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ИССЛЕДОВАНИЕ КИНЕТИЧЕСКИХ И СТРУКТУРНЫХ ХАРАКТЕРИСТИК МЕМБРАН В ПРОЦЕССЕ ЭЛЕКТРОДИАЛИЗНОЙ ОЧИСТКИ МЕДЬСОДЕРЖАЩИХ РАСТВОРОВ

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В данной работе приведены проанализированные результаты исследования структурных и кинетических характеристик ионообменных мембран МА-40, МК-40, PC Acid 60 и СМ(Н) (RALEX®) при разделении и концентрировании компонентов медьсодержащих растворов в процессе электродиализа. В частности, определены размеры аморфных и кристаллических областей структуры материала мембран, рентгеновская степень кристалличности, рассчитаны коэффициенты задержания мембранами катионов Cu^{2+}, и анионов NO_3^- и SO_4^{2-}. Для мембран МА-40 и МК-40 обнаружено, что в процессе насыщения водой структура материала мембран изменяется, что в свою очередь может влиять на кинетику массопереноса через мембрану, но при этом наблюдается малая чувствительность к механической и термической нагрузкам при циклических условиях эксплуатации. Это подтверждается совпадением углов дифракции, при которых наблюдаются пики сухих и водонасыщенных образцов мембран, и незначительным изменением рентгеновской степени кристалличности материала мембран в пределах 5-7% в меньшую сторону для водонасыщенных образцов. Данные, которые были получены в ходе выполнения работы, свидетельствуют об относительно хорошей очистке электродиализом медьсодержащих растворов от посторонних ионов (NO_3^- и SO_4^{2-}) при высоких показателях концентрирования катионов Cu^{2+}. В первой секции электродиализного аппарата удерживаются чуть меньше 90% катионов Cu^{2+}, при этом анионов NO_3^- и SO_4^{2-} в ней остается значительно меньше – около 16,7% и 25,7% соответственно. При этом стоит отметить, что с целью максимального повышения эффективности концентрирования требуется дальнейшие исследования оптимальных параметров электродиализа.

Ключевые слова: электродиализ, ионообменные мембраны, медьсодержащие растворы, структурные характеристики, кинетические характеристики

STUDY OF KINETIC AND STRUCTURAL CHARACTERISTICS OF MEMBRANES IN PURIFICATION PROCESS OF COPPER-CONTAINING SOLUTIONS BY ELECTRODIALYSIS

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This paper presents the analyzed results of the study of structural and kinetic characteristics of ion-exchange membranes MA-40, MK-40, PC Acid 60 and CM(H) (RALEX®) during separation and concentration of copper-containing solutions in the electrodialysis process. In particular, the sizes of amorphous and crystalline regions of the membrane material structure and the degree of crystallinity were determined, the retention factor of cation Cu^{2+} and anions NO_3^- and SO_4^{2-} were calculated. It was found that for mA-40 and MK-40 membranes the structure of the membrane material changes during water saturation, what in turn can affect the kinetics of mass transfer through the membrane. In addition a low sensitivity of membrane material to mechanical and thermal loads under cyclic operating conditions is detected. This is confirmed by the coincidence of diffraction angles at which peaks of dry and water-saturated samples of membranes are observed, and by a non-significant change in the x-ray degree of crystallinity of the membrane material within 5-7% in the smaller side for water-saturated samples. The data, which were obtained during this work, indicate a relatively good cleaning of copper-containing solutions by electrodialysis from extraneous ions (NO_3^- and SO_4^{2-}) at high values of Cu^{2+} cations concentration. In the initial section, slightly less than 90% of Cu^{2+} cations are retained, while NO_3^- and SO_4^{2-} anions remain significantly less – about 16.7% and 25.7%, respectively. It should be noted that the most optimal parameters of electrodialysis are required for maximizing the efficiency of concentration.

Key words: electrodialysis, ion exchange membranes, copper-containing solutions, structural characteristics, kinetic characteristics

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INTRODUCTION

During a long period electrodialysis was mainly used for desalination and demineralization of water. However, in recent decades there is an interest in the use of electrodialysis in other sectors of industry related to the separation and purification of liquid and gas mixtures: hydrometallurgy, electroplating, food processing, medicine, etc. [1-5].

One of the main directions of electrodialysis processes development as well as devices design improvement and process parameters optimization is increasing in plant capacity. The analysis of the kinetic and structural membrane characteristics allows to study the possibility of process parameters optimizing because the structure and composition of membranes define the basis of processes mechanisms taking place in the system "membrane – environment" [6-9]. In addition, the ratio of crystal and amorphous phases, which is found in this study, is important for the mechanical properties of polymer membranes, which may be associated with such operating characteristics as permeability decreasing rate during experimental studies [10].

