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ВЛИЯНИЕ АРМИРОВАНИЯ НА СВОЙСТВА ГЕТЕРОГЕННОЙ БИПОЛЯРНОЙ МЕМБРАНЫ

Биполярные мембраны используются в электромембранных технологиях, при помощи которых производятся кислоты и гидроксиды из соответствующих солей. Свойства биполярной мембраны существенно влияют как на сам процесс электродиализа, так и на конструкцию электродиализатора. Целью настоящего исследования является сравнение трех типов биполярных мембран. Первая мембрана была положена в основу обеих последующих. Она представляет собой экструдированную гетерогенную мембрану, изготовленную на соэкструзионной линии. Второй образец мембраны подвергся в прессе армированию при помощи двух полипропиленовых текстилей. Третий образец обрабатывали в прессе при тех же самых условиях, как описано выше, за исключением того, что армирующий материал не был использован. У подготовленных мембран сравнивались размерные и весовые изменения в процессе набухания, электрохимические свойства и полученные параметры технологических испытаний на устройстве EDBM-Z. Использование армирующей ткани в биполярной мембране в значительной степени влияет на направление набухания мембраны. В то время как неармированные мембраны больше набухают по площади, армированные мембраны наиболее набухают в толщину. Это существенно меняет их транспортные свойства, которые влияют как на форму кривой вольтамперной характеристики, так и на результаты технологических испытаний. Мембраны, армированные тканями, показывают при испытаниях более высокую эффективность и низшее потребление энергии на 23%, чем подвергшиеся прессованию мембраны. Транспортировка соли на единицу площади мембраны для обоих типов мембран одинаковая. Экструдированная мембрана без последующего прессования показывает по всем наблюдаемым параметрам значительно худшие величины.

Ключевые слова: биполярная мембрана, катионная смола, анионная смола, анод, катод, ионы солей, полиэтилен

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INFLUENCE OF REINFORCEMENT ON THE OF HETEROGENEOUS BIPOLAR MEMBRANE PROPERTIES

Bipolar membranes are used in electromembrane technologies with the help of which acids and hydroxides are produced from the corresponding salts. Properties of the bipolar membrane have significant impact as on electrodialysis process itself, so on the structure electrodialysis apparatus. The purpose of this study is to compare three types of bipolar membranes. The first membrane was the basis for the next two. It represents a heterogeneous extruded membrane made in the co-extrusion line. The second sample of the membrane was exposed to reinforcement in molding machine with the use of two polypropylene textiles. The third sample was treated in the molding machine under the same conditions except for using reinforcing material. We prepared membranes compared The said prepared membranes were thoroughly compared in relation of dimentional and weight changes during swelling, electrochemical properties and parameters of technological tests with the use of EDBM-Z device. The use of reinforcing fabric in the bipolar membrane greatly affects the direction of the membrane swelling: membranes without reinforcing swell larger in size; reinforced membranes – grow in thickness. This significantly change their transport properties, which affect both the shape of the current-voltage characteristic curve, and the results of technological tests. Membranes reinforced with fabrics when tested show higher efficiency and 23% lower energy consumption in comparison with the membranes subjected to compression. Transporting of salt per area unit of the membrane for the both types of membranes is same. Extruded membrane without subsequent compaction shows much worse values for all the observed parameters.

Keywords*:* bipolar membrane, cationic resin, anionic resin, anode, cathode, salt ions, polyethylene

INTRODUCTION

The bipolar membranes (BM) consist of two layers of ion-exchange material. One layer is a cation exchanger, and the second one an anion exchanger. After application of suitable voltage dissociated water concentrates on a boundary line of this two phases (Fig. 1). Cation exchange bed produces transporting of proton to cathode and anion exchange bed moves hydroxyl ion to anode. This phenomenon is the basis of electrodialysis technology with bipolar membrane t (EDBM) in production of acids and bases for the corresponding salts.

