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РЕГЕНЕРАЦИЯ УЛЬТРАФИЛЬТРАЦИОННЫХ МЕМБРАН В ПРОЦЕССЕ РАЗДЕЛЕНИЯ ВОДОМАСЛЯНЫХ СИСТЕМ

А.В. Маркелов, А.В. Соколов

Александр Владимирович Маркелов (ORCID 0000-0001-7125-6570)*, Александр Викторович Соколов (ORCID 0000-0001-9124-6329)

Кафедра автомобильного транспорта, Ярославский государственный технический университет, Московский просп., 88, Ярославль, Российская Федерация, 150023
E-mail: aleksandr203.37@mail.ru *, sokolovalexandre@mail.ru

Для очистки отработанных нефтесодержащих продуктов предложено использовать баромембранный процесс. В качестве фильтрующих элементов использовались трубчатые ультрафильтрационные полимерные мембраны с активным слоем из фторопласта с добавками из диатомита и керамические мембраны на основе Al_2O_3. В процессе эксплуатации мембранных элементов происходит падение удельной производительности, связанное с их загрязнением. Загрязнение ультрафильтрационных мембран рассматривается как необратимое отложение высокомолекулярных веществ, нефтепродуктов и жиров на поверхности или в порах мембраны. Однако при правильной технической эксплуатации возможно увеличение срока службы мембран. В работе рассматриваются две стратегии технического обслуживания мембранных элементов, основанных на их промывке (регенерации) через определенные промежутки времени. Выбор способа и режима регенерации мембранных элементов определяется в зависимости от типа материала мембран и от конкретных рабочих условий их эксплуатации. Для регенерации полимерных трубчатых мембран, ввиду их анизотропной конструкции и непрочной связи между активным (тонкопористым) и дренажным (крупнопористым) слоями предлагается химический способ промывки с использованием в качестве реагента керосина. Для керамических мембран, ввиду особенности их производства путем спекания при высоких температурах, возможны два способа регенерации: физический и химический. Физический способ заключался в использовании обратной промывки мембранных пор пермеатом. Эксперименты показали, что наиболее эффективным методом регенерации мембранных элементов при падении удельной производительности на 30% является обратная промывка. Этот метод не требует использования дополнительных реагентов и, следовательно, не предполагает их последующей утилизации.

Ключевые слова: отработанные масла, ультрафильтрация, полимерные и керамические мембраны, удельная производительность, регенерация мембран

REGENERATION OF ULTRAFILTRATION MEMBRANES IN THE PROCESS OF SEPARATING OIL-WATER SYSTEMS

A.V. Markelov, A.V. Sokolov

Alexandr V. Markelov (ORCID 0000-0001-7125-6570)*, Alexandr V. Sokolov (ORCID 0000-0001-9124-6329)

Department of Road Transport, Yaroslavl State Technical University, Moskovsky ave., 88, Yaroslavl, 150023, Russia
E-mail: aleksandr203.37@mail.ru *, sokolovalexandre@mail.ru

It is proposed to use a baromembrane process for the purification of spent oil-containing products. Tubular ultrafluorescence polymer membranes with an active layer of fluoroplast with diatomite additives and ceramic membranes based on Al_2O_3 were used as filter elements. During the operation of membrane elements, there is a drop in specific productivity associated with their contamination. Contamination of ultrafiltration membranes is considered as irreversible deposition of high-molecular substances, petroleum products and fats on the surface or in the pores of the membrane. However, with proper technical operation, it is possible to increase the service life

of the membranes. The paper considers two strategies for maintenance of membrane elements based on their washing (regeneration) at certain intervals. The choice of the method and mode of regeneration of membrane elements is determined depending on the type of membrane material and on the specific operating conditions of their operation. For the regeneration of polymer tubular membranes due to their anisotropic design and the fragile connection between the active (fine-porous) and drainage (coarse-porous) layers, a chemical washing method using kerosene as a reagent is proposed. For ceramic membranes, due to the peculiarities of their production by sintering at high temperatures, two regeneration methods are possible: physical and chemical. The physical method consisted in using permeate backwash of membrane pores. Experiments have shown that it is most effective to regenerate membrane elements with a 30% drop in specific productivity by backwash, since it does not require the use of additional reagents and their subsequent disposal.

Key words: waste oils, ultrafiltration, polymer and ceramic membranes, specific productivity, membrane regeneration

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INTRODUCTION

Enterprises of construction, motor transport, chemical, textile, engineering and other industries are sources of pollution of water bodies and soils with organic substances that are difficult to oxidize [1, 2].