The aim of this work is to study the structural and kinetic characteristics of ion-exchange membranes MA-40, MK-40, PC Acid 60 and CM(H) (RALEX) in the separation and concentration of copper-containing solutions during electrodialysis, the study of which will help to determine and use the optimal parameters of the most effective electrodialysis process in the separation of copper-containing solutions.

EXPERIMENTAL PART

Domestic ion-exchange membranes MK-40, MA-40 produced by "Schekinoazot" Ltd., as well as PCAcid 60 and CM(H) (RALEX), which are produced by the German company PCCell GmbH and the Czech company MEGA respectively, were chosen for the research. The choice is justified by the fact that these membranes are widely used in industry and therefore research will be relevant.

Heterogeneous ion-exchange membranes MK-40 and MA-40 are a mechanical composition consisting of particles of milled granulated ionite in size from 5 to 50 μm, which are pressed into the low-pressure polyethylene matrix and reinforced with capron fabric [11, 12]. Membranes PC Acid 60 and CM(H) (RALEX), in turn, are made of polyethylene and dispersed particles of the ionite, the size of which no more than 10-12 μm, by the method of hot rolling [13]. In more detail, the characteristics of the membranes are presented in table 1 [14-16].

One of the stages of the research work was the study of kinetic characteristics of ion-exchange membranes PC Acid 60 and CM(H) (RALEX) in the process of electrodialysis separation of copper-containing solution. The studies were carried out on an electrodialysis plant comprising a membrane cell and four independent lines – one for dilute and electrode washing and two are for the concentrate of the substances from which the initial solution is purified.

Table 1

Characteristics of membranes

Таблица 1. **Характеристики мембран**

Membrane type	MA-40	МК-40	PC Acid 60	CM(H) (RALEX®)
Thickness, mm	0.3-0.5	0.3-0.5	0.16-0.2	< 0.45
Tensile strength, Mpa, not less than	11.9	11.9	0.39-0.49	-
Surface electrical resistivity: Ohms per cm², max	10.0	11.0	2	8
Ionic group	$-N_3-$	SO_3H	-	$R-SO_3$
Ionic form - counter ion	Cl^-	Na^+	Cl^-	Na^+
Inert binder	polyethylene	polyethylene	polyethylene	polyethylene
Reinforcing material	polyamide	polyamide	polyester	polyester

As a model solution was selected an aqueous solution of $CuSO_4$ (0,01 M/l) with addition of 0,012 M/l of disodium salt of ethylenediaminetetraacetic acid $C_{10}H_{14}N_2Na_2O_8 \cdot 2H_2O$ (EDTA-Na_2) to prevent precipitation of metals and for creating complex cations, which had to retain the cations of the metals in the primary section for diluate. Besides, $NaNO_3$ (0,05 M/l) was added to the solution for a more stable electrodialysis process as well as a few drops of HNO_3 for the pH level decreasing. This solution was initially placed into the section for diluate; deionized water was added to the concentrate section and $NaSO_4$ (0,5 M/l) was added to the electrode washing section. The initial volume of liquid in each section was 5 liters.

The separation process was carried out at a voltage of 30 V. During the experiment samples of 50 ml were collected from all sections every 15 min to measure the substance concentration.

After the concentration measuring retention factors R were calculated for each membrane for the relevant ions by the equation [20]:

$$R = \left(1 - \frac{C_f}{C_{in}}\right) \cdot 100\%, \quad (1)$$

where C_f – the concentration of the solute in the final solution, kg/m^3; C_{in} – the concentration of the solute in the initial solution, kg/m^3.

Another stage of the work was the structural characteristics study of the samples of ion-exchange membranes MA-40 and MK-40. These membranes were chosen to study structural characteristics due to their greater availability and due to the similarity with the membranes PCAcid 60 and CM(H) (RALEX), as they have the same inert binder.

Prior to the experimental studies, two samples were prepared for each type of membrane – one dry and one water-saturated. These samples were analyzed using diffractometer Dron-3. Radiographs were recorded in the region of high angles of 2θ (2° to 40°), and the radiation was CuKα (λ = 1,54 Å).

Then the size of amorphous and crystalline regions was determined by the Scherrer equation [17, 18]:

$$L = \lambda \left(\beta cos \frac{2\theta}{2}\right)^{-1}, \quad (2)$$

where λ – the wavelength of x-ray radiation, 2θ – the angle of diffraction, β – the width of the reflex at half-height of the peak.

