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Baromembrane technologies for purification of industrial wastes, using pulse water treatment

In this article the data of the purification of water steam condensate of the LLP «Pavlodar Petrochemical Plant» is considered. In the course of research multifunctional ion exchangers for pre-filters synthesized and were studied. A pilot reverse osmosis unit was produced with a capacity of 600 L per hours, and had been tested to clean steam condensate using a «Unit of Electromagnetic Treatment» (UET) and without it. The optimal treatment mode of purification was selected to the separation of the concentrate 0.1 m³ per hour. It was found that after desalination of water acidity value of pH changed to acidic medium (permeate) or to the basic medium in the concentrate tract, which associated by decreasing or increasing hydrocarbonate ions, accordingly, in permeate and concentrate. It was noted that the using of the UET at the inlet, led to a slight decreasing of the operating pressure inside the reverse osmosis unit, while on the reverse osmosis unit without UET it corresponded to the calculated value. It was established that the membranes used in testing by using the UET practically have no deposits on themselves, and without it they have a noticeable amount of contamination on their surface. Thus, using the UET in the process of reverse osmosis desalination can significantly increase the service life of membranes and maintain their selectivity, and the proposed method allows to achieve the required water purification standards set by the plant.

Keywords: anion exchanger, static exchange capacity, process flow, membrane elements, selectivity, pilot reverse osmosis unit, electromagnetic water treatment device, water sample, test protocol, antiscalant.

Introduction

Rapid industrialization and economic growth have significantly contributed to human welfare in the recent decades in contributing to industrial pollution and eradication of the natural resources around the world. In particular, the generation of large amounts of industrial effluents has considerably stressed the available water resources, thus raising a great concern not only in developing countries, but also worldwide. There are a number of pieces of evidence for subsequent toxic effects on aquatic organisms when industrial effluents are discharged into the environment without an effective treatment [1, 2].

The environmentally friendly methods recently used for obtaining water are ion exchange and reverse osmosis. These technologies can assure an obtainment of deeply purified water, with content of impurities much below the maximum permissible values according to hygienic standards and even below the limits of detection [3, 4].

Nowadays the membrane and sorption technologies are one of the most progressive and developing branches in chemical engineering [5, 6]. High efficiency of the using membrane processes in various industrial technologies, and their environmental compatibility contribute to rapid growth of the scientific and applied research. Also, the number and range of manufactured membranes and units are increased, as well as it associated in the funding allocated for these purposes in all economically developed countries. Membrane technology in its modern form has been entering in our life since the 70s of the 20th century.

One of the reasons for the intensive development of the membrane technology is its relatively low energy costs of separation processes [7,8]. In the forecasts of the world economy development, the membrane technologies are characterized as technologies of the future. The volume of their utilization in the economically developed countries increases annually by 20–25 % [9]. Thus, membranes in reverse osmosis and ultra-filtration, as compared with the other methods (evaporation, freezing, distillation), make it possible to carry out dehydration and concentration of the product, dispose of impurities, isolate the necessary substances and purify solutions. Herewith, the energy consumption is significantly reduced.

Among the membrane processes, which have been intensively developing in the recent years, baromembrane processes, such as reverse osmosis, ultra-, micro- and nano-filtration, hold a special place, which is connected with related to their universality and a wide range of application [10, 11]. They are characterized by

such features as low energy consumption, simplicity of the equipment design, an ability to work at the ambient temperature.

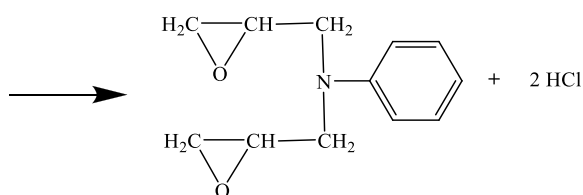
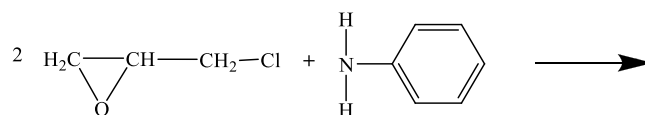
The Republic of Kazakhstan has the largest mineral and raw material resources and does not have sufficient sources of fresh water. At the same time, a number of regions of our country have large reserves of underground waters, which are not used for the needs of water supply due to the high content of salts, dissolved in water. These waters can become sources of water supply only in case of their further desalination.

