

ЕҢБЕК ҚЫЗЫЛ ТУ ОРДЕНДІ  
«Ә. Б. БЕКТҰРОВ АТЫНДАҒЫ  
ХИМИЯ ҒЫЛЫМДАРЫ ИНСТИТУТЫ»  
АКЦИОНЕРЛІК ҚОҒАМЫ

# ҚАЗАҚСТАННЫҢ ХИМИЯ ЖУРНАЛЫ

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## ХИМИЧЕСКИЙ ЖУРНАЛ КАЗАХСТАНА

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## CHEMICAL JOURNAL of KAZAKHSTAN

АКЦИОНЕРНОЕ ОБЩЕСТВО  
ОРДЕНА ТРУДОВОГО КРАСНОГО ЗНАМЕНИ  
«ИНСТИТУТ ХИМИЧЕСКИХ НАУК  
им. А. Б. БЕКТУРОВА»

**2 (66)**

АПРЕЛЬ – ИЮНЬ 2019 г.  
ИЗДАЕТСЯ С ОКТЯБРЯ 2003 ГОДА  
ВЫХОДИТ 4 РАЗА В ГОД

АЛМАТЫ  
2019

A. B. YESZHANOV<sup>1,2</sup>, I. V. KOROLKOV<sup>1,2</sup>, Y. G. GORIN<sup>1,2</sup>, O. GÜVEN<sup>3</sup>,  
M. V. ZDOROVETS<sup>1,2</sup>, S. S. DOSMAGAMBEVA<sup>2</sup>

<sup>1</sup>Institute of Nuclear Physic of the Republic of Kazakhstan, Almaty, Republic of Kazakhstan,

<sup>2</sup>L. N. Gumilyov Eurasian National University, Astana, Republic of Kazakhstan,

<sup>3</sup>Hacettepe University, Ankara, Turkey

## ENHANCEMENT OF MEMBRANE DISTILLATION OF PET TRACK-ETCHED MEMBRANES BY HYDROPHOBIZATION WITH DICHLORODIMETHYLSILANE

**Abstract.** Recent reports from the United Nations show that approximately 1.8 billion people live in countries that experience an absolute shortage of water resources. Due to global population growth and industrialization, it is expected that water consumption will lead to a 40% shortage of fresh water by 2030. Desalination by membrane distillation can help to reduce lack of water by extracting fresh water from polluted sources, including seawater, and wastewater. In this study, new types of membranes have been tested for this purpose. Hydrophobic track-etched membranes (TeMs) based on poly(ethyleneterephthalate) (PET) were prepared using covalent binding of silicon monomer such as dichlorodimethylsilane (DCDMS). Efficiency of hydrophobization was increased by preliminary oxidation by hydrogen peroxide treatment. Pre-oxidation of PET TeMs leads to an increase in water contact angle (CA) up to 102°. The effect of reagent concentration, reaction time were studied by scanning electron microscope (SEM), Fourier-transform infrared spectroscopy (FTIR), gravimetrically, water- and gas-flow rate, contact angle measurements. The performance of the modified membranes was evaluated using direct contact membrane distillation (DCMD) process. The results show maximum water flux of 17 kg/m<sup>2</sup>·h during 6h of saline solution operation at 15 g/l with efficiency up to 98 %.

**Keywords:** PET, track-etched membranes, dichlorodimethylsilane, membrane distillation, hydrophobization, water desalination, surface hydrophobicity.

As a result of natural and human induced factors, the water consumption in the world is constantly increasing, especially in densely populated regions. The problem of lack of fresh water is no longer the prerogative of African countries, but also it can be relevant for Central Asian region. As a result of the combination of anthropogenic and natural factors, more than 70% of Kazakhstan territory is subject to desertification processes to varying degrees. At the moment, there is an acute shortage of water resources in Kazakhstan for the needs of industry and agriculture, and for drinking water supply. The importance of this problem was also noted by the President Nursultan Nazarbayev in the message "Strategy Kazakhstan-2050: a new political course of the prosperous country", that one of the challenges of the current century will be the shortage of drinking water [1].

The main pollutants of water resources are petroleum products, phenols, easily oxidized organic substances and heavy metal ions. In addition, man-made radionuclides entering the groundwater as a result of nuclear dumps, accidents at

nuclear power plants, discharges of liquid radioactive waste of some industries, make it unsuitable for use without proper treatment.