Heterogeneous bipolar membranes, manufactured by OOO «MemBrain», have been made without using of some reinforcing fabric. Prepared in such a way membranes have several "inappropriate" properties. The first is too high brittleness in dry state making handling of the membrane and it's formatting to be hindered. Absence of some reinforcing fabric also significantly affects the mechanical properties of the membranes in their swollen state. Membranes are very soft. It causes their bending and extrusion. Another disadvantage of unreinforced membrane is their swelling. Membranes without reinforcement change their size (first of all parameters of their surface area - length and width of the membrane). This manifests itself not only in the primary swelling, but also at changing of the ionic form of the ion exchange particles in the membrane. Due to the last two disadvantages it is especially difficult to use them in EDBM plants. Membranes in these plants bend, clog chambers and inlets. This leads to overflowing between separate circuits. As a result, reduces the effectiveness EDBM process decreases.

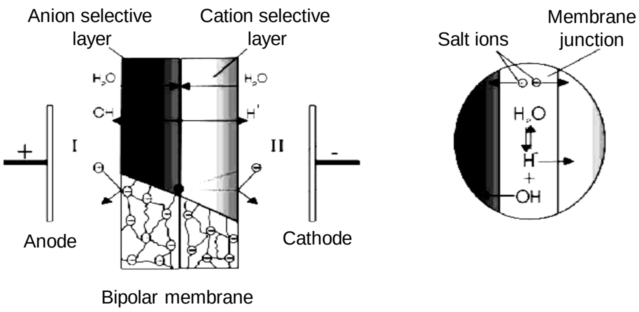

Fig. 1.Diagram of the bipolar membrane [1]
Рис. 1.Схема биполяной мембраны [1]

EXPERIMENTAL

Sample preparation

Bipolar membranes are made of finely reground mixture of ion exchange resins and a bounding agent, as a rule it is polyethylene. Washed and dried ion exchanger was pulverized in a vibratory mill «Vibrom 42S» so that at least about 99% of particles have a size less than 100 microns. Ion-exchange resins were then blended with the linear polyethylene of low density at weight ratio of 3:2 in continuous mixer «Xinda SJW-45». Manufactured granulated mixture was then extruded in order to obtain a form of a flat membrane of 0.35 mm thick in co-extrusion line. Membranes were divided into three samples. The first of these (designated as BM1) was not subjected to any further processing. Two others were treated on a laboratory press «Presshydraulika ZHOT60MT» at 140 °C during 20 min without pressure, the next 10 min at the same temperature under the pressure of 50 atm. Cooling of the sample to a temperature of 50 °C was executed without pressure reducing. The second sample (marked as BM1-P) was only extruded under the conditions mentioned above. In the third sample (designated as BM1-T) under the above conditions were pressed-in two polypropylene fabrics «Sefar IEM-05-195/70» of 70 micron [2], one on each side.

BM specifications

Changing of the membrane size after swelling were performed on the samples of 10×10 cm size, which were dried at 105 °C and then swollen in demineralized water.

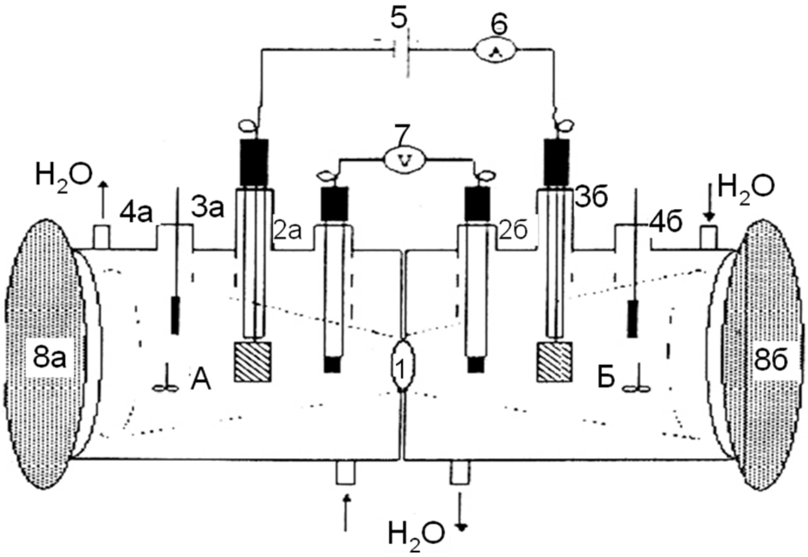

Fig. 2. The electrochemical cell for measuring V-A characteristics. A, B - conical chamber s with solution of 0.5 M KCl; 1 - membrane sample; 2a, 2б - reference electrodes Ag / AgCl; 3a, 3б - Pt-electrodes; 4a, 4б - thermometers; 5, 6, 7 - connection of potentiostat; 8a, 8б - clamping plates
Рис. 2. Электрохимическая камера для измерения V-A характеристики: А, Б – конические камеры с раствором 0,5M KCl; 1 – мембранный образец; 2а, 2б – эталонные электроды Ag/AgCl; 3а, 3 б – Pt-электроды; 4а, 4б – термометры; 5, 6, 7 – подключение потенциостата; 8а, 8б – зажимные пластины

