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APPLICATION OF TUBULAR POLYSULFONE MEMBRANES TO DECOLORIZATION OF TEXTILE EFFLUENTS

The separation characteristics of tubular polysulfone membranes formed on the surface of porous polymethyl methacrylate bars has been presented. The effect of the concentration of some dyes on the transport and separation properties of the membranes has been analysed. The gel bath consisting of 30% sulphuric acid solution causes a nearly twofold rise of the hydraulic permeability. The efficiency of ultrafiltration was tested on samples of wastewater coming from two processes, dyeing and rinsing of textiles. After 150 hours of operation at 0.18 MPa, the volume flux of the permeate varied from 0.4 m³/m² a day to 0.8 m³/m² a day for the dyeing bath and the rinsing bath, respectively, and the efficiency of TOC removal ranged from 65 to 75% and from 45 to 55% for the dyeing bath and the rinsing bath, respectively.

1. INTRODUCTION

Some of the wastewaters produced as a result of industrial activities (especially those from the manufacture of organic dyes, from the textile industry, from paper-mills or leather-processing plants, etc.) contain coloured matter, a category of low-biodegradability organics. A considerable part of this pollution comes from the textile industry, wherein the effluents from dyeing and printing processes account for some 30% of the total wastewater volume [8]. The effluent from a dye-house is a mixed brew of spent water (from dyeing, fixation, washing and rinsing), residual coloured matter and chemicals (acids, alkalies, redox substances, penetrants, salts, etc.).

Intensely coloured wastewaters discharged into the waterways make the recipient stream unfit for domestic or industrial uses. Waters containing dyes in high concentrations reduce the transmission of light, thus disturbing aquatic plant growth

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and exerting a harmful effect on fish. Moreover, some of the dyes entering the watercourse are classified as toxic pollutants.

The most common methods of treating dye-bearing wastewater are chemical precipitation [13], [14], activated sludge process [3], [7], [10], chlorination [6], [12], ozonation [6], [12], and adsorption on activated carbon [4], [7], [13], [14].

Chemical precipitation involves iron salts or alum as coagulants which may be aided with lime, whenever necessary. The process yields a dichromate COD removal of 44 to 62% and a colour removal of 62.8 to 81.5% [13], [14]. There are disadvantages, i.e., high consumption of chemicals, precipitation of large quantities of bottom sediment, and rise of salinity level. Furthermore, the coagulation process is inefficient when the wastewater under treatment contains direct dyes and vat dyes [8].

The activated sludge process is reported to give trouble primarily because many of the dyes present in the feed are toxic, and the colour removal efficiency is as low as from 0 to 20% [3], [7]. However, making use of the sorption properties of the biomass brings about an increase in the degree of removal which varies from 65 to 80% and from 30 to 50% for coloured matter and dichromate COD, respectively [10].

Chlorination of the effluent from dyeing processes is effective only for certain groups of dyes (e.g., acid dyes or reactive dyes), and becomes inefficient in the presence of direct dyes [12].

In the past few years ozonation has begun to gain popularity as applied to the treatment of waters containing dyes. The process yields either partial or complete oxidation of dye particles, the colour removal efficiency ranging from 76 to 96% [6].

The application of activated carbon for colour removal generally fails because of the poor sorbing capacity of this medium [19]. The activated-carbon bed procedure has one disadvantage more: there is no adsorption of direct dyes [4].

Taking into account the shortcomings of conventional treatment methods, pressure membrane processes (ultrafiltration and reverse osmosis) have been suggested as a means of treating a bath which contains high-molecular-weight dyes. When applying membrane processes, both water and dyes may be recovered from the waste stream and rendered suitable for re-use [9], [18]. Since direct dyes are claimed to be less sensitive to treatment than other dyes, they have been given particular attention in the study.

2. EXPERIMENTAL

2.1. PREPARATION OF POLYSULFONE SOLUTION

The polysulfone solution was prepared by the Koenst-Mitchell method [5] from an aromatic P 3500 Union Carbide polysulfone (referred to as PS) and a dimethylformamide solvent (referred to as DMF). The concentration of PS in the solvent-polymer solution amounted to 12.5 and 13.75 wt.%. Mixing was carried out at 293 K for 96 hours.

