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Electrochemical and Transport Characteristics of Anion Exchange Membranes in Succinic Acid Solutions

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Abstract: The physicomechanical and transport characteristics of heterogeneous anion exchange membranes Ralex AMH-Pes and MA-41, and homogeneous anion exchange membranes AHT and MA-1 in solutions of sodium chloride and succinic acid are studied. It was found that in succinic acid solutions, the concentration dependence of the specific electrical conductivity of anion-exchange membranes has an atypical course compared with the dependence measured in solutions of strong electrolytes. This is due to a change in the pH and ionic composition inside the membrane compared to the external working solution. The concentration dependence of the integral coefficient of diffusion permeability of anion-exchange membranes in succinic acid solution has a decreasing character. This dependence can be explained by the pH shift inside the membrane to a more alkaline region when diluting the external solution, while the equilibrium of the succinic acid dissociation reaction inside the membrane shifts towards the formation of a two-charge succinate anion. With an increase in the proportion of double-charged ions in the membrane phase, an increase in electrostatic forces capable of attracting ions of the opposite charge sign occurs. At the same time, the concentration of co-ions in the membrane phase increases. This effect leads to an increase in the rate of diffusion transfer of the succinic acid molecule with a decrease in its concentration in the working solution.

The study of the mass transfer of succinic acid through anion-exchange membranes and the volt-ampere characteristics (CVC) of the anion-exchange membranes AHT and MA-1 showed that succinic acid is capable of being intensively transferred through anion-exchange membranes in the super-limit current mode, as a result of the development of electroconvection at the ion-exchange membrane/diffusion layer interface..

Keywords: aion-exchange membrane; ion transport; current-voltage characteristic; water-splitting; weak electrolyte; succinic acid

1. Introduction

Succinic acid is used in various fields: food industry (as an antioxidant and acidity regulator, food additive E363); agriculture (plant growth regulator, food additive for animals); chemical industry, as a precursor to many chemicals that meet the principles of "green chemistry" (environmentally friendly solvents demethylsuccinate and 1,4-butanediol, as well as biodegradable polymers polybutylene succinate, Bionelle, etc.) [1,2]. But the most important use of succinic acid has been found in medicine and pharmacology as: sedative; antispasmodic; antithyroid; anti-hopping agent[3,4]. Traditionally, succinic acid is obtained in a petrochemical process by catalytic hydrogenation of maleic anhydride obtained from butane [5]. However, succinic acid, which is used in the food industry and medicine, is produced exclusively through biotechnological production [1,6,7,8]. With this method of production, succinic acid is formed in the form of salts and it must not only be extracted from an enzymatic mixture containing also proteins and saccharides, but also converted into acid form for further use[9,10].

Various methods are used to extract succinic acid from the enzymatic mixture: baromembrane processes [11] recrystallization [12], desalination [13], precipitation and extraction [10,15]. A promising method is the bipolar electrodialysis method, since it allows not only to extract succinic acid salts from a mixture, but also to obtain succinic acid in molecular form [14,16]. As shown by the authors of [14] who studied the process of conversion of succinic acid salts by bipolar electrodialysis with different configurations of the electrodialysis apparatus and the arrangement of membranes, the most effective process is provided by the configuration in which succinate anions are transferred through the anion exchange membrane from the salt to the acid tract. In this case, the efficiency of the processing process will be largely determined by the properties of the anion exchange membrane in the processed solution.

Succinic acid is a weak electrolyte. Weak electrolytes and ampholites, depending on the pH in the solution, can be in various forms: molecular and/or ionic (single and multicharged). For this reason, the electrochemical and transport properties of ion-exchange membranes in solutions of weak electrolytes differ from those in solutions of strong electrolytes and depend on pH. Thus, a decrease in pH leads to a decrease in the flow of carbonate anions through ion-exchange membranes due to the formation of carbonic acid [17]. A decrease in pH from 6 to 2.5 leads to a 2-fold decrease in tartrate mass transfer [18]. This is due to the fact that the two-charge anion $C_4H_4O_6^{2-}$ transforms into single-charge $C_4H_5O_6^-$ and molecular C4H6O6 forms. In [19] the transport of H_3BO_3 through anion exchange membranes was studied depending on the pH of the solution. It was found that with an increase in pH from 3 to 9, the boron flux increased by about 18 times, but a further increase in pH leads to a decrease in boron transfer.

The effect of the pH of the solution inside the ion exchange membrane on diffusion transfer is considered in [20,21]. The authors [20] studying the diffusion transfer of amino acids through ion-exchange membranes, revealed the mechanism of "facilitated" transfer of amino acids through anion–exchange membranes in OH^- form. Bipolar charged amino acid ions interact with OH^- ions and turn into anions, which are counterions for the anion exchange membrane. In this case, the flow of amino acid anions increases, compared with the diffusion flow in the membrane in the chloride form. A similar mechanism of "facilitated" diffusion was observed by the authors for ammonium cations [21] by investigating the diffusion permeability of anion-exchange membranes in solutions of ammonium chloride.

The current-voltage characteristics (CVC) of ion-exchange membranes in solutions of some weak electrolytes differ from the CVC in solutions of strong electrolytes. The authors of the works [22,23,24,25,26] studying the properties of ion-exchange membranes in phosphoric acid solutions at pH = 4.7 by voltammetry, two sections of the limiting current were observed on the CVC. Authors [22,23,24,25] This effect was explained as follows: the flow of electric current through the membrane system leads to a decrease in the concentration of single-charge phosphate ions in the emitting diffusion layer. This is manifested in the appearance of the first limiting current. A further increase in the current above the limit occurs due to the dissociation of water, while OH^- ions are transferred to the membrane phase, which leads to an increase in pH inside the membrane. In this case, single-charge phosphate ions, crossing the solution/membrane boundary, turn into a two-charge form. The second limiting current is associated with the almost complete transition of the membrane into the form of double-charged phosphate anions. An increase in current above the second limit is possible due to two mechanisms: the transition of the membrane into the form of three-charged phosphates, as well as the dissociation of water.

