

Concentration of organic contaminants by ultrafiltration

Katarzyna Majewska-Nowak*, Malgorzata Kabsch-Korbutowicz,
Tomasz Winnicki

*Institute of Environment Protection Engineering, Wroclaw University of Technology,
Wybrzeze Wyspainskiego 27, 50-370 Wroclaw, Poland
Tel. +48 71 3203639; Fax +48 71 3282980; email: katarzyna.majewska-nowak@pwr.wroc.pl*

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Abstract

The results of concentration experiments on model dye solutions containing anionic surfactant and mineral salts were reported. The objective of the study involved recovery of valuable substances and water reuse. The membrane efficiency as well as dye and surfactant rejections during concentration tests were evaluated. The Mollsep Fiber (Nadir) modules with membranes made of polyethersulfone (10 and 30 kDa) were used in the experiments. The anionic surface active agent (sodium dodecyl sulphate) and sodium chloride (NaCl) were added to the dye (Direct Black) solutions. The concentration experiments were conducted at 0.1 MPa. The effect of concentration factor on the volume flux of dye solutions, dye and surfactant rejection was studied. It was found that membrane permeability and dye separation factor was kept constant during concentration process for all experimental solutions. Surfactant retention was highly influenced by solution composition and subjected to great variations. Nevertheless, it was possible to receive two streams in UF process: concentrated dye solutions and water containing anionic surfactant (SDS).

Keywords: Ultrafiltration; Concentration process; Anionic dye; Sodium dodecyl sulfate; Concentration factor

1. Introduction

Many industries, such as dyestuffs, textile, paper and laundry generate a considerable amount of colored wastewater. It is quite obvious that public perception of water quality is highly influenced by the color. The presence of very small amounts

of organic dyes in water, even at concentrations less than 1 g/m³, is visible and undesirable [1]. More than 700,000 tones of dyes are produced annually and two per cent of this production are discharged directly to water streams [2]. Because of increasingly stringent restrictions on the organic content of industrial effluents it is necessary to eliminate organic load from wastewater before it is discharged.

*Corresponding author.

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Furthermore, used dye and rinsing baths may contain also high quantities of auxiliary organic and mineral compounds (detergents, salts, acids, hydroxides, dispersing and complexing agents, etc.). Purification of these complex effluents is therefore highly advisable, in view of the decrease of wastewater volume, as well as the reuse of valuable substances. It should be pointed out that discharging the wastewater purified with the use of conventional technologies (coagulation, adsorption, active sludge [1,3]) is rather inefficient and contributes to the lost of valuable raw materials.

Membrane technology is being increasingly used in the treatment of textile and laundry wastewater. Although a number of studies have been carried out involving the application of pressure membrane processes in water reuse [4–10], only a few papers deal with recovery of valuable components from exhausted dye or rinsing baths, mostly with the use of nanofiltration [11–14]. The possibility of concentrating dye solutions, as well as saving water, auxiliaries (mostly salts) and energy by utilization of commercially available nanofiltration membranes has been showed [13]. It is interesting to note that in principle there is no literature available on the application of low-pressure membrane processes (microfiltration, ultrafiltration) in the industrial reuse of valuable chemical compounds of exhausted dye or rinsing baths. Merely the recovery of polymeric dyes and low-soluble dyes (indigo) in ultrafiltration process was reported as an efficient process in industrial applications [14]. Also Porter and Gomes [15] found (in laboratory experiments) that it was possible to retain anionic dyes by ceramic microfilters and polymeric ultrafiltration membranes.

It should be stressed out that satisfactory applications of membrane processes to the treatment of complex dye effluents requires wastewater fractionation into three streams (at least): concentrate — rich in organic compounds, permeate — rich in mineral salts, and pure water permeate. Adoption of such a procedure needs