Currently, most of the ongoing researches in the field of water purification is aimed on the development of environmentally friendly, energy-saving and efficient systems based on membrane separation processes [2-4].

There are various types of membranes used for these purposes: isotropic, anisotropic, composite, flat, tubular, fiber, woven, sintered or molded.

One of the most important and defining characteristics is the membrane material. Currently vast majority of the membranes on the market are made from polymeric materials. The main materials for the manufacture of flat polymer membranes are cellulose acetates, aromatic polyamides, fluoropolymers, poly(ethylene terephthalate) (PET), polyacrylonitrile, polyamides, polyimides, polyethylene, polypropylene and some other polymers [5].

One of the methods for producing so-called ion-track membranes is the irradiation of a polymer film. Its macrostructure is formed by irradiation with accelerated ions followed by physicochemical treatment, ultraviolet (UV) irradiation and chemical etching. This results to the formation of track-etched membranes (TeMs).

A characteristic feature and advantage of TeMs is regular pore geometry with the ability to control their number per unit area and a narrow pore size distribution. This in turn, provides a given selectivity and specific performance of membrane [6, 7].

Currently PET films are one of the most widely used materials for the preparation of TeMs. The main characteristics of film materials based on PET should include high strength and optical characteristics, low swelling and shrinkage, thermal stability, chemical indifference towards conjugate media [8-10].

Despite the fact that the surface of PET TeMs has medium-hydrophobic properties, for using them in the process of membrane distillation (MD), it is necessary to improve the water-repellent properties of such membranes. One of the most frequently used methods of material hydrophobization is the creation of a thin polymer film layer of hydrophobic nature on their surface. In this case, two problems can be solved simultaneously: a change in the surface energy of the TeMs and a decrease in the average pore diameter to the optimum value. However, most of the methods used do not satisfy the needs of MD, since the created hydrophobic layer is susceptible to washing away with prolonged contact with water. The most commonly used are polysiloxane polymers and copolymers with a pronounced hydrophobic nature. The problem is instability of the connection of these polymers with the underlying surface of the membrane. Thus the hydrophobization of PET TeMs for such applications is an important task <sup>[11-19]</sup>.

The new generation of membranes will provide access to alternative sources, providing water purification unsuitable for irrigation, industrial and domestic use. Industrial application of the membrane distillation process will help to solve the problems of water scarcity, environmental pollution, inconsistencies between water supply and water demand [20].

In this paper, we consider method of hydrophobization based on covalent binding of dichlorodimethylsilane (DCDMS) on PET TeMs. The effect of pre-oxidation of PET TeMs and further silanation on the contact wetting angle will be studied in comparison with the etched samples. The obtained hydrophobic membranes were tested in desalination process.

## EXPERIMENTAL

*Chemicals.* DCDMS, sodium hydroxide, *o*-xylene, 2-propanol, hydrogen peroxide, hydrochloric acid were purchased from Sigma-Aldrich and used as received. Deionized water (18.2 M $\Omega$ ) was used in all experiments.

*Preparation of the membrane.* PET films with a thickness of 12  $\mu\text{m}$  («Mitsubishi Polyester Film», Germany) were irradiated in the DC-60 accelerator by Ar ions with an energy of 1.75 MeV/nucleon and ion fluence of  $1 \cdot 10^8$  ion/cm<sup>2</sup>. After the etching process in a 2.2 M solution of NaOH at 85°C, membranes with pore sizes of 200–250 nm were obtained. Etched membranes were dried and kept between paper sheets at room conditions.

*Hydrophobization of PET TeMs.* To increase efficiency of covalent binding of silanes, pre-oxidation of PET TeMs in the H<sub>2</sub>O<sub>2</sub>/UV system was applied [8]. For this purpose, PET TeMs samples (5×7.5 cm<sup>2</sup>) were oxidized in a solution of 0.3M H<sub>2</sub>O<sub>2</sub> at pH = 3 (HCl). Oxidation was carried out for 180 min under UV-irradiation with 190 W mercury lamps at 254 nm. After the oxidation the samples were washed with deionized water twice, dried in air at room temperature.

Hydrophobization of PET TeMs was achieved by immersion of samples into the solution of DCDMS in 2-propanol or *o*-xylene with various concentrations (0.01M, 0.1M, 1M, 5M) during 1h – 48h. At the end of these periods membranes were quickly removed from the reaction medium rinsed with the solvent and dried.

