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ENVIRONMENTAL PROTECTION: FROM SORBENTS TO MEMBRANES



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ENVIRONMENTAL PROTECTION: FROM SORBENTS TO MEMBRANES

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Колективна монографія «Environmental Protection: from Sorbents to Membranes» - під ред. проф. Н. Кабай, докт. хім. наук Ю. С. Дзязько, проф. М. Арда, докт. хім. наук К.О. Каздобіна - 108 с.

Колективна монографія включає статті за матеріалами українсько-турецького семінару, який було проведено 11 листопада 2016 р. на базі Інституту загальної та неорганічної хімії ім. В. І. Вернадського НАН України за участю фахівців Егейського університету (Турецька республіка) та за підтримки Відділення хімії НАН України, Відділу міжнародних зв'язків НАН України, а також Наукової та Технічної дослідної Ради Турецької республіки TÜBİTAK. У монографії наведено результати досліджень, які направлені на вилучення токсичних і цінних компонентів із розчинів техногенного та природного походження, зокрема з біологічних рідин, із застосуванням сорбційних та мембранних методів. Результати робіт можуть бути застосовані у водопідготовці, хімічній, фармацевтичній, харчовій промисловості. Особливу увагу приділено синтезу нових матеріалів для процесів розділення та впливу структури цих матеріалів на їх функціональні властивості: проникну здатність, електропровідність, каталітичну спроможність тощо.

Монографія є корисною для фахівців в області мембранних та сорбційних технологій, а також для студентів вищих навчальних закладів.

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ION EXCHANGE MEMBRANES AND ACTIVATED CARBON

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Abstract. *Application of standard contact porosimetry to investigations of activated carbon, polymer ion exchange membranes and composites based on them is considered. The method allows us to research porous structure of swollen materials in a wide diapason of pore radii (from 1 nm to 100 μ m), to recognize mechanism of composite formation, to find location of inorganic particles in one or other type of pores of the polymers, to estimate hydrophilic and hydrophobic porosity.*

Keywords: *ion exchange membranes, organic-inorganic composites, activated carbon, standard contact porosimetry, hydrophilic and hydrophobic pores.*

Introduction. Functional properties of ion exchange membranes (IEMs) and activated carbon (AC) are determined, particularly, by their porous structure, which can be investigated with various methods [1-5]. The information content of widespread porosimetry techniques is limited. Regarding polymer IEMs, their structure is labile, it is formed mainly during swelling and strongly depends on liquid or temperature. This makes it impossible the application of mercury porosimetry, BET and microscopy methods. No swelling is characteristic for AC, but these materials contain both hydrophilic and hydrophobic pores, which cannot be recognized with the mentioned methods. These two types of pores affect functional properties of AC. Moreover, the pore size is within a very wide diapason (4-5 orders) for IEMs and AC.

Experimental. Such untraditional method as standard contact porosimetry (MSCP) is the most informative and correct technique for investigations of IEMs and AC [1-5]. MSCP gives a possibility to keep temperature, which is equal to that in fuel cells or electrodialysis cells. Similar working liquid (electrolyte solution or water) can be also used [2, 3]. Thus, porous structure that is realized under the real operating conditions can be determined. The MSCP allows us to obtain pore size distributions in the maximal diapason: from 1 nm to 3×10^5 nm (five orders of magnitude).

Results and discussion. *Homogeneous ion exchange membranes.* These materials can be considered as solid polymer electrolytes containing only one

phase (ion-exchanger). As shown in example of MA-100 anion exchange membrane, very insignificant swelling occurs in octane (Fig. 1, [3]). It means practically no porosity in this medium similarly to air-dry state. In aqueous medium, considerable porosity is formed in the region of pores, a radius of which is less than 10 nm. The most known homogeneous materials are proton conductive perfluorinated membranes of Nafion type containing sulfo groups. Porous structure of these materials is formed only in water or in water vapour due to hydration of functional groups [6]. Water molecules move apart the polymer chains producing pores.

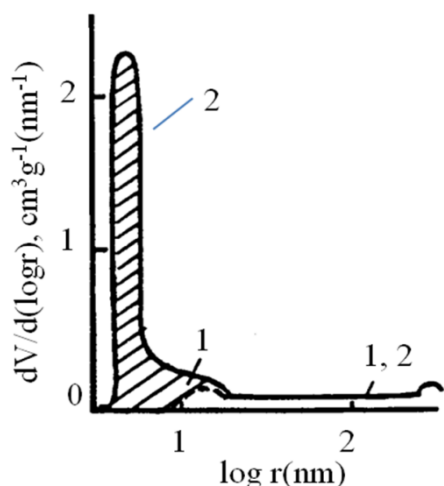


Fig. 1. Differential Pore Size Distributions for Homogeneous MA-100 Membranes (Adapted from [3]). Working Liquids Are Water (1) and Octane (2).