2.2. FORMATION OF MEMBRANES

The membranes were formed on the surface of 25 cm long bars made of polymethyl methacrylate. The casting procedure consisted of the following steps: immersion of the sinter in the polymer solution which had a temperature of 298 K, exposure of the bar and the cast film to the ambient air for 0 to 15 s, and immersion in a gel bath having a temperature of 298 K. The gel bath consisted of distilled water or aqueous solutions of sulphuric acid at a concentration of 7.5; 15.0; 22.5; 30.0, and 37.0 wt.%. After two hours of immersion in the gel bath the membranes were rinsed with distilled water. Distilled water was also used for the storage of the membranes.

The three sequences of membranes obtained via the above procedure are characterized in tab. 1.

Table 1

Casting parameters								
Sequence Series		PS concentra- tion in casting solution wt.%	Time of solvent evaporation s	H_2SO_4 con- centration in gel bath wt.%	Average membrane thickness in series μm			
I	1	13.75	0	58 <u>-</u>	180			
	2	13.75	15		150			
II	1	12.5	0		140			
	2	12.5	15	·	100			
III	1	12.5	0	7.5	125			
	2	12.5	0	15.0	105			
	3	12.5	0	22.5	100			
	4	12.5	0	30.0	100			
	5	12.5	0	37.5	100			

2.3. EXPERIMENTAL APPARATUS

The system for ultrafiltration tests is shown in fig. 1. The membrane module 2 of an effective surface of 49.2 cm^2 is embedded in the lower part of a 1.2 dm³ volume pressure apparatus 1 made of metaplex. Pressure is generated by feeding the apparatus with an inert gas (nitrogen) from a cylinder 9. The stub pipes 4 serve for collection of permeate samples. A Dispenser 344 plunger micro-pump 6 provides circulation between the feeding tank 5 and the apparatus 1. Systems of that design enable a constant concentration of the liquid to be kept in the ultrafiltration cell 1. A magnetic stirrer 3 permits a complete mix and prevents excess dye deposition on the membrane surface.



Fig. 1. Apparatus for the testing of membrane modules by ultrafiltration 1 - pressure apparatus, 2 - bar covered with membrane, 3 - magnetic stirrer, 4 - outlet of permeate, 5 - feeding tank, 6 - pump, 7 - venting valve, 8 - permeate, 9 - gas cylinder, 10 - pressure regulator, 11 - pressure reducing valve, 12 - manometer, 13 - stop valve

2.4. CHARACTERIZATION OF DYES

Seven organic dyes have been selected for the study taking into account the extent of their industrial uses, water-solubility, and variety of molecular weight, particular consideration being given to direct dyes which are difficult to degrade. They are the following: Direct Red 6BL (m.w. 1048), Direct Green G (m.w. 878), Direct Black Meta (m.w. 781), Reactive Yellow D-GR (m.w. 594), Eriochrome Black T (m.w. 461), Acid Orange (m.w. 327) and Methyl Red (m.w. 291).

While the first three direct dyes are widely used in dye-houses [16], the last three have come into general use as tracers in analytical chemistry. The tracer-dyes have been selected because of their low molecular weight. Quests for industrial low-molecular-weight dyes have failed. Reactive Yellow has been considered in this study because it is quite frequently applied for the dyeing of textiles.

Model solutions were obtained by dissolving the dye sample in distilled water (except Methyl Red which had been dissolved in ethyl alcohol prior to distilled water treatment).

2.5. TRANSPORT AND SEPARATION PROPERTIES

The transport properties of the membranes were determined in relation to both distilled water and dye solution. When related to distilled water, the transport properties were examined at a pressure of 0.18 MPa, after the volume flux of water through the membrane had become stable. Basing on the measured rate of water flow through the membrane, the volume flux was determined.