The calculation of the x-ray degree of crystallinity (DC) was carried out by the method of S.L. Aggarwal and G.P. Tilley [19]:

$$CK = \frac{I_c}{I_c + I_a} \cdot 100\%, \quad (3)$$

where I_c – the integral intensity of the crystalline phase; I_a – the integral intensity of the amorphous phase.

RESULTS AND DISCUSSIONS

Analysis of ion concentrations in samples and calculation of retention factors (table 2 and Fig. 1) revealed that the creation of complex Cu – EDTA-Na_2 cations allows to hold a little less than 90% Cu^{2+} cations in the diluate section, while the amount of anions NO_3^- and SO_4^{2-} are significantly less – about 16.7% and 25.7%, respectively.

This indicates a relatively good purification from extraneous ions and the possibility of electrodialysis application for the simultaneous concentration of valuable Cu^{2+} cations and their purification from extraneous ions, in particular NO_3^- and SO_4^2

The following x-ray diffractograms for membrane dry and water saturated samples were obtained as a result of experiments, which were carried out to study the structural characteristics of chosen membranes (Fig. 2).

Table 2

Contents of the studied components in the section for diluate before and after the experiment

***Таблица 2*. Содержание исследуемых компонентов до и после проведения эксперимента в секции для дилюата**

Time, s	Concentration, mg/l	%
Cu^{2+}		
0	460	100
2700	440	89.91
NO_3^-		
0	3715.24	100
2700	658.2	16.65
SO_4^{2-}		
0	1182.2	100
2700	322.64	25.65

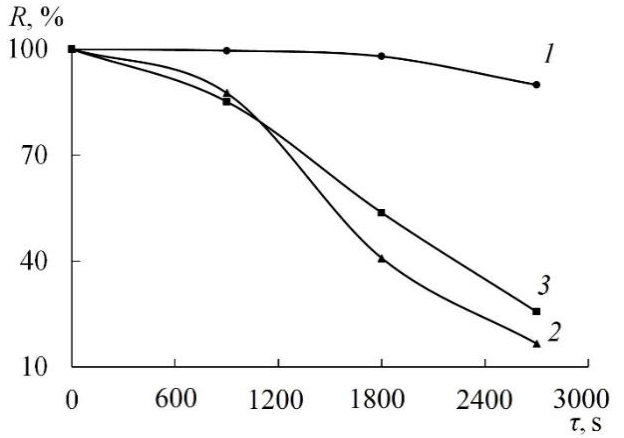

Fig. 1. The dependence of the retention factors of ion-exchange membranes on time: 1 – cations Cu^{2+} by anion exchange membrane PC Acid 60, 2 – anions NO_3^- by cation exchange membrane CM(H) (RALEX®), 3 – anions SO_4^{2-} by cation exchange membrane CM(H) (RALEX®)

Рис. 1. Зависимости коэффициента задержания ионообменных мембран от времени: 1 – катионов Cu^{2+} анионообменной мембраной PCAcid 60, 2 – анионов NO_3^- катионооменной мембраной CM(H) (RALEX®), 3 – анионов SO_4^{2-} катионооменной мембраной CM(H) (RALEX®)

The diffractograms for the dry and water-saturated samples of the MA-40 membrane depict that they have two pronounced maximum at diffraction angles of 21° and 23.5° and one unobtrusive maximum at an angle of 20°. In the dry sample the maximum at an angle of 21° is more intensive, while the peaks of both samples at an angle of 23.5° are the same. The hardly noticeable maximum (20°) is more intensive in the water-saturated sample.

All peaks obtained for the membrane MK-40 samples are the same as for the membrane MA-40, excepting the amorphous peak located at an angle of diffraction 19° instead of 20°. It is worth noting that all the peaks on the diffractogram for the dry sample are more intensive than for the water-saturated one.

The detected coincidence of diffraction angles indicates a low sensitivity of macromolecules to mechanical and thermal workloads under cyclic operating conditions.

Parameters of supramolecular structure of MA-40 and MK-40 membranes are calculated and presented in the table 2.

The data, which are obtained as a result of the DC calculation according to the equation (2), have shown that for the dry sample of the MA-40 membrane DC is 70.66% and for the water-saturated one it is 63.58%. For the MK-40 membrane these parameters are 68.26% and 63.81% respectively. The obtained DC results do not contradict the literature data as DC of polyethylene, which is an inert binder in membranes, varies within 40-80% [21, 22].

