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Separation of oil-containing effluents of construction industry enterprises

A.V. Markelov[1], D.V. Volkov[1], E.B. Korobova[2]

Alexander V. Markelov
[1]Yaroslavl State Technical University, Yaroslavl, Russia
aleksandr203.37@mail.ru

Danil V. Volkov
[1]Yaroslavl State Technical University, Yaroslavl, Russia
volkovdv@ystu.ru

Elena B. Korobova
[2]Limited Liability Company "Transportnaya Lisingovaya Companiya", Yaroslavl region, Shchedrino, Russia
lena-korobova@inbox.ru

The paper considers the influence of different input parameters on the ultrafiltration process of oily wastewater from reinforced concrete structures manufacturing plants, which are water-oil systems. Also we defined quantitative and qualitative indicators of the wastewater composition as the concentration of pollutants, particle sizes of pollutants, density, hydrogen index, specific electrical conductivity (SEC), etc. Based on this analysis, we determined the choice of material and physical characteristics of the membranes, the development of technological processes for the treatment of oily wastewater for specific production conditions. The study contains data on quantitative and chemical analysis of oily wastewater; an experimental installation for separation into components of water-oil systems; the effect of operating pressure, medium temperature, and flow velocity on the ultrafiltration process kinetics; experimental studies on separation of oily wastewater.

Key words: oily waste water, ultrafiltration, ceramic membranes

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НАУЧНАЯ СТАТЬЯ
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Разделение нефтесодержащих стоков предприятий строительной отрасли

А.В. Маркелов[1], Д.В. Волков[1], Е.Б. Коробова[2]

Александр Владимирович Маркелов
[1]Ярославский государственный технический университет, Ярославль, Российская Федерация
aleksandr203.37@mail.ru

Данил Владимирович Волков
[1]Ярославский государственный технический университет, Ярославль, Российская Федерация
volkovdv@ystu.ru

Елена Борисовна Коробова
[2]Общество с ограниченной ответственностью «Транспортная лизинговая компания», Ярославская область, пос. Щедрино, Российская Федерация
lena-korobova@inbox.ru

Исследовано влияние различных входных параметров на процесс ультрафильтрации нефтесодержащих сточных вод предприятий по производству железобетонных конструкций, представляющих собой водомасляные системы. Определены концентрация загрязняющих веществ в нефтесодержащих сточных водах, размеры частиц загрязнений, плотность, водородный показатель, удельная электрическая проводимость (УЭП). На основе проведенного анализа получены аргументированные ответы на вопросы, связанные с выбором материала и физических характеристик мембран, разработке технологических процессов по очистке нефтесодержащих сточных вод для конкретных производственных условий. Создана экспериментальная установка по разделению на компоненты водомасляных систем, позволяющая оценить влияние рабочего давления, температуры среды и скорости потока на кинетику процесса ультрафильтрации.

Ключевые слова: нефтесодержащие сточные воды, ультрафильтрация, керамические мембраны

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INTRODUCTION

The construction industry, which consumes a huge amount of water resources, effluents of various compositions and with different physico-chemical properties are formed [1-4].

The reuse of wastewater in the water recycling system reduces discharges into water bodies. According to the principle of permissible use in recycling water supply wastewater is divided into groups A and B [1-4].

Group A includes wastewater can be used in the general water recycling system, i.e. contaminated only with easily oxidised substances[1-4] – after cooling of surface refrigerators and condensers, glands of pumps and other units of various machines, condensates from steam injectors, water steam distillation, settling water of distillate products, etc. Water of this type can be discharged to the sewerage system of technological units through local settling tanks. These waters may also include rainwater, water from flushing of floors, technological and construction sites. Water of this type must be routed to the recycling water system via settling tanks.

Group B is wastewater which cannot be diverted into the general water recycling system. These waters are polluted with non-volatile salts, acids, alkalis, and oil products (OP), as well as organic substances with increased solubility in water or resistance to oxidation by air oxygen. Wastewaters of this group are divided into four types according to the composition of pollutants and the nature of their effect on water bodies and aquatic organisms [1- 4].