That is why, the development of methods for producing new membranes, which are the basis of the desalination equipment, is of a big practical importance [12].

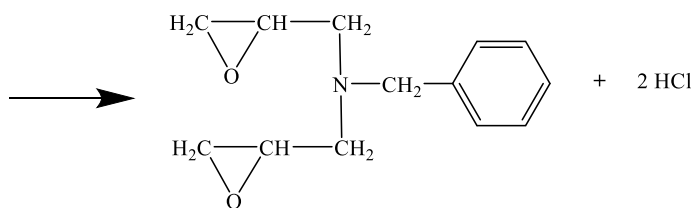
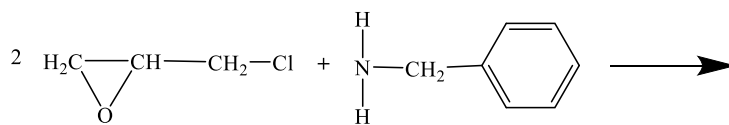
This work aims at the purification of the water steam condensate of Pavlodar Petrochemical Plant LLP (PPCP LLP), using the reverse osmosis method, combined with an electromagnetic water treatment device (EWTD) for ensuring a minimum volume of the concentrate (no more than 10 %), and preventing an intense salt deposition on the reverse osmosis membranes.

Experimental

Polyfunctional anion exchangers, based on aniline (A), benzylamine (BA) and polyethyleneimine (PEI) were obtained by the condensation with PEI of epoxyamines, synthesized from A or BA, and ECH. First, glycidyl derivatives of amines (epoxyamines) were synthesized from aniline (A) or benzylamine (BA), and epichlorohydrin (ECH) in the presence of NaOH at the temperature of 50 °C for 6 hours:

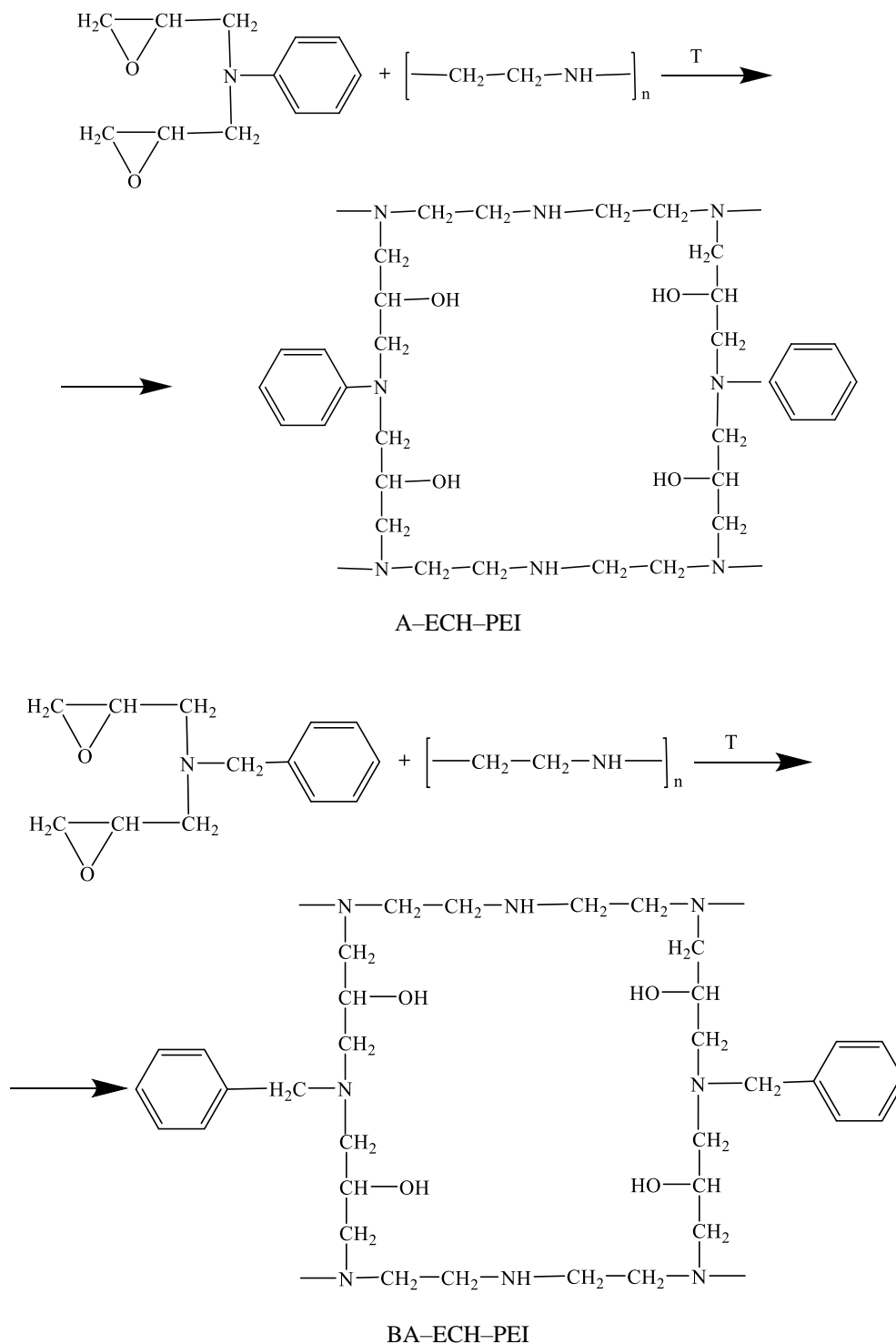


A-ECH



BA-ECH

The polycondensation with PEI was carried out in a solution of dimethylformamide (DMF) at different mass ratios, at the temperature of 60–65 °C for 5–6 hours, after which the reaction mass was baked at the temperature of 100 °C for 16–24 hours.



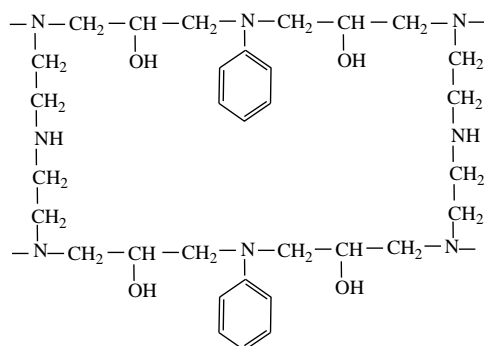
The elemental composition of anion exchangers (found/calculated), % for A-ECH-PEI: C — 73,32/73,86; H — 17,60/17,34; N — 5,89/5,60; O — 3,19/3,20 and BA-ECH-PEI: C — 70,72/70,92; H — 17,61/17,48; N — 7,81/8,09; O — 3,86/3,51.

The study of the main physical and chemical characteristics of ion exchangers, based on A-ECH-PEI and BA-ECH-PEI has been carried out according to the GOSTs 19180–73, 10898.1–74 and GOST 10898.5–74 on the enlarged batches, obtained under the optimal conditions.