The CVC of the anion exchange membrane in a mixture of acetic, malic and citric acids also has two points of tilt change [27]. The authors [27], also associate the first limiting current with a decrease in the concentration of the substance in the diffusion layer, the presence of the second limiting current is explained by a change in the ionic forms of the components of the mixture, leading to an increase in the resistance of the system.

Also, two limiting currents are observed for anion-exchange membranes in individual solutions of weak organic acids: citric (at pH=7.8) and oxalic (at pH=6.8), whereas at other pH values, the CVC of the membranes does not differ from the CVC of strong electrolytes [28]. Having studied the volt-ampere characteristics and frequency spectra of the electrochemical impedance of ion-exchange membranes, the authors [28] concluded that the presence of the first limiting current is not associated with diffusion limitations, but is explained by a change in the ionic conductivity of a thin reaction layer near the membrane. The fact is that at pH values of 7.8 and 6.8, citric and oxalic acids, respectively, are in solution mainly in the form of a two-charge anion. The flow of an electric current leads to a decrease in the pH value in the primembrane region from the giving side of the anion exchange membrane. This effect leads to a shift in equilibrium towards the single-charge ionic form and an increase in the resistance of the electromembrane system, which is reflected in the form of a plateau of the first limiting current. The authors attribute the presence of the second limiting current to the depletion of the diffusion layer.

The transfer of weak electrolytes in extreme and ultra-extreme modes also has a number of features. In the works [29,30,31,32] The so-called "barrier effect" was discovered by those who studied the electrodialysis of carbonate anions, tartrate ions, amino acid ions and ammonium cations. The essence of which lies in the fact that in extreme current regimes, the flow of ions of a weak electrolyte decreases, compared with other ions, due to their interaction in the primembrane layer with hydrogen cations, which are formed as a result of intense dissociation of water molecules, with the formation of an uncharged molecular form [29,30]. In the case of amino acids that are ampholytes, the barrier effect is associated with the recharging of amino acid ions at the surface of ion-exchange membranes, which blocks their further transfer through the membranes (circulation effect). In this case, the amino acid remains in the desalination chamber, while other ions pass into the concentration chamber [31].

Thus, the properties of ion-exchange membranes in solutions of weak electrolytes differ from those in solutions of strong electrolytes, and the transfer of weak electrolytes through ion-exchange membranes is a complex process and depends on the pH of both the equilibrium solution and inside the membrane itself.

The aim of this work is a comparative study of the electrochemical properties of anion–exchange heterogeneous and homogeneous membranes and the determination of the mechanism of ions transport in solutions of a strong electrolyte – sodium chloride and a weak electrolyte - succinic acid.

2. Materials and Methods

Sodium chloride (NaCl), nitric acid with a standard titer (HNO_3), sodium hydroxide with a standard titer (NaOH), succinic acid ($C_4H_6O_4$) were purchased from Chemmed (Moscow, Russia). Deionized water was used in all experiments.

Objects of the Study

The objects of the study were the following anion exchange membranes: heterogeneous anion exchange membrane MA-41 (Shchekinoazot LLC, Russia) and heterogeneous anion exchange membrane Ralex AMH Pes (MEGA a.s., Czech Republic). These membranes consist of a polystyrene matrix cross-linked with devenylbenzene containing quaternary amino groups. To give the membranes mechanical strength, they are reinforced with filaments: nylon (MA-41) and polyestersulfone (Ralex AMH Pes). Anion exchange homogeneous membrane AHT (LANRAN, People's Republic of China). Currently, there is no information about the nature of fixed groups and the AHT membrane matrix in the open literature. Also, as part of the work, the anion-exchange homogeneous membrane MA-1 was studied, developed by employees of the Department of Physical Chemistry of Kuban State University [33]. This membrane is chemically a copolymer of N,N-diallyl-N,N-dimethylammonium chloride and ethyl methacrylate. Quaternary ammonium groups in the structure of pyrrolidine cycles formed as a result of polymerization of N,N-diallyl-N,N-dimethylammonium chloride, rigidly bound to the polymer matrix, act as an ion-exchange fragment in this membrane.

Succinic acid is a weak electrolyte capable of dissociating in two steps to form single and/or double-charged anions [34]:

$$C_4H_6O_4 \rightleftarrows C_4H_5O_4^- + H^+ \qquad K_1 = \frac{[C_4H_5O_4^-][H^+]}{[C_4H_6O_4]} = 5.67\cdot10^{-5}\ \text{mol/L} \qquad (1)$$

$$C_4H_5O_4^- \rightleftarrows C_4H_4O_4^{2-} + H^+ \qquad K_2 = \frac{[C_4H_4O_4^{2-}][H^+]}{[C_4H_5O_4^-]} = 2.31\cdot10^{-6}\ \text{mol/L} \qquad (2)$$

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Thus, depending on pH, succinic acid in solution can be in various forms, Figure 1.

