

A Study on Kinetic Characteristics of Reverse Osmosis Separation of Wastewater Containing Heavy Metals Ions

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Abstract

The paper considers the problem of wastewater treatment in electroplating industry. Commonly used methods of wastewater treatment were presented and the advantage of membrane method was justified. The main kinetic characteristics of reverse osmosis separation of solutions which include diffusion, hydrodynamic permeability and detention coefficient were considered. The effect of concentration, temperature and pressure on basic kinetic characteristics of reverse osmosis separation of solutions was estimated. The results of an X-ray study of the degree of crystallinity of MGA-95 membrane were described. X-ray diffraction patterns of the polymer membrane samples in dry and swollen forms were obtained. The degrees of crystallinity of the membrane were calculated and analyzed.

Keywords

Electroplating industry; crystallinity degree; kinetic characteristics; membranes; X-ray scattering.

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Introduction

When used in production processes, water is contaminated with various organic and mineral substances, including toxic and poisonous ones, capable of destroying any biological life in natural waters or amenable industrial wastes. One of the sources of environmental pollution by harmful substances is electroplating industry [1].

The main type of electroplating industry wastes are wash waters containing a large amount of heavy metal ions. To reduce the amount of wash water heavy metals up to the maximum permissible concentrations (MPC), a closed water supply system must be used, that is, wash waters, subjected to purification from impurities, must be returned into the production process, while extracted impurities must be buried or recycled. Thus, electroplating wastewater treatment is one of the most urgent problems.

According to production processes and the equipment used, methods of electroplating wastewater treatment can be classified into:

- mechanical / physical (sedimentation, filtration, evaporation);
- chemical (reactant treatment);

- coagulation-flotational (flotation, flocculation, coagulation);
- electrochemical (electroflotation, electrodialysis, electrolysis);
- sorptional (sorption filters, ion exchange filters);
- membrane (ultrafiltration, nanofiltration, reverse osmosis, electrodialysis);
- biological ones[1].

If there are significant amounts of industrial wastewater to be purified at waste treatment plants, it is reasonable to apply the electrochemical and membrane methods of water purification, and the overall wastewater treatment system must combine different technologies: pre-treatment reagent, electroflotation, filtration, sorption, membrane concentration, vacuum evaporation.

Today, membrane water treatment systems are one of the most effective and high-quality methods of wastewater purification. They are more productive and constitute a set of special semi-permeable membranes separating the filtrate from the treated slurry. The liquid portion of the contaminated substance passes through the septum while mechanical particles are trapped by a membrane filter.

In contrast to traditional methods that require large areas, a multi-step processing technology, and

a significant number of operational personnel, membrane technologies have the following advantages:

- 1) compact equipment (for example, one module of ultrafiltration with a diameter of 250 mm and a length of 1700 mm has a capacity of 2.5 up to 7 m³/h);
- 2) ease of capacity expansion due to a modular design of the equipment;
- 3) reliable barrier filtration;
- 4) sufficiently low power consumption;
- 5) the possibility of obtaining high-quality filtration in one step of water treatment (production of drinking water from surface water and groundwater);
- 6) minimal use of chemicals;
- 7) the ability to automate the processes of treatment and monitoring the water quality;
- 8) rapidly advancing technology (the emergence of chemically resistant membranes).

Costs per 1 m³ of water treated by membrane technology, compared with traditional technologies, are steadily reducing. If ten years ago they were several times greater, at present they have significantly become less.

The most important kinetic characteristics that determine the rate of mass transfer in the reverse osmosis processes are the following: diffusion, hydrodynamic permeability and rejection coefficient [2–4]. Modern membrane separation processes of industrial solutions and effluents must meet strict requirements on the separating ability of membranes. Therefore, the study of the kinetic characteristics of the membranes and their testing are of scientific and practical importance for designing and constructing the membrane elements and devices.

The mechanism of separation of aqueous solutions by semi-permeable membranes still remains controversial [5]. At the moment, there are several hypotheses to explain mass transfer of substances through semi-permeable membranes. One of the main hypotheses is that of the role of the influence of crystalline and amorphous regions on the permeability of substances. In work [5] the role of the capillary bound water in the membrane is based on the idea that there exist crystalline and amorphous regions in the membranes, wherein the crystalline regions do not quite obviously affect the transfer of water and solute. Water molecules penetrating into the amorphous regions are linked by hydrogen bonds with functional groups of the polymer. The resulting layer of bound water has an ordered structure and is not able to dissolve. Moreover, this hypothesis does not consider the correlation between the amorphous and crystalline regions of semi-permeable membranes, and crystalline region of membranes is not studied from the standpoint of its effect on mass transfer.

The formation of steady hydrogen bonds with surface pore ions in the amorphous regions and defects

of the membrane crystallites lead to a greater or lesser extent, to the disruption of the supramolecular structure of the polymer membrane. Therefore one of the goals of this work is to study X-ray spectra of the composite semi-permeable MGA-95 membrane.

