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KATARZYNA MAJEWSKA-NOWAK*, TOMASZ WINNICKI*

THE EFFECT OF DYE CONCENTRATION ON MEMBRANE PERFORMANCE IN THE ULTRAFILTRATION PROCESS

Polysulfone membranes were synthesized and their properties were studied to check the usability of this type of membranes in discolouration of aqueous solutions of organic dyes by ultrafiltration. The experiments were carried out under static conditions using seven organic dyes of molecular weights ranging between 291 and 1048.2. The pressure applied varied from 0.5 to 2.05 MPa and the dye concentration in the aqueous solution from 100 to 1000 gm^{-3}. The influence of the dye concentration on the ultrafiltration efficiency was determined. It has been found that in case of high-molecular-weight dyes (above 800) the separation properties of the membranes do not depend on the dye concentration, while for low-molecular-weight dyes, the elimination coefficient decreases with the increasing dye concentration. It has been also observed that the membrane transport properties worsen with the increasing dye concentration.

1. INTRODUCTION

Among the various industrial wastewaters the treatment of which is difficult, those containing hardly degradable dyes are especially troublesome. The main difficulty is the development of one efficient and economically justified method of dye removal. This is caused by quick changes in techniques of production and the fact that the technological processes differ in their kinds and various quantities of dyes are used. Hence the composition of wastewaters is subject to variations (e.g. the concentration of dyes in textile dyeing effluents can vary from 10 to 10000 gm^{-3} [5]). Conventional treatment methods, used so far to remove hardly degradable dyes from industrial wastewaters, have not yielded satisfactory discolouration effects. Thus, the need for more efficient techniques has directed the attention of many investigators to the application of membrane processes (such as ultrafiltration), which involve the selection action of semipermeable

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* Institute of Environment Protection Engineering, Technical University of Wrocław, Wybrzeże Wyspiańskiego 27, 50-370 Wrocław, Poland.

membranes to separate organic dyes from aqueous solutions. Membrane processes not only permit the reduction of the pollution load, but they also allow reuse of the water purified in this way and the recovery of some other valuable substances (e.g. dyes).

The selection of membranes for application to ultrafiltration is determined by the ability of the membrane to retain solute molecules of a given size and by the intensity of solvent permeability. The most popular are cellulose acetate membranes, but they can work in a limited range – at temperatures up to 308 K [2], and pH from 3 to 7 [12]. At the present time, polysulfone membranes are considered to be the most promising among the variety of types employed, as they may be used in almost whole range of pH [12], as well as at elevated temperatures (up to 378 K) [1]. They are also resistant to oxidation with chlorine [12]. Hence, they show very good physicochemical properties, but, so far, their applications in nonmodified form have been limited to the separation of very high-molecular-weight compounds (above several thousand) [12, 14]. All attempts to make polysulfone membranes able to retain compounds of a molecular weight below 1000 have failed [12].

The investigations reported in this paper are the continuation of our previous studies [10, 11]. Our preliminary results have shown that polysulfone membranes yield a complete retention of organic dyes of a molecular weight higher than 800, but are not able to retain dyes of a molecular weight below 300. It has been also observed that transport and separation properties of the membranes depend mainly on the casting parameters. Our preliminary studies were performed on model solutions of selected organic dyes in concentration of 100 gm^{-3}. Taking into account the variability of the composition of dye effluents, the main purpose of the studies presented in this paper was to determine the influence of dye concentration on the ultrafiltration efficiency.

All our investigations aim at evaluation of the usefullness of polysulfone membranes for discolouration and concentration of aqueous solutions of organic dyes by ultrafiltration process in order to apply these membranes in dye wastewater treatment.

2. EXPERIMENTAL

2.1. PREPARATION OF POLYSULFONE MEMBRANES

The casting solution, i.e. 15 weight percent of P 3500 aromatic polysulfone (Union Carbide) dissolved in dimethylformamide (DMF), was prepared according to the procedure described by KOENST and MITCHELL [7]. The membranes were cast by means of a typical device consisting of a horizontal glass plate and a movable frame with two micrometric screws. The upper ends of the screws were fastened to the frame, and the lower ones to the casting knife. The screws were used to adjust the distance between the knife edge and the glass surface, and, consequently, the membrane thickness. The frame put in motion by a small electric motor moved slowly along the levelled bench and the knife spread the solution uniformly allover the glass surface. The speed of the frame movement was also adjusted.