The investigation of the transport properties in relation to the dye solutions, as well as the separation properties of the membranes began immediately, following completion of the procedure of transport properties determination (in relation to water). The experiments involved aqueous solutions wherein dyes occurred at four concentrations: 100, 250, 500 and 750 g/m³. The ultrafiltration process was carried out at 0.18 MPa, after a steady flow had been achieved.

Dye concentrations in the experimental solutions were measured by means of a Carl-Zeiss-Jena spectrophotometer at various wavelengths.

2.6. ULTRAFILTRATION OF TEXTILE EFFLUENTS

The efficiency of the ultrafiltration process was studied with respect to two types of 5 dm^3 wastewater samples – an exhausted dyeing bath with Direct Black RT as a dyeing agent, and the effluent from primary rinsing after dyeing with Direct Black RT. Samples had been taken from a jigger operated in the finishing department of a Hemp and Flax Processing Plant in Poland.

Prior to the ultrafiltration run, each sample was pretreated while passing through the anthracite-sand bed. The ultrafiltration process covered a period of about 150 hours and involved a pressure of 0.18 MPa. In routine analyses degree of dilution yielding complete colour removal, dichromate COD and TOC, both in the feed and the filtrate, were determined. TOC determination was carried out in a TOCA Beckman 915 A apparatus by the standard method [15]. The dilution method was applied to determine the specific colour of the wastewater (the sample was diluted until a complete removal of coloured matter was achieved). The dilute sample was compared to a standard one (distilled water).

3. DISCUSSION OF RESULTS

3.1. SEPARATION CHARACTERISTICS

The separation factor was related to the molecular weight of the dye for membranes which had been cast from a solution containing 12.5% and 13.75% of PS by solvent evaporation taking 0 and 15s (membranes of sequence I and membranes of sequence II, respectively, tab. 1). The plots are shown in fig. 2.

The separation characteristics provide data for determining two major parameters which are of importance to the future applications of the membranes – cut off, i.e., the lowest molecular weight of the compounds completely retained by the membrane, and the maximum molecular weight of the compounds passing through the membrane (MMWp). As shown by the plots in fig. 2, both cut off and MMWp depend on the PS concentration in the casting solution and on the time of solvent evaporation from the membrane surface. The increase of PS concentration and the decrease of evaporation time account for a shift of the separation characteristics to the left, which is an indication that cut off and MMWp values have decreased. Hence, it may be expected that cut off and the MMWp values for membranes prepared at a PS concentration of 12.5% and a solvent evaporation time of 0 to 15s vary from



Fig. 2. Elimination coefficient versus molecular weight of dye for membranes cast from 12.5 (A) and 13.75 (B) wt.% PS solution

Solvent evaporation time: 0s (1), 15s (2)

900 to ~ 2500 and from 100 to 300, respectively. Cut off and MMWp values for membranes cast at a PS concentration of 13.75% and an evaporation time of 0 to 15 s range from 750 to 1100 and from 150 to 250, respectively. The relationship between casting conditions and separation properties is strongly influenced by the membrane structure.

The shape of the separation characteristics provides some information on the pore size distribution in the membrane. As the angle of inclination of the curve increases, the distribution of the pore size becomes smaller. On the basis of the shape of the plots in fig. 2, it may be concluded that neither PS concentration nor solvent evaporation time has a distinct effect on the distribution of the pore size in the membrane (the angle of inclination is almost identical for all the plots).

3.2. EFFECT OF DYE CONCENTRATION ON MEMBRANE PROPERTIES

The results are plotted in figs. 3 and 4. PS concentrations in the casting solutions amounted to 12.5% and 13.75% (no solvent evaporation being applied). The membranes obtained via this route belong to sequences I and II, series 1 (tab. 1). Each dye solution contained one of the four dyes: Direct Green, Direct Black, Eriochrome Black or Acid Orange. The data in figs. 3 and 4 show that the increase in dye concentration reduces membrane permeability. This finding holds specifically for high-molecular-weight dyes (fig. 3). The contribution of low-molecular-weight dyes to the deterioration of transport properties is poor. High concentrations of those dyes have only a slight effect on membrane permeability, if at all.