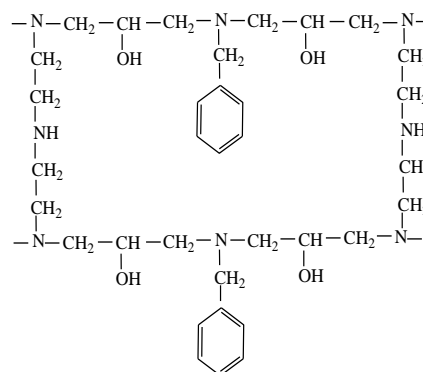
Results and Discussion

In the course of our studies, we have synthesized and investigated polyfunctional ion exchangers for pre-filters.

On the basis of the chemical analysis, the structure of the synthesized polymers may be represented as follows:



A-ECH-PEI



BA-ECH-PEI

Table 1 shows the main physicochemical characteristics of the synthesized anion exchangers A-ECH-PEI and BA-ECH-PEI.

Table 1

Main physicochemical characteristics of the synthesized anion exchangers

Anion exchangers based on	COE _{HCl} , mg-equ/g	V _{sp} , ml/g	Chemical stability in solutions, %			Thermal stability in water, %
			5 n H ₂ SO ₄	5 n NaOH	10 % H ₂ O ₂	
A-ECH-PEI	4.83	4.5	92.5	94.9	70.1	95.0
BA-ECH-PEI	8.95	5.7	97.9	98.7	72.0	94.1

Note. COE_{HCl} — static exchange capacity of an anion exchanger by 0.1 n solution of HCl; V_{sp} — specific volume of an ion exchanger.

Tests of efficiency of applying the electromagnetic water treatment technology have been carried out in the process of the reverse osmosis desalination of the water steam condensate of PPCP LLP. It has been found that with a decrease in the volume of the concentrate, the concentration of all salts in it increases, namely, the hardness salts in the concentrate form microcrystals with their isolation on the surface of a membrane element. As a result the productivity of the unit sharply decreases and the membrane elements should be «chemically» washed or replaced. This problem may be solved either with the help of reagents, introduced into the initial water, or with the help of magnetic pulse treatment of water in the process of reverse osmosis. To solve this problem it is necessary to create the conditions, when only the emerging microcrystals of the hardness salts will be in water, as if on a «magnetic pad», without isolating them on the membrane surface.

An electromagnetic transducer is designed for cleaning and protection against the deposits of the hardness salts. It can be used both independently and as an addition to the existing water treatment systems. The control unit has a printed board with a powerful electromagnet and a microprocessor, which generates electromagnetic pulses and controls the change in the frequency range from 1 kHz to 25–50 kHz. Electromagnetic waves of various lengths, amplitude and frequency, which vary over time, are transmitted through the wire-transmitters. An electromagnetic pulse is concentrated in the volume of water flowing in the pipeline where the coil is installed. Under the effect of electromagnetic waves, divalent cations of calcium and magnesium lose an ability to interact with anions and form insoluble compounds. The converted calcium and magnesium cations lose an ability to crystallize and precipitate. Herewith, the chemical and mineral composition of water does not change.

According to the goal to be sought, a set of documents for the test bench has been developed, including two reverse osmosis units, one of which is equipped with an electromagnetic water treatment device. A process flow has been developed, according to the fact that the initial water enters the tank, where from the water is

supplied to the two reverse osmosis units with a capacity of 600 l/h. In front of a high pressure pump — after the tie-in point of the recirculation line — an electromagnetic water treatment device is installed on the pipeline of one unit. The test bench operates in a closed circuit, i.e. permeate and brine return to their initial capacity. The controlled parameters of the test bench are: the capacity of permeate, concentrate, recirculation line, differential pressure at each circuit, TDS in the water steam condensate before the treatment, in permeate and concentrate thrice a day.

During the next researches, tests have been conducted for the purification of the steam condensate, taken from PPCP LLP, using EWTD and without it. An assembled pilot reverse osmosis unit with a capacity of 600 l/h is shown in Figure 1.