Based on the previous results [10, 11], membranes 65–115 μm thick were cast at two different initial temperatures of casting solution (303 and 318 K). The membranes were then left at room temperature for 60 seconds to evaporate the solvent from their surfaces.

2.2. TESTING DEVICE

The polysulphone membranes prepared according to the method described above were tested under static conditions. The testing device (fig. 1) is a pressure apparatus *2* made of stainless steel. The membrane *11* of the effective surface area of 15.9 cm^2 was fixed in the lower part of the apparatus and supported by a porous PVC sinter *13*. The solution to be tested was fed to upper part of the apparatus through an inlet pipe *3*. To obtain and maintain the pressure required, nitrogen was supplied from a cylinder *7* and let through a reducing valve *6* to an inlet *4*.

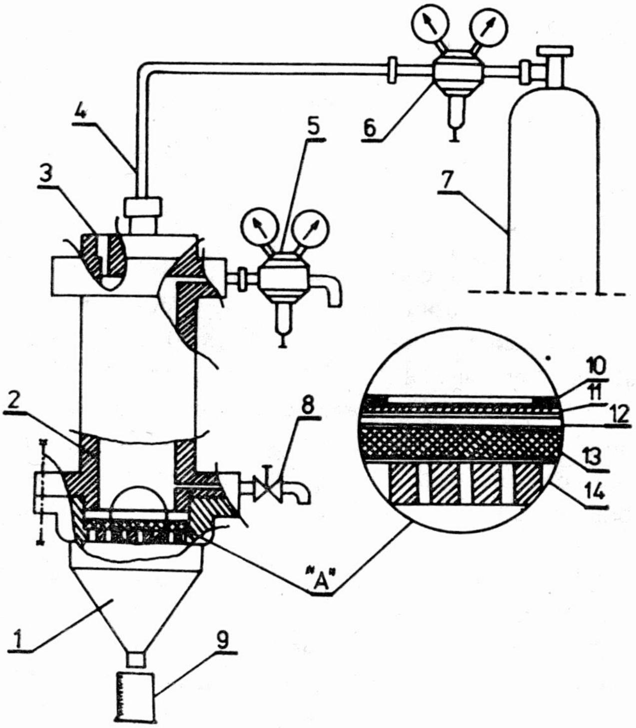


Fig. 1. Apparatus for membrane testing under static conditions

1 – outlet of permeate, *2* – pressure apparatus, *3* – stub pipe for solution supply. *4* – stub pipe for gas supply, *5*, *6* – reducing valves, *7* – gas cylinder, *8* – needle valve, *9* – receiver, *10* – rubber gasket, *11* – membrane, *12* – filter paper, *13* – sinter, *14* – grid

Rys. 1. Aparat do testowania membran w warunkach statycznych

1 – odprowadzenie permeatu, *2* – aparat ciśnieniowy, *3* – króciec doprowadzający roztwór, *4* – króciec doprowadzający gaz, *5*, *6* – reduktory ciśnienia, *7* – butla z gazem, *8* – zawór iglicowy, *9* – odbieralnik, *10* – uszczelka gumowa, *11* – membrana, *12* – bibuła filtracyjna, *13* – spiek, *14* – ruszt

2.3. TESTING METHOD

Three membranes, differing in casting parameters, thus also in structure (tab. 1), were tested. The membrane porosity was calculated from the difference between the masses of wet and dry membrane. Mean membrane pore radius was determined by two methods: 1) using Poiseuille equation [3], assuming tortuosity factor equal to 1.64 [15] and 2) calculating Knudsen gas permeability coefficients and membrane geometry coefficients from the measurements of membrane gas permeability (in the way described in [15]). The results obtained by these two methods were almost the same.

Table 1

Characteristics of the experimental membranes
Charakterystyka membran użytych w badaniach

Membrane no.	Membrane thickness μm	Temperature of casting solution K	Mean membrane pore radius nm	Membrane porosity	Volume flux of water at 2·5 MPa m³/m²d
1	65	303	0.60	0.66	0.05
2	85	318	1.10	0.72	0.12
3	115	318	1.70	0.77	0.17

The membrane samples were conditioned prior to each experimental cycle. In this process distilled water was let through the membrane under a pressure of 2.5 MPa, until a steady volume flux was achieved, usually after 30 to 80 h. The values of the steady volume fluxes of water at a pressure of 2.5 MPa are given in tab. 1.