Pore size distributions have been also obtained for Nafion 112, Nafion 115, Nafion 117 as well as for Russian analogue of these membranes (MF-4SK), water was used as a working liquid [6]. Porosity of these IEMs was shown to be provided mainly by micro- and mesopores, a radius (r) of which is less than 10 nm. This structure is so-called as "nanostructure" or "ion exchanger phase". The curves of pore size distribution for all mentioned membranes are close to each other in the nanostructure region. It is assumed that the nanostructure is determined by similar chemical nature of the membranes. Based on the investigations of other IEMs, this conclusion can be related for all ion exchange membranes. The pore size

distributions are rather different in the region of $r=10-500$ nm. These pores are evidently affected by the procedures of membrane preparation, which are different for various type of IEMs.

Any change of the synthesis procedure affects porous structure of IEMs [7]. For instance, increasing in ion exchange capacity of the MF-4SK membrane from 0.71 to 1.02 mg-eq g⁻¹ causes almost doubling porosity value. The cumulative volume of micro- and mesopores in ethanol is higher in two times than that in water. Macropores are practically absent in two cases. The information about the membrane structure is important for methanol-air fuel cell. The swelling degree of the membranes depends also on nature of counter-ions. The MF-4SK membrane shows a decrease of porosity (about 30%) after loading with Na⁺. The volume of pores decreases sufficiently, when inorganic counter-ions are substituted by organic species [3].

Heterogeneous membranes. These IEMs contain not only ion-exchanger but also inert components. For example, Fig. 2 illustrates differential pore size distributions for the MC-40 membranes containing different amount of divinylbenzene (DVB) and polyethylene particles as a binder [8]. The curves are

characterized by two maxima: the first peak is in the region of micro- and mesopores (from <1 nm to 100 nm), the second one is attributed to macropores ($r=300-3000$ nm). Pores of the first type are formed due to swelling of ion exchanger phase, voids of the second type are due to inert polyethylene phase. As seen, a growth of DVB content causes an increase of the second maximum, the first peak becomes smaller.

Organic-inorganic membranes. These membranes are probably the widespread type of composite IEMs. One way to prepare them is to insert inorganic particles into preliminarily formed polymer membranes. Since porous structure of ion exchange polymers are complex, the incorporated particles can be precipitated in one or other types of pores depending on their size. The particle size is determined by Ostwald-Freundlich equation [9]:

$$\ln \frac{C}{C_{\infty}} = \frac{\beta v_m \sigma \cos \varphi}{RT r} \quad (1)$$

Here C is the compound concentration in the solution, C_{∞} is the concentration of saturated solution, β is the shape factor of particles, v_m is the molar volume of the compound, σ is the surface tension of the solvent, φ is the wetting angle, R is the gas constant, T is the temperature, r is the particle radius. The particles, a radius of which is less than the r value, are dissolved and reprecipitated as larger formations. This thermodynamic equation allows

us to control the modification conditions and to form purposefully smaller or larger particles of the modifier (usually it is inorganic ion-exchanger) [10]. The equation was adapted taking into consideration ion exchange properties of the polymer [11].

The composite IEMs were obtained by modification of heterogeneous membranes with zirconium hydrophosphate (MC-40 cation exchange membrane) and hydrated zirconium dioxide (MA-40 anion exchange material). In order to establish the interrelation between structure of the modified polymers and their functional properties, following parameters have been introduced: (i) a ratio of volumes of pores of the first and the second type (α), (ii) a ratio of volumes of micro- and mesopores of the first type (γ) [12]. A change of these parameters, which is caused by the inorganic particles, has been

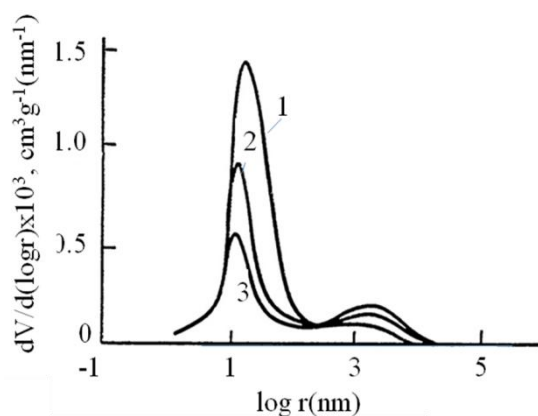


Fig. 2. Differential Pore Size Distributions for Heterogeneous MK-40 Membranes Obtained by Copolymerization of Styrene with DVB (Adapted from [8]). The Content of DVB: 2 % (1), 4 % (2) and 8 % (3).