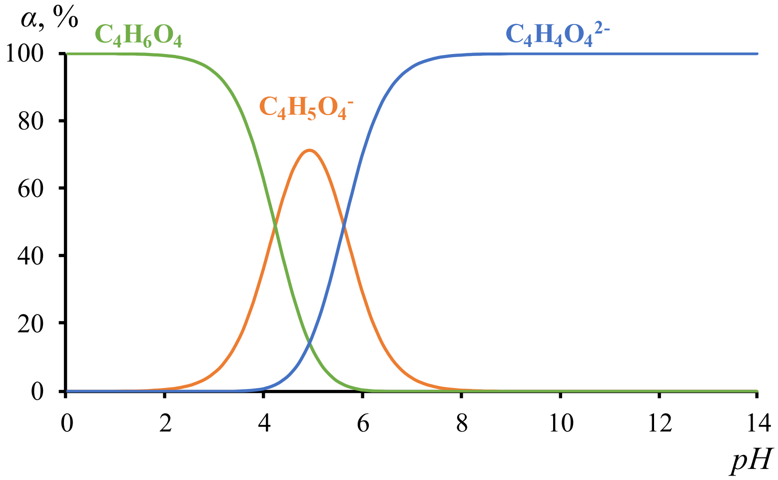

Figure 1. Distribution of succinic acid forms depending on pH

At pH < 3, succinic acid is almost all in molecular form. At pH = 4.8, the maximum content of the single-charge form is observed, at this pH value, the mass transfer of succinic acid is most effectively carried out under electrodialysis conditions: the transfer of 1 C of electricity is accompanied by the transfer of approximately 1 mole of matter. For this reason, the properties of anion-exchange membranes were subsequently studied at this pH value. At pH > 7, the double charged anion becomes the dominant form.

2.4. Investigation of the physico-mechanical and transport-structural characteristics of anion exchange membranes

The following were experimentally investigated: exchange capacity, true density, moisture content, swelling, electrical conductivity, as well as diffusion permeability. The exchange capacity, density, swelling and moisture capacity of anion exchange membranes were determined by standard methods [35]. The swelling of ion-exchange membranes was determined in solutions of 0.5 M NaCl and 0.5 M succinic acid at pH = 4.8.

The electrical conductivity of anion exchange membranes was studied by the mercury contact method [35] (using a potentiostat-galvanostat P-45X "Electrochemical instruments") in solutions of sodium chloride (0-0.2 M) and succinic acid pH = 4.8 (0-0.1 M).

The diffusion permeability of anion exchange membranes was studied in a non-flowing cell, Figure 2. The cell consists of two chambers separated by a test membrane (1). The chamber (2) was filled with a test solution of sodium chloride (0.1-1.5 M) or succinic acid (0.1-0.6 M) at pH = 4.8, the chamber (3) was filled with 100 ml of distilled water, into which a conductometric cell with a thermal sensor was immersed (4) and a combined pH electrode (5). Mixing of the solution to reduce the diffusion layers is provided using agitators (6). During the experiments, the value of the electrical conductivity, temperature and pH of the solution in the chamber (3) was recorded using an Expert 002 conductometer, an Expert 001 ionomer manufactured by Econix-Expert LLC, a personal computer and Expert 00x software.

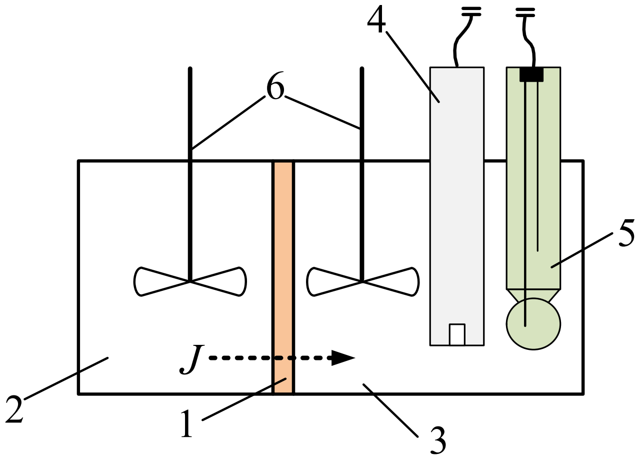

Figure 2. Diagram of a cell for studying the diffusion permeability of ion exchange membranes: 1 – the membrane under study; 2 – a chamber with a working solution; 3 – a chamber with distilled water; 4 – a conductometric cell; 5 – a combined glass electrode for pH measurement; 6 – agitators

The value of the integral coefficient of diffusion permeability was determined by the formula (3):

$$P_m = \frac{Vl}{SC}\frac{dC}{dt}, \tag{3}$$

where P_m – is the integral coefficient of diffusion permeability, m²/s; V – is the volume of the chamber (3), m³; l – is the membrane thickness, m; S – is the working area of the membrane, m²; C – is the concentration of the test solution in the chamber (3), mol/m³; $\frac{dC}{dt}$ – the rate of concentration change in the chamber (3).

The sodium chloride content in the chamber (3) was determined by the calibration dependence of the electrical conductivity on the concentration. Difficulties may arise in the conductometric determination of the content of succinic acid and its anions in the chamber (3), since it is a weak electrolyte and its concentration will depend not only on electrical conductivity, but also on pH. In the course of the study, the approach described in [36], was used, according to which the specific electrical conductivity of a dilute solution is related to the concentration of dissolved particles according to the Kohlrausch equation, written in the following form:

$$\varkappa = \frac{F^2}{RT}\sum_{i=1}^{n} z_i^2 D_i C_i, \tag{4}$$

where: $\varkappa$ – is the specific electrical conductivity of the solution, Cm/m; F – is the Faraday number Kl/mol; R – is the universal gas constant, J·mol⁻¹·K⁻¹; z_i, D_i и C_i – are the charge, diffusion coefficient and concentration of the i-th component.

Four types of charged particles $C_4H_5O_4^-$, $C_4H_4O_4^{2-}$, OH^-, H^+ and Na^+ can be present in our system (since NaOH is used to correct the pH of the initial solution), between which the condition of electroneutrality is observed:

$$[Na^+]+[H^+]=[C_4H_5O_4^-]+2[C_4H_4O_4^{2-}]+[OH^-]. \tag{5}$$

Solving a system of equations (1), (2), (4), and (5), at known values of $\varkappa$, pH, it is possible to find the concentration of succinic acid in the chamber (3) (both total and its forms separately).In calculations, the diffusion coefficient of the single-charge succinate anion was taken to be

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$D(C_4H_5O_4) = 9.68 \cdot 10^{-10}$ m²/s, and the double-charge $D(C_4H_4O_4^{2-}) = 7.58 \cdot 10^{-10}$ m²/c [34]. The total concentration of all forms of succinic acid found in this way was used to calculate the coefficient of diffusion permeability according to formula 3.