In the experiments involved seven organic dyes of molecular weights between 291 and 1048.2 were used (tab. 2). Four of them (direct red, direct green, direct black, and reactive yellow) are commercial azo dyes most often employed in textile industry (in dyehouses). The other dyes (tab. 2) are typical indicators applied in analytical chemistry.

To examine the membrane properties the following five concentrations of selected organic dyes (tab. 2) were chosen: 100, 200, 400, 600, and 1000 gm^{-3}. The ultrafiltration tests were carried out at the pressures: 0.5, 1.0, 1.5, 2.0, and 2.5 MPa. Transport properties of the membranes were determined by measuring the volume fluxes of dye solutions at given pressure. The hydraulic permeability was calculated from the equation:

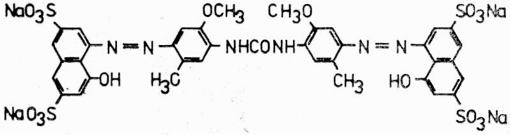
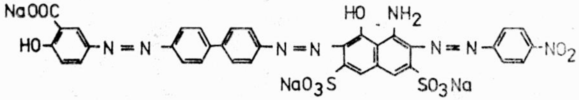
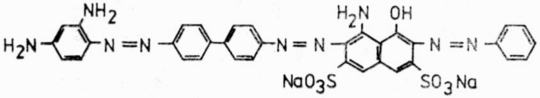
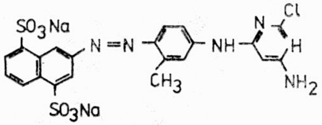
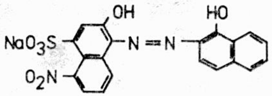
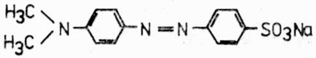
$$L_p = \frac{J_v}{\Delta P}$$

where:

L_p – hydraulic permeability, $m^3N^{-1}s^{-1}$,
J_v – volume flux, m^3m^{-2} day^{-1} ($= 1.16 \times 10^{-5} m^3m^{-2}s^{-1}$),
ΔP – pressure difference, MPa.

Table 2

Characteristics of the dyes used in the ultrafiltration tests
Charakterystyka barwników użytych w badaniach

Name of dye	Molecular weight	Classification	λ_{max} nm	Symbol	Structure
Direct Red 79 **(C.I.* 29065)**	1048.2	disazo direct	555	DR	
Direct Green 81 **(C.I.* 30315)**	878.1	trisazo direct	390	DG	
Direct Black 38 **(C.I.* 30235)**	781.2	trisazo direct	570	DB	
Reactive Yellow	593.6	monoazo reactive	430	RY	
Eriochrome Black T	461.0	monoazo	560	EB	
Acid Orange 52 **(C.I.* 13025)**	327.0	monoazo acid	485	AO	
Methyl Red	291.0	monoazo	495	MR	

* Colour Index.

Separation properties of the membranes were determined by measuring the elimination coefficient R_s defined as:

$$R_s = \frac{C_1 - C_2}{C_1} \cdot 100$$

where:

R_s — elimination coefficient, %,
C_1 — dye concentration in feed solution, gm^{-3},
C_2 — dye concentration in permeate solution, gm^{-3}.

Concentrations of dyes in model solutions were determined colorimetrically at the appropriate wavelengths λ_{max} (tab. 2), using a Carl-Zeiss-Jena Spectrophotometer.

3. RESULTS AND DISCUSSION

3.1. EFFECT OF THE DYE CONCENTRATION ON THE TRANSPORT PROPERTIES OF THE MEMBRANES

The relationship between the hydraulic permeability and the dye concentration in model solution for the membranes tested are given in fig. 2.

It has been found that with the increasing dye concentration the membrane hydraulic permeability decreases. This tendency becomes more evident for the dyes of high molecular weights. In the case of direct red, the increase in the dye concentration from 100 to 1000 gm^{-3} yielded 15 to 30% drop in the hydraulic permeability (depending on the membrane properties — tab. 1). For low-molecular-weight dyes the worsening of the membrane transport properties at higher dye concentrations is much smaller or it does not occur.