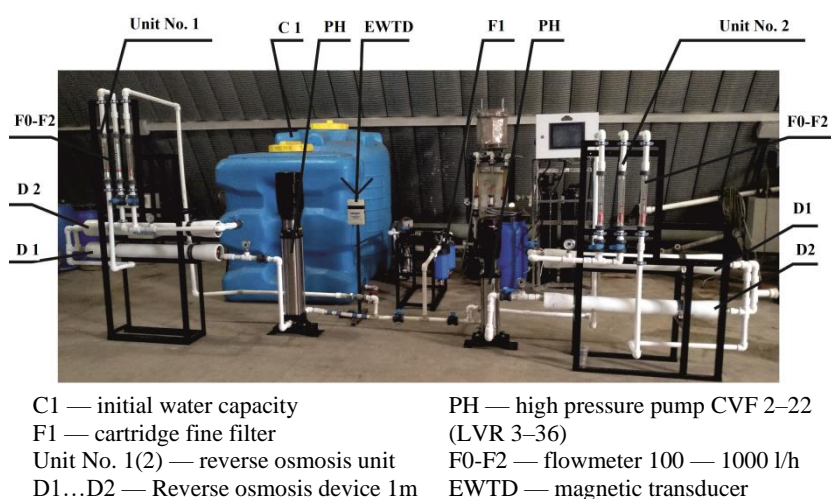


Figure 1. General view of a pilot reverse osmosis unit with a capacity of 600 l/h

Sampling of the steam condensate from PPCP LLP, amounting to 2500 L has been carried out. The obtained samples have been submitted for a complete chemical analysis to an independent certified testing laboratory of «Kazecologiya» Scientific Research and Production Center LLP (Almaty). According to the test protocol, the composition of the obtained water samples is as follows (Table 2).

Table 2

Data of the chemical analysis of the steam condensate samples from PPCP LLP

Parameters	Content
Salt content, mg/dm ³	238
Total hardness, mg-equ/dm ³	3.0
Iron, mg/dm ³	50
Oil products, mg/dm ³	0.8
pH — value	7.24

The required standards for the purified steam condensate requested by PPCP LLP are presented in Table 3.

Table 3

Required standards for the purified water steam condensate

Product	Parameters	Standard
Water steam condensate	Salt content, mg/dm ³	No more than 40
	Total hardness, mg-equ/dm ³	No more than 5
	Iron, mg/dm ³	No more than 0.5
	Oil products, mg/dm ³	No more than 0.3
	pH — value	6.8–7.5

Tables 4, 5 show the results of the tests, carried out on the reverse osmosis unit, combined with EWTD and without it.

During the tests at the pilot units, the optimal mode for desalinating the water steam concentrate has been selected, whose gist is as follows: a water flow with a capacity of $0.7 \text{ m}^3/\text{h}$ has been supplied to the inlet of the high-pressure pump of the reverse osmosis units, the permeate output has been $0.6 \text{ m}^3/\text{h}$, the discharge into the concentrate circuit has been $0.5 \text{ m}^3/\text{h}$, of which $0.4 \text{ m}^3/\text{h}$ has been fed back to the inlet to the high pressure pump via the recirculation circuit. Thus, the concentrate discharge has been equal to $0.1 \text{ m}^3/\text{h}$. Also, in the course of the repeated tests, the optimal volume of water flow in the recirculation circuit of the reverse osmosis units ($0.4 \text{ m}^3/\text{h}$) has been chosen.

It should be noted, that pH of the initial water has been equal to 7.24, after desalination of the water, pH has shifted to the acidic medium (permeate) and to the alkaline medium on the concentrate circuit, which is connected with a decrease and, respectively, an increase of hydrocarbonate ions in permeate and concentrate. It has been noted, that the use of EWTD at the inlet leads to a slight decrease in the working pressure inside the reverse osmosis unit, while at the reverse osmosis unit without EWTD, the working pressure corresponds to the calculated one. This effect is explained by the effect of electromagnetic waves on the ability of divalent calcium and magnesium cations to interact with anions to form insoluble compounds and their subsequent precipitation on the working surface of the reverse osmosis membranes (which, as a rule, leads to an increase in the working pressure as the membrane surface becomes polluted).