found to reflect ion movement through the composites. Primary nanoparticles in pores of the first type improve ion transport due to additional counter-ions. Small aggregates (up to 200-300 nm) in pores of the second type are filters against co-ions and uncharged particles (for instance, organics). Large agglomerates can be also formed in pores of the second type, a size of which is several microns. They deteriorate functional properties of IEMs. Thus, the modification procedure has to provide no agglomerate formation.

The modification procedure [12] allows us to precipitate only primary nanoparticles and their small aggregates inside heterogeneous IEMs (Table 1). The primary particles cause a decrease of α and γ parameters indicating location inside pores of the first type. The inorganic cation-exchanger, such as zirconium hydrophosphate, increases the membrane potential in neutral media. Amphoteric hydrated zirconium dioxide deteriorates charge selectivity under these conditions. Thus, at least one side of the composite ion-exchange membrane has to be in a contact with acidic solution. It is necessary to provide anion exchange ability of the modifier. Lower electrical conductivity of the composite IEMs than that of the pristine membranes is due to barrier function of the aggregates in pores of the second type.

Table 1: Characteristics of IEMs (Adapted from [12])

Membrane	α	γ	Potentiometric transport number (0.5 M/1 M NaCl)	Electrical conductivity, mOhm⁻¹ m⁻¹ (0.5 M NaCl)
MK-40, pristine	1.85	1.59	0.92	160
MK-40, composite	1.76	1.50	0.94	30
MA-40, pristine	1.35	0.98	0.96	27
MA-40, composite	1.11	0.91	0.92	22

The membranes were applied to desalination of biological liquids, for instance, milky whey. The advantages of organic-inorganic membranes over polymer IEMs are no concentration polarization, when the current is lower than the limiting value [12], higher desalination degree [13] and current efficiency [12, 13], faster removal of hardness, hydrophosphate and dihydrophosphate ions than cations of alkaline metals and chloride anions [13]. At last, organic-inorganic IEMs are stable against fouling with organics.

Composite polymer membranes. Polymer fibrous composites are relatively new IEMs. Combination of different fibrous fillers and ion exchangers allows us to obtain materials with a wide diapason of properties. Cation exchange materials of Polycon type are obtained with a polycondensation method: strongly acidic ion-exchanger is formed between the fibers as well on outer surface of the fibrous membranes (Fig. 3). Synthesis procedure involves impregnation of the membrane with a solution of monomer followed by

pressing. Exothermic heating of the material occurs during pressing, this affects structure of the membrane being formed.

Hydrophilic and hydrophobic pores of composite membranes, which are determined by pressure during synthesis, were investigated in [14] using octane and water. The method of standard contact porosimetry allows us to determine a wetting angle for water (θ) [1-4]. As shown, porous structure of swollen Polycon-type membranes involves pores due to fibers and ion exchange filler. In general, fibers contain hydrophilic micropores ($\theta \approx 0^\circ$), hydrophilic-hydrophobic mesopores ($\theta < 90^\circ$) and hydrophobic meso- and macropores ($\theta > 90^\circ$) [14]. The ion exchange filler contains only completely hydrophilic micro- and mesopores. The values of specific surface area are different ($70\text{--}480\text{ m}^2\text{ g}^{-1}$) for various Polycon membranes containing an equal amount of the ion-exchanger (90 mass %). However, exchange capacity is similar for all samples ($2.6\text{--}2.8\text{ mg-eq g}^{-1}$). This is explained by different orientation of functional groups relatively to polymer chains. The orientation provides different distributions of water molecules and different nanostructure of the filler.