The decrease in membrane permeability with the increasing concentrations of solution is explained by the increasing osmotic pressure of the latter, thus by the decreasing the driving force of the process (difference in the pressures at both sides of the membrane) and, consequently, in the decrease of the membrane permeability. Thus, it might be expected that the worsening of the membrane transport properties would be more pronounced for low-molecular-weight dyes, because of the higher osmotic pressure of the solution containing these dyes. However, as shown by the experimental results, the decrease in the hydraulic permeability for high-molecular-weight dyes was much greater than it had been expected. This can be explained by the almost complete retention of these dyes by the membranes. Hence, the dye concentration in the solution layer at the membrane surface (which is due to concentration polarization) is much higher than that in an analogical layer formed during ultrafiltration of low-molecular-weight dyes (which, into a considerable degree, passed through the membranes). To avoid these undesirable effects it is therefore necessary to carry out further investigations under dynamic conditions (i.e. with intensive mixing of the solution at the membrane surface).

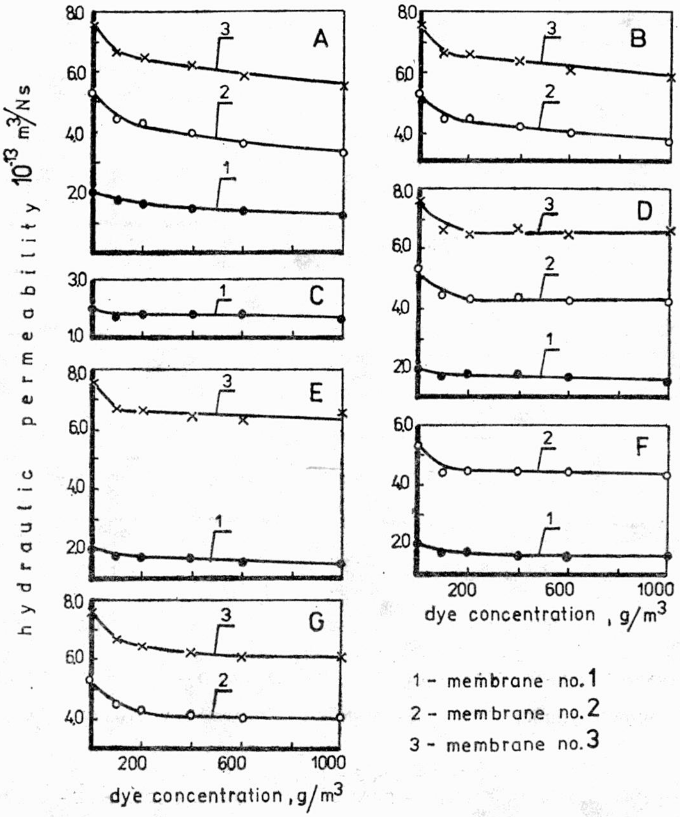


Fig. 2. Hydraulic permeability versus dye concentration in aqueous solution for various dyes
A – DR, *B* – DG, *C* – DB, *D* – RY, *E* – EB, *F* – AO, *G* – MR

Rys. 2. Zależność przepuszczalności hydraulicznej od stężenia barwnika w roztworze wodnym dla różnych barwników
A – DR, *B* – DG, *C* – DB, *D* – RY, *E* – EB, *F* – AO, *G* – MR

3.2. EFFECT OF THE DYE CONCENTRATION ON THE MEMBRANE SEPARATION PROPERTIES

The relationship between the dye elimination coefficient and the dye concentration in model solution for the membranes tested are given in figs. 3, 4, and 5.

It has been observed that the elimination coefficient for dyes of higher molecular weights (direct red, direct green, and direct black) is in principle independent of the dye concentration. It becomes clear if we assume that particles of these dyes have much greater sizes than the pore diameters in the membrane (e.g. the particle radius of direct green is equal to 1.5 nm [4]). This assumption is supported by very high elimination coefficients (90–100%). It should be pointed out that in aqueous solutions dissociations