The operation of road construction equipment, vehicles and equipment leads to the formation of a large amount of used motor, transmission, industrial and hydraulic oils [3]. The consumption of motor oils tends to increase due to the growth of the vehicle fleet, which is the main supplier of this type of waste, therefore, in the developed countries of Europe and North America, a number of resource-saving and organizational and economic measures have been developed aimed at reducing the increase in its consumption. The content of up to 90% of the oil base in used motor oils compared to 10-20% in crude oil, leads to a lower cost of regenerated oil compared to fresh oil production. For this reason, used motor oils in developed countries are considered as a raw material base for the production of petroleum products [4, 5].

In order to organize low-waste production, it is proposed to regenerate spent toxic waste using membrane separation processes, such as microfiltration (MF) and ultrafiltration (UF), which are more efficient and economical than conventional separation methods. Recently, all over the world, baromembrane (BM) technologies have been used for desalination of sea water to drinking water, wastewater treatment in order to isolate valuable components, for concentration, purification and separation of solutions of macromolecular compounds in various industries [6].

The economic feasibility of using ultrafiltration to separate oil-water systems is largely limited by the phenomenon of blockage of a semi-permeable partition [7].

Without the development of special operating conditions for membranes, they will be clogged in a short time. Identification and quantification of contaminants are necessary to elucidate the fouling mechanism and membrane regeneration strategy [8-10].

A wide variety of methods are used to combat the deposition of solutes on the membrane. The main methods include chemical methods - washing with the help of reagents, and physical ones - changing the hydrodynamics of the separated flow, ultrasonic and vibration exposure, backwashing [7, 11-16].

The choice of the method and mode of regeneration of membrane elements is determined depending on the specific conditions of their operation.

A feature of the design of polymer tubular membranes is the impossibility of using backwash, but only washing with the use of chemical reagents.

For ceramic membranes, both methods of regeneration are possible [16-21].

MATERIALS AND METHODS

Used Lukoil (M-14D2) engine oil was used to conduct experiments using the method of reagent and non-reagent flushing. Samples were taken from diesel locomotives after 300 h of operation in the maintenance areas at the Operational Ivanovo Depot of the Northern Railway of Russian Railways. The physical and chemical properties of used oil are presented in Table 1.

Table 1
Characteristics of engine oil M-14D2 and waste oil
***Таблица 1*. Характеристики моторного масла М-14Д2 и отработанного масла**

Defined indicator, unit	Oil M-14D2	Oil spent
Kinematic viscosity at 100 °C, mm^2/s	13.32	13.77
Flash point in an open crucible, °C	258	225
Hydrogen index, pH	not standardized	7.72
Base number in mg/KOH per 1 mg of oil	14.3	9.02
Mass fraction of water, %	traces	traces
Optical density, cm^{-1}	not standardized	234
Mass fraction of mechanical impurities, %	absence	0.04
Density actual at 24.5 °C, kg/m^3	889	895

Before separation, engine oils were pretreated by coagulation by introducing a 50% aqueous solution of diaminometanal (0.5% wt.) and centrifugal separation (separation factor K_p = 8000). The analysis of suspended particles in used oil after pretreatment is shown in Fig. 1 [10, 22].

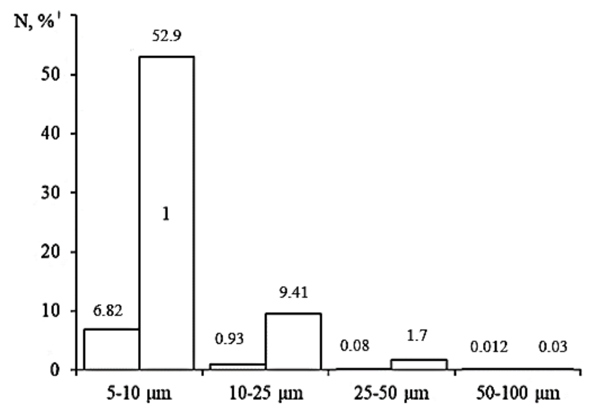

Fig. 1. Distribution of suspended particles in waste oil after pretreatment
Рис. 1. Распределение взвешенных частиц в отработанном масле после предварительной обработки

As a filtering material, ultrafiltration ceramic monotube membranes with a selective layer based on Al_2O_3 and a pore size of 0.01 µm, manufactured by Cer amicfilter LLC, Moscow, were used.

BACKWASH METHOD

Currently, special attention is directed to the study of a method for controlling deposits in membrane processes using backwashing. This method does not involve the use of chemical reagents and synthetic detergents and requires minimal changes in the design of apparatus for the separation of solutions.

Backwashing consists in supplying purified oil to the annulus of the membrane module through the permeate line. At the same time, the purified oil penetrates through the membrane in the opposite direction (relative to the filtration mode) – from the outside to the inside. The flow of waste oil separation and backwashing is schematically shown in Fig. 2.