Several electrochemical methods are known to characterize bipolar membranes. We have used current-voltage characteristic [3] because of its simplicity and ease of interpretation. Firstly the samples were swollen in deionized water, and then were soaked in 0.5 M KCl solution in which the membranes were measured. Each membrane was clamped between two electrochemical chambers of conical shape (Fig. 2) and then measured with the use of four electrodes method in electro-dynamic mode by potentiostat «Biologic SP-300». Sample area constituted 0.74 cm^2. The measurements were performed at 25 ± 0.5 °C with the measuring range of 0–60 mA (potentiostat limited capacity for a total of 10 V) and current increase rate of 100 μA/s. The working electrodes were platinum, reference electrode – Ag/AgCl (1 M KCl).

Technological tests with the use of EDBM-Z device

Standardized test characterizes the membrane during the electrodialysis process taking into account standard conditions in the device. The purpose of this test is to simulate the conditions of the process on a large industrial unit and controlling the behavior of individual membranes depending on these conditions.

BM were tested with the use of EDBM-Z device. Active area of one membrane constituted 64 cm^2. Membranes swelled in demineralized water prior to being placed in the device. Block diagram of the device is stated in Figure 3. The cell was drawn tight in order to avoid any infiltrations. Before testing overflows between the individual chambers at half rate of the flow were determined. If overflow were higher than 10 ml/min, the cell was folded over and drawn tight repeatedly. During the test, the desalination of sodium sulfate takes place. In the acid loop Sulfuric acid forms in the acid loop, and the base circuit forms sodium hydroxide.

During testing, voltage, passing current, conductivity and pH of all the four circuits were controlled. The following parameters were determined from the data obtained:

- weight of the salt flow J (g/(m^2 h)) is determined in accordance with the equation (1):

$$J = \frac{\Delta m}{A \cdot \Delta t} = \frac{\Delta m}{N \cdot w \cdot l \cdot \Delta t}, \qquad (1)$$

where Δm – weight loss of salt in the product; A – effective area of a membrane; N - the number of

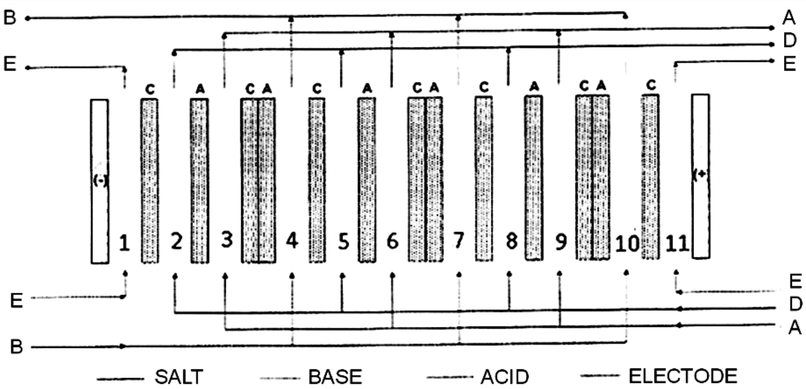

Fig. 3. Connection scheme of three circuit EDBM
Рис. 3.Схема подключения трехконтурного EDBM

Table 1.