Experimental

The study of diffusion permeability and sorption capacity of reverse osmosis membranes depends on the type of membrane, solutions, and concentration. To quantify the diffusion permeability, the diffusion permeability coefficient P_d was introduced.

Salts of chromium and copper being the main components of electroplating wastes were used as test solutions. Cellulose acetate membranes MGA-95 and MGA-95P were employed. The study to determine the diffusion membrane permeability was carried out using a setup, the diagram of which is shown in Fig. 1. The setup consists of a three-chambered thermostated cell I-II-III, the chambers of which are separated by membranes 1 and 2, which are fixed by means of grids 15 made of plexiglass, containers for stock solutions 3–5 and waste solutions 6–8, magnetic stirrers 9–11. To maintain the required temperature of solutions, coil heat exchangers 19–21 are integrated in each chamber, in which water circulates from the thermostats 25–27. The temperature in chambers I-II-III is controlled by means of potentiometers 22–24 connected to thermocouples 16–18 having calibration HC. The volume of cell chambers is $0.4 \cdot 10^{-3}$ m³. The working area of the membrane is $2.67 \cdot 10^{-6}$ m². The chambers I-II-III are made of polymethylmethacrylate.

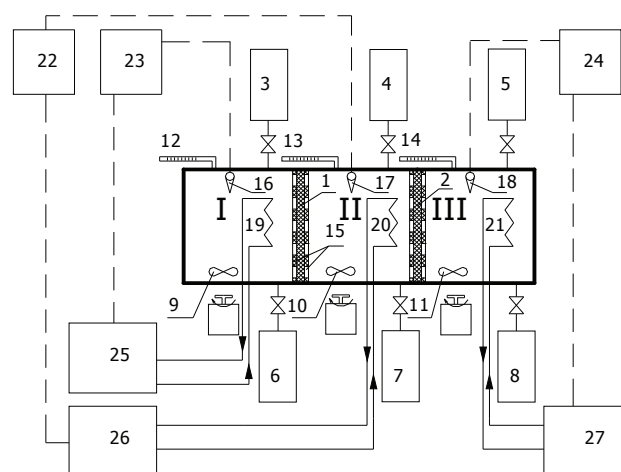


Fig.1. Diagram of the setup to study diffusion flux:

- 1, 2 – membranes; 3–5 – containers for stock solutions;
 6–8 – containers for waste solutions; 9–11 – magnetic stirrers;
 12–14 – measuring capillaries; 15 – grids; 16–18 – thermocouples;
 19–21 – coil heat exchangers; 22–24 – potentiometers;
 25–27 – thermostats

The study to determine the diffusion permeability was carried out according to the following procedure. Pre-prepared membranes were placed in a three-chamber cell. Then the chambers I–III were filled with a solution of the required concentration.

To establish a steady diffusion flow, the solutions were kept in the cells for a long time (10–14 hours), and then were discharged. After the cell chambers (I–III) were rinsed with distilled water for 15 min, they were filled with solution of the required concentration from the containers of stock solutions 3–5. Then magnetic stirrers were switched on for intensive mixing the solutions during the experiment.

As a result of the preliminary calibration experiments the optimal time of the experiment was found to be equal to 5 hours. After the experiment the samples were taken from chambers I–II–III, through the containers of waste solutions 6–8. After determining the concentrations of the solutions before and after the experiment membrane diffusion permeability was calculated.

Diffusion permeability coefficient P_d was calculated using the formula

$$P_d = \frac{C_2 V_2 \delta}{(C_1 - C_2) F_m \tau}, \quad (1)$$

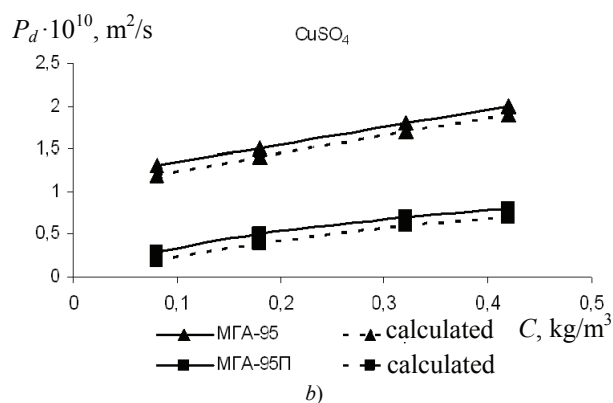
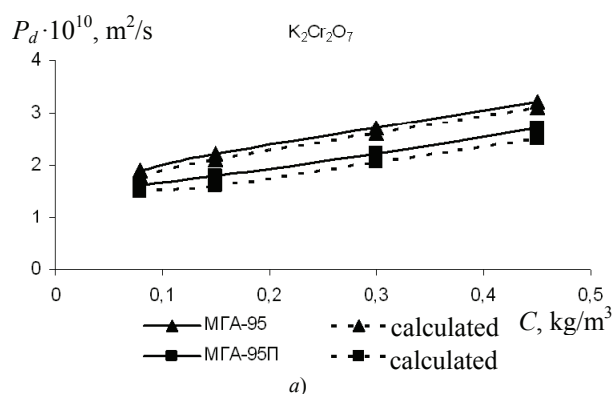