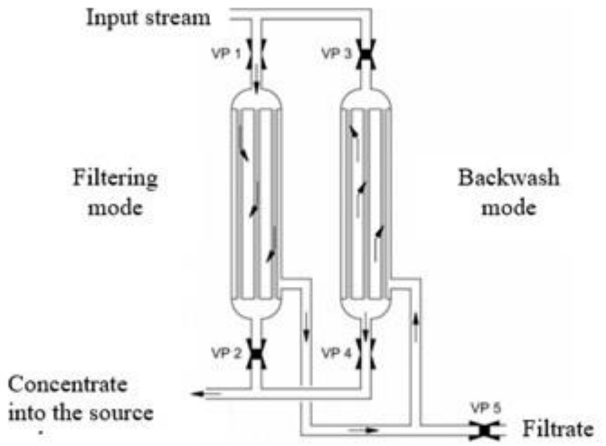

Fig. 2. Scheme of flow movement during separation waste oils and membrane backwash
Рис. 2. Схема движения потоков при разделении отработанных масел и обратной промывки мембран

Passing through the membrane at ΔP_{op} = 0.2 MPa, the purified oil destroys the accumulated deposits on the inner surface of the tubular element, and is carried back to the original reservoir by the waste oil flow at ΔP = 0.1 MPa. Using this technology, it is possible to regenerate membrane modules one by one without stopping the separation process in automatic mode, which increases the productivity of the plant and significantly reduces the cost of operating the equipment.

REAGENT WASHING METHOD

Experiments on the reagent cleaning of membranes were carried out with kerosene, in which the products of aging oils dissolve well.

Washing with kerosene was carried out for 1 min according to the method of a fixed drop in productivity (by 30%). To do this, the waste oil separation process is turned off, the membrane module is connected to the reagent flushing line (Fig. 3).

RESULTS AND DISCUSSION

BACKWASHING OF MEMBRANES

The specific productivity of the installation at the beginning of the experiment K_0 was 14.8 $dm^3/(m^2h)$ at a temperature T = 363 K.

The study of the frequency of washing was carried out according to two methods: by the time of separation and by reaching a drop in specific productivity by 30%.

In the first case, filtration was carried out for 60 min, after which the membranes were backwashed. The total time of the experiment was 240 min. The experimental results are presented in Table 2 and Fig. 4.

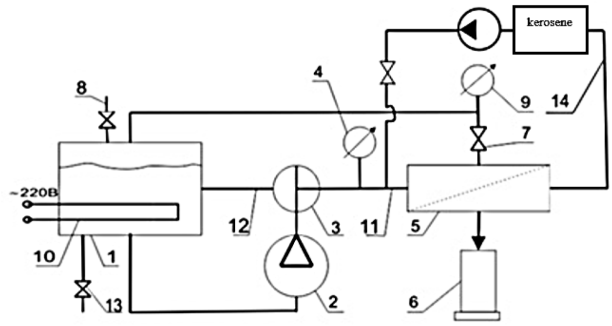

Fig. 3. Schematic diagram of the installation with a reagent flush line:1 - reservoir with used engine oil; 2 - pump, 3 - three-way valve; 4.9 - pressure gauges; 5 - membrane elements; 6-dimensional flask with purified oil; 7 - shutoff valves; 8 - tap for filling used oil; 10 - electric heater; 11 - pressure line; 12 - bypass line; 13 - drain valve; 14 - reagent flush line

Рис. 3. Принципиальная схема установки с линией реагентной промывки: 1 – резервуар с отработанным моторным маслом; 2 – насос, 3– трехходовой кран; 4,9 – манометры; 5 – мембранные элементы; 6 –мерная колба с очищенным маслом; 7 – запорная арматура; 8 – кран для залива отработанного масла; 10 – электрический тэн; 11 – напорная магистраль; 12 - байпасная магистраль; 13 – вентиль сливной; 14 – линия реагентной промывки

Table 2

The results of the study of the frequency of flushing at a fixed time

***Таблица 2.* Результаты исследования периодичности промывки по фиксированному времени**

Duration of the experiment τ, min.	Unit productivity, dm^3/h	Unit productivity Ki, $dm^3/(m^2h)$	K_i/K_0
0	4.9	14.8	1.00
60	1.48	4.46	0.30
Regeneration by backwash, τ = 1min, ΔP_{op} = 0.2 MPa			
60	4.8	14.1	0.95
120	1.57	4.97	0.34
Regeneration by backwash, τ = 1 min, ΔPop = 0.2 MPa			
120	4.02	12.07	0.82
180	1.57	4.97	0.34
Regeneration by backwash, τ = 1min, ΔP_{op} = 0.2 MPa			
180	3.4	10.21	0.69
240	1.31	3.95	0.27

In the second case, backwashing was carried out after a drop in productivity by 30% in the same mode as in the first method. The results of the experiments are shown in Table 3.

For the convenience of comparing the experimental data obtained by two methods of backwashing and presented in Tables 1 and 2, graphs are plotted in Fig. 4.