Petroleum products are the most hazardous substances of anthropogenic origin. The operation of construction, road and motor-tractor machinery, enterprises producing reinforced concrete structures and sand-lime bricks, machine-building industry, petrol stations, as well as accidents at oil storages and refineries, pipelines lead to pollution of water resources and create a serious ecological threat to any region [6-12].

Wastewater may contain gasoline, kerosene, fuel and lubricating oils, benzene, toluene, xylenes, fatty acids, phenols, glycerides, steroids, pesticides, and organometallic compounds [6-12], which is about 90% of the total amount of all organic impurities.

Wastewater is divided into low-concentrated and concentrated wastewater in terms of petroleum products concentration. Low-concentrated wastewater is produced when metal products are washed after heat treatment and canning. At many enterprises concentrated oil-containing wastewater is diluted with a large amount of conditionally clean water and becomes low-concentrated. The content of oil in them varies from 10 to 500 mg·dm^{-3} [1, 13, 14].

Oil-contaminated wastewater mainly contains used cleaning solutions and used cutting fluids (coolants). They are oil-in-water emulsions consisting of industrial oils, asidol, ethylene glycol, sodium nitrite, etc. [1, 6-14]. Indeed, coolants contain various stabilisers as well as a large number of additives (anticorrosive, antibacterial, extreme pressure, etc.) [13, 14].

The main reasons for changing coolants are the presence of large amounts of suspended solids (metal dust, abrasive particles), loosening and rotting. Emulsion heterogeneity indicates the significant presence of "alien" oil, while the occurrence of a putrid (hydrogen sulphide) odour indicates the water-oil emulsion (WME) is affected by bacteria [13, 14].

According to the Federal Waste Classification Catalogue, waste water-oil emulsions are Hazard Class III wastes [1]. According to the order of the Ministry of Natural Resources of the Ministry of Natural Resources (MNR) of Russia from June 15, 2001 No. 511, the multiplicity of dilution with clean water, in which there is no harmful effect on hydrobiota, is 101-1000 times. Such wastewater can be treated by mechanical, physico-chemical, chemical, and biological methods [6-14].

Coarse impurities (suspended solids, floating oil products, etc.) are removed mechanically.

These include sedimentation, filtration, and centrifugation. The finely dispersed, colloid-dissolved, and dissolved impurities are separated by coagulation, flotation, sorption, aeration, ion exchange (physical-chemical methods), ultrafiltration, electrodialysis, ozonation, reagent treatment, softening, etc. (chemical methods) [6-13].

Currently, membrane methods of liquid separation have been greatly developed. They are characterized by simplicity of hardware design, low energy consumption, non-reactivity, etc. [13-17].

The purpose of this work is an experimental assessment of oily wastewater purification possibility from construction industries by ultrafiltration.

THE EXPERIMENTAL PART

Semi-permeable polymer ultrafiltration membranes of tubular type manufactured by RPA Vladipor made of fluoroplast, polysulfone, polyestersulfone, polysulfonamide, polyvinyl chloride, modified polyvinyl chloride, manufactured according to TS 6559-88, 605-221-734-83, 655-4-88, and ceramic monotube membranes with a selective layer based on Al_2O_3 manufactured by LTD Keramikfilter were used as the filter material (Russia, Moscow).

Wastewater from steaming chambers and forming stations of reinforced concrete structures containing oil products (emulsol) was used as an object of investigation. The samples were taken at the company for production of reinforced concrete structures of CBS Holding (Ivanovo, Russia).

To conduct experimental studies on the baromembrane separation of water-oil systems, a laboratory installation was created (Fig. 1).