Fig. 2. The dependence of the diffusion permeability coefficient on the solutions concentration :
a – $K_2Cr_2O_7$; b – $CuSO_4$

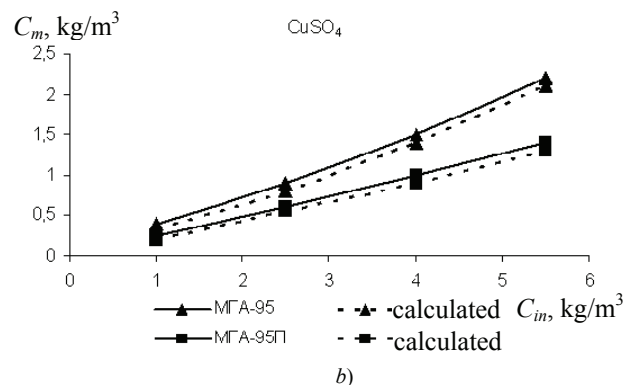
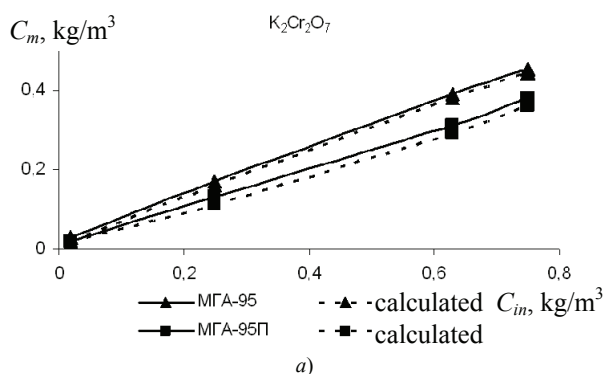


Fig. 3. Membranes sorption isotherms for solutions:
a – $K_2Cr_2O_7$; b – $CuSO_4$

where V_2 – volume of the solution in chamber II; δ – thickness of the swollen membrane; F_m – the working area of the swollen membrane; $C_{1,2}$ – dissolved solid concentration in chambers I and II; τ – the time of the experiment.

Dependences of the diffusion permeability coefficient on the concentration of chromium and copper solutions are shown in Fig. 2.

The analysis of the obtained dependences shows that the diffusion permeability of copper and chromium increases with increasing concentration. This can be explained by the nature of solutes. Fig. 3 presents sorption isotherms of MGA-95, MGA-95P membranes for aqueous solutions of copper and chromium.

As seen from sorption isotherms, sorption capacity of the membrane increases with increasing concentration. On the basis of experimental data on diffusion permeability and the sorption capacity, the experimental diffusion coefficient of copper and chromium salts through membranes was calculated by the following formula:

$$D_d = \frac{P_d C_{init}}{\bar{C}}, \quad (2)$$

wherein \bar{C} – the concentration of substances in the membrane.

Table 1

The values of the diffusion coefficients

Membrane	0Solution	Concentration C_{init} , kg/m ³	Diffusion coefficient D_d , 10^{10} , m ² /s
MGA-95	CuSO ₄	0.08	4.61
		0.16	4.80
		0.36	5.18
		0.42	5.27
	K ₂ Cr ₂ O ₇	0.08	3.81
		0.15	4.70
		0.31	5.88
		0.45	7.18
MGA-95II	CuSO ₄	0.08	2.30
		0.16	2.55
		0.36	2.80
		0.42	2.92
	K ₂ Cr ₂ O ₇	0.08	4.63
		0.15	5.15
		0.31	6.15
		0.45	7.08

Table 1 shows the diffusion coefficient values for test solutions.

According to the data presented in Table 1, with an increase in concentration in the stock solution, the diffusion coefficient increases for the two types of solutions and membranes. The increase in the diffusion coefficient with increasing concentration is characteristic of strongly diluted solutions, which include test solutions [6, 7].

The experimental data were used to calculate approximation dependences of the diffusion coefficient on the concentration of test solutions

$$D_d = b C^n, \quad (3)$$

where b , n – empirical coefficients, the values of which are shown in Table 2.

The study of the hydrodynamic permeability and detention coefficients was carried out on the setup shown in Fig. 4.