Fig. 3. Volume flux versus dye concentration in aqueous solution for dyes of various molecular weights: 878.1 (1), 781.2 (2), 461 (3), 327 (4)

Measurements were performed for membranes cast from 12.5 (A) and 13.75 (B) wt.% PS solution



Fig. 4. Elimination coefficient versus dye concentration in aqueous solution for dyes of various molecular weights: 878.1 (1), 781.2 (2), 461 (3), 327 (4)

Measurements were performed for membranes cast from 12.5 (A) and 13.75 (B) wt.% PS solution

The decrease of membrane permeability with the increasing dye concentration can be attributed to the rise in the osmotic pressure of the solution, i.e., to the decreasing driving force (pressure difference between the two compartments separated by the membrane). Taking into account the increased osmotic pressure of solutions containing low-molecular-weight dyes, it might be expected that increased low-molecular-weight dye concentrations will bring about a greater deterioration of transport properties than they did. The drop of the volume flux for highmolecular-weight dyes (which was greater than the expected value) may be explained as being a consequence of an almost complete retention by the membranes as well as of the concentration polarization phenomenon. The dye concentration in the solution film forming at the membrane surface was much higher than during the ultrafiltration of low-molecular-weight dyes which generally pass through the membrane. The degrees of separation of high-molecular-weight dyes are only slightly influenced by their concentrations (fig. 4). This is not surprising when the dye particle size is assumed to be greater than the membrane pore diameter. An over ninety-percent separation seems to support this assumption (the lack of complete removal should be attributed to the distribution of pore size). The slight decrease in the separation factor for Direct Green and Direct Black with the increasing dye concentration may be explained in terms of the sorption-capillary hypothesis [11]. According to this theory, a thin layer of pure solvent forms on the surface of a membrane placed in the aqueous solution of an electrolyte. The increase in dye concentration decreases the thickness of that layer and increases the number of pores available to single, non-aggregated dye particles, thus contributing to a deterioration of the separation factor.

The separation factors for low-molecular-weight dyes (Eriochrome Black, Acid Orange) tend to increase as the dye concentration increases from 50 to 400 g/m³, and from there decreases with the increasing concentration value. Monoazo acid dyes show a tendency to aggregate in aqueous solutions at concentrations higher than 0.6 mol/m³ [2] (which is equivalent to concentrations greater than 200 g/m³ at molecular weight approaching 300). Increase in dye particle size, as a result of aggregation, accounts for the rise of the separation factor in the initial range of concentration. When dye concentration increases, the thickness of the solvent (water) layer near the membrane surface decreases to deteriorate the selectivity of the membrane.

3.3. EFFECT OF SULPHURIC ACID CONCENTRATION IN GEL BATH ON MEMBRANE PROPERTIES

One of the objectives of this study was to improve the permeability of the membranes without deteriorating their separation properties. Taking into account the promising results reported by TWEDDLE and co-workers [17], aqueous solutions of sulphuric acid were used as the gelating medium. The relationship between volume flux, separation factor and sulphuric acid concentration is plotted in fig. 5. The dye involved was Direct Black Meta at a concentration of 100 g/m^3 .

Sulphuric acid concentration in the bath varied from 7.5 to 37.5 wt.%. Further increase brought about some undesirable changes ("disappearance" of the cast film in the acid solution, evident deterioration of permeability, fig. 5).

Analysis of the plots in fig. 5 shows that as the sulphuric acid concentration in the gel bath increases, so does the volume flux of both water and dye solution. This is so until a certain value of sulphuric acid concentration (i.e., 30 wt.%) is achieved. From there, volume flux begins to fall rapidly. The volume flux for membranes gelated in 30% sulphuric acid solution is approximately twice as high as that for water-gelated membranes. The dye separation factor varies from 93.4% (when only water gel bath is involved) to 99% (when H_2SO_4 concentration in the gel bath amounts to 37.5 wt.%).