*Membrane characterization.* Chemical changes on the membrane surfaces were studied by taking FTIR spectra using Agilent Cary 600 Series Spectrometer with Single Reflection Diamond ATR accessory (GladiATR, PIKE). JEOL JSM-7500F scanning electron microscope was used to characterize pore diameters and morphology after PET TeMs modification. Water contact angle of the samples was measured using Digital Microscope 1000x at room temperature.

*Direct contact membrane distillation (DCMD).* Direct contact membrane distillation (DCMD) was used to determine separation performance of hydrophobized PET TeMs. DCMD rig was described in our previous paper [19, 20]. In brief, the modified membrane was placed in a cell for MD process, the flow rate on permeate and feed side was controlled and kept at 227±3 ml/min and 453±3 ml/min respectively using Easy load Cole-Parmer Masterflex L/s 77200-62, these parameters provide optimal performance and lowest temperature polarization. The temperature at the feed side was 85 °C and at the permeate side 10 °C. The permeate flux was measured by weighing the mass of liquid on permeate side

at fixed time intervals (30 sec) on a balance ( $\pm 0.01$  g). The efficiency of salt rejection was evaluated conductometrically using Hanna Instruments HI2030-01.

## RESULTS AND DISCUSSION

*Preparation and characterization of hydrophobic membrane.* Hydrophobization of the surface was achieved by creating on the surface of PET TeMs covalent bonds along the terminal OH groups with DCDMS.

The reaction is based on the chemical inertness of the Si-C bonds and the high reactivity of the Si-Cl bonds, which easily undergoes by hydrolysis and interaction with the OH-rich surface of PET TeMs. Schematically, the covalent attachment process of DCDMS is presented in figure 1.

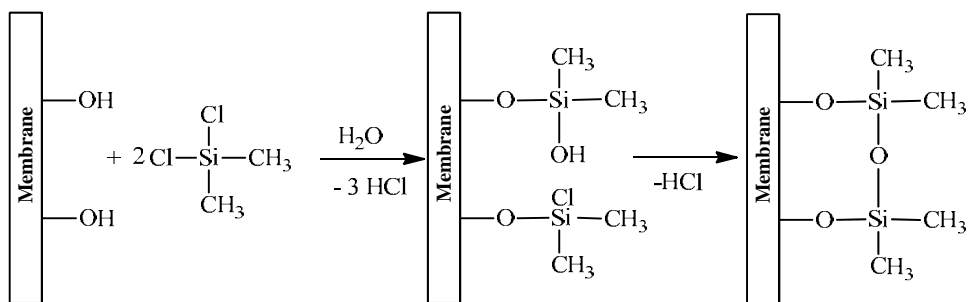


Figure 1 – Scheme of the hydrophobization of surfaces of PET TeMs

The experiment was carried out under different conditions by changing the solvent (*o*-xylene, 2-propanol), concentration of DCDMS (0.01M; 0.1M; 1M; 5M) and reaction time (from 1 h to 48 h). The results are shown in table 1.

Table 1 – Results of hydrophobization PET TeMs by DCDMS

No	Time, h	Solvent	Concentration, M	Water contact angle, °
1	0	–	–	49±4
2	1	<i>o</i> -xylene	1	70±4
3	6	<i>o</i> -xylene	1	73±3
4	16	<i>o</i> -xylene	1	79±2
5	24	<i>o</i> -xylene	1	84±4
6	48	<i>o</i> -xylene	1	84±2
7	24	<i>o</i> -xylene	0.1	76±4
8	24	<i>o</i> -xylene	0.01	74±4
9	24	<i>o</i> -xylene	5	75±5
10	24	2-propanol	0.1	72±5

According to the obtained data, it was concluded that *o*-xylene is a suitable solvent and it was chosen for further work with samples in other conditions. Images of drops on the surface of the membranes after modification in *o*-xylene under various conditions are presented in figures 2, 3.