Table 2

Approximation dependences of the diffusion coefficient

Membrane	Solution	$b \cdot 10^{10}$	n
MGA-95	CuSO ₄	5.34	0.06
	K ₂ Cr ₂ O ₇	8.44	0.31
MGA-95II	CuSO ₄	3.22	0.13
	K ₂ Cr ₂ O ₇	7.13	0.17

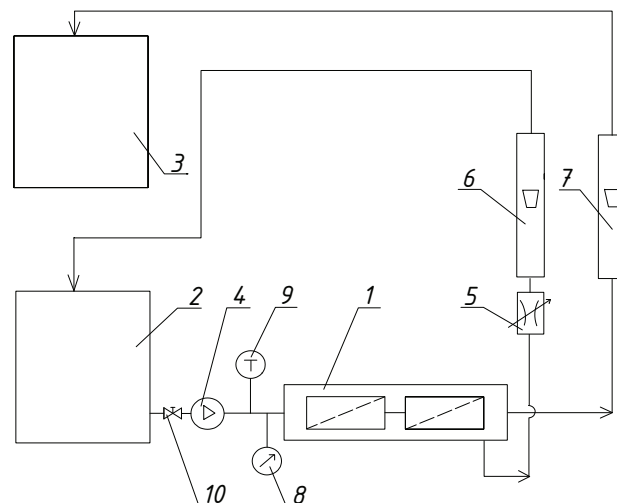


Fig. 4. Diagram of the reverse osmosis setup to study the detention and hydrodynamic permeability coefficients of membranes

The setup consisted of the reverse osmosis module 1, a stock solution container 2, a permeate container 3, a pump 4, a throttle 5, rotameters for permeate 6 and permeate 7, a manometer 8, a thermometer 9, valve 10. The main valve separating element of the setup is a reverse osmosis module 1, which is equipped with two reverse osmosis roll units PSD-E-6.5 / 900A with MGA-95 membranes. Experimental studies on the reverse osmosis setup were carried out as follows. From container 2 the stock solution was pumped into the reverse osmosis module 1 with ND-2.5 type of a plunger pump 4, which supplied the solution in the range from 0 to 2.5 m³/h and set pressure up to 6 MPa. The required operating pressure of the solution in the reverse osmosis module was set up with a throttle 5. The pressure was controlled by the electric contact manometer 8. The consumption of the solution was controlled by the rotameter 6, and the temperature was measured with the thermometer 9. The consumption of permeate after the reverse osmosis module was controlled by the rotameter 7 and collected in the container 3.

Experimental studies were conducted by varying the concentration, the solution flow rate and temperature. To determine the average values, a series of three experiments was conducted.

The value of the hydrodynamic permeability coefficient K was calculated from the dependence [2, 8]

$$K = \frac{V/(F_m \tau)}{P}, \quad (4)$$

where K – coefficient of hydrodynamic permeability of the membrane; V – volume of the collected permeate, m³; τ – the time of the experiment; P – working pressure.

Detention coefficient was determined by the formula

$$k = 1 - \frac{C_{\text{per}}}{C_{\text{init}}}, \quad (5)$$

where k – detention coefficient; C_{per} – the concentration of the solute in the permeate, kg/m^3 ; C_{init} – the concentration of the solute in stock solution, kg/m^3 .

The results of experimental studies of the detention and hydrodynamic permeability coefficients of MGA-95 membrane are shown in Fig. 5–7.

The experiments revealed the following regularities. When the concentration of the solutions for all the substances under study increases, the detention and hydrodynamic permeability coefficients remain practically constant, which is characteristic of reverse osmosis separation of strongly diluted solutions, which include test solutions [3].

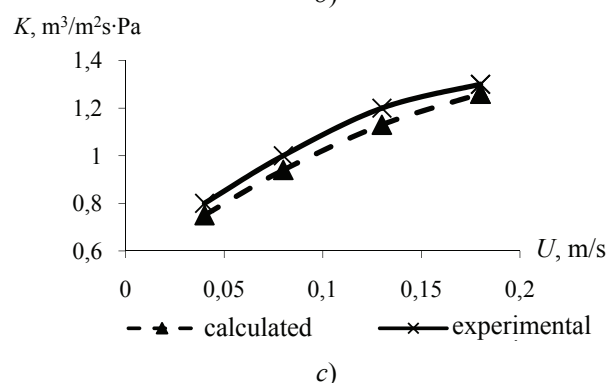
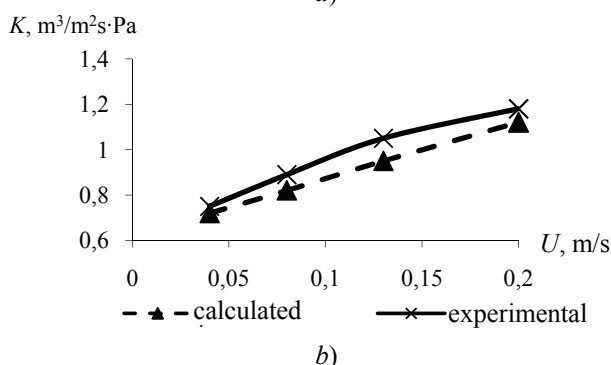
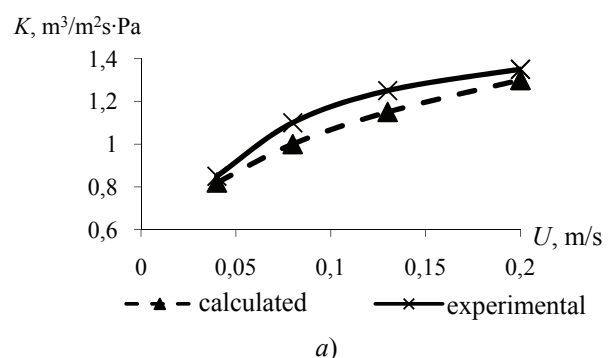