Fig. 5. Volume flux and elimination coefficient versus H_2SO_4 concentration in gel bath 1 - distilled water, 2 - Direct Black Meta aqueous solution

A gel bath containing sulphuric acid (optimum acid concentration approaches 30 wt.%) yields a satisfactory improvement of membrane properties. This is in agreement with the data reported by TWEDDLE and co-workers [17].

The phenomena involved may be (at least partly) explained in terms of the BOKHORST theory [1]. We can expect that the presence of sulphuric acid in the bath accounts for a considerable rise in the diffusion rate of the gelating agent, which makes the polymer precipitate rapidly in the superficial layer of the cast film. This gives a reduced number of large pores in the upper part of the membrane and facilitates solvent washout from the lower part where, as a result of a higher concentration of the polymer at the moment of gelation, a structure with a great number of small pores is formed. A membrane displaying a small number of large pores in its upper part, and a great number of small pores in its lower part has high porosity and, consequently, high permeability and selectivity. On the other hand, membranes of that kind have the disadvantage of being prone to clogging (fig. 5 and paragraph 3.4 of this paper).

There is one more factor that contributes to the increase of membrane permeability with the increasing sulphuric acid concentration in the gel bath - the membrane thickness (tab. 1). Unfortunately, the available casting method eliminates thickness control (and that is why it calls for improvements).

The rapid drop in permeability for the sulphuric acid concentration of 40 wt.% (which indicates a decrease in membrane porosity) is far from being fully understood. This phenomenon is likely to be associated with some complex interactions among the polymer chains, the solvent and non-solvent, or - last but not least - with the mechanism governing the sulfonation reaction.

3.4.TREATMENT OF EFFLUENTS FROM DYE HOUSES BY ULTRAFILTRATION

The membranes used in this study exhibited a relatively high permeability and a high dye separation factor. PS concentration in the casting solution and sulphuric acid concentration in the gel bath amounted to 12.5% and 30 wt.%, respectively. No

solvent evaporation was carried out. The membranes formed via the above route belong to the series 4 of sequence III (tab. 1). Some major parameters of the waste streams from the textile dyeing with Direct Black RT (after pretreatment on an anthracite-sand bed) are listed in tab. 2.

-		1	1		0
1	a	b	1	е	2

Effluent from dyeing process using Direct Black RT						
Exhausted dye bath	Primary rinsing bath					
4852	57.6					
756	11.9					
1:30,000	1:100					
45,000	510					
	ess using Di Exhausted dye bath 4852 756 1:30,000 45,000					

Volume flux was related to the time of membrane operations. The plots are given in fig. 6. The curves in figs. 7–9 provide information on the behaviour of individual pollutants in the permeate and on the efficiencies of their removal as a function of time.



Fig. 6. Volume flux versus time of membrane operation for textile effluents containing Direct Black RT A – exhausted dyeing bath, B – primary rinsing bath

As shown by these data, the permeability of the ultrafiltration membranes decreases rapidly in the initial stage for both effluents. This should be attributed to the following two factors — the tendency of the membranes to clogging, and the phenomenon of concentration polarization. Frequent washing of the membranes was found to be advantageous, allowing recovery of the volume flux which approached its original value.

Apart from the rapid drop at the beginning of the process, membrane permeability remains almost constant throughout, amounting to 0.4 m^3/m^2 a day



Fig. 7. COD in filtrate and COD elimination versus time of membrane operation A – exhausted dycing bath, B – primary rinsing bath



Fig. 8. TOC in filtrate and TOC elimination versus time of membrane operation A – exhausted dyeing bath, B – primary rinsing bath



Fig. 9. Dilution required for complete colour removal in concentrate (1) and in filtrate (2) versus time of membrane operation

A - exhausted dyeing bath, B - primary rinsing bath

and $\sim 0.8 \text{ m}^3/\text{m}^2$ a day for the exhausted dye bath and the primary rinse, respectively. The distinct difference in the volume flux between the two waste streams is not surprising, when taking into account the dye concentrations involved.

