determination of the kinetics of electrode processes. The value of half-wave potentials $E'_{1/2}$ in a series of related compounds is characterized not only by the ability for electro-reduction of this group, but also by the electronic and stereo interactions of the latter with the polarographic inactive part of the molecule, the mutual influence of atoms and groups in the molecule.

We conducted a polarographic study of benzene-polycarboxylic acids such as in ethylene glycol solution as in water (**Figures 5–7**). All the four acids are polarographically active and give diffusion, irreversible waves directly proportional to the bulk concentration. The benzene-polycarboxylic acids studied by us with symmetric arrangement of carboxyl groups in water solutions give single-wave polarograms with following values of half-wave potentials: trimesinic –1.73 and mellitic –1.62V. As for trimellitic and pyromelitic acids in aqueous solutions, it gives two-wave polarogram with following values of half-wave potentials, respectively, $E'_{1/2}$ and $E''_{1/2}$: trimellitic –1.27 and –1.60, and pyromelitic –1.10 and –1.68V. It should be noted that all of the studied benzene-polycarboxylicacids in ethylene glycol give single-wave polarograms with following values of half-wave potentials: trimellitic –2.0, trimesinic –1.85, pyromelitic –1.9, and mellitic –2.1V. The inhibiting factor of the discharge of acid molecules in the ethylene glycol at the more negative potential is the high viscosity of this solvent.

The pyromelitic acid, which has four carboxylic groups, was shown to have the different behavior from other acids. Namely, on the curves ate appearing maxima of the first kind such as in aqueous solutions in ethylene glycol.

They have the shape of an acute peak and arise in diluted Solutions of supporting electrolytes. Mairanovsky and coworkers [16] explain the main reason for the appearance of maxima by the mixing of the solution near the surface of the mercury drop electrode. The reason for the mixing is the unevenness of the polarization of the surface of the electrode, which is the result of fluid motion near the surface of the mercury drop. In spite of differences in explaining the mechanism of formation of the maximum, all the authors agree, that the maxima are caused by the influence of electric charge on the mercury electrode. Therefore, polarographic maximum of the first kind cannot be formed or must disappear at the zero-charge potential of the mercury dropping electrode. To suppress the maximum, a surface-active substance – gelatin was added to the solution.

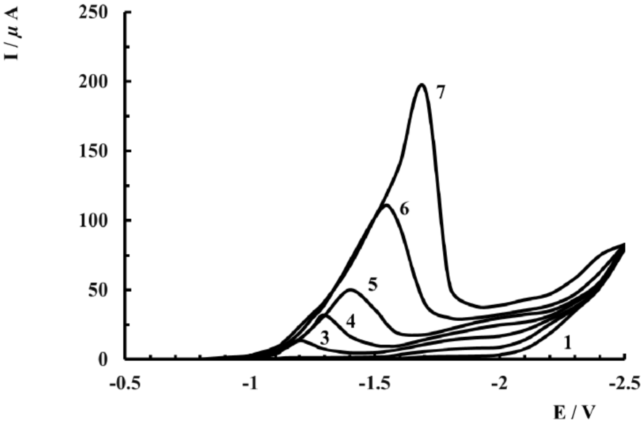


Fig. 5: Polarographic curves of reduction of following concentrations of pyromelitic acid in 0.5M $NaClO_4$ aqueous solutions: 1 – 0, 2 – $3.8\cdot10^{-4}$, 3 – $1.2\cdot10^{-3}$, 4 – $2.2\cdot10^{-3}$, 5 – $3.2\cdot10^{-3}$, 6 – $3.5\cdot10^{-3}$, and 7 – $4.0\cdot10^{-3}$M.

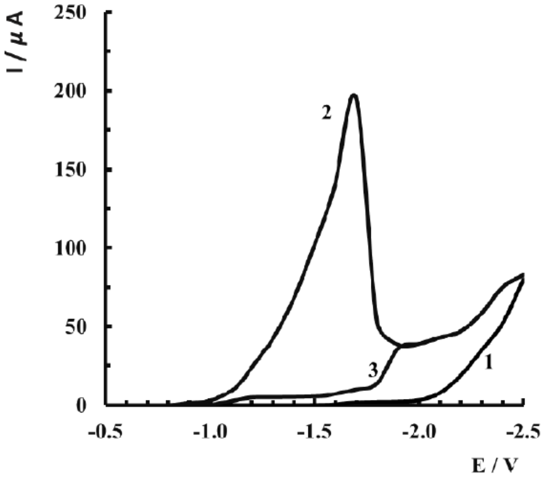


Fig. 6: Polarographic curves of reduction of following concentrations of pyromelitic acid in 0.5 M NaClO4 aqueous solutions: 1 – 0, 2 – 4.0•10–3, and 3 – 4.0•10–3(+gelatin) M.