Fig. 5. The dependence of the hydrodynamic permeability coefficient of MGA-95 membrane on the solution flow rate: a – $\text{K}_2\text{Cr}_2\text{O}_7$; b – CuSO_4 ; c – NiCl_2

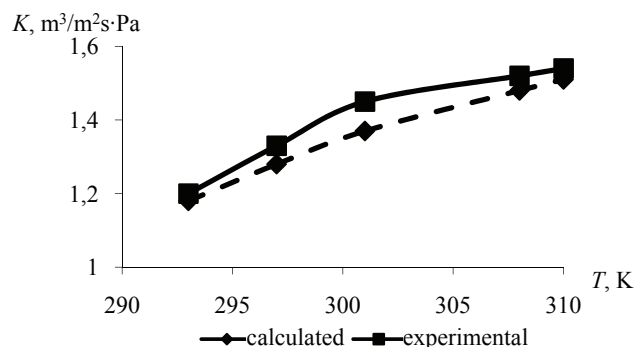


Fig. 6. The dependence of the hydrodynamic permeability coefficient of MGA-95 membrane on the temperature of the solution $\text{K}_2\text{Cr}_2\text{O}_7$

With the increase in the solution flow rate in the intermembrane channel, detention and hydrodynamic permeability coefficients slightly increase. This is due to the fact that with increasing flow rate of the solution over the membrane, the effect of concentration polarization reduces [8–10].

The rise in temperature of separated solutions also causes an increase in the hydrodynamic permeability coefficient of membranes since the viscosity of the solutions decreases, which improves the separation process.

Taking into account the obtained dependences of the hydrodynamic permeability of MGA-95 membrane on pressure, solution flow rate and temperature for calculating the values, the following approximation expression was obtained:

$$K = \frac{G}{k(P - \Delta\pi)\alpha \text{Re}^n (T/T_0)^m}, \quad (6)$$

where k , a , m , n are empirical coefficients; T_0 , T are the reference (we have adopted 293 K) and the operating temperature of a separated solution.

To calculate the detention coefficient, we used a modified formula by B.V. Derjaguin, N.V. Churaev, G.A. Martynov and V.M. Starov [2], which was obtained on the basis of the theory of convective diffusion:

$$k = 1 - \frac{1}{1 + (\gamma - 1) \left[1 - \exp\left(-\frac{vh}{D_m}\right) \right] \exp\left(-\frac{v\delta}{D_0}\right)}, \quad (7)$$

wherein D_0 , D_m – diffusion coefficients in the free volume and in the membrane, respectively, which are dependent on the solute concentration and temperature of the solution; h – membrane thickness; v – filtration rate; δ – thickness of the laminar sub-layer of the solution at the membrane surface; γ – the quantity which takes into account sorption properties of the membrane.

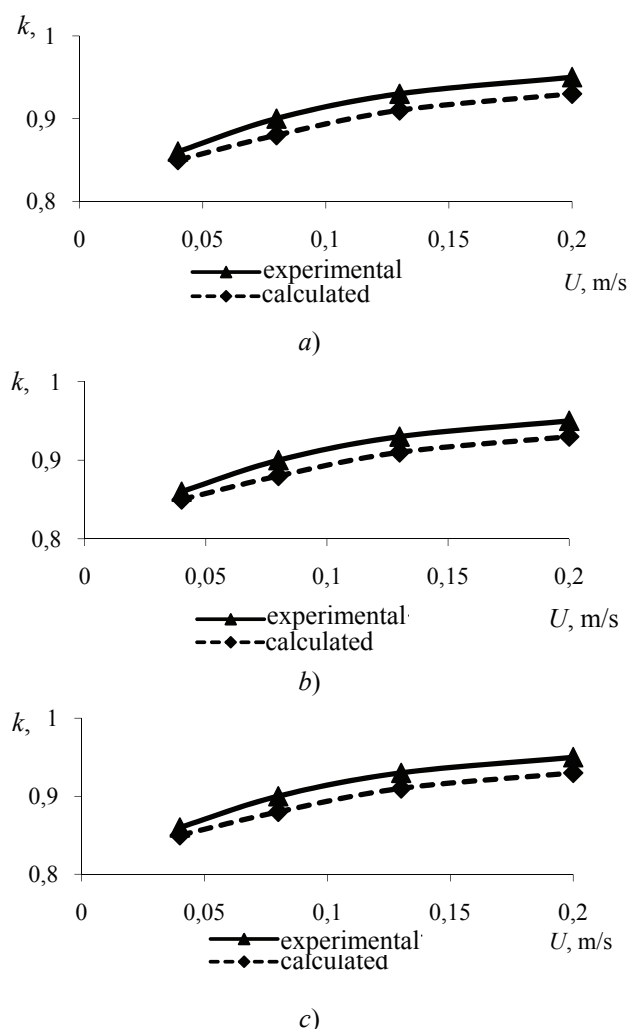


Fig. 7. The dependence of the detention coefficient of MGA-95 membrane on the solution flow rate:
a – $K_2Cr_2O_7$; *b* – $CuSO_4$; *c* – $NiCl_2$