It has been found, that a pressure decrease is connected with a change in the salt content of the tested water. The water temperature also has an indirect effect. With a temperature increase of the water for osmosis, the medium becomes less viscous and dense, as a result, more mobile. This leads to the increase of the filtration rate, which negatively affects the membrane life. To ensure the maximum possible mode of operation of the membranes, it is necessary to have a temperature range between $15\text{--}20 \text{ }^\circ\text{C}$.

The following statement is actual for expressing the dependence of the diffusion rate of water molecules through the membrane walls, upon changing of the water temperature before osmosis: when the solution temperature changes by one degree (decreases or increases), the specific flow rate of the medium reaches 3 % of the set value. This change causes a negative effect on the filtration processes on the industrial scale, as the osmosis treatment units require constant capacity. The osmosis capacity is temperature dependent as follows: a decrease in the temperature reduces the capacity, and when it reaches $4 \text{ }^\circ\text{C}$, the rate drops approximately twice.

Upon the completion, the membrane elements have been removed for visual inspection and determination of the degree of microcrystal contamination. Figures 2–3 present the photographs of the membrane samples, using EWTD and without it.



Figure 2. Photo of the membrane samples after the tests, using EWTD



Figure 3. Photo of the membrane samples after the tests, without using EWTD

Table 4

Results of the tests of the steam condensate samples on the reverse osmosis unit, combined with EWTD

Date	Time	ΔP_{in} , bar	ΔP_{out} , bar	Q_{perm} , m ³ /h	Q_{conc} , m ³ /h	Q_{rec} , m ³ /h	TDS, (mg/l)			T, °C			pH		
							initial	perm.	conc.	initial	perm.	conc.	initial	perm.	conc.
09.12.19	9.00	6.8	6.8	0.5	0.07	0.3	20.2	12.0	143.7	7	7	7	7.24	6.31	6.93
09.12.19	12.00	6.8	6.8	0.5	0.07	0.3	20.2	11.9	143.7	7	8	8	7.24	6.34	6.93
09.12.19	15.00	6.7	6.7	0.5	0.07	0.3	20.2	11.9	143.7	8	8	8	7.24	6.32	6.93
10.12.19	9.00	6.8	6.7	0.5	0.07	0.3	20.2	12.2	144.0	6	6	6	7.24	6.31	6.92
10.12.19	12.00	6.8	6.8	0.5	0.07	0.3	20.2	11.7	144.2	6	7	7	7.24	6.34	6.96
10.12.19	15.00	6.7	6.7	0.5	0.07	0.3	20.2	12.0	143.7	8	8	8	7.24	6.34	6.93
11.12.19	9.00	6.8	6.7	0.5	0.07	0.3	20.2	12.0	143.7	8	8	8	7.24	6.34	6.93
11.12.19	12.00	6.7	6.7	0.5	0.07	0.3	20.2	11.9	143.7	8	8	8	7.24	6.32	6.94
11.12.19	15.00	6.8	6.7	0.5	0.07	0.3	20.2	11.7	144.0	8	8	8	7.24	6.32	6.92
12.12.19	9.00	6.8	6.8	0.5	0.07	0.3	20.2	12.0	144.2	7	7	7	7.24	6.34	6.96
12.12.19	12.00	6.7	6.7	0.5	0.07	0.3	20.2	11.7	143.7	7	7	7	7.24	6.34	6.93
12.12.19	15.00	6.8	6.8	0.5	0.07	0.3	20.2	12.0	144.1	7	8	8	7.24	6.31	6.93
13.12.19	9.00	6.8	6.8	0.5	0.07	0.3	20.2	11.7	143.7	8	8	8	7.24	6.34	6.93
13.12.19	12.00	6.7	6.7	0.5	0.07	0.3	20.2	11.9	143.7	8	8	8	7.24	6.31	6.93
13.12.19	15.00	6.7	6.7	0.5	0.07	0.3	20.2	12.2	144.2	8	8	8	7.24	6.31	6.93