2.5. Investigation of the volt-ampere characteristics of anion exchange membranes and electromass transfer in solutions of sodium succinate

The voltage characteristics (VAC) of anion exchange membranes were studied on the cell shown in Figure 3. The cell consists of electrode chambers (EC), buffer chambers (BC), desalination chambers (DC) and concentration chambers (CC), between which the membrane under study (*A*) is located.

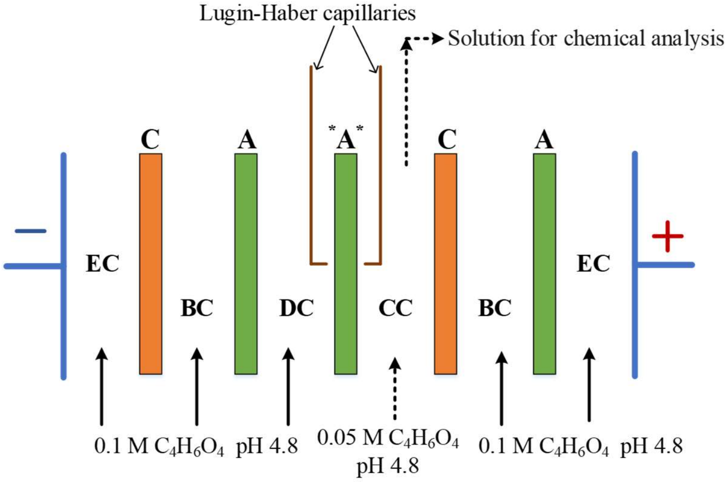

Figure 3. The scheme of the cell for the study of CVC anion exchange membranes and electromass transfer: *A* – the membrane under study; C and A – auxiliary cation and anion exchange membranes; EC - electrode chambers; DC – desalination chamber; CC – concentration chamber; BC – buffer chamber

A solution with a total content of 0.1 mol/l and pH=4.8 of all forms of succinic acid was supplied to the desalination chamber and buffer chambers, with a circulation rate of 5 ml/min. The composition of the solution and the pH were kept constant. 0.05 M succinic acid solution with pH =4.8 was fed into the concentration chamber at a rate of 1 ml/min. This concentration and rate are chosen to accumulate in one pass through the concentration chamber the amount of succinic acid required for determination by titration. A 0.1 M succinic acid solution with pH=4.8 circulated in the electrode chambers at a rate of 90 ml/min. A high flow rate is necessary for the effective removal of gases from the electrode chamber formed as a result of electrode reactions.

A constant current density was provided using a laboratory direct current source B-5-50 (RIAP, Moscow, Russia) The potential drop on the membrane under study (AHT or MA-1) was recorded using Luggin-Haber capillaries connected to the membrane and silver chloride electrodes ESr-10101, as well as the liquid analyzer Expert 001 of ECONIX-EXPERT LLC, Russia. Heterogeneous ion exchange membranes Ralex CMH Pes and anion exchange membranes Ralex AMH Pes were used as auxiliary ion exchange membranes.

The total CVC of the anion exchange membrane was recorded in a quasi-stationary state, which was then processed according to the method described in [37]. The essence of the method consists in subtracting from the total CVC membrane, the effective resistance consisting of the ohmic resistance of the membrane, the membrane /solution boundaries, as well as the resistance of the solution between the measuring capillaries and the membrane under study. The effective resistance is found experimentally and numerically equal to the tangent of the angle of inclination of the initial "ohmic" section of the dependence of the applied current (I) on the voltage drop across the membrane ($\Delta\varphi$).

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Such a current-voltage characteristic is called "reduced" and makes it possible to more clearly distinguish the density of the limiting current of the ion exchange membrane.

Also, during the experiments, samples were taken at the outlet of the desalination chamber to determine the concentration of succinic acid. The concentration of succinic acid was determined by titration with a solution of 0.05 M NaOH. The essence of the method is that the pH of the analyzed sample was adjusted to 3.6 using 0.1 M nitric acid solution and then titrated with 0.05 M sodium hydroxide solution to pH = 9.2 [38]. The concentration of all forms of succinic acid, in terms of molecular form, in the sample is determined by the formula:

$$C(C_4H_6O_4) = \frac{C(NaOH)V(NaOH)}{2V_{al}}, \tag{6}$$

where: $C(C_4H_6O_4)$ – is the concentration of succinic acid, mol/l; V_{al} – is the volume of the aliquot, ml; $C(NaOH)$ – is the concentration of sodium hydroxide, mol/l; $V(NaOH)$ is the volume of the titrant used for titration, ml.

According to the data obtained, the total flow of succinic acid through the anion exchange membrane under study was calculated:

$$J = \frac{\Delta C(C_4H_6O_4)t}{SV} \tag{7}$$

where: $\Delta C(C_4H_6O_4)$ – is the change in the concentration of succinic acid in the desalination chamber, mol/l; V is the sample volume, ml; S=4 is the working area of the membranes, cm^2; t is the time for which the sample volume V is taken, min.

3. Results and discussion

3.1. The results of the study of the physico-mechanical and transport-structural characteristics of anion exchange membranes

Table 1 presents the results of determining the exchange capacity, moisture content and swelling of anion exchange membranes in solutions of sodium chloride and succinic acid. Data analysis shows that homogeneous AHT and MA-1 membranes have the lowest exchange capacity compared to heterogeneous membranes MA-41 and Ralex AMH-Pes. Such differences are related to the specific composition and chemical structure of the polymers that make up the membranes.