The structure of the MGA-95 composite membrane was studied by X-ray large angles scattering. X-ray-diffractometric measurements were carried out at large angles 2θ between 2° – 40° on a DRON-3 diffractometer 3 shown in Fig. 8, with automatic PC recording in reflection geometry. CuK_α ($\lambda = 1.54 \text{ \AA}$) radiation was used. Monochromatization was ensured by Ni-filter.

The obtained diffraction patters (Fig. 9) have blurred low-intensive peaks with maxima at angles 2θ approximately equal to 8.4° , 16.5° , 22.5° , 25.5° , thus it can be easily seen that there is a redistribution of the intensity and an increase in the scattering diffusion component in the sample membrane saturated with water (Fig. 9, curve *b*). However, the position of maxima of X-ray scattering at angles of 16.5° , 22.5° , 25.5° for the membrane samples in different states remains almost unchanged. At the same time, maximum at an angle of 8.4° does not only considerably expand, but there observed its displacement into the region of



Fig. 8. DRON-3 diffractometer

large angles of 9.6° for the sample membrane saturated with water.

A comparison of our results with experimental data in [11] suggests that the X-ray scattering curves are typical of cellulose acetates formed from solutions. Crystallographic calculations for reflections at angles $2\theta = 16.5^\circ$, 22.5° , 25.5° performed by the Bragg equation

$$d = \lambda (2 \sin \theta)^{-1} \quad (8)$$

give the following values of interplanar distances $d_{16.5} = 0.597 \text{ nm}$; $d_{22.5} = 0.439 \text{ nm}$; which agrees within 10 % error with the parameters of crystal lattice of cellulose α_1 -phase in the diffraction of atoms located in crystallographic planes (100); (010).

The interplanar distance for reflection at an angle 25.5° equals $d_{25.5} = 0.387 \text{ nm}$. This corresponds to half of the long diagonal of the parallelepiped of the unit cell in plane (001) with the sides $a = 0.597 \text{ nm}$, $b = 0.439 \text{ nm}$, and the angle between them $\gamma = 81.3^\circ$, that can be attributed to the diffraction reflection from the atoms of cellulose in plane (110) [12]. Thus, reflections in these diffraction angles should be clearly

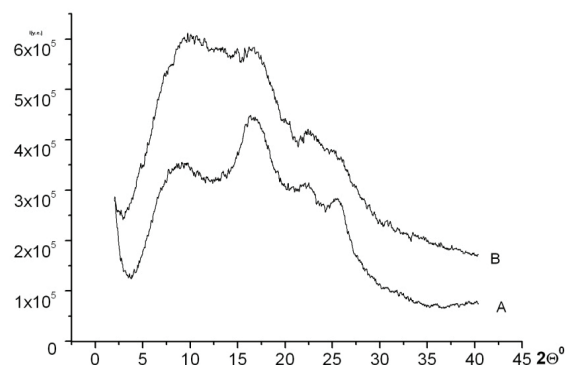


Fig. 9. The X-ray diffraction patterns of the MGA-95 polymer membrane samples obtained in the reflection geometry:
a – dry membrane; *b* – membrane swollen with water

identified as the structural state of the crystalline portion of cellulose acetate. As for the reflection at an angle of 8.4° , it is noted in [12] that the formation of mesophases with increasing concentration of the polymer causes an increase in the intensity of X-ray reflections in the angular range 2θ , about $7\text{--}8^\circ$ and a decrease in the intensity of the reflections in the region of angles $20\text{--}24^\circ$, responsible for the crystallization of the polymer. Therefore, reflection $2\theta = 8.4^\circ$ is referred to the diffraction of atoms of amorphous mesophase of a special type of structural ordering of a supramolecular helix with a specific orientation, i.e. the structure of the substrate of MGA-95 composite membrane of cellulose acetate, is likely to be regarded as a two-phase system of the crystalline and amorphous mesophases.

The calculated intermolecular distances for angles 8.4° and 9.6° were found to be equal 1.17 nm – for dry samples and 1.03 nm for swollen membrane samples.

To understand the physics of structural transformations in the membrane when it is swollen, more detailed measurements and theoretical analysis were carried out using the program Origin 6.0 of diffraction maxima at $2\theta = 8.4^\circ$ and 16.5° (Fig. 10).