Table 5

Results of the tests of the steam condensate samples on the reverse osmosis unit

Date	Time	ΔP_{in} , bar	ΔP_{out} , bar	Q_{perm} , m ³ /h	Q_{conc} , m ³ /h	Q_{rec} , m ³ /h	TDS, (mg/l)			T, °C			pH		
							initial	perm.	conc.	initial	perm.	conc.	initial	perm.	conc.
09.12.19	9.00	7.0	7.0	0.5	0.07	0.3	20.2	12.0	142.4	7	7	7	7.24	6.40	6.98
09.12.19	12.00	7.0	7.0	0.5	0.07	0.3	20.2	12.0	142.4	7	8	8	7.24	6.37	6.98
09.12.19	15.00	6.9	6.9	0.5	0.07	0.3	20.2	12.0	142.4	8	8	8	7.24	6.39	6.98
10.12.19	9.00	7.0	6.9	0.5	0.07	0.3	20.2	12.3	142.1	6	6	6	7.24	6.40	6.99
10.12.19	12.00	7.0	7.0	0.5	0.07	0.3	20.2	11.8	142.0	6	7	7	7.24	6.37	7.1
10.12.19	15.00	6.9	6.9	0.5	0.07	0.3	20.2	12.0	142.4	8	8	8	7.24	6.37	6.98
11.12.19	9.00	7.0	6.9	0.5	0.07	0.3	20.2	12.0	142.4	8	8	8	7.24	6.37	6.98
11.12.19	12.00	6.9	6.9	0.5	0.07	0.3	20.2	12.0	142.4	8	8	8	7.24	6.39	7.2
11.12.19	15.00	7.0	6.9	0.5	0.07	0.3	20.2	11.8	142.1	8	8	8	7.24	6.39	6.99
12.12.19	9.00	7.0	7.0	0.5	0.07	0.3	20.2	12.0	142.0	7	7	7	7.24	6.37	7.1
12.12.19	12.00	6.9	6.9	0.5	0.07	0.3	20.2	11.8	142.4	7	7	7	7.24	6.37	6.98
12.12.19	15.00	7.0	7.0	0.5	0.07	0.3	20.2	12.0	142.1	7	8	8	7.24	6.40	6.98
13.12.19	9.00	7.0	7.0	0.5	0.07	0.3	20.2	11.8	142.4	8	8	8	7.24	6.37	6.98
13.12.19	12.00	6.9	6.9	0.5	0.07	0.3	20.2	12.0	142.4	8	8	8	7.24	6.40	6.98
13.12.19	15.00	6.9	6.9	0.5	0.07	0.3	20.2	12.3	142.0	8	8	8	7.24	6.40	6.98

It is seen in the above photographs, that the membranes, which have been tested, using EWTD, practically have no deposits thereon, and those, tested without using EWTD, have a noticeable contamination on their surface. Hence it follows, that the use of EWTD in the process of the reverse osmosis desalination can significantly increase the service life of the membranes, and maintain their selectivity.

Conclusions

Thus, a highly effective technology for purifying the water steam condensate of Pavlodar Petrochemical Plant LLP has been developed on the basis of the reverse osmosis method, combined with the electromagnetic water treatment device. During the tests at the pilot units, the optimal mode for desalinating the water steam concentrate has been selected. It has been established, that the use of this technology, which is based on pulsed water treatment technique, makes it possible to minimize the volume of the concentrate and contributes to preventing an intense salt deposition on the reverse osmosis membranes. Herewith, a high efficiency of water purification and an increase in the concentration coefficient are achieved and therefore service life of these membranes is increased with preservation of their highly selective properties.