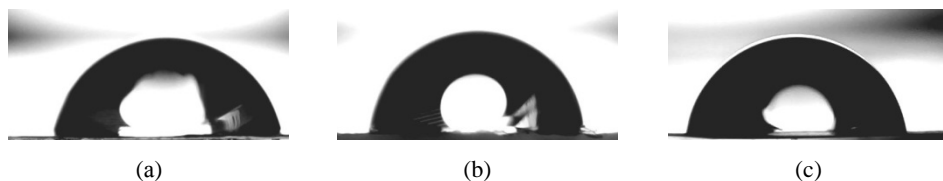


Figure 2 – Images of drops on modified PET TeMs at a concentration of DCDMS in *o*-xylene of 0.01M (a), 1M (b) and 5M (c)

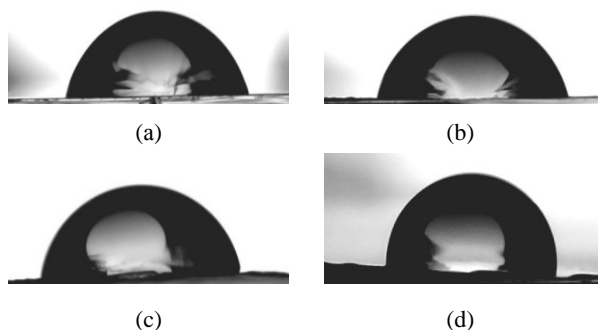


Figure 3 – Images of drops on modified PET TeMs at a concentration of DCDMS of 1 M and a reaction time of 1h (a), 6h (b), 16h (c) and 48h (d)

It can be seen that, the DCMDS system in *o*-xylene with a concentration of 1M and time of 24 h is the most optimal for achieving the hydrophobic properties of the PET TeMs surface. At the same time, it should be noted that a decrease in the CA with an increase in the concentration of the DCMDS to 5M is probably associated with the predominant interaction of silanes with each other than with the surface of the membrane. To increase efficiency of covalent binding of silanes, pre-oxidation of PET TeMs in the  $H_2O_2/UV$  system was used [8]. Pre-oxidation lead to increasing chemical active groups on the surface that potentially can improve density of hydrophobic layer. In turn, this will lead to an increase in the value of the contact angle. The obtained results are presented in table 2.

The obtained data indicate that pre-oxidation leads to an increase in the CA to  $102^\circ$ . This is probably due to the creation of active reaction center on the surface of the membrane that occur after oxidation. Thus, research on the hydrophobization of etched and oxidized PET TeMs in DCMDS was carried out, and the optimum process conditions were determined. Studies have shown that pre-oxidation leads to an increase in the CA to  $102^\circ$ .

Table 2 – Experimental data on the wetting angle of oxidized and etched PET TeMs by DCDMS

No	Sample name	Concentration, M	Time, h	Water contact angle, °	Effective pore size, nm
1	PETeMsox.	1	1	75±3	189±5
2	PET TeMsetched	1	1	70±4	191±6
3	PETeMsox.	1	3	78±2	184±5
4	PET TeMsetched	1	3	71±1	182±6
5	PETeMsox.	1	5	79±1	171±5
6	PET TeMsetched	1	5	78±1	172±5
7	PETeMsox.	1	24	102±2	165±6
8	PET TeMsetched	1	24	84±2	164±6

Modified membranes were studied by FTIR-ATR spectroscopy. Typical FTIR-ATR spectra of pristine and modified PET TeMs as well as DCDMS for comparison are shown in figure 4. The comparison of the modified and non-modified PET TeMs spectra shows us differences confirming the presence of DCDMS on the membrane surface. The main absorption peaks related to PET are  $3432\text{ cm}^{-1}$  (O–H),  $2972\text{ cm}^{-1}$  (C–H),  $2910\text{ cm}^{-1}$  (C–H),  $1715\text{ cm}^{-1}$  (C=O),  $1470$ ,  $1430$ ,  $1409\text{ cm}^{-1}$  (aromatic vibrations of benzene), C(O)–O bonds of ester groups ( $1238\text{ cm}^{-1}$ ),  $980\text{ cm}^{-1}$  (O–CH<sub>2</sub>). In the modified samples, the valence vibrations of Si–CH<sub>3</sub> and Si–O bonds at  $1118$  and  $1045\text{ cm}^{-1}$ , the deformation vibrations of the Si–C bond at  $987\text{ cm}^{-1}$ , which are related to DCDMS, are observed.