extensive investigations on process efficiency, depending on the molecular interactions between components of the treated solutions. However, only a few papers are focused on the dye and surfactant retention from aqueous dye-detergent mixtures. Most of the investigations deal with MEUF (micellar enhanced ultrafiltration) [16–18], in which only successful organic dye removal is important, whereas data on surfactant rejections are passed over. Generally, in MEUF complete surfactant retention is assumed, because surfactant concentrations are enough high to form large micells. If the surfactant concentration is below the critical micellar concentration (CMC), what usually occurs in real waste effluents, small surfactant monomers appear in the treated solutions. In such a case, the mechanism governing the dye and surfactant retention will be the results of the molecular interactions between the solution components, as well as the interactions between the dye/surfactant molecules and membrane material. The steric and charge effects may lead to unexpected high retentions of solutes. The influence of charge shielding, due to presence of mineral salts should also be taken into account. Van der Bruggen et al. [12] found that the ionic strength as well as the presence of cationic surfactants in dye baths had only a minor influence on cationic dye retention. On the contrary, Khamis et al. [19] found that the presence of dyes and surfactants of the same charge in the treated solutions had positive effect on membrane retention. Tang and Chen [7] confirmed that dye rejection remained almost constant regardless of what salt concentration is used. However, Akbari et al. [20] observed a distinct decrease of dye separation factor with the increasing concentration of mineral salts in the treated solution. Koyuncu et al. [21] showed that various mechanisms (cake formation or adsorption) were responsible for flux decline during nanofiltration of dye and salt mixtures characterized by different ionic strength.

Taking into consideration the poor literature data on applying low-pressure membrane

processes to recovery of valuable compounds from dye exhausted baths, it is advisable to perform ultrafiltration (UF) concentration experiments on aqueous mixtures containing dye, surfactant, and mineral salt. In this paper the effect of solution composition on anionic dye and anionic surface active agent (sodium dodecyl sulphate — SDS) retentions in the course of concentration process with the use of ultrafiltration was described. It should be emphasized that, on the contrary to MEUF process, in the reported experiments the SDS concentration was below the critical micelle concentrations.

2. Materials and methods

2.1. Modules

Commercially available Mollsep Fiber (Nadir) ultrafiltration modules were used in the concentration experiments. The modules were of capillary type and contained membranes made of polyethersulfone. Technical parameters of the experimental modules applied in the semi-pilot tests are given in Table 1.

Table 1
Characteristics of the Mollsep Fiber (Nadir) ultrafiltration modules

Parameter	Module type	
	Mollsep Fiber FUS0181	Mollsep Fiber FUS03C1
Configuration	Capillary	
Membrane material	Polyethersulfone	
Cut off (kDa)	10	30
Effective surface area (m ²)	0.26	0.14
Capillary length (mm)	280	280
Internal capillary diameter (mm)	0.8	1.2
Operational conditions		
Maximum temperature	60°C	
pH	1–12	

2.2. Ultrafiltration process

Concentration experiments, as well as the determination of transport and separation properties of ultrafiltration modules, were carried out using the system presented in Fig. 1. The installation enabled membrane testing in various module configuration. The set-up operated in concentration mode as well as at constant concentration of circulated solution (via permeate recirculation between the feeding tank and the membrane module).

The installation of the cross-flow system incorporated the following major parts: ultrafiltration module (1), hydraulic membrane pump Milroyal C — Dosapro Milton Roy (3), 25 μm prefilter (5), feeding tank (2) and permeate tank (10). Additional sensors protected the setup against the unexpected pressure increase. Experimental solutions were passing through a 25 μm cartridge filter prior to ultrafiltration module. The effective volume of the set-up amounted to 80 dm³.

The concentration experiments together with the evaluation of membrane transport and separation properties were conducted at 0.05, 0.1, and 0.15 MPa and cross flow rate 2–2.5 m/s.

Prior to each cycle, the membrane module has been treated with water at 0.10 MPa, until the constant permeate volume flux was established.

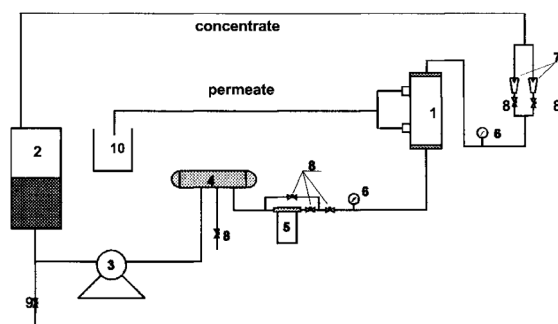


Fig. 1. Semi-pilot UF set-up (1: membrane module; 2: feeding tank; 3: pump; 4: pressure accumulator; 5: preliminary filter 25 μm ; 6: manometer; 7: rotameters; 8: control valves; 9: drain valve; 10: permeate tank).