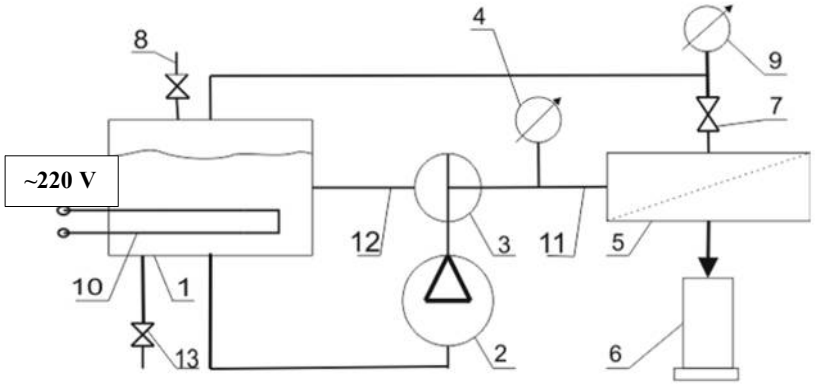
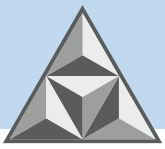


Fig. 1. Schematic diagram of a laboratory installation for the separation of water-oil emulsions:
1 – a tank with spent engine oil; 2 – a pump; 3 – a three-way valve; 4, 9 – pressure gauges, respectively, at the inlet and outlet of the filter module; 5 – ultrafiltration module; 6 – dimensional flask with permeate; 7 – shut-off valves; 8 – a tap for filling waste oil; 10 – heating device; 11 – pressure line; 12 – bypass line; 13 – drain valve

Рис. 1. Принципиальная схема лабораторной установки по разделению водомасляных эмульсий:
1 – резервуар с отработанным моторным маслом; 2 – насос; 3 – трехходовой кран; 4, 9 – манометры на входе и выходе из фильтрующего модуля; 5 – ультрафильтрационный модуль; 6 – мерная колба с пермеатом; 7 – запорная арматура; 8 – кран для залива отработанного масла; 10 – нагревательный прибор; 11– напорная магистраль; 12 – байпасная магистраль; 13 – сливной вентиль

The initial solution for separation is pumped by pump 2 from tank 1 to the tubular membrane element 5. The ultrafiltrate (permeate) is collected in a volumetric flask 6. The solution is heated with electric heater 10. Pressure in the system is controlled by manometers 4, 9. To supply the working solution to the membrane element the pump is switched on and the handle of three-way valve 3 is turned. To change the pressure in the system turn the handle of the three-way valve making the connection of the main pressure line 11 with the bypass line 12.

The concentrate is returned to tank 1 by opening shut-off valve 7. Drain the concentrated



solution from the original tank 1 after separation by means of valve 13.

This laboratory installation allowed conducting studies of wastewater treatment from petroleum products under various process modes: pressure drop Δp = 0.1-0.6 MPa; solution temperature t = 20-90 °C, separated flow velocity V = 1-5 m·s^{-1}.

For the physico-chemical analysis, water-emulsion effluents of the holding of the Combine of Building Structures (Russia, Ivanovo) containing grease for molds and formwork of the Polyplast Forms brand type 3 were used.

According to TS 0258-038-58042865-2009 Polyplast Form type 3 contains in its composition mineral oils, corrosion inhibitors, active film-forming additives, hydrocarbons (paraffins, isoparaffins, naphthenes, oleic acid), organic alcohols (isopropyl, butyl) [18]. Polyplast Form type 3 is a ready-to-use universal grease for all kinds of forms (horizontal and vertical application). It is manufactured by Polyplast-UralSib, LTD (Russia, Pervouralsk, Sverdlovsk region).

In order to obtain reliable results three wastewater samples were taken from the storage tanks of the production base of Combinat of Building Structures (CBS, Russia, Ivanovo) for the manufacture of reinforced concrete structures in reusable metal moulds before discharge of such water to the wastewater treatment plant. The results of the physico-chemical analysis of samples No. 1, No. 2, No. 3 are presented in Table 1.