The moisture content of the anion exchange membranes Ralex AMH Pes and MA-41 in the form of succinic acid is 2 times less than in the form of Cl$^-$, and for the membrane AHT and MA-1 by 3.6 and 1.2 times, respectively. Firstly, this may be due to the fact that the chloride anion is more hydrated and has a larger effective radius compared to succinate anions. Secondly, larger succinate anions can displace water from the ion channels of membranes.

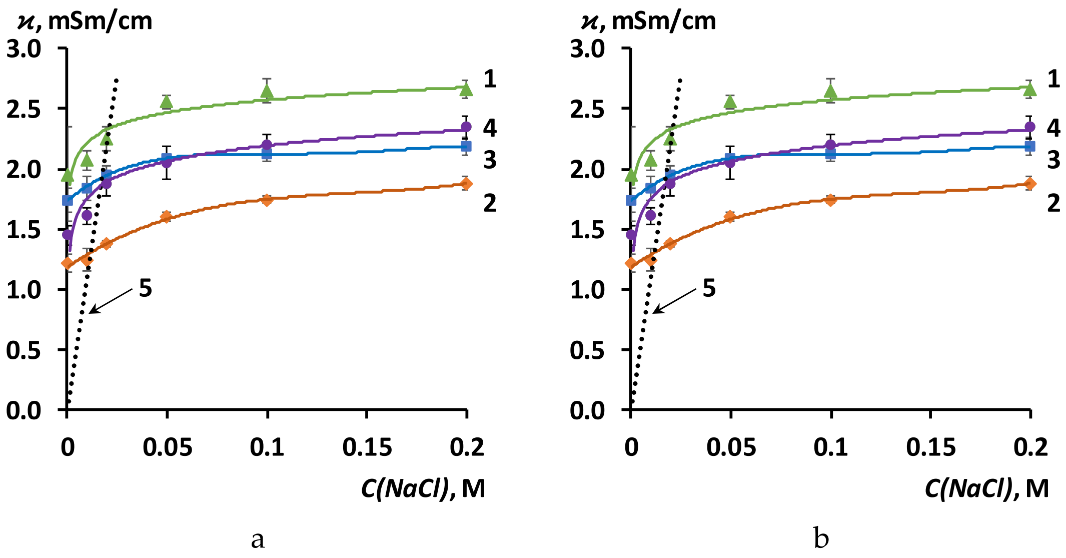
The change in the linear dimensions of anion-exchange membranes in a solution of sodium chloride is also greater than in a solution of succinic acid. At the same time, the length of anion-exchange membranes in both sodium chloride solution and succinic acid solution varies slightly within 5.68%, whereas the change in membrane thickness in both solutions is significant (9.94-23.25%).

Table 1. Exchange capacity, moisture capacity and swelling of anion exchange membranes in solutions of sodium chloride and succinic acid.

Membrane		Ralex AMH Pes	MA-41	AHT	MA-1
Exchange capacity, mmol/g(wet)		1.05±0.06	0.74±0.04	0.49±0.05	0.34±0.01
Density, g/cm^3		1.15±0.02	1.14±0.02	1.03±0.02	1.16±0.02
	NaCl	62.6±0.80	72.3±0.60	80.5±0.30	53.1±0.90

Moisture capacity, %	$C_4H_6O_4$		40.0±0.90	32.9±0.20	22.8±0.30	44.3±0.20
Swelling (change in linear dimensions), %	NaCl	thickness	23.3±0.10	10.91±0.09	11.42±0.10	14.3±0.10
		length	3.24±0.08	5.7±0.10	4.05±0.04	12.4±0.10
	$C_4H_6O_4$	thickness	21.9±0.10	9.9±0.10	8.1±0.10	5.8±0.10
		length	2.08±0.04	3.15±0.06	1.9±0.10	6.7±0.10

Figure 4a shows the concentration dependences of the electrical conductivity of anion exchange membranes in sodium chloride solutions. Data on the specific electrical conductivity of anion-exchange membranes were processed within the framework of a microheterogenic model [35], Table 2. According to this model, the ion-exchange membrane is represented as two pseudophases: the gel phase (inert polymer, polymer chains, hydrated fixed groups) and the phase of the electron neutral solution (an equilibrium solution filling the pores of the membrane).



a b

1 – Ralex AMH Pes; 2 – MA-41; 3 – AHT; 4 – MA-1; 5 – data for the electrical conductivity of the solution

Figure 4. Concentration dependences of specific electrical conductivity (a) and integral coefficient of diffusion permeability (b) of anion-exchange membranes in sodium chloride solutions.

Table 2. Exchange capacity, moisture capacity and swelling of anion exchange membranes in solutions of sodium chloride and succinic acid.

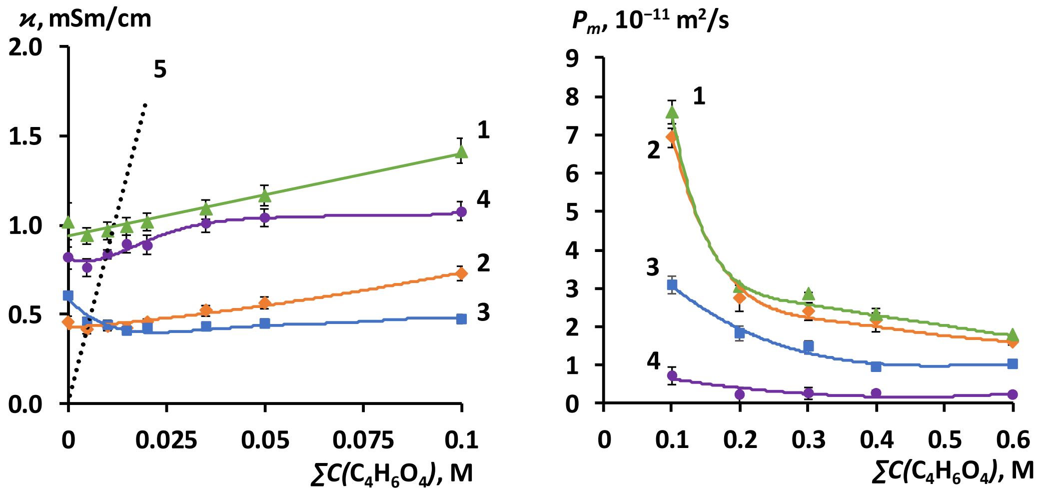
Membrane	$\varkappa_{iso}$, mSm/cm	f_2	α	G, 10^{-15} $m^5 \cdot mol^{-1} \cdot s^{-1}$
Ralex AMH Pes	2.20	0.12	0.39	7.86
MA-41	1.30	0.15	0.28	1.37
AHT	1.90	0.06	0.38	4.55
MA-1	1.70	0.12	0.51	6.86