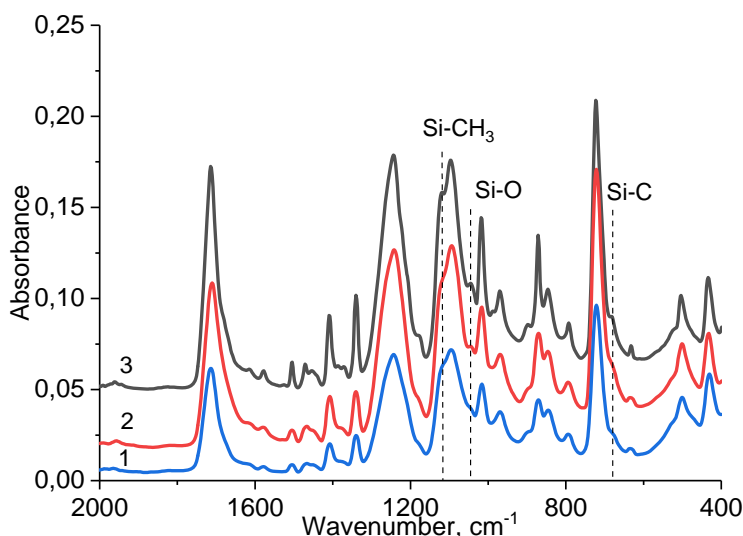


Figure 4 – FTIR-ATR spectra of 1 – pristine PET TeMs, 2 – DCDMS-PET TeMs (reaction time 24h), 3 – DCDMS-PET TeMs (reaction time 48h)

The surface of the modified membranes was also studied by SEM.

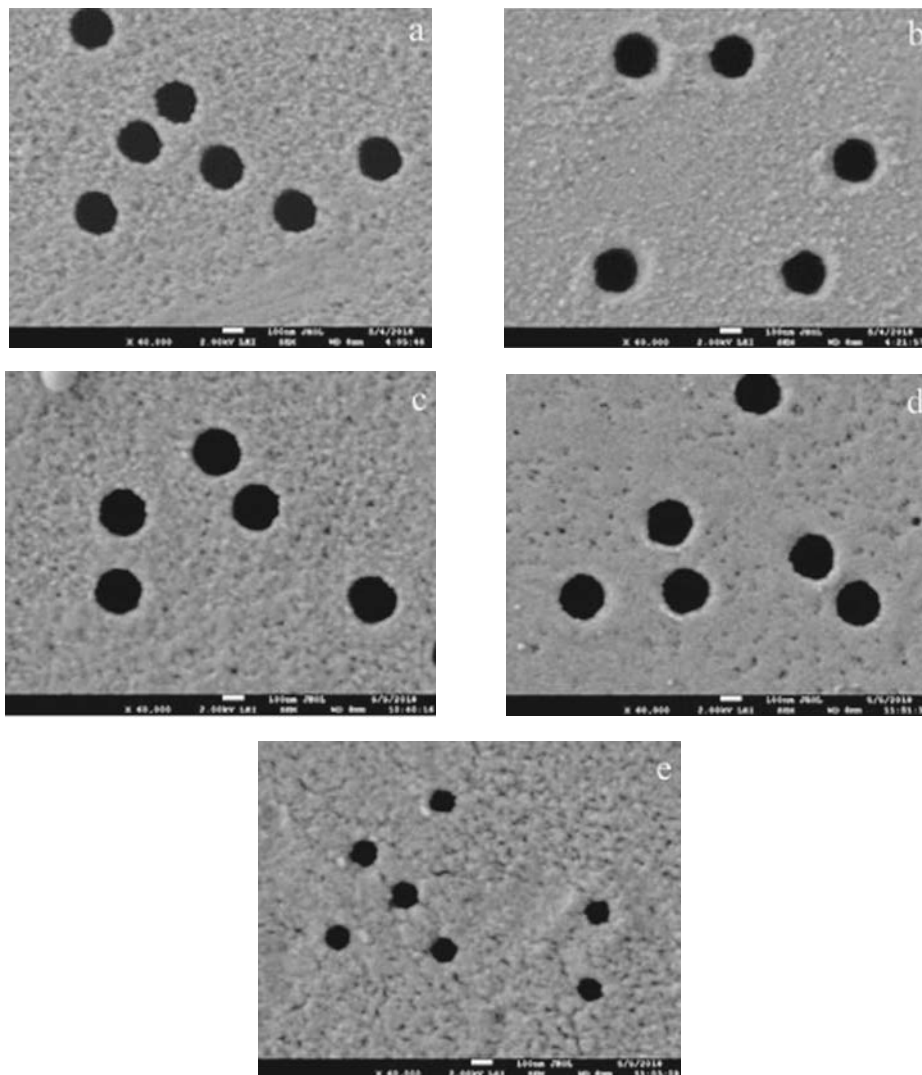


Figure 5 – SEM images of PETTeMs surface after modification of DCDMS depending on the reaction time of 1h (a), 6h (b), 16h (c), 24h (d) and 48h (e)

SEM images showed us that increasing reaction time led to decreasing pore size, that is also consistent with the results on gas permeability. The decrease in pore diameter is due to swelling of the PET TeMs.