Table 1. Results of physico-chemical analysis of the water-oil emulsions of the holding KSK (Ivanovo, Russia)
Таблица 1. Результаты физико-химического анализа ВМЭ холдинга КСК (Россия, г. Иваново)

Indicator	Standard	*With* ± Δ mg·dm^{-3}, P = 0.95, n = 2		
		Sample No. 1	Sample No. 2	Sample No. 3
pH,	6.0-9.0	8.3±0.1	8.2±0.1	8.9±0.1
Suspended substances, mg·dm^{-3}	300	2316±463	572±114	1554±233
Dry residue, mg·dm^{-3}	3000	20500±4100	16233±3247	19900±3980
Chemical oxygen consumption, mg O_2·dm^{-3}	500	18760±3752	15600±3120	18215±3643
Content:	Standard*			
Nitrate ions, mg·dm^{-3}	Not regulated	183.0±36,6	51.6±10.3	54.6±11.0
Nitrite ions, mg·dm^{-3}	Not regulated	4.61±0.92	2.12±0.42	2.91±0.582
Sulphate ions, mg·dm^{-3}	300	188.0±37.6	71.8±14.4	121±24
Phosphate ions, mg·dm^{-3}	Not regulated	13.10±2.62	15.20±3.04	24.30±4.86
Chloride ions, mg·dm^{-3}	1000	202±40	175±35	280±56
Fluoride ions, mg·dm^{-3}	Not regulated	36.5±7.3	39.4±7.9	54.6±11.0
Petroleum products, g·dm^{-3}	10	10935±1094	9290±929	14400±2880
Fats, mg·dm^{-3}	50	16316±3263	10071±2014	10100±2525
Anionic surfactants, mg·dm^{-3}	10	2.20±0.44	0.440±0.088	3.15±0.79
Nonionic surfactants, mg·dm^{-3}	Not regulated	6187±1237	4083±817	4711±942

* – permissible concentrations of pollutants in wastewater allowed to discharge into wastewater disposal systems, approved by Resolution No. 644 of the Government of the Russian Federation dated 29.07.2013 "On Approval of the rules of cold water supply and sanitation" [19].

The main components in the waste water composition are: suspended substances, oil products, grease, non-ionic surface-active substances. In samples No. 1, No. 2, No. 3 the concentration of pollutants exceeds the permissible concentration for discharge into the sewage system regarding suspended solids by 1.9-8.0 times, regarding the parameter dry residue by 5-7 times, chemical oxygen demand by 31-38 times, oil products content by 900-1400 times, fat content by 200-326 times.

Oil-containing wastewater stratifies into components over time and the particles become larger. Therefore, particle size measurement was carried out twice - immediately after formation and after 3 months. The acoustic method based on measuring the degree of attenuation of the ultrasonic signal was used [20]. The results are tabulated (Table 2) and illustrated in Fig. 2.

Table 2. Average values of the particle size of the dispersed phase of emulsions
Таблица 2. Средние значения размера частиц дисперсной фазы эмульсий

Type of emulsion	Particle size (main peak) D_h, nm	Particle size (second peak) D_h, nm
Sample of used oil-water emulsion immediately after formation	56±2	144±4
Sample of the spent water-oil emulsion after settling for 3 months	113±3	959±25

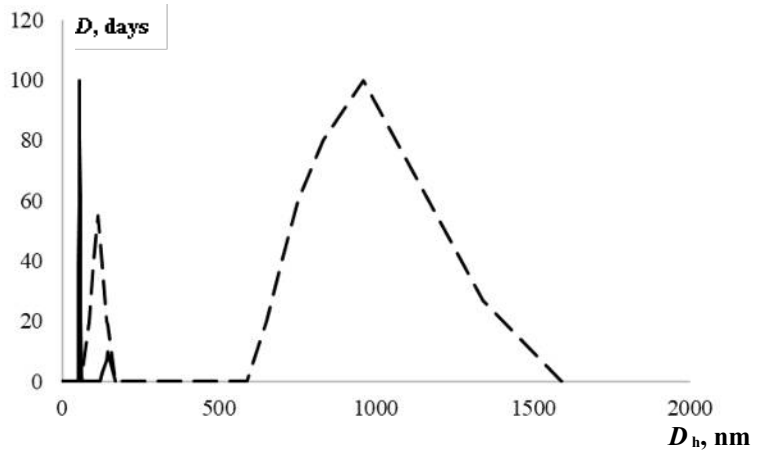


Fig. 2. Comparison of the particle size distribution of oil-containing wastewater dispersed phase: solid line – immediately after formation; dotted line – after destruction (stratification) after 3 months