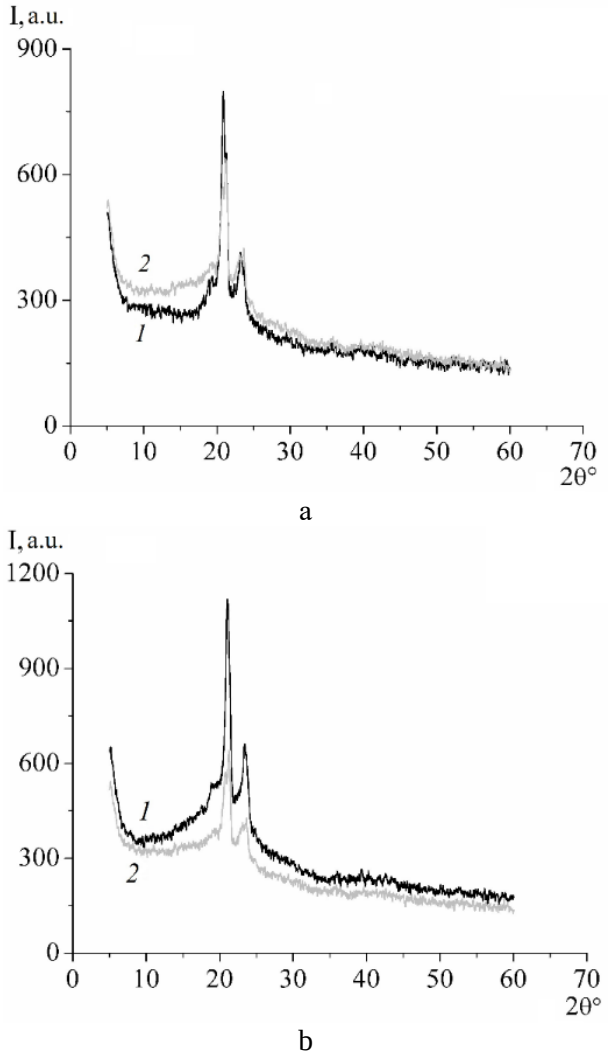

Fig. 2. X-ray diffractograms of samples of ion-exchange membranes MA-40 (a) and MK-40 (б) in geometry for reflection in dry (1) and water-saturated (2) membrane states

Рис. 2. Рентгеновская дифрактограмма образцов ионообменных мембран МА-40 (а) и МК-40 (б) в геометрии на отражение при сухом (1) и водонасыщенном (2) состояниях мембраны

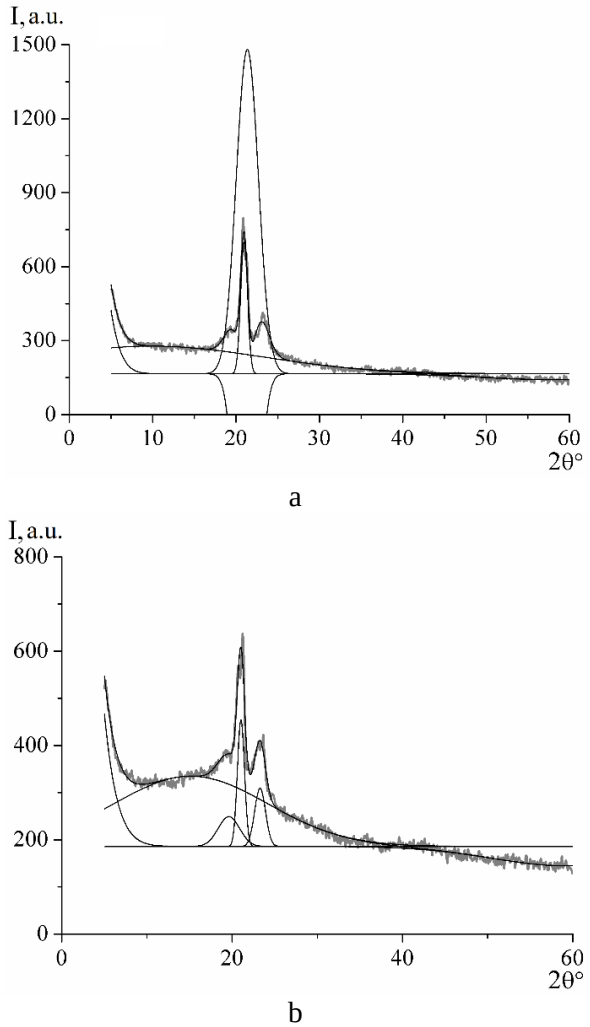

Fig. 3. Results of full-profile analysis of experimental diffractogram for dry (a) and water-saturated (б) samples of MA-40 membrane
Рис. 3. Результаты профильного анализа экспериментальной дифрактограммы для сухого (а) и водонасыщенного (б) образцов мембраны МА-40

Table 3

Structural parameters of MA-40 и MK-40 membranes
Таблица 3. **Структурные характеристики мембран МК-40 и МА-40**