The obtained values of the parameters f_2 (the proportion of the equilibrium solution in the membrane phase) and α (the mutual arrangement of the conductive and non-conductive phases) for the membranes MA-41 and Ralex AMH Res are in good agreement with the literature data [39,40].

The value of the f_2 parameter for the AHT membrane is 2 times lower than the corresponding parameter for heterogeneous ion-exchange membranes based on a polystyrene matrix and a MA-1 membrane (Table 2). This explains the low diffusion permeability of these membranes (Fig. 4b, curve 3), since the main diffusion transfer of the electrolyte molecule is carried out in pores filled with an electroneutral solution. According to the microheterogenic model, the closer the parameter α (responsible for the mutual arrangement of phases) is to 1, the higher the diffusion permeability of the ion exchange membrane at the same values of f_2 (if α = 1, then the arrangement of phases is parallel, therefore, there are through pores in the membrane). The α parameter of the Ralex AMH Res, MA-41 and AHT membranes is close to 0.3, while for the MA-1 membrane it is 0.51. The parameter *G*, which characterizes the diffusion permeability of the gel phase of the membrane, was also evaluated. For the Ralex AMH Res membrane, the *G* parameter has the highest value among the studied membranes and is 7.86×10^{-15} $m^5\times mol^{-1}\times s^{-1}$. The *G* parameter for the MA-1 membrane is also high and is 6.86×10^{-15} $m^5\times mol^{-1}\times s^{-1}$. Thus, the high proportion of the inter-gel space in the structure, the highest value of the parameter α and the high mobility of co-ions in the gel phase of the MA–1 membrane explains the high values of the integral coefficient of diffusion permeability of this membrane in NaCl solutions (2.3×10^{-11} - 5.2×10^{-11} m^2/s), Fig. 4 b curve 4.

The microheterogenic model makes it possible to describe and predict well the properties of ion-exchange membranes in solutions of strong electrolytes, but at the moment it cannot be applied to describe the characteristics of membranes in solutions of weak electrolytes, since the latter are capable of being in various forms depending on the pH in solution.

Figure 5a shows the results of measuring the specific electrical conductivity of ion-exchange membranes in succinic acid solutions at pH=4.8. Data analysis shows that in the region of very dilute succinic acid solutions (0-0.005 M), an increase in the specific electrical conductivity of heterogeneous Ralex AMH Res and MA-41 membranes (curves 1 and 2) is observed with a decrease in the concentration of the solution. Then the dependence of the electrical conductivity on the concentration increases linearly. The dependence of $\varkappa$ on $C(C_4H_6O_4)$ for AHT membranes at concentrations from 0 to 0.015 M has a decreasing character and with a further increase in acid concentration, the value of electrical conductivity reaches a plateau. The electrical conductivity of the MA-1 membrane in succinic acid solutions increases at concentrations from 0.005 to 0.05 M with further access to the plateau, Fig. 5a, curve 4. As shown in the works, [41,42,43] Having studied the electrical conductivity of ion-exchange membranes in solutions of ampholites and weak electrolytes, the electrical conductivity of ion-exchange membranes in these solutions depends not only on the pH of the external solution, but also on the pH of the solution inside the membrane itself. This difference in the pH of the solution inside the ion exchange membranes differs from the pH of the external solution, due to the Donnan exclusion of hydrogen cations from the phase of the anion exchange membrane or hydroxyl anions from the phase of the cation exchange membrane [42,44,45,46]. Moreover, with dilution of the external solution, there is an increase in the Donnan exclusion of co-ions from the membrane phase and an increase in the pH difference between the internal and external solutions. A change in the pH inside the membrane with a change in the concentration of the external solution leads to a shift in the ionic equilibrium between the forms of succinic acid (equations (1) and (2)) having different mobility, which leads to an atypical dependence of the electrical conductivity of anion-exchange membranes on the concentration, Figure 5a.



a

b

1 – Ralex AMH Pes; 2 – MA-41; 3 – AHT; 4 – MA-1; 5 – data for the electrical conductivity of the solution

Figure 5. Concentration dependences of specific electrical conductivity (a) and integral coefficient of diffusion permeability (b) of anion-exchange membranes in succinic acid solutions at pH=4.8.

When comparing the concentration dependence of the specific electrical conductivity of anion-exchange membranes in a solution of sodium chloride (Fig. 4 a) and succinic acid (Fig. 5 a), it can be seen that in solutions of a strong electrolyte, the value of ϰ decreases in a series: Ralex AMH Pes > AHT ≈ MA-1 > MA-41, whereas in solutions of a weak electrolyte this series looks like this: Ralex AMH Pes > MA-1 > MA-41> AHT. The electrical conductivity of the ion exchange membrane in solutions of strong electrolytes depends on the exchange capacity, parameters f_1, f_2, α. The ratio of these parameters leads to the fact that in a solution of sodium chloride, the electrical conductivity of Ralex AMH Pes membranes has a maximum, MA-41 minimum, and AHT and MA-1 intermediate values, Fig. 4a. In solutions of weak electrolytes, the pH value inside the ion exchange membrane is also added to this set of parameters. The exchange capacity of the AHT and MA-1 membranes is lower than the exchange capacity of the Ralex AMH Pes and MA-41 membranes (Table 1). The higher the exchange capacity of the anion exchange membrane, the stronger the Donnan exclusion of hydrogen cations from the membrane phase and, accordingly, the higher the pH value inside the membrane itself. An increase in pH leads to the formation of a double-charged succinic acid anion with greater mobility. This effect, as well as the ratio of the structural parameters of anion-exchange membranes balanced with succinic acid solution, can explain the course of the dependence of electrical conductivity on the concentration of the external solution.