Рис. 2. Сравнение распределения размера частиц дисперсной фазы сточных нефтесодержащих вод: сплошная линия – сразу после образования; пунктирная линия – после разрушения (расслоения) через 3 мес.

Thus, the size of the contamination particles was 0.05-0.15 μm. To remove such particles, membrane elements with a pore size of the active layer of 0.01-0.05 μm were selected.

RESULTS AND DISCUSSION

Figure 3 shows the results of a study of the permeability of polymer membranes for 8 hours. For the experiment we use a model oil-in-water solution with a concentration of petroleum products of 500 mg·dm^{-3}.

Figure 4 shows the results of experimental studies on the effect of the duration of the process on the retention capacity of membranes with the same model solution.

The analysis of the curves shown in Fig. 3 shows a 50-60% drop in permeability 4 hours after the start of the separation process, then stabilization is observed. This phenomenon is associated with shrinkage of the membranes and the formation of a layer on the membrane surface.

Selectivity increases by 2-5% over time (see Fig. 4), which is also explained by a decrease in the average pore size as a result of shrinkage of the membranes and the formation of a layer on the surface.

Increasing the temperature of the separated solution is the main way to intensify the process on the one hand and reducing selectivity on the other.

The effect of temperature on the productivity and selectivity of the process is shown in Fig. 5. The studies were carried out for the most productive membranes made of fluoroplast and polysulfonamide. The temperature of the solution varied from 293 to 323 K with an interval of 10 degrees.

Conditions for conducting experimental studies:
– model solution with a concentration of petroleum products 500 mg·dm^{-3};
– working pressure 0.4 Mpa.

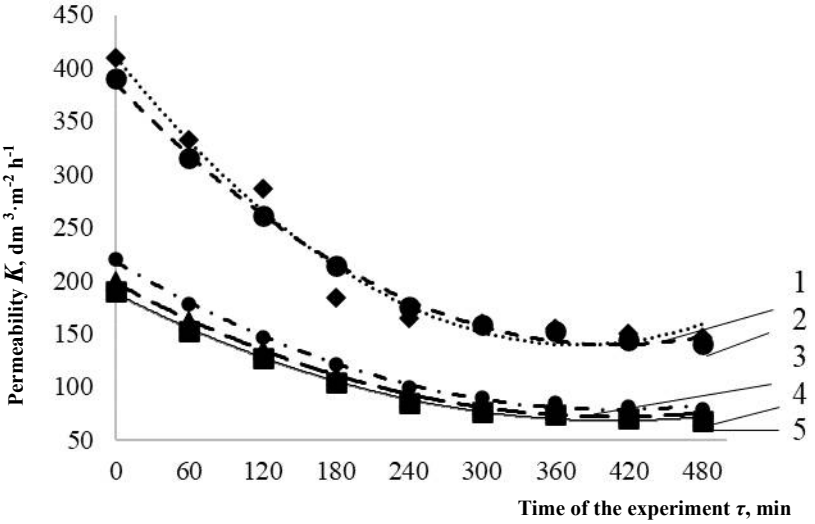


Fig. 3. Dependence of polymer membrane selectivity on time at t = 323 K, petroleum products concentration 500 mg·dm^{-3}, ΔP = 0.4 MPa: 1 – phtoroplast; 2 – polysulfonamide; 3 – polysulfone; 4 – polyvinyl chloride; 5 – polyestersulfone (curves obtained by approximation of data by the least squares method)