Conditions of the standard three-circuit test of EDBM
***Таблица 1.* Условия стандартного трехконтурного теста EDBM**

Parameter	Parameter value
Composition	-CM_AM_BM_CM_AM_BM_CM_AM_BM_CM+
Salt circuit (S)	0.6 l Na_2SO_4 10 g/l
Inlet of acid circuit (A), base (B)	0.3 l of demineralized water
Electrode circuit (E)	0.3 l Na_2SO_4 10 g/l
Stream S, A, B	25 l/h
Stream E	50 l/h
Voltage	3 V/BM (9 V/ cell without electrodes)
Active area of the BM	192 cm^2
Total installed area of membranes	1153.6 cm^2
Completion of the test	90% demineralization of S circuit
Separator thickness	0.8 mm

membrane pairs; w – the effective width of the membrane; l – the effective length of the membrane; Δt – time of 95% desalination;

- time t (min) which resulted in decrease of the product conductivity d of 90%;

- the specific energy consumption E (W·h/kg) at a ratio for1 kg of salt being transferred from the product:

$$E = \frac{U \cdot Q}{\Delta m}, \qquad (2)$$

where U – the applied voltage; Q – used electric charge; Δm – amount of salt being transferred from the product;

- current efficiency η (%) was determined according to the equation:

$$\eta = \frac{\nu_K \cdot z_K \cdot F \cdot \Delta n}{N \cdot Q} \cdot 100\%, \qquad (3)$$

where νK – stoichiometric ratio of cations; ZK – valence of cations; F – Faraday constant; Δn – amount of substance transferred salt from the product; N – number of bipolar membranes-chambers (chamber: SM_AM_VM); Q – electric charge having being used.

RESULTS AND DISCUSSION

The samples were prepared in accordance with the parameters given above. Sample of the BM1-T fabric was completely laminated, whereby it was confirmed that the selected parameters of pressing are sufficient. Definite complications were brought in with brittleness of the samples without fabric in a dry state. These complications were most pronounced at the determination of dimensional change upon swelling, when dried membrane should be straighten and measured. Dimensions of the membranes are listed in Table 2 and fit in with the expected values. Most of all increases the area of swollen membranes without processing by pressing. The use of reinforcing fabric in the bipolar membrane largely affects the direction of the membrane swelling. While unreinforced membrane swells with increasing its area, reinforced membrane mostly swell in thickness.

The membrane thickness also affects its electrochemical behavior. Current-voltage specifications were changed for each sample membrane (Fig. 4). The diagram shows huge difference between different types of membranes. The slope of the linear part beginning approximately at voltage of about 2 volts characterizes the intensity of water decomposition in the bipolar membrane. In this regard, we can say that water decomposition proceeded most rapidly on the sample pressed membrane P-BM1. This is probably due to the thickness of the membrane, which affects both ions transporting out of the membrane, and water transporting the water at the phase boundary.

Table 2.

Changes after the swelling of membranes

***Таблица 2.* Изменения после набухания мембран**

Designation	Parameter value in dry and swollen state (Δ – percentage increase relative to dry state)			
	Width s, cm	Length d, cm	Thickness tl, мм	Weight m, g
BM1	9.85→11.45 (Δ=16.2%)	9.95→11.55 (Δ=16.1%)	0.342→0.433 (Δ=22.0%)	4.456→7.542 (Δ=69.3%)
BM1-P	9.90→11.5 (Δ=16.2%)	9.85→11.40 (Δ=15.7%)	0.346→0.428 (Δ=19.2%)	4.613→7.330 (Δ=58.9%)
BM1-T	9.95→11.35 (Δ=14.1%)	9.85→11.15 (Δ=13.2%)	0.349→0.464 (Δ=33.1%)	3.839→6.289 (Δ=63.8%)

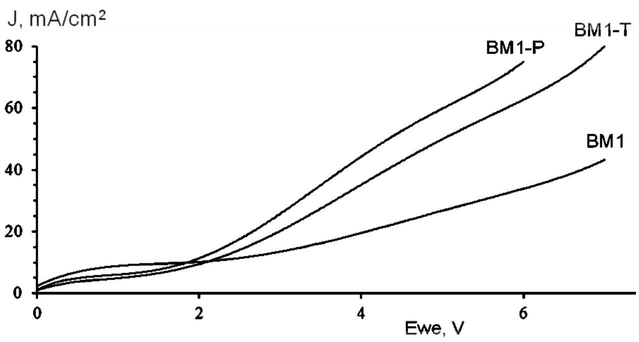

Fig. 4. The current-voltage characteristics
Рис. 4. Вольт-амперная характеристика

Behavior of membranes at voltage in the range from 0.2 to 0.8 V, before starting of intensive water splitting depends on the bipolar membranes flux. We can see that the bipolar membrane with T-VM1 fabric lets pass the least quantity of ions in this voltage range. This phenomenon is consistent with the results of technological tests (Table 3).