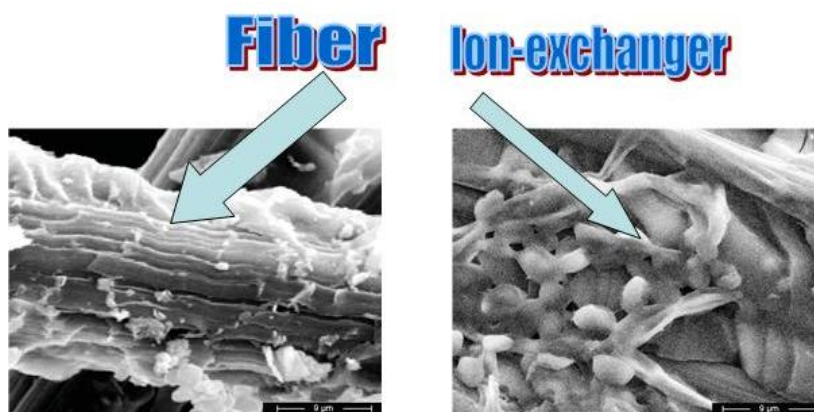


Fig. 3. SEM Images of Polycon Membrane (Adapted from [14]).

Pore corrugation. The method of standard contact porosimetry allow us to recognize pore corrugation: this feature of porous structure of the IEMs affects their electrical conductivity [15]. Homogeneous MA-100 anion exchange membrane and home-made heterogeneous cation exchange membranes were investigated [16]. The last samples were produced by copolymerization of methacrylic acid with DVB in a solution of polyethylene (a binder) in m-xylene.

Fig. 4a illustrates integral energetic adsorption-desorption isotherms measured in water for the MA-100 membrane, integral pore size distributions obtained during evaporation of water or decane and the difference between these curves. A change of the membrane volume, which was measured with a micrometer during water evaporation, is also given. During swelling in water, the pore volume increases in 9 times mainly due to pores of the first type attributed to nanostructure. The volumes of pores of the second type are practically similar, when water or decane are used as working liquids. The main important result is a coincidence of the curves in the region of nanostructure and small hysteresis in the region of macropores.

Similar family of curves are given in Fig. 4b for the home-made membrane. The curves for water adsorption and desorption also coincide in the region of nanostructure, however, the hysteresis is much significant comparing with homogeneous MA-100 membrane. Thus, pores of the first type, which are formed in water, are not corrugated. At the same time, macropores are corrugated. Since corrugation means also pore tortuosity, the highest values of electrical conductivity and diffusion coefficients should be found for homogeneous membranes, in which macropores are practically absent. This conclusion is assumed to be common for all ion exchange membranes. Particularly this assumption explains very high conductivity of Nafion membranes, which are used in low-temperature fuel cells.

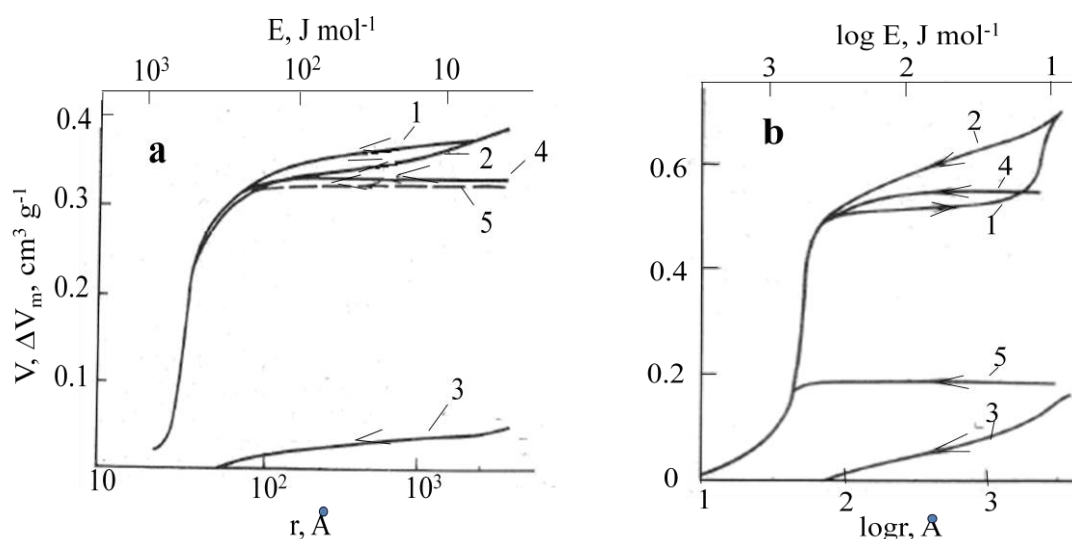


Fig. 4. Integral Energetic Isotherms of Water Adsorption (1) and Desorption (2), Integral Pore Size Distributions Measured in Water (2) or Decane (3) during Evaporation. The Curve (4) Is the Difference of Curves (2) and (3), the Curve (5) Is a Change of the Membrane Volume during Release of Water from Pores. The Data Are Given for the MA-100 Anion Exchange Membrane (a) and for the Membrane Produced by Copolymerization of Methacrylic Acid with DVB (b). Adapted from [16].