It was established that the best approximation of these peaks is provided by bimodal Gaussian function (see. Fig. 10), and the calculated parameters of the supramolecular structure of the membrane samples are presented in Table 3.

On the basis of these calculations we can ascertain that in the process of swelling the supramolecular structure of the membrane material changes owing to deforming both the crystalline and amorphous phases of the sample.

Therefore, X-ray degree of crystallinity was calculated. Since the reflection at an angle of 16.5° is responsible for scattering of the atoms in the crystallographic plane (100) of the crystallite unit cell, and at an angle of 8.4° – for the amorphous component of the structure, X-ray degree of crystallinity was calculated by formula [13]

$$CD = \frac{I_{16.5} - I_x}{I_{16.5}} \cdot 100\%, \quad (9)$$

where $I_{16.5}$ – (peak intensity) is the distance from the baseline to the top of the diffraction peak at an angle $2\theta = 16.5^\circ$; I_x – (minimal intensity) is the distance from the baseline to the dispersion curve minimum between the diffraction peaks at angles $2\theta = 8.4^\circ$, 16.5° (see. Fig. 10) (the angle at which two bimodal Gaussian function curves overlap for a dry membrane $I_x = 13.4^\circ$, for a swollen membrane $I_x = 13.9^\circ$). Crystallite size, i.e. the coherent scattering region (CSR) was determined by the known Scherrer formula [13]

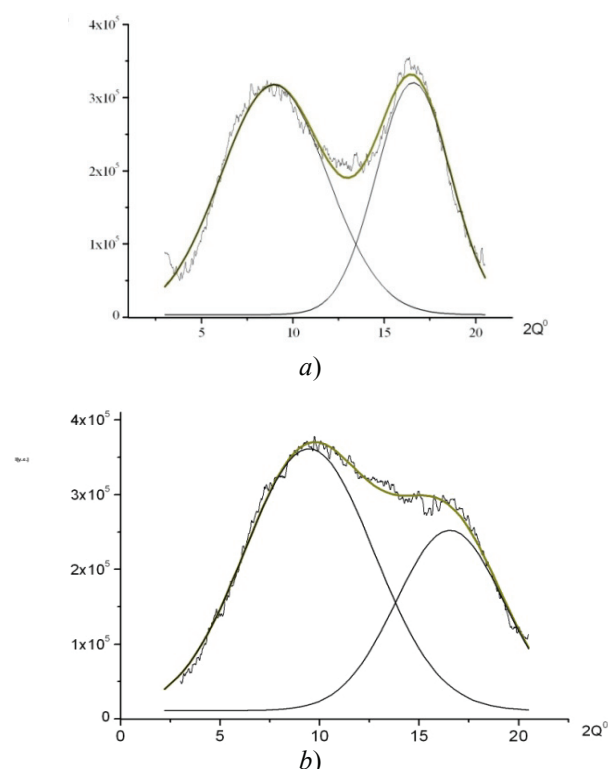


Fig. 10. The diffraction maxima at scattering angles:
a – $2\theta = 8.4^\circ$; b – $2\theta = 16.5^\circ$

(A – dry membrane; B – membrane swollen with water;
thin solid lines – bimodal Gaussian and their superposition)

Table 3

**Parameters of the supramolecular structure of the MGA-95 membrane sample
in the analysis of the diffraction maxima at angles $2\theta = 8.4^\circ$, $2\theta = 16.5^\circ$**

MGA-95	Crystal diffraction maxima				Amorphous diffraction maxima				X-ray degree of crystallinity, %
	peak position 2θ	half-width of the peak β , °	integrated half-width β_i , °	L_{cr} , nm	peak position θ	half-width of the peak β , °	integrated half-width β_i , °	L_{am} , nm	
Dry	16.5°	3.7	4.6	2.4	8.4°	5.4	6.9	1.7	70
Water-saturated	16.5°	5.8	7.3	1.5	9.5°	6.5	8.2	1.4	40

$$L_{cr} = \lambda \left(\beta \cos \frac{2\theta}{2} \right)^{-1}. \quad (10)$$

The values of the crystallinity degree (CD) obtained by this method were found to be equal to 70 % for dry membranes and 40 % – for swollen membranes.

The results of calculating the CD indicate that the proportion of amorphism in the swollen sample membrane has almost doubled. This change in CD can be explained by the adsorption properties of water molecules.

That is, water molecules, having a strong tendency to donor-acceptor interactions with the potential-forming ions of free surfaces of capillary-porous medium have wedging anisotropic effect on intermolecular membrane heterostructure, reducing the crystallites, perpendicular to the axis *c* of the crystal lattice.