The study of the diffusion permeability of ion-exchange membranes in succinic acid solutions at pH=4.8 showed the presence of properties of the studied ion-exchange membranes in these solutions, compared with the properties in strong electrolytes Fig. 4 b. The concentration dependence of the integral coefficient of diffusion permeability of anion-exchange membranes in succinic acid solution has a decreasing character. This is also explained by the pH shift inside the membrane to a more alkaline region when the external solution is diluted, while the equilibrium inside the membrane shifts towards the formation of a two-charge form.

With an increase in the proportion of double-charged ions in the membrane phase, an increase in electrostatic forces capable of attracting ions of the opposite charge sign occurs, thus, an increase in the concentration of co-ions in the membrane phase occurs. The scheme of transport of succinic acid forms in the pore of the anion-exchange membrane is shown in Figure 6.

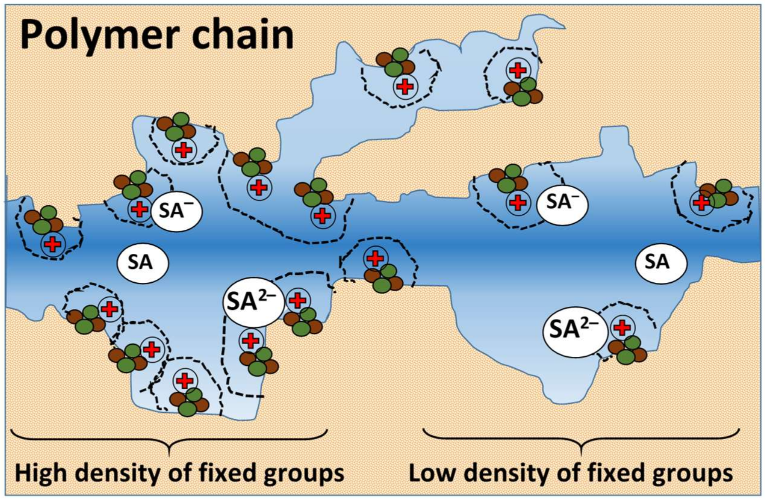


Figure 6. Scheme of the structure of pores of a homogeneous anion exchange membrane in a solution of succinic acid at pH=4.8

This leads to an increase in the rate of diffusion transfer of the succinic acid molecule with a decrease in its concentration in the working solution. Also, as shown in [47] succinic acid is prone to dimerization in aqueous solutions. Moreover, with increasing concentration, the proportion of dimers in the external solution and in the solution enclosed inside the membrane increases. This effect, along with a decrease in the proportion of the double-charged form of acid, can also lead to a decrease in the transfer of succinic acid through the ion-exchange membrane with an increase in its concentration. The authors of the work established similar effects of reducing the transfer of weak electrolyte through the ion exchange membrane with an increase in its concentration in the studied solution due to the formation of associates using the example of phenylalanine [48].

The values of the diffusion permeability coefficients of the Ralex AMH Res and MA-41 anion exchange membranes have similar values in a weak electrolyte solution from $7.5 \cdot 10^{-11}$ to $1.8 \cdot 10^{-11}$ m^2/s, depending on the concentration of the solution under study. Currently, there are no model representations that would allow us to describe the structure of ion-exchange membranes in solutions of weak electrolytes. The close value of the P_m parameter for Ralex AMH Pes and MA-41 membranes can be explained by several factors. Firstly, by such a ratio of the values of the structural parameters of these membranes in succinic acid solution, which ensure the same permeability (the mutual location of the sites of conductive and non-conductive phases, the presence of through pores, the proportion of the equilibrium solution inside the membranes). In addition, the exchange capacity of the MA-41 membrane is 30% lower than that of the Ralex AMH Pes membranes, therefore, the pH and ionic composition inside these membranes will differ. Also, a different value of the exchange capacity will lead to different motility of the co-ions in the gel phase. Thus, the close value of the integral coefficients of diffusion permeability of Ralex AMH Pes and MA-41 membranes can be explained by a combination of various factors. For homogeneous anion-exchange membranes, the values of the integral coefficient of diffusion permeability in succinic acid solutions are lower compared to the heterogeneous membranes studied in this work (2.5-1.7 times for the AHT membrane and 10.5-7.8 times for the MA-41 membrane). The low diffusion permeability makes the use of these membranes promising in the processes of electrodialysis processing of solutions containing succinic acid and other organic acids.

3.2. The results of the study of the volt-ampere characteristics of anion exchange membranes and electromass transfer in solutions of sodium succinate

Figure 7 shows CVCs of AHT and MA-1 anion-exchange membranes in succinic acid solution.