Permeate volume fluxes and retention coefficients were determined with respect to experimental dye and surfactant after a steady conditions of flow were achieved.

Permeate volume flux (J) was calculated as follows:

$$J = \frac{V}{t \cdot A}, \text{ m}^3/\text{m}^2 \text{ day}$$

where V is permeate volume (m^3), t stands for time (day), and A denotes the effective membrane surface area (m^2).

The experiments on stability of tested membrane modules in concentration of model solutions were carried out at initial feed volume 55 dm^3 . After each hour of the test the permeate flux was measured and concentrations both of anionic dye and anionic surfactant were analysed in the permeate and concentrate.

The degree of solution concentration was established in terms of the concentration factor (CF) calculated by virtue of:

$$\text{CF} = \frac{V_0}{V_t}$$

where V_0 denotes the initial volume of concentrated solution (i.e. 55 dm^3) and V_t is the volume of concentrate after time t . The concentration process was carried out until the volume of concentrate in the feeding tank reached $6\text{--}9 \text{ dm}^3$.

2.3. Experimental solutions

Transport and separation properties of the Mollsep Fiber (Nadir) modules were determined towards distilled water, aqueous solutions of organic dye (Direct Black), aqueous solutions of organic dye and sodium dodecyl sulphate (SDS), and aqueous solutions containing dye, SDS and sodium salt (NaCl).

The concentration tests were carried out with aqueous solutions of Direct Black, aqueous

solutions containing SDS and dye, and aqueous solutions containing dye, SDS and sodium salt (NaCl).

In the aqueous solutions dye and surfactant concentration was equal to 100 g/m^3 . The initial concentration of SDS was below its critical micelle concentration ($\text{CMC} < 2257 \text{ g/m}^3$) [22]. The molecular formula of the detergent was as following: $\text{CH}_3(\text{CH}_2)_{11}\text{OSO}_3\text{Na}$, and the molecular weight of SDS amounted to 288.38 Da . The molecular formula of Direct Black was as following: $\text{C}_{34}\text{H}_{25}\text{N}_9\text{O}_7\text{S}_2\text{Na}_2$ and the molecular weight of the dye was equal to 781.2 Da .

Dye concentration in aqueous solutions was determined spectrophotometrically at a wavelength of 585 nm corresponding to the maximum absorbance of the sample.

The SDS concentration in the feed and the permeate was determined based on the colour reaction (Rhodamine G6 as indicator) and spectrophotometric measurements of the absorbance at a wavelength of 565 nm .

Salt concentration in the tested solutions was equal to 1 kg/m^3 .

3. Results and discussion

3.1. Transport and separation properties of the Mollsep Fiber (Nadir) UF modules

The experiments were aimed at evaluating the efficiency of concentration of dye-detergent aqueous solutions by ultrafiltration. The effect of the solution composition on the process productivity was verified. Two capillary membrane modules (Mollsep Fiber Nadir) characterized by different cut-off values (10 and 30 kDa) were used in the tests.

Prior to concentration process, basic transport and separation properties of the modules were determined. The permeate volume fluxes at three values of transmembrane pressure (0.05 , 0.10 , and 0.15 MPa) are shown in Fig. 2.

The volume flux of distilled water amounted to 3.65 and $5.15 \text{ m}^3/\text{m}^2 \text{ day}$ (at 0.15 MPa) for

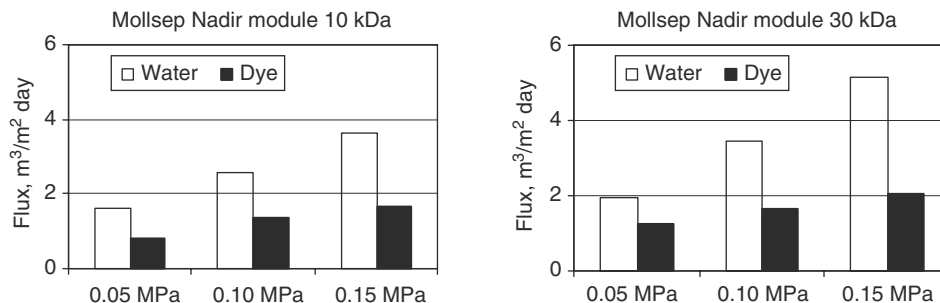


Fig. 2. Volume flux of water and dye solution at three various transmembrane pressures for Mollsep Nadir modules.