Рис. 3. Зависимость селективности полимерных мембран от времени при температуре 323 К, концентрации нефтепродуктов 500 мг·дм$^{-3}$, ΔP = 0.4 МПа: 1 – фторопласт; 2 – полисульфонамид; 3 – полисульфон; 4 – поливинилхлорид; 5 – полиэфирсульфон (кривые получены аппроксимацией данных методом наименьших квадратов)

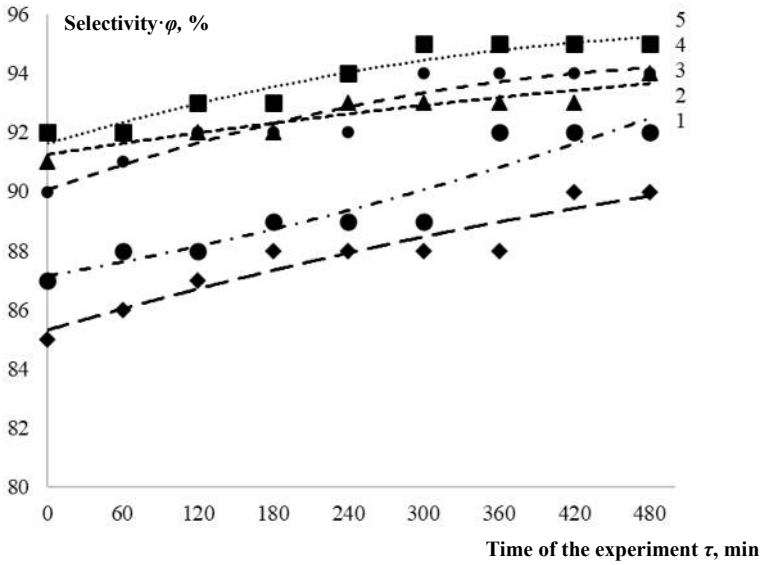
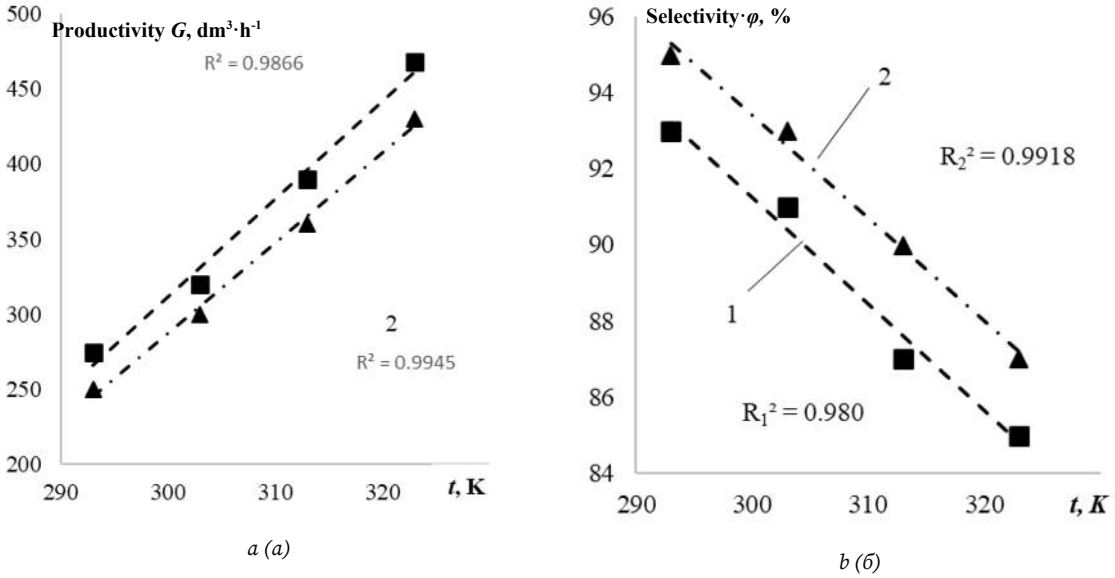