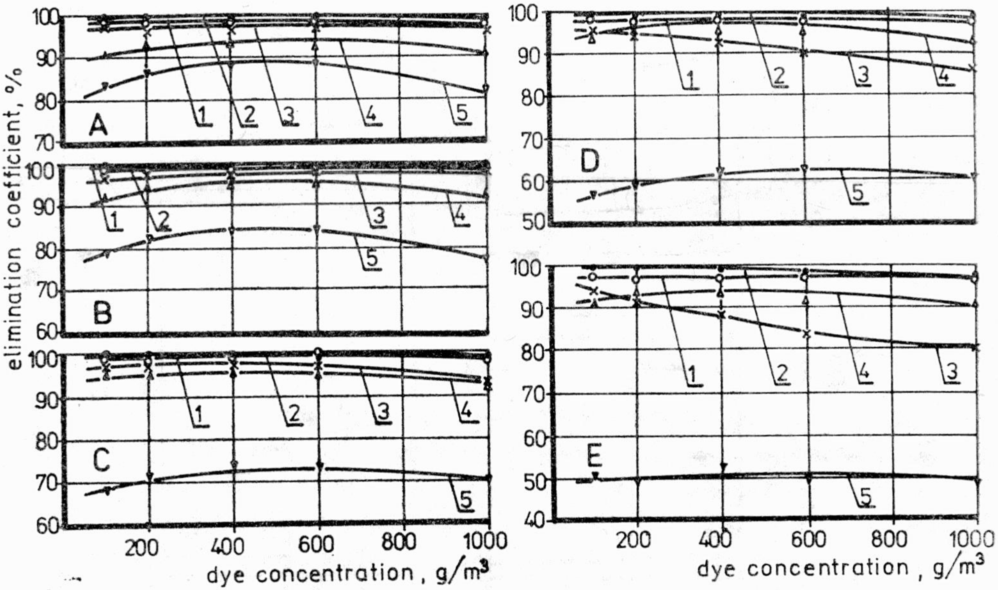


Fig. 3. Elimination coefficient versus dye concentration in aqueous solution for various dyes

1 – DR, *2* – DB, *3* – RY, *4* – EB, *5* – AO

Measurements were performed at: *A* – 0.5 MPa, *B* – 1.0 MPa, *C* – 1.5 MPa, *D* – 2.0 MPa, *E* – 2.5 MPa for membrane no. 1

Rys. 3. Zależność współczynnika eliminacji od stężenia barwnika w roztworze wodnym dla różnych barwników

1 – DR, *2* – DB, *3* – RY, *4* – EB, *5* – AO

Pomiary wykonano przy ciśnieniach: *A* – 0,5 MPa, *B* – 1,0 MPa, *C* – 1,5 MPa, *D* – 2,0 MPa, *E* – 2,5 MPa dla membrany nr 1

of direct dyes result in coloured anions readily aggregable characterized by a high aggregability at dye concentration higher than 0.4 mol m^{-3} [6,13] (this corresponds to concentrations higher than 400 gm^{-3}, when the molecular weight is of the order of 800–1000). In this case, the maximum sizes of the created dye aggregates can vary from 10 to 100 nm [8].

It has been found that in the case of reactive yellow, the elimination coefficient decreases with the increasing dye concentration. This effect becomes more pronounced at higher pressures. Reactive yellow, as its name implies, belongs to the group of reactive dyes, hence, its particles are not able to aggregate in aqueous solutions [13]. The decrease of the elimination coefficient with the increasing dye concentration can be explained by assuming the sorptive-capillary hypothesis [9], according to which, a thin layer of solvent is formed on the surface of the membrane immersed in aqueous electrolytic solution. With the increasing dye concentration the thickness of the solvent decreases, the number of pores accessible to the dye particles increases leading to the decrease of the dye elimination coefficient.

It has been found that the elimination coefficient of low-molecular-weight dyes (acid orange and methyl red) increases with the increasing dye concentration up to the value