10 kDa Mollsep Nadir module and 30 kDa Mollsep Nadir module, respectively. When dye containing solution was passing through the module, a considerable drop in membrane permeability was observed for both modules. This effect was most pronounced for the highest pressure applied. The results obtained indicated that the modules were susceptible to membrane fouling. The 30 kDa Mollsep Nadir module exhibited only a slight higher permeate flux (for dye solution) than 10 kDa Mollsep Nadir module, although the initial values of water flux for both modules were more diverse.

The separation properties towards organic dye (Direct Black) for both modules at three various transmembrane pressures are given in Fig. 3. It was found that the modules exhibited excellent dye rejection (almost 100%), irrespective of the pressure applied. This finding was in agreement with our previous study [23] obtained for flat polytetrafluoroethylene Nadir membranes.

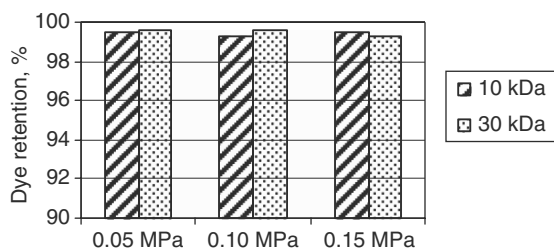


Fig. 3. Dye (Direct Black) retention at three various pressures for Mollsep Nadir modules (10 and 30 kDa).

According to our earlier experiments carried out for flat Nadir membranes [22], the SDS rejection coefficient for PES membranes of 10 and 30 kDa should be expected in the range of 60–68% and 27–30% (at $\Delta P = 0.2$ MPa), respectively.

Taking into account the dye (Direct Black) and SDS retention values obtained in the current and former investigations, it was advisable to perform concentration experiments with the aim of receiving two streams: concentrate — rich in organic dye, permeate — rich in surface active agent (SDS) and/or mineral salt (NaCl).

3.2. Concentration experiments

3.2.1. Module permeability in the course of concentration process

Transport and separation properties of Mollsep Nadir modules were evaluated at constant concentration of dye in the treated solutions, i.e. the permeate was recirculated to the feeding tank. In order to verify the efficiency of concentration process, as well as the possibility of separation dye-surfactant mixtures into useful streams, dye aqueous solutions differing in the composition were subjected to ultrafiltration, which was performed without permeate recirculation. The concentration process was carried out until the volume in the feeding tank reached 6–9 dm³, approximately, i.e. the concentration factor (CF)

took the maximum value of 5.6–9.5, respectively (the final concentrate volume depended on the process mode).

The effect of concentration factor on the volume flux of dye solutions in the concentration process for two Mollsep Nadir modules (10 and 30 kDa) is presented in Fig. 4. Generally, it was found that after initial, rather insignificant, drop in volume flux, the module permeability was kept almost constant during the concentration process. It is worth noting that the Mollsep Nadir

module characterized by 30 kDa cut-off value exhibited membrane permeability at the initial stage of concentration process equal to about $1 \text{ m}^3/\text{m}^2 \text{ day}$. This value is quite similar to volume flux noted for 10 kDa Mollsep Nadir module, although water permeabilities of these modules determined at the preliminary tests differed in a greater extent (Fig. 2). It also turned out that the decrease in module efficiency during the concentration process was a little greater for 30 kDa Mollsep Nadir module than for 10 kDa Mollsep