Membrane sample	The position of the maximum 2θ, °	The half-width of peak β, rad	*L*, nm
MA-40			
Crystalline phase			
Dry	21.5	0.44	14.4
Water-saturated		0.46	13.77
Dry	23	0.61	5.22
Water-saturated		0.71	4.49
Amorphous phase			
Dry	20	0.14	13.11
Water-saturated		0.43	4.27
MK-40			
Crystalline phase			
Dry	21.5	0.32	19.8
Water-saturated		0.47	13.48
Dry	23	0.81	3.93
Water-saturated		0.7	4.55
Amorphous phase			
Dry	19	0.14	11.03
Water-saturated		0.17	9.08

It is necessary to note that the DC values of dry and water-saturated membrane samples of each type are smaller for water-saturated ones in the range of 5-7%. This fact confirms the previously mentioned assumption about low sensitivity of membrane material macromolecules to mechanical and thermal loads under cyclic operating conditions.

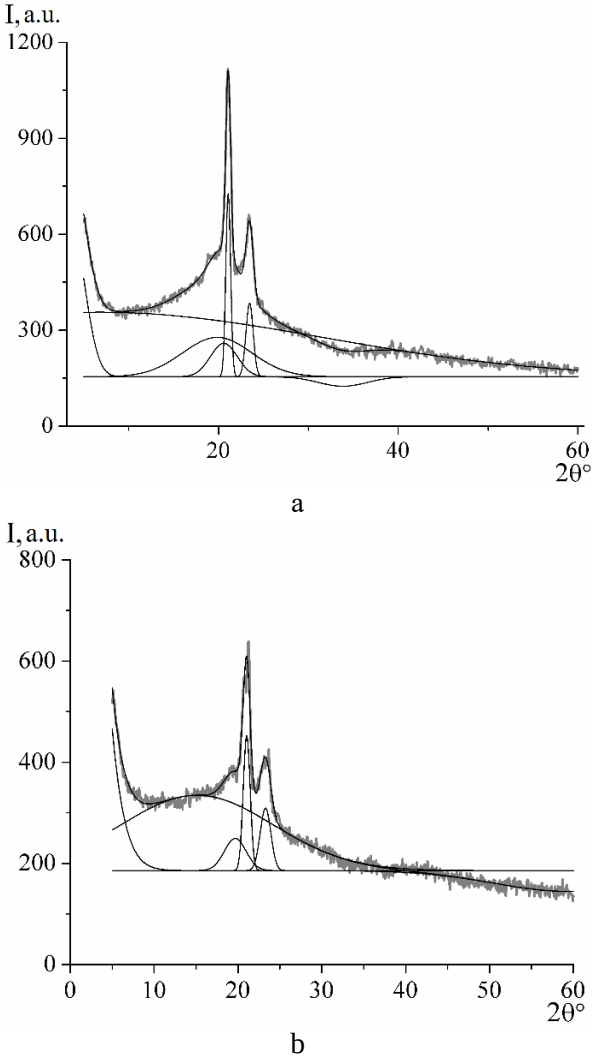

Fig. 4. Results of full-profile analysis of experimental diffractogram for dry (a) and water-saturated (б) samples of MK-40 membrane
Рис. 4. Результаты профильного анализа экспериментальной дифрактограммы для сухого (а) и водонасыщенного (б) образцов мембраны МК-40

The full-profile analysis of the dry and water-saturated samples was performed to quantify the observed changes in the MA-40 and MK-40 polymer membranes, which are caused not only because of the multilayered composition, but also because of the variety of possible conformations of polyethylene macromolecules. To do this the background of incoherent scattering from the primary x-ray diffractograms was previously deducted by traditional method: by drawing a tangent to the points of the x-ray scattering intensity curve at the angles 2θ from 10° to 40°. The profiles of wide-angular scattering and the possible conformations of the polyethylene macromolecules are presented in Fig. 3 and 4. Features of x-ray spectra in the specified range of diffraction angles were simulated by five or six gaussians (see Fig. 3 and 4).

CONCLUSIONS

The obtained data demonstrate a relatively good purification of copper-containing solutions from extraneous ions by electrodialysis.

The structure of ion-exchange membranes MK-40 and MA-40 material varies slightly within 5-7% in the process of water saturation. This fact and the detected coincidence of diffraction angles of membrane dry and water-saturated sample peaks indicate a low sensitivity of the membrane material macromolecules to mechanical and thermal workloads under cyclic operating conditions.

The results of the study can be part of further and deeper studies of the optimal parameters of electrodialysis, which will maximize the efficiency of concentration.

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