The direct proportionality between the heights of diffusion currents and the concentrations of our acids such as in water as in ethylene glycol allows us to determine their concentrations. As an example, we present the polarographic curves of mellitic acid in an aqueous solution and the calibration graph was bolted from it (Figure 8).

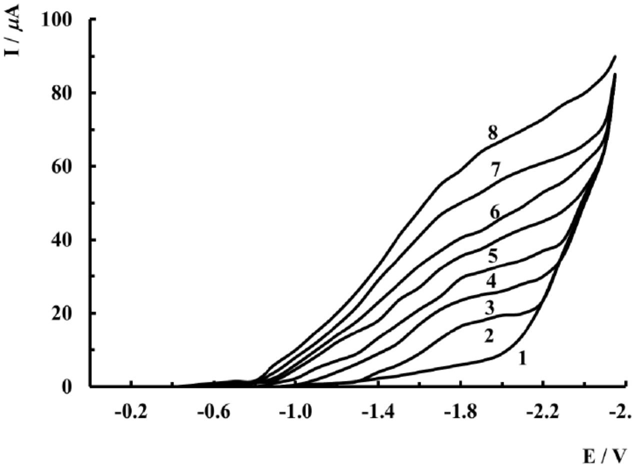


Fig. 7: Polarographic curves of reduction of following concentrations of mellitic acid in 0.5M NaClO4 aqueous solutions: 1 – 0, 2 – 8.0•10–4, 3 – 1.4•10–3, 4 – 1.9•10–3, 5 – 2.3•10–3, 6 – 2.7•10–3, 7 – 3.0•10–3, and 8 – 4.0•10–3M.

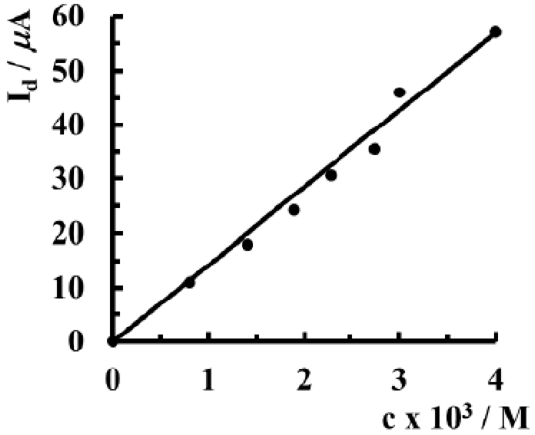


Fig. 8: Dependence of height of limit current on concentration for mellitic acid in 0.5M NaClO4 aqueous solutions at E = –1.9V.

3 Conclusions

The adsorption of four benzene-polycarboxylic acids with a different content of number and location carboxylic groups in various solvents on Hg was studied by measuring the differential capacity as a function of the electrode potential. It was shown that all acids studied by us show adsorption ability in both aqueous and ethanol solutions.

The values of the significant parameters characterizing the processes taking place in the double electrical layer in various solvents indicate that all four acids have differential capacities slightly different from each other. The comparison of their behavior in water and ethanol solutions showed that in the anode side they are adsorbed from ethanol solutions better than from water and in the cathode side they adsorbed worse. It should be noted that no one acid shows adsorption ability in ethylene glycol solutions. The reason for this, by our opinion, is superficial activity of the ethylene glycol with high value of viscosity of ethylene glycol that prevents the movement of large molecules of the benzene-tetracarboxylic acids to the electrode surface.

Analysis of the experimental data and the calculated by Frumkin–Damaskin theory the values of parameters are show that during the adsorption process, probably, several effects are superimposed on each other:

Expulsion of adsorbed molecules from bulk solution; Effects of strong π-electronic interaction of aromatic ring with positively charged electrode surface; and Effect of electrostatic interaction between carboxyl groups.

It has been shown that the acids studied by us exhibit polarographic activity and give diffusion waves directly proportional to concentrations, which makes it possible to determine their concentration.

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