Activated carbon. These sorbents are applied to solution of following tasks: prevention of emission of harmful gases into the atmosphere, purification of waste waters, production of pure water using capacitive deionization (CDI) etc. The CDI method requires the most hydrophilic electrodes, this property can be estimated with a method of standard contact porosimetry. Here the results of investigations of porous structure and hydrophilic-hydrophobic properties of following AC are given: CH900 (Curaray Co, Japan), VISKUMAK (Neorganica LTD, RF), SAIT (SAIT Co, South Korea). The first two samples are textiles, the electrode of SAIT type was produced by pressing of powder of activated carbon with a binder (polytetrafluorethylene).

Integral and differential pore size distributions for the carbon materials were obtained using water or octane (Fig. 5a-c). These distributions are plotted vs effective pore radius (r^*) that is determined as [2– 5]:

$$r^* = \frac{r}{\cos \theta} \quad (2)$$

where r is the true value of pore radius. The θ – $\log r$ curves were also plotted (Fig. 5d). Since octane wets all materials almost perfectly, $r^* \approx r$ for this liquid. The difference between the maximal content of octane and water corresponds to hydrophobic pores. All investigated carbon electrodes were found to include both hydrophilic and hydrophobic pore volumes (hydrophilic and hydrophobic specific surface area, Table 2). The wetting angle distributions show hydrophobicity for all electrodes, especially for the SAIT material containing particles of hydrophobic binder. Moreover, complex θ – $\log r$ dependences are due to inhomogeneous distribution of surface groups in pores of different size.