The removal efficiencies achieved for the exhausted dye bath and for the primary rinse are as follows: dichromate COD, from 60 to 75% and from 45 to 60%, respectively, and TOC, from 65 to 75% and from 45 to 55%, respectively. No clear correlation was found between separation factor and membrane operation time either for dichromate COD or TOC. The dilution at which complete colour removal is obtained has been decreased significantly - from 1:30,000 to about 1:4,000 and from 1:100 to about 1:20, respectively.

Although the treatment effects can be considered satisfactory, they are not as good as it has been expected. This is likely to be the consequence of insufficient mix at the membrane surface (concentration polarization). To eliminate the shortcoming, it is necessary to start investigations on modification of the design of the membrane module.

4. CONCLUSIONS

1. The degree of dye retention by polysulfone membranes cast on the surface of polymethyl methacrylate bars is strongly correlated with the molecular weight of the dye. Depending on the casting parameters, the membranes are able to yield a complete removal of organic dyes having molecular weights which range between 750 and 2,500.

2. Increase of dye concentration in the aqueous solution accounts for the deterioration of membrane permeability and separation factor. It should be noted that the separation factor generally decreases for low-molecular-weight dyes to remain on a constant level for high-molecular-weight ones.

3. Application of a gel bath involving sulphuric acid of ~ 30 wt.% concentration brings about a considerable improvement of both transport properties and separation factor.

4. Polysulfone membranes cast on the surface of polymethyl methacrylate bars may be of utility as applied to the ultrafiltration of effluents from dyeing processes. Dichromate COD and TOC removal efficiencies achieved by this method range from 50 to 70% (irrespective of the length of membrane operation) at a volume flux between 0.4 and 0.8 m^3/m^2 day and at pressure of 0.18 MPa.

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ZASTOSOWANIE RUROWYCH MEMBRAN POLISULFONOWYCH Do odbarwiania ścieków włókienniczych

Przedstawiono charakterystyki eliminacyjne rurowych membran polisulfonowych formowanych na powierzchni porowatych prętów z polimetakrylanu metylu. Przeanalizowano wpływ wybranych barwników na własności transportowe i separacyjne tych membran. Stwierdzono, że kąpiel żelująca w postaci 30% roztworu kwasu siarkowego powoduje blisko dwukrotny wzrost przepuszczalności hydraulicznej membran. Zbadano efektywność ultrafiltracji ścieków pochodzących z procesów farbowania i płukania tkanin. Po 150 godzinach pracy pod ciśnieniem 0.18 MPa, strumień objętościowy permeatu wynosił od $0.4 \text{ m}^3/\text{m}^2\text{d}$ dla kąpieli farbiarskiej do $0.8 \text{ m}^3/\text{m}^2\text{d}$ dla kąpieli płuczącej, zaś stopień usunięcia OWO zmieniał się od 65 do 75% dla kąpieli farbiarskiej i od 45 do 55% dla kąpieli płuczącej.

ПРИМЕНЕНИЕ ТРУБНЫХ ПОЛИСУЛЬФОНОВЫХ МЕМБРАН ДЛЯ ОТБЕЛИВАНИЯ ТЕКСТИЛЬНЫХ СТОКОВ

Представлены элиминационные характеристики трубных полисульфоновых мембран формированных на поверхности пористых стержнев из полиметакрилата метила. Проанализовано влияние избранных красителей на транспортные и сепарационные свойства этих мембран. Доказано, что студенетельная ванна в виде 30% раствора серной кислоты вызывает почти двойное повышение гидравлической проницаемости мембран. Исследовано эффективность ультрафильтрации стоков из процессов крашения и полоскания тканей. После 150 часов работы под давлением 0,18 МПа, объемный поток пермеата составлял от 0,4 м³/м²д для красительной ванны до 0,8 м³/м²д для полоскательной ванны степень удаления ОWO же изменилась от 65 до 75% для красительной ванны и от 45 до 55% для полоскательной ванны.