*Transport properties of hydrophobized PET TeMs after DCMD process.* Membrane distillation was carried out in direct contact mode in a laboratory setup [19]. The membrane distillation characteristics of the modified membranes were evaluated using NaCl solution with concentrations of 15 g/l. In the MD process,



the efficiency of mass transfer and salt removal was evaluated, as shown in figure 6. A gradual decrease in productivity occurred in the process. Deviations in performance are most likely due to the inconstancy of the temperature gradient.

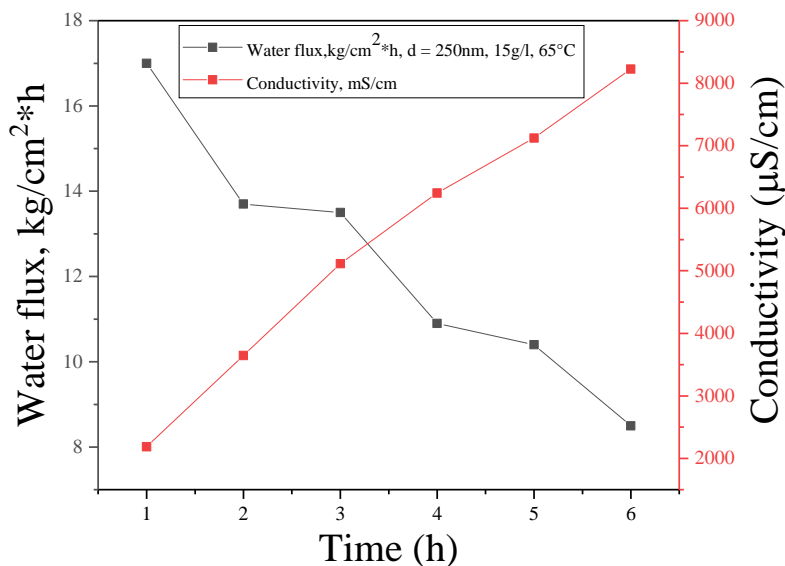


Figure 6 – MD water flux and the distillate electrical conductivity during continuous DCMD tests using hydrophobized PET TeMs (DCDMS) for NaCl solution with concentration of 15 g/l with pore diameters of 250 nm

Average water flux for the solution of NaCl (15g/l) is 17 kg/m<sup>2</sup>·h. The degree of salt rejection for a solution with a concentration of 15 g/l was 98%. It is worth noting that wetting and contamination are one of the main factors contributing to reduced productivity. Results show us that hydrophobized PET TeMs have a high potential to use them for membrane distillation and need to be further studied.

**Conclusions.** In this work, PET TeMs was modified by covalent binding of DCDMS with improved surface hydrophobicity. FTIR, and contact angle analysis confirm the success coating of DCDMS on the membrane using *o*-xylene as a solvent. The optimal parameters (DCDMS system in *o*-xylene with concentration 1M for 24h) showed, that using pre-oxidation PET TeMs leads to CA 102°, while non-oxidized samples have CA - 84°. Thus, technologically convenient methods of hydrophobization by covalent binding of DCDMS to the surface of PET TeMs were investigated. Then modified membranes with pore size of 200±50 nm were tested in DCMD process for water desalination. The average results reaches maximum water flux of 17 kg/m<sup>2</sup>·h 15 g/l NaCl solution operation with efficiency up to 98%. The results show the possibility of using hydrophobized PET TeMs in the method of water purification by membrane distillation.