Table 3

Results of backwash experiments up to a 30% drop in performance

***Таблица 3.* Результаты экспериментов при обратной промывке до падения производительности на 30%**

The duration of the experiment τ, min.	Unit productivity, dm^3/h	Specific unit productivity K_i, $dm^3/(m^2h)$	K_i/K_0
0	4.9	14.8	1.00
45	3.43	10.29	0.69
Regeneration by backwash, τ =1min, ΔP_{op} = 0.2 MPa			
45	4.86	14.578	0.98
88	3.4	10.2	0.69
Regeneration by backwash, τ = 1 min, ΔP_{op} = 0.2 MPa			
88	4.85	14.55	0.98
130	3.39	10.18	0.69
Regeneration by backwash, τ =1min, ΔP_{op} = 0.2 MPa			
130	4.84	14.51	0.98
175	3.38	10.16	0.69
Regeneration by backwash, τ = 1 min, ΔP_{op} = 0.2 MPa			
175	4.83	14.5	0.98
215	3.38	10.14	0.69
Regeneration by backwash, τ =1min, ΔP_{op} = 0.2 MPa			
215	4.83	14.48	0.98
255	3.38	10.14	0.69

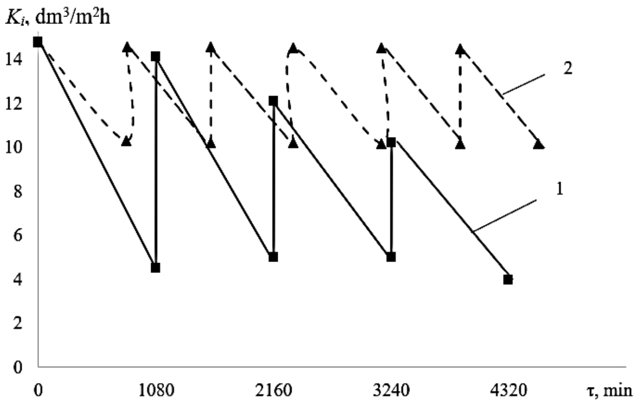

Fig. 4. Comparison of the values of the specific productivity of membranes after backwashing: 1 - by the fixed time method; 2 - by the method of fixed performance drop

Рис. 4. Сравнение значений удельной производительности мембран после обратной промывки: 1 – методом фиксированного времени; 2 – методом фиксированного падения производительности

As can be seen from the graphs in Fig. 2, the second method is the most effective (according to a fixed drop in productivity), because allows you to restore productivity by 98% over a long period of operation.

The fixed time method leads to an irreversible drop in productivity, after which it is necessary to dismantle the tubular elements and regenerate them by annealing. This type of regeneration is possible, but it requires more time, stops the separation process and greatly increases operating costs.

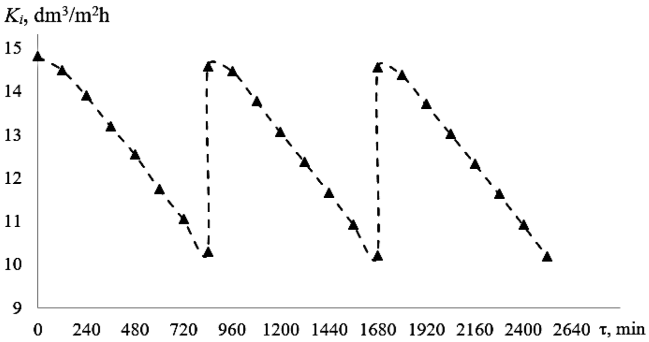

Fig. 5. Results of chemical washing of ceramic tubular membranes at a temperature of 293 K, washing pressure ΔP_{prom} = 0.15 MPa
Рис. 5. Результаты реагентной промывки керамических трубчатых мембран при температуре 293 К, давлении промывки $\Delta P_{пром}$ = 0,15 Мпа

REAGENT WASHING

The specific productivity of the installation at the beginning of the experiment K0 was 14.8 $dm^3/(m^2h)$ at a temperature T = 363 K.

The study of the frequency of flushing was carried out after reaching a drop in specific productivity by 30%. The results of the reagent wash experiments are shown in Fig. 5.

The reagent wash results show good efficiency. The specific productivity was restored to 98% of the original value.

CONCLUSIONS

As a result of experiments on washing membrane elements, it was concluded that backwashing for ceramic membranes after a drop in productivity by 30% is the most effective and is recommended for use in the engineering design of ultrafiltration separation devices. used oils.

The disadvantage of the method using a reagent for washing is the additional cost of purchasing a solvent, the need for its disposal, as well as stopping the separation process.

Авторы заявляют об отсутствии конфликта интересов, требующего раскрытия в данной статье.

The authors declare the absence a conflict of interest warranting disclosure in this article.

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