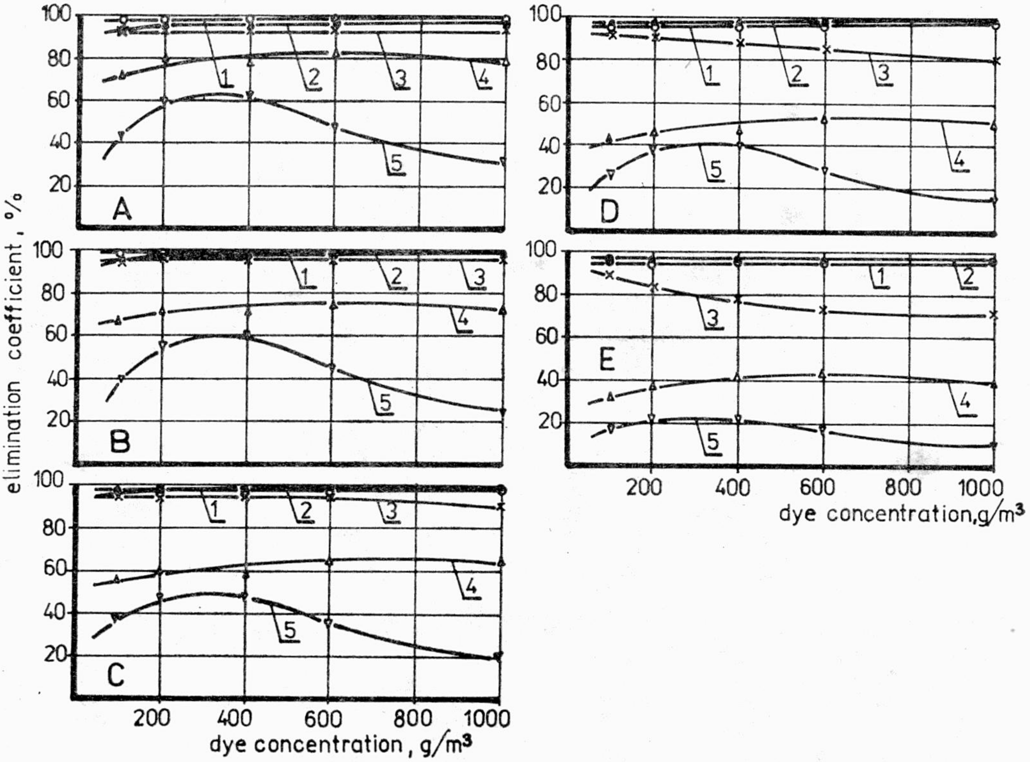


Fig. 4. Elimination coefficient versus dye concentration in aqueous solution for various dyes

1 – DR, *2* – DG, *3* – RY, *4* – AO, *5* – MR

Measurements were performed at: *A* – 0.5 MPa, *B* – 1.0 MPa, *C* – 1.5 MPa, *D* – 2.0 MPa, *E* – 2.5 MPa for membrane no. 2

Rys. 4. Zależność współczynnika eliminacji od stężenia barwnika w roztworze wodnym dla różnych barwników

1 – DR, *2* – DG, *3* – RY, *4* – AO, *5* – MR

Pomiary wykonano przy ciśnieniach: *A* – 0,5 MPa, *B* – 1,0 MPa, *C* – 1,5 MPa, *D* – 2,0 MPa, *E* – 2,5 MPa dla membrany nr 2

of 400 $\mathrm{gm^{-3}}$, thereupon it decreases. This relationship is much more pronounced for methyl red than for acid orange. Similar tendency is observed for eriochrome black in the case of the membrane of a low volume flux (at the given pressure) (tab. 1), and the elimination coefficient achieves its maximum at the concentration of 600 $\mathrm{gm^{-3}}$ (fig. 3). In the case of the membrane of high volume flux (at the given pressure) (tab. 1), the elimination coefficient of this dye decreases with the increasing dye concentration (fig. 5). The particles of monoazo acid dyes are aggregable in aqueous solutions, at the dye concentration higher than 0.6 mol $\mathrm{m^{-3}}$ [6] (this corresponds to the concentrations higher than 200 $\mathrm{gm^{-3}}$ at the molecular mass of the order of 300). The increase in sizes of penetrating particles (due to dye aggregation) increases the dye elimination in the initial range of concentrations, at higher dye concentrations the thickness of the water layer at the membrane surface decreases and the membrane selectivity is worse. According to KU-