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**Резюме**

*А. Б. Есжанов, И. В. Корольков, Е. Г. Горин,  
О. Гювен, М. В. Здоровец, С. С. Досмагамбетова*

**ПЭТФ ТРЕКТИК МЕМБРАНАЛАРДЫҢ  
МЕМБРАНАЛЫҚ СҮЗУ ҮШІН  
ДИХЛОРИДИМЕТИЛСИЛАН ГИДРОФОБИЗАЦИЯЛАУ**

Қазіргі уақытта Біріккен Ұлттар Ұйымының деректері бойынша шамамен 1,8 миллиард адам су ресурстарының жетіспеушілігін сезінетін елдерде тұрады. Жаһандық халық санының өсуімен индустрияландырудың арқасында 2030 жылға қарай суды тұтыну тұщы судың жетіспеушілігіне әкеледі деп күтілуде. Мембраналық айдау арқылы тұзсыздандыру ластанған көздерден тұщы суларды, соның ішінде теңіз суын, сарқынды суларды және радиоактивті су. Су ресурстарының негізгі ластағыштары – мұнай өнімдері, фенолдар, пестицидтер, оңай тотыққан органикалық заттар және ауырметалдар иондары. Қазіргі уақытта суды тазарту саласында жүргізіліп жатқан зерттеулердің басым бөлігі мембраналық бөлу процес-терінен егізделген экологиялық таза, энергияны үнемдейтін және тиімді жүйелерді дамытуға бағытталған. Бұл зерттеу үшін мембрананың жаңа түрлері тексерілді. Гидрофобты тректік мембраналарды (ТМ) полиэтилентерефталат (ПЭТФ) негізделген кремний мономер дихлоридиметилсилан (ДХДМС) сияқты ковалентті байланыстыру арқылы алынды. Гидрофобизацияның тиімділігі сутегі асқынының ерітіндісінде тотықтырумен алдын-ала көбейді. РЕТ ТМ-нің тотығуы алдын-ала контакті ылғалдылық бұрышын (КЫБ) 102°-ге дейін арттырады. Реакциялық компоненттердің концентрациясының әсері, КЫБ-дың өзгеруіне реакция уақыты электрондық сканерден өтетін электрондық микроскоп (СЭМ), ИҚ-спектроскопия көмегімен зерттелді. Түрлендірілген мембраналардың өнімділігі мембраналық сүзу (МС) арқылы бағаланды. Ең жоғарғы нәтижелер су өнімділігі 15г/л NaCl концентрациясы кезінде 98,36% дейін тазалау тиімділігімен 6 сағат бойы 17 кг/м<sup>2</sup>-сағ дейін жетеді.

**Түйін сөздер:** ПЭТФ, тректік мембраналар, дихлоридиметилсилан, мембраналық сүзу, гидрофобты түрлендіру, суды тазарту, беттік гидрофобизация, ковалентті байланыстыру.

**Резюме**

*А. Б. Есжанов, И. В. Корольков, Е. Г. Горин,  
О. Гювен, М. В. Здоровец, С. С. Досмагамбетова*

**ГИДРОФОБИЗАЦИЯ ПЭТФ ТРЕКОВЫХ МЕМБРАН  
ДИХЛОРИДИМЕТИЛСИЛАНОМ  
ДЛЯ МЕМБРАННОЙ ДИСТИЛЛЯЦИИ**

В настоящее время, по данным Организации Объединенных Наций, примерно 1,8 миллиарда человек живут в странах, испытывающих абсолютную нехватку водных ресурсов. Из-за глобального роста населения и индустриализации ожидается, что к 2030 году потребление воды приведет к 40% дефициту пресной воды. Опреснение путем мембранной дистилляции может помочь уменьшить нехватку воды за

счет извлечения пресной воды из загрязненных источников, включая морскую воду, сточные воды и радиоактивная вода. Для этой цели в работе были протестированы новые типы мембран. Гидрофобные трековые мембраны (ТМ) на основе полиэтилентерефталата (ПЭТФ) были получены методом ковалентного связывания кремниевого мономера, такого как дихлордиметилсилан (ДХДМС). Эффективность гидрофобизации повышалась путем предварительного окисления в растворе перекиси водорода. Предварительное окисление ПЭТФ ТМ приводит к увеличению контактного угла смачивания (КУС) до 102°. Влияние концентрации реагирующих компонентов, времени реакции на изменение КУС изучали с помощью сканирующего электронного микроскопа (СЭМ), ИК-спектроскопии с Фурье-преобразованием. Производительность модифицированных мембран оценивали с использованием процесса мембранной дистилляции (МД). Результаты показывают, что максимальная производительность воды достигает 17 кг/м<sup>2</sup>·ч в течение 6 ч при концентрации 15 г/л NaCl с эффективностью очистки до 98,36%.

**Ключевые слова:** ПЭТФ, трековые мембраны, дихлордиметилсилан, мембранная дистилляция, гидрофобная модификация, очистка воды, гидрофобизация поверхности, ковалентное связывание.