As a result of full-profile analysis of X-ray patterns in the region of angles 16.5°; 21.5°; 25.5°, responsible for X-ray scattering maxima from the crystallographic planes (100); (010); (110) of a unit cell, according to the program Origin 6.0, the values of half-widths β of the peaks for dry 3.7°; 2.5°; 3.2° and 5.8°; 3.7°; 5.7° swollen membranes were obtained. According to formula (10) the parameters of the crystallites were determined. So the dimensions of the crystallites vary from 2.4 nm to 1.5 nm in a direction of plane (100), from 3.5 nm to 2.4 nm in plane (010), from 2.8 nm to 1.6 nm in plane (110). These values suggest an anisotropic reduction, i.e. crystallites “fraying”, thereby increasing the amorphous substrate effect on the transmembrane processes. This explanation is consistent with the conclusions of the authors of [14]. Reflection shift at an angle $2\theta = 8.4^\circ$ towards larger angles – 9.6° and increase in the peaks half-width β are due to conformational changes in macromolecular cellulose acetate mesophase, which is confirmed by the intermolecular distance *d* calculated by formula (1), which varies from 1.17 nm in the dry membrane sample to 1.03 nm in a swollen membrane sample.

This microdistortion of supramolecular structure can be explained according to [11] by a straightening (stretching) of macromolecules of cellulose acetate.

Estimation of the degree of crystallinity, crystallite size of membrane samples before and after swelling and the type of X-ray patterns proves that adsorbed water causes a destructive effect on the structural ordering of acetate cellulose MGA-95 membrane basis, thus lengthening the mesophase macromolecules, causing internal stress and deformation of the membrane.

The results of specific effects arising from the interaction between penetrant and polymer membrane matrix should be taken into account in the technological process of wastewater treatment.

Conclusion

1. Basic kinetic characteristics of reverse osmosis separation of solutions, which include diffusion, hydrodynamic permeability and detention coefficient were considered.

2. The effect of the concentration, temperature and pressure on the main kinetic characteristics of reverse osmosis separation of solutions was estimated.

3. X-ray diffraction patterns of the polymer membrane samples in dry and swollen form were obtained. The degree of crystallinity of the membrane was calculated and analyzed.

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Membrane Technology for Solutions Separation of Galvanic Production Process Water

Designated purpose, application area

Rational use and protection of water resources is a problem of great importance. Galvanic production is one of the most dangerous sources of surface and underground water pollution because of large volume of wastewater which contains contaminants of heavy metals, inorganic acids and alkali, surfactants and other toxic compounds. Contact with untreated or inadequately treated sewage and other waste containing non-ferrous metals in water bodies has caused great damage to the national economy and the environment. One of the solutions of this problem is the sewage treatment that achieves the least negative impact on the environment.

Today membrane systems are among the most effective methods of wastewater treatment. They are the most productive and constitute a set of special



Reverse osmosis device

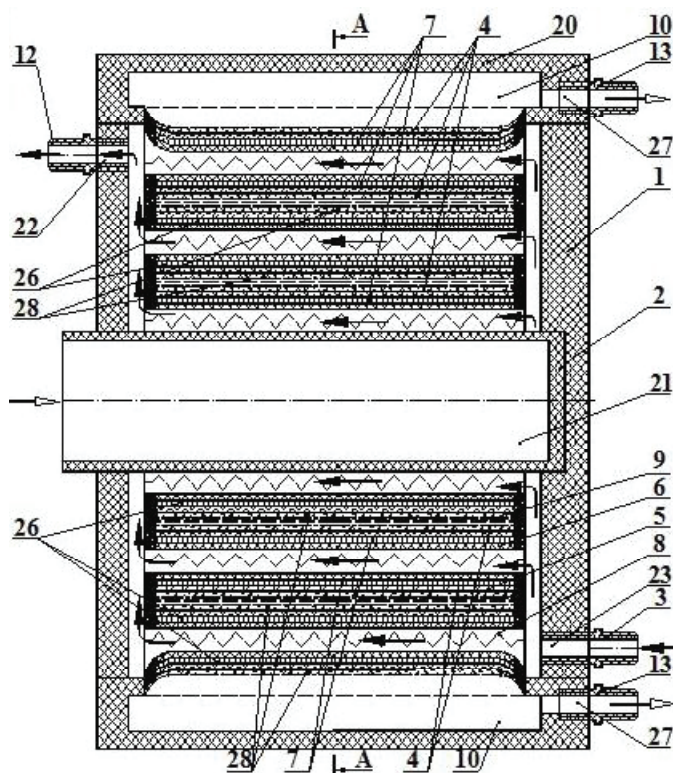
semipermeable membranes separating the filtrate from the cleaning solution. The solvent passes through the diaphragm and the solutes are retained by membrane filter. Compared with traditional methods, membrane technologies require more compact equipment, low power consumption, drinking and industrial water obtaining, minimal use of chemicals and the ability to automate processing and monitoring of water quality.

Specifications

Designed electrical baromembrane device cleans and concentrates industrial solutions, differentially allocates ions of multicomponent systems and obtains highly purified substances.

Patent documentation

Patent № 2326721 "Electrical Baromembrane Roll Type Device". Authors: Lazarev S.I., Abonosimov O.A., Ryabinskiy M.A.



Membrane roll type device

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