All the values of Table 3, were figured out of the changes in salt concentration in salt (product) limits. Bipolar membranes without press treatment are used for desalination approximately two times longer than the other two samples. Lower flow of salt (J) of the product is consistent to it too. As for the current efficiency and energy consumption, they are roughly comparable to the pressed version. The bipolar membrane with embedded textile achieve the same salt flow and time of 90% desalting, but with lower power consumption.

Table 3.

Results of standardized technologies of testing

***Таблица 3.* Результаты стандартизированных технологий испытаний**

Sample	$t_{90\%}$, min	η, %	E, W·h/kg	J, g/(h·m^2_{BM})
BM1	121.4	54.3	2160.3	165.7
BM1-T	58.3	70.6	1604.6	335.2
BM1-P	60.4	56.9	1992.0	332.5

Membrane methods distinguish by geometrical shape during operation. With a wide range of mechanical and electrochemical characteristics of modern ion-exchange membranes, the choice of technology of their implementation is multi-plane.

The easiest way is membrane electrolysis. At the initial electrolysis diaphragm is replaced by ion exchange membrane [4]. Electrolysis of water or other redox reactions occur successfully at the electrodes due to selective ion transporting. In membrane electrolysis, each block may consist of only two compartments and the two electrodes. This technique requires significant expenditures for specific electrodes and high demands on their chemical resistance [4, 5]. This method is an effective solution for the base synthesis (when only acid disengage from the salt or only a base with a high degree of purity).

Electrodialysis is a "transition" technology between membrane electrolysis and electrodialysis. [4] In three-section chamber saline solution is injected between the membranes. The cations are transported through the cation exchange membrane to the cathode. Anions are transported through the anion exchange towards the anode. The resulting parent solution is desalted. The final concentration of the products is limited by water transport and chemical resistance of the electrodes. Such shortcomings as leakage of protons and the rate of ion exchange membranes are successfully compensated by the modern membranes.

In the light of foregoing bipolar membranes in electrodialysis are the most attractive technology for solutions partitioning. In this work we propose [4, 6-8] options of installation of bipolar membranes in the cell. The main directions of the membrane arrangement variations depending on the separation task are stated in table 4.

The selectivity of the bipolar membrane is strongly dependent on the concentration of salts, acids and bases. This fact leads to limitations in the performance of the membrane. The main advantage of bipolar membranes is the efficient use of energy. Economically attractive are bipolar membrane technology for the simultaneous concentration of acid and alkali.

Table 4.

Methods for efficient installation of membranes

***Таблица 4.* Способы эффективной установки мембран**

Membrane location	Purpose of the process	Source
Three-section cell	Separation relatively concentrated salt solutions to acids and bases	[4]
Two-section cell	Disengagement acidic or alkaline component of the solution [4]	[4]
Configuration with two monopolar membranes	increases the ratio of acid (or alkali) to salt: salt splitting, acid/base restoration [6-8]	[6-8]

Double exchange reactions (metathesis-electrodialysis) can be carried out only with the correct configuration of monopolar membranes. Bipolar membranes alternately placed between the two electrodes. In contrast to the classical metathesis, metathesis-electrodialysis is not an equilibrium process, which allows to allocate (remove) readily soluble contaminants. Its disadvantage is relatively low concentration of the product solution, as far as salts crystallization in the unit can cause damage of the membrane [4]. The concentration of the product is limited by the speed of water transporting, and degree of purification of limited with selectivity of the membrane and the reverse diffusion [9]. Particular influence at operation shows polymeric binder [11-14].

CONCLUSIONS

There were prepared samples of laminated heterogeneous bipolar membranes were prepared. Reinforcing fabric does did not affect the boundary layer and increases increased the permeability of the bipolar membranes. Textiles BM improved mechanical properties in the dry state. Membranes with reinforcing fabric swelled more considerably in thickness than the in square. This is largely affected their transport properties. At voltages, leading to the degradation of water, reinforced diaphragm reached the lower flows than the extruded pressed membrane without fabric. At lower voltages reinforced membrane exhibited very low permeability. The combination of the last two results arising from the current-voltage characteristics, was demonstrated when technological tests. Reinforced membranes were desalted approximately at the same time as the compressed ones without fabric, with the same mass of salt flow of salt, but with a higher current efficiency of the cell.

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