Fig. 4. Dependence of polymer membrane selectivity on time at t = 323 K, petroleum products concentration 500 mg·dm^{-3}, ΔP = 0.4 MPa: 1 – phtoroplast; 2 – polysulfonamide; 3 – polysulfone; 4 – polyvinyl chloride; 5 – polyestersulfone (curves obtained by approximation of data by the least squares method)

Рис. 4. Зависимость селективности полимерных мембран от времени при температуре 323 К, концентрации нефтепродуктов 500 мг·дм$^{-3}$, ΔP = 0.4 МПа: 1 – фторопласт; 2 – полисульфонамид; 3 – полисульфон; 4 – поливинилхлорид; 5 – полиэфирсульфон (кривые получены аппроксимацией данных методом наименьших квадратов)

The analysis of the dependencies presented in Fig. 5 allows us to observe the expected response

of the membrane–solution system to temperature changes. With a change in temperature to 323 K, the permeability predictably increased by 40%, and the selectivity decreased from 93-95 to 85-87%. This is due to a decrease in the viscosity of the solution and an increasing slip of dirt particles through a semi-permeable partition.



a (а) *b (б)*

Fig. 5. The effect of the solution temperature on the permeability (*a*) and selectivity of the process (*b*); 1 – polysulfonamide; 2 – fluoroplast (curves are obtained by data approximation by the least squares method)

Рис. 5. Влияние температуры раствора на проницаемость мембран (*а*) и селективность процесса (*б*); 1 – полисульфонамид; 2 – фторопласт (кривые получены аппроксимацией данных методом наименьших квадратов)

The linear approximation of the experimental data (Fig. 5) gives a high confidence value R^2 = 0.98-0.99. Thus, the effect of temperature on permeability and selectivity can be described by the equation [21]:

$$y = a \cdot x + b, \quad (1)$$

where *a, b* are the approximation coefficients; *x* is the variable parameter, temperature.

Coefficients *a* and *b* were calculated according to the formulas [21]:

$$a = \frac{n \cdot \sum_{i=1}^{n} x_i \cdot y_i - \sum_{i=1}^{n} x_i \cdot \sum_{i=1}^{n} y_i}{n \cdot \sum_{i=1}^{n} x_i^2 - \left(\sum_{i=1}^{n} x_i \right)^2}; \quad (2)$$

$$b = \frac{\sum_{i=1}^{n} y_i - a \cdot \sum_{i=1}^{n} x_i}{n}, \quad (3)$$

where *n* is the number of experiments.

As a result of calculations, we have obtained equations adequately describing the effect of temperature:

for selectivity (see Fig. 5, *a*)

$$\varphi_1 = 6{,}49\,t - 1635{,}7\,; \quad (4)$$

$$\varphi_2 = 6\,t - 1513; \quad (5)$$

for permeability (Fig. 5, *b*)

$$G_1 = -0.28\,t - 175.24\,; \quad (6)$$

$$G_2 = -0.27\,t - 174.41\,. \quad (7)$$

The obtained patterns adequately describe the change in permeability and selectivity from temperature in the range from 373 to 323 K.

CONCLUSIONS

We develop the installation for separation of water-oil systems using polymer and ceramic membranes. The composition of the tested systems was determined using certified methods through the use of modern analytical equipment. The spent emulsion contains particles of 56 and 144 nm in size, and after its destruction, particles grow to 113-959 nm.

Membranes made of fluoroplast and ceramics are advisable to use when separating used engine oils. Theproductivity of such membranes depends on the structure of the microporous layer and its properties. With an increase in the concentration of petroleum products in the water-oil emulsion to g/dm^3, the specific capacity of the membranes decreases by 40-50%, which is associated with an increase in the viscosity of the liquid phase.

It was also revealed that with an increase in the temperature of the separated solution (from 313 to 383 K), the ultrafiltration process is intensified by 8 times.

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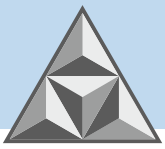

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