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Суды импульстік өңдеуде қолдана отырып, өнеркәсіптік ағындарды тазартудың баромембранды технологиялары

Мақалада «Павлодар мұнай-химия зауыты» ЖШС су бу конденсатын тазарту бойынша деректер келтірілген. Жүргізілген зерттеулер барысында алдын-ала тазалау сүзгілеріне арналған полифункционалды иониттер синтезделген және зерттелген. Өнімділігі 600 л/с пилоттық керіосмостық кондырғы дайындалды, онда электромагнитті өңдеу құралын (ЭӨҚ) қолдана отырып және онсыз бу конденсатын тазалау бойынша сынақтар жүргізілген. Тазалаудың оңтайлы режимі таңдап алынған, ол бойынша концентратты шығару 0,1 м³/сағ. құрайды. Суды тұщыландырғаннан кейін рН қышқыл ортаға (пермеат) және концентратты жолда сілтілі ортаға ығысуы орын алды, бұл пермеат пен концентраттағы гидрокарбонат-иондардың азаюымен және тиісінше ұлғаюымен байланысты. ЭӨҚ-ны кіреберісте пайдалану керіосмос кондырғысының ішіндегі жұмыс қысымының аздап төмендеуіне әкелді, ал ЭӨҚ-сыз керіосмос кондырғысында ол есептелгенге сәйкес келді. ЭӨҚ-ны қолдану арқылы сынау процесінде пайдаланылатын мембраналардың іс жүзінде шөгінділері жоқ, ал ЭӨҚ-ны пайдаланбаса олардың бетінде ластанудың елеулі мөлшері бар екені анықталған. Сонымен, керіосмос тұзсыздандыру процесінде ЭӨҚ-ны қолдану мембраналардың қызмет ету мерзімін едәуір арттырып, олардың селективтілігін сақтауға мүмкіндік береді, ал ұсынылған әдіс зауыт орнатқан суды тазарту стандарттарына қол жеткізуге көмектеседі.

Кілт сөздер: анионит, статикалық алмасу сыйымдылығы, технологиялық схема, мембрандық элементтер, селективтілік, пилоттық керіосмос кондырғысы, электромагниттік су тазарту құрылғысы, сынақ есебі, антискалант.

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Баромембранные технологии очистки промышленных стоков с применением импульсной обработки воды

В статье приведены данные по очистке водяного парового конденсата ТОО «Павлодарский нефтехимический завод». В ходе проведенных исследований синтезированы и исследованы полифункциональные иониты для фильтров предварительной очистки. Изготовлена пилотная обратноосмотическая установка, производительностью 600 л/ч, на которой проведены испытания по очистке парового конденсата с применением прибора электромагнитной обработки (ПЭО) и без него. Подобран оптимальный режим очистки, по которому сброс концентрата составлял 0,1 м³/ч. Найдено, что после опреснения воды происходил сдвиг pH в кислотную среду (пермеат) и в щелочную среду на концентратном тракте, что связано с уменьшением и, соответственно, увеличением гидрокарбонат-ионов в пермеате и концентрате. Отмечено, что использование ПЭО на входе привело к незначительному уменьшению рабочего давления внутри обратноосмотической установки, в то время, как на обратноосмотической установке без ПЭО, оно соответствовало расчетному. Установлено, что мембраны, используемые в процессе испытаний с применением ПЭО, практически не имеют на себе отложений, а без использования ПЭО обладают заметным количеством загрязнений на их поверхности. Таким образом, использование ПЭО в процессе обратноосмотического опреснения позволяет значительно увеличить срок службы мембран и сохранить их селективность, а предложенный способ помогает достичь требуемых норм очистки воды, выведенных заводом.

Ключевые слова: анионит, статическая обменная емкость, технологическая схема, мембранные элементы, селективность, пилотная обратноосмотическая установка, прибор электромагнитной обработки воды, проба воды, протокол испытаний, антискалант.

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