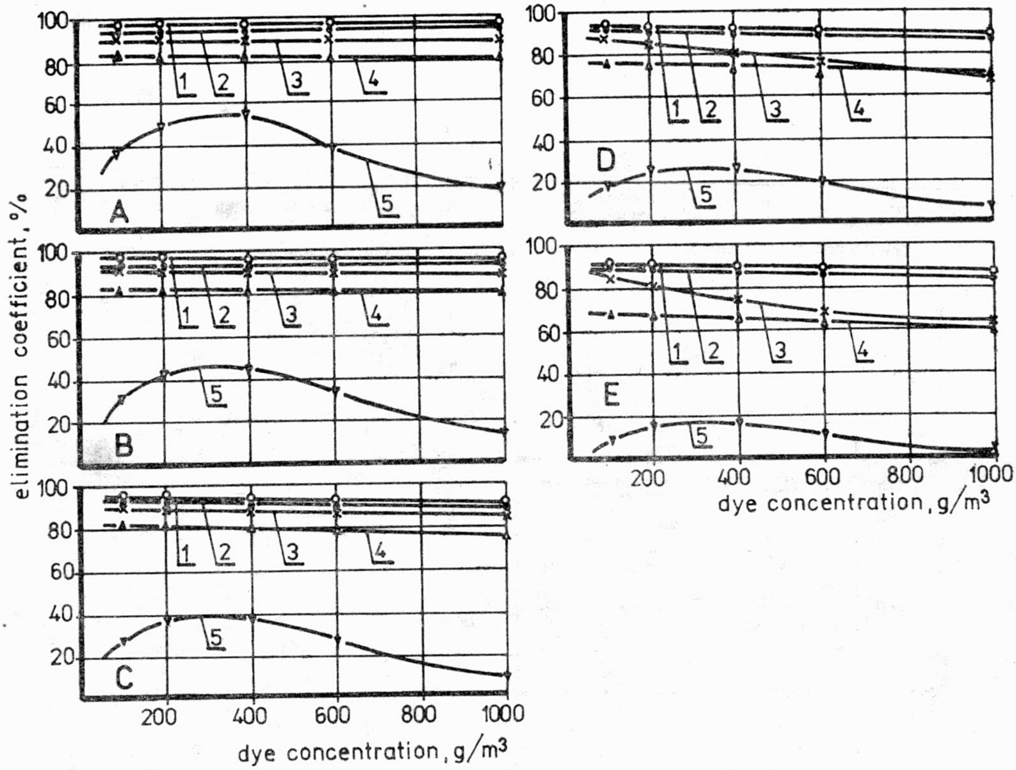


Fig. 5. Elimination coefficient versus dye concentration in aqueous solution for various dyes

1 – DR, *2* – DG, *3* – RY, *4* – EB, *5* – MR

Measurements were performed at: *A* – 0.5 MPa, *B* – 1.0 MPa, *C* – 1.5 MPa, *D* – 2.0 MPa, *E* – 2.5 MPa for membrane no. 3

Rys. 5. Zależność współczynnika eliminacji od stężenia barwnika w roztworze wodnym dla różnych barwników

1 – DR, *2* – DG, *3* – RY, *4* – EB, *5* – MR

Pomiary wykonano przy ciśnieniach: *A* – 0,5 MPa, *B* – 1,0 MPa, *C* – 1,5 MPa, *D* – 2,0 MPa, *E* – 2,5 MPa dla membrany nr 3

KUSHKINA et al. [8], at low dye concentration and when the sizes of dye aggregates are comparable or smaller than the pore diameter in the membrane, the water layer is strongly bound with the membrane surface, then the diffusive permeation of dye aggregates is insignificant in comparison to capillary flow of water under the pressure gradient. The maximum elimination is the more shifted toward lower concentration, the smaller is aggregability of the dye. The continuous decrease of the eriochrome black elimination with the increasing dye concentration (in the case of high-hydraulic-permeability membrane, fig. 5) may be due to the fact that despite the aggregation of dye particles, the dye aggregate sizes are too small, compared to the pore diameter in the membrane.

As it follows from the experimental results (figs. 3, 4, and 5) the pressure acting on the membrane has no significant effect on the degree of elimination for high-molecular-weight dyes (above 800). It is obvious, because aggregates of these dyes are much greater in size than the pore diameter in the membrane. For low-molecular-weight dyes, the increase in the pressure applied decreases the elimination coefficient. In this case the increasing pressure is followed by the increasing hydraulic permeability of membrane. Hence, the dye concentration in the solution layer at membrane surface increases, and the elimination coefficient decreases.

4. CONCLUSIONS

1. Polysulfone membranes cast on a glass surface can be applied in removal of organic dyes from aqueous solutions by ultrafiltration method. The membranes are able to retain completely the dyes of molecular masses higher than 800.