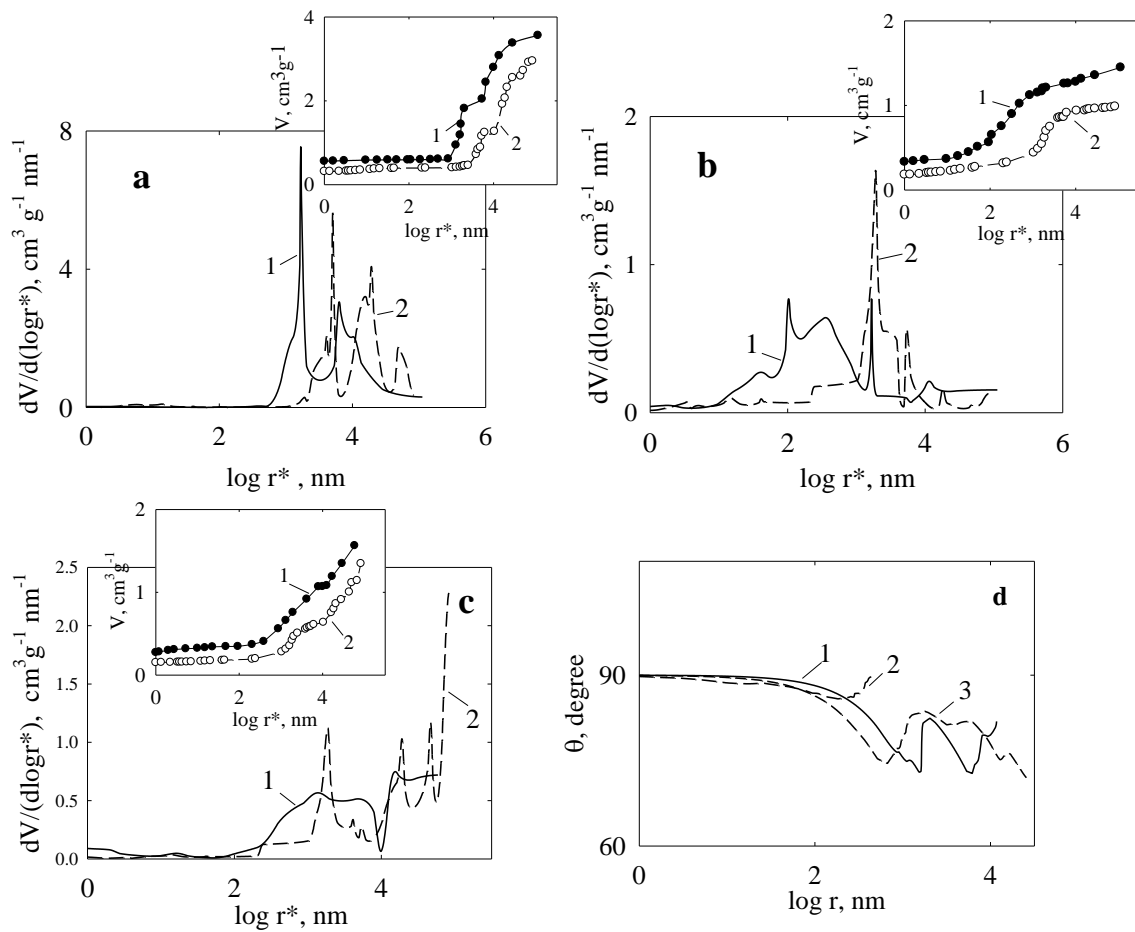


Fig. 5. Differential Pore Size (a-c) and Wetting Angle (d) Distributions Measured in Octane (a-c, 1) and Water (a-c, 2) for the CH900 (a, d1), SAIT (b, d2) and VISKUMAK (c, d3) Electrodes. Insertions: Integral Pore Size Distributions (a-c).

A very wide diapason of pore size is characteristic for the electrodes (from $r < 1$ nm to $100 \mu\text{m}$), pores are both hydrophilic and hydrophobic. The values of specific surface area are rather high, they are sufficiently different from each other. The magnitudes of hydrophilic and hydrophobic porosity are also various.

The data obtained in octane media allow us to make main conclusions. A total diapason of pore size is very wide: the r values are less than 1 nm for the smallest pores and larger than $100 \mu\text{m}$ for the largest voids. Micropores

($r < 1\text{ nm}$) as well as large macropores ($r > 100\text{ }\mu\text{m}$) are attributed for all materials. Pores, a radius of which is 1-100 nm, are practically absent in the CH900 and VISKUMAK textiles. All types of pores have been found for the SAIT electrode.

Table 2. Characteristics of Porous Structure of Carbon Electrodes

Electrode	Specific surface area (m^2g^{-1})		Ratio of hydrophilic and total surface areas	Porosity ($\text{cm}^3\text{cm}^{-3}$)		
	Total	Hydrophilic		Total	Hydrophilic	Hydrophobic
CH900	1520	850	0.56	0.850	0.786	0.064
VISKUMAK	600	416	0.70	0.729	0.623	0.106
SAIT	940	520	0.55	0.715	0.490	0.225

Total porosity is rather high for all electrodes. A comparison of the data, which were obtained in water and octane media, shows higher volume of hydrophilic pores than that for hydrophobic voids. The magnitudes of total specific surface are 600-1520 m^2g^{-1} , particularly the values of 420-850 m^2g^{-1} are due to hydrophilic pores. The last values are very important, since electric double layer, which determines main characteristics of CDI processes, is realized in these pores. The largest contribution of hydrophilic regions into total surface area has been found for the VISKUMAK textile. Regarding the CH900 ACT textile and the SAIT composite, the ratios of hydrophilic and total surface areas are sufficiently lower and close to each other.

Conclusions. The MSCP allows us to investigate porous structure of polymer IEMs in a very wide diapason of pore radius unlike, for instance, a method of water adsorption-desorption isotherms. In opposite to mercury porosimetry, this method can be applied to swollen materials, i.e. they can be researched under real conditions. Inert voids and pores that are responsible for ion exchange are recognized by this manner. The information about the influence of synthesis conditions on structure of IEMs and composites based on them can be obtained. Particularly inversion of polymer chains in polymer composite membranes and location of inorganic particles in one or other types of pores of the polymers have been found. The method also allows us to research hydrophilic-hydrophobic properties of AC, this is especially important for CDI processes.

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УДК 544.23.022+544.726

ІОНООБМІННІ МЕМБРАНИ ТА АКТИВОВАНЕ ВУГІЛЛЯ

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Резюме. Розглянуто застосування методу еталонної контактної порометрії для дослідження активованого вугілля, полімерних іонообмінних мембран та композитів на їх основі. Метод дозволяє вивчати пористу структуру у широкому діапазоні радіусів пор (від 1 нм до 100 мкм), встановлювати механізм формування композитів, ідентифікувати неорганічні частинки у тих або інших порах полімерів, оцінювати гідрофільну та гідрофобну пористість.

Ключові слова: іонообмінні мембрани, органо-неорганічні композити, активоване вугілля, еталонна контактна порометрія, гідрофільні та гідрофобні пори.