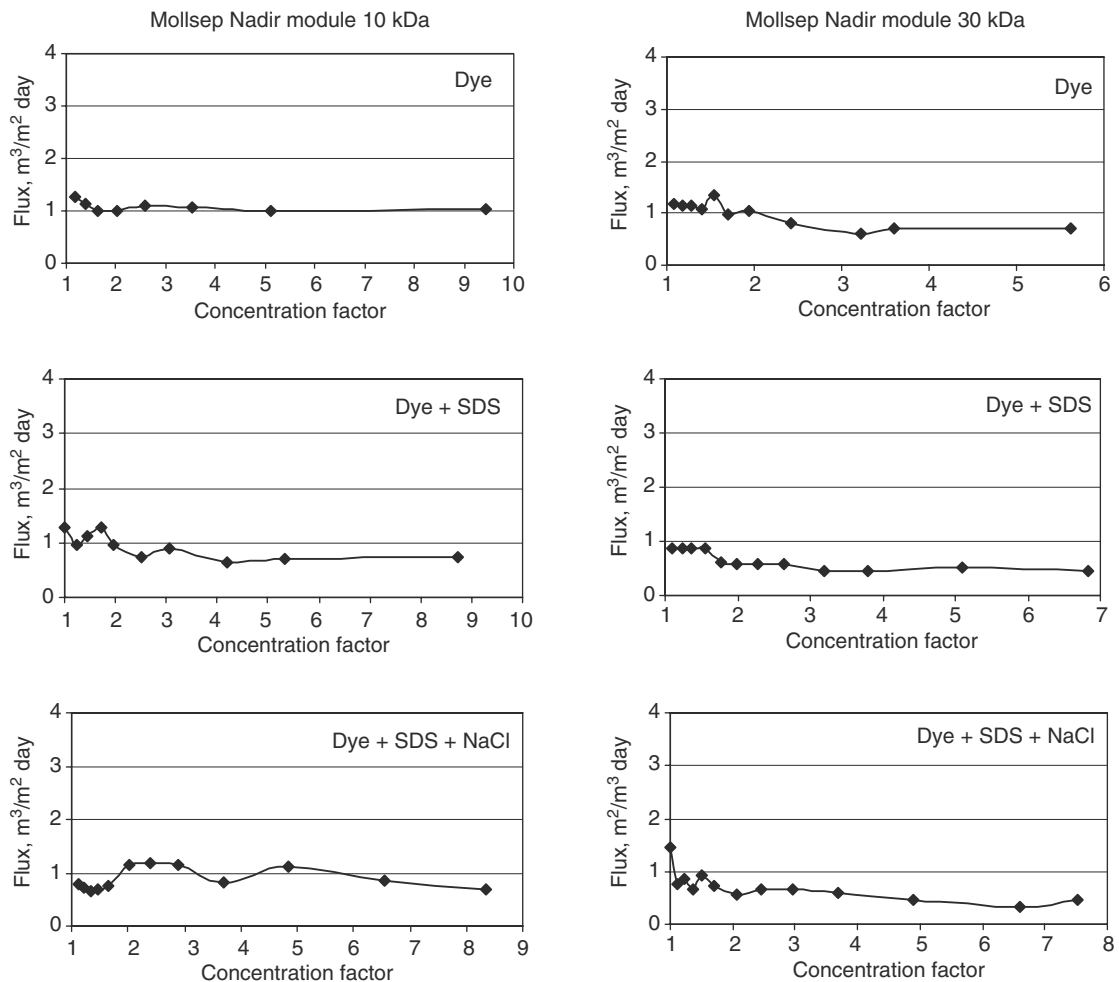


Fig. 4. The effect of concentration factor and dye solution composition on volume flux of Mollsep Nadir modules (10 and 30 kDa) ($\Delta P = 0.10 \text{ MPa}$).

Nadir module. This was an indication that the former module was more susceptible to adsorptive fouling than the second one.

An insignificant diversity in flux values, depending on the solution composition, was observed for tested modules. This finding was more pronounced for 30 kDa Mollsep Nadir module. The presence of SDS in dye solution caused a slight decrease in permeate flux (in relation to solution containing only organic dye). The lowest flux values measured at the end of

concentration process were characteristic of an aqueous mixture of dye, SDS and salt.

3.2.2. Efficiency of dye removal in the course of concentration process

The effect of concentration factor on the efficiency of dye removal is given in Figs. 5 and 6. The permeate quality (Fig. 5) as well as the percentage removal of dye (Fig. 6) in the course of ultrafiltration concentration was evaluated.