2. The transport properties of the membranes are slightly influenced by the dye concentration in the aqueous solution. In general, the hydraulic permeability decreases with the increase dye concentration, this relationship being more pronounced for the dyes of higher molecular masses.

3. In the case of high-molecular-weight dyes (above 800) neither the dye concentration in the aqueous solution, nor the pressure acting on the membrane exerts a significant effect on the degree of dye elimination. For low-molecular-weight dyes the increase of the dye concentration and the pressure is accompanied with the decrease of the elimination coefficient.

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WPŁYW STĘŻENIA BARWNIKÓW NA EFEKTYWNOŚĆ ULTRAFILTRACYJNEGO ODBARWIANIA ICH WODNYCH ROZTWORÓW

Przedstawiono sposób wytwarzania membran polisulfonowych oraz opisano badania nad ich właściwościami. Badania te miały na celu sprawdzenie przydatności membran do ultrafiltracyjnego odbarwiania wodnych roztworów barwników organicznych. W układzie statycznym wykorzystano siedem barwników organicznych o masie cząsteczkowej 291–1048,2. Zastosowano ciśnienia 0,5–2,5 MPa; stężenia barwników w roztworach modelowych zmieniano w zakresie 100–1000 gm^{-3}. Określono wpływ stężenia barwników na efektywność ultrafiltracji. Stwierdzono, że w przypadku barwników o dużej masie cząsteczkowej (ponad 800) właściwości separacyjne membran nie zależą od stężenia barwników, natomiast w przypadku barwników o małej masie cząsteczkowej zwiększenie stężenia barwnika powoduje zmniejszenie współczynnika eliminacji. Zaobserwowano również pogarszanie się właściwości transportowych membran wraz ze zwiększaniem stężenia barwników.

EINFLUß DER FARBSTOFFKONZENTRATION AUF DIE EFFEKTIVITÄT DER ULTRAFILTRATIONSENTFÄRBUNG VON WÄSSRIGEN FARBSTOFFLÖSUNGEN

Die Methode der Herstellung von Polysulphonmembranen sowie die Untersuchungen an den Membraneigenschaften wurden beschrieben. Die Untersuchungen bezwecken die Brauchbarkeitsprüfung der Membranen zur Ultrafiltrationsentfärbung von wässrigen organischen Farbstofflösungen. In statischen System wurden sieben organische Farbstoffe mit dem Molekulargewicht 291–1048, 2 ausgenutzt. Es wurde der Druck von 0,5–2,5 MPa angewendet, die Farbstoffkonzentration in Modellösungen ändert sich innerhalb von 100-1000 gm^{-1}. Der Einfluß der Farbstoffkonzetration auf die Effektivität der Ultrafiltration wurde bestimmt. Es wurde festgestellt, daß im Falle der Farbstoffe mit hohem Molekulargewicht (über 800) die Separationseigenschaften der Membranen von der Farbstoffkonzentration unabhängig sind, bei Farbstoffen mit niedrigen Molekulargewicht beeinflußt die Steigung der Farbstoffkonzentration die Minderung des Eliminierungskoeffizienten. Es wurde auch die Verschlechterung von Transporteigenschaften der Membranen bei der Steigerung der Farbstoffkonzentration beobachtet.

ВЛИЯНИЕ КОНЦЕНТРАЦИИ КРАСИТЕЛЕЙ НА ЭФФЕКТИВНОСТЬ УЛЬТРАФИЛЬТРАЦИОННОГО ОБЕСЦВЕЧИВАНИЯ ИХ ВОДНЫХ РАСТВОРОВ

Представлен способ изготовления полисульфоновой мембраны, а также описаны исследования по их свойствам. Эти исследования имели своей целью проверить пригодность мембран для ультрафильтрационного обесцвечивания водных растворов органических красителей. В статической системе было использовано семь органических красителей с молекулярной массой 291–1048,2. Были применены давления 0.5–2.5 МПа; концентрации красителей в модельных растворах изменяли в пределах 100–1000 моль$^{-3}$. Определено влияние концентрации красителей на эффективность ультрафильтрации. Выявлено, что в случае красителей с большой молекулярной массой (свыше 800) сепарационные свойства мембран не зависят от концентрации красителей, в случае же красителей с малой молекулярной массой повышение концентрации красителя вызывает уменьшение коэффициента элиминации. Было замечено также ухудшение транспортных свойств мембран с повышением концентрации красителей.