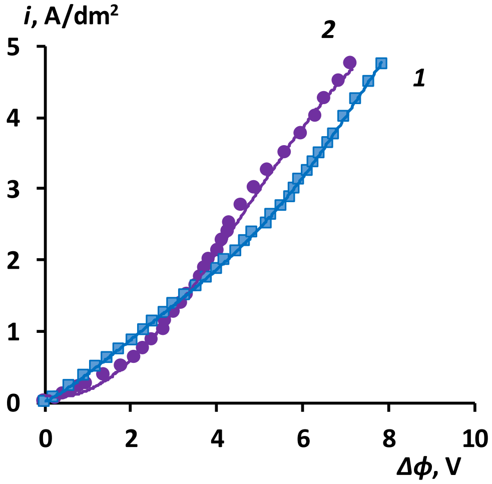


Figure 7. The CVCs of membranes in 0.1 M succinic acid solution at pH=4.8: 1 – AHT, 2 – MA-1

The form of the current-voltage curves obtained in the solution of a weak electrolyte salt differs from the usual form known for the solution of a strong electrolyte. On the classical current-voltage curve of an ion-exchange membrane, as a rule, three sections corresponding to different dominant ion transport mechanisms are easily distinguished. The first section of the CVC is traditionally called "ohmic". It corresponds to the underlimiting current mode of ion transfer, when ions are transferred from the solution volume to the membrane surface by an electrodiffusion mechanism. With the development of concentration polarization at the surface of the ion exchange membrane, from the depleted layer, the concentration of counterions tends to zero. The current density that corresponds to the onset of this state is called the maximum. In this case, the limiting stage of ion mass transfer through the membrane is the transfer of ions by a diffusion mechanism from the volume of the solution to the surface of the membrane. Unlike electrode systems, in electromembrane systems, it is possible to increase the current density above the limiting value due to the development of the so-called conjugate effects of concentration polarization [49]. The increase in current above the limit in this area can be explained by the manifestation of electroconvection or catalytic water-splitting.

In the case of the results obtained in the solution of succinic acid salt, we see a nonlinear behaviour of the system starting from the smallest currents. Increasing of the potential drop leads to current increase in the whole investigated range. The conditional transition to the overlimiting state can be determined by the change in the slope of the curve, but this method is not precise enough.

Such behaviour of the studied homogeneous membranes can be explained by a large number of conjugated processes at the membrane/solution interface, as well as in the membrane volume itself. Such mechanisms include a possible change in the form of succinate ion from a single-charged to a double-charged one when it enters the membrane, causing a conjugate flux of hydrogen ions directed towards the solution [50]; such transformations cause a rearrangement of concentration profiles inside the membrane and, as a consequence, a change in its electrical conductivity.

When the potential drop reaches 3 V or more, an increase in the current growth rate is observed for the MA-1 membrane. Analysis of the dependence of the total flux of succinic acid particles also shows an increase in the flux compared to the AHT membrane (Figure 8).

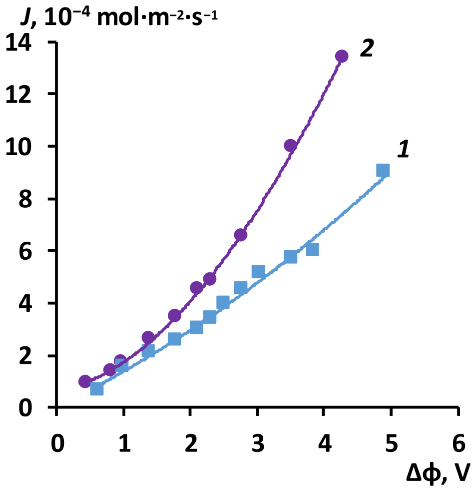


Figure 8. Dependence of the total flow of all forms of succinic acid on the density of the potential jump for membranes: 1 – AHT and 2 – MA-1

It is known from previous studies of the electrochemical properties of the MA-1 membrane in solutions of strong electrolytes that this membrane is characterised by a rather short plateau of the limiting current and a rapid transition to the overlimiting state with development of a non-equilibrium electroconvection [51,52]. This allow to facilitate the delivery of a substance from the depth of the solution to the membrane surface. For the AHT membrane, this mechanism is less pronounced, and as a result, the dependence of succinate ion flux on the potential jump is almost linear in the whole range of current densities studied.

The analysis of Fig. 7 and 8 shows that at the same current density, the mass transfer of succinic acid using the MA-1 membrane proceeds more intensively than when using the AHT membrane. The low diffusion permeability and high electromass transfer of succinic acid make the MA-1 membrane promising for use in the processes of electromembrane production of succinic acid from fermentation mixtures.

4. Conclusions

In the course of the work, the main physical-mechanical and transport-structural characteristics of the anion-exchange heterogeneous membranes Ralex AMH Pes and MA-41, as well as homogeneous anion-exchange membranes AHT and MA-1 in solutions of sodium chloride and succinic acid were determined. Analysis of the data on electrical conductivity and diffusion permeability in solutions of sodium chloride within the framework of the microheterogeneous model showed that the proportion of interspiral spaces in the membranes Ralex AMH Pes and MA-1 is 0.12, in the membrane MA-41 - 0.15. While the proportion of the intergel in the AHT membrane is two times less and is 0.06, which affects its low diffusion permeability. The parameter α, reflecting the relative position of the conducting and non-conducting phases, is in the range of 0.28-0.51 for all membranes, which indicates a chaotic arrangement of the gel and interspiral phases. It is shown that the properties of anion-exchange membranes in succinic acid solutions differ from the properties in solutions of strong electrolytes, which is due to the ability of weak electrolytes to change their ionic form depending on pH, including inside the ion-exchange membrane.

The study of the current-voltage characteristics and electromass transfer in electromembrane systems with AHT and MA-1 anion-exchange membranes showed that succinic acid can be intensively transferred through anion-exchange membranes in an extreme mode. This effect can be explained by the development of electroconvection at the membrane/diffusion layer interface. Due to the occurrence of electroconvection, the dependence of the succinate ion flux on the potential jump is almost linear in the entire studied range of current densities.

The data analysis shows that the MA-1 membrane is the most promising for its use in the process of extracting succinic acid from the fermentation mixture, since it has low diffusion permeability, sufficiently high electrical conductivity and high electrotransport characteristics in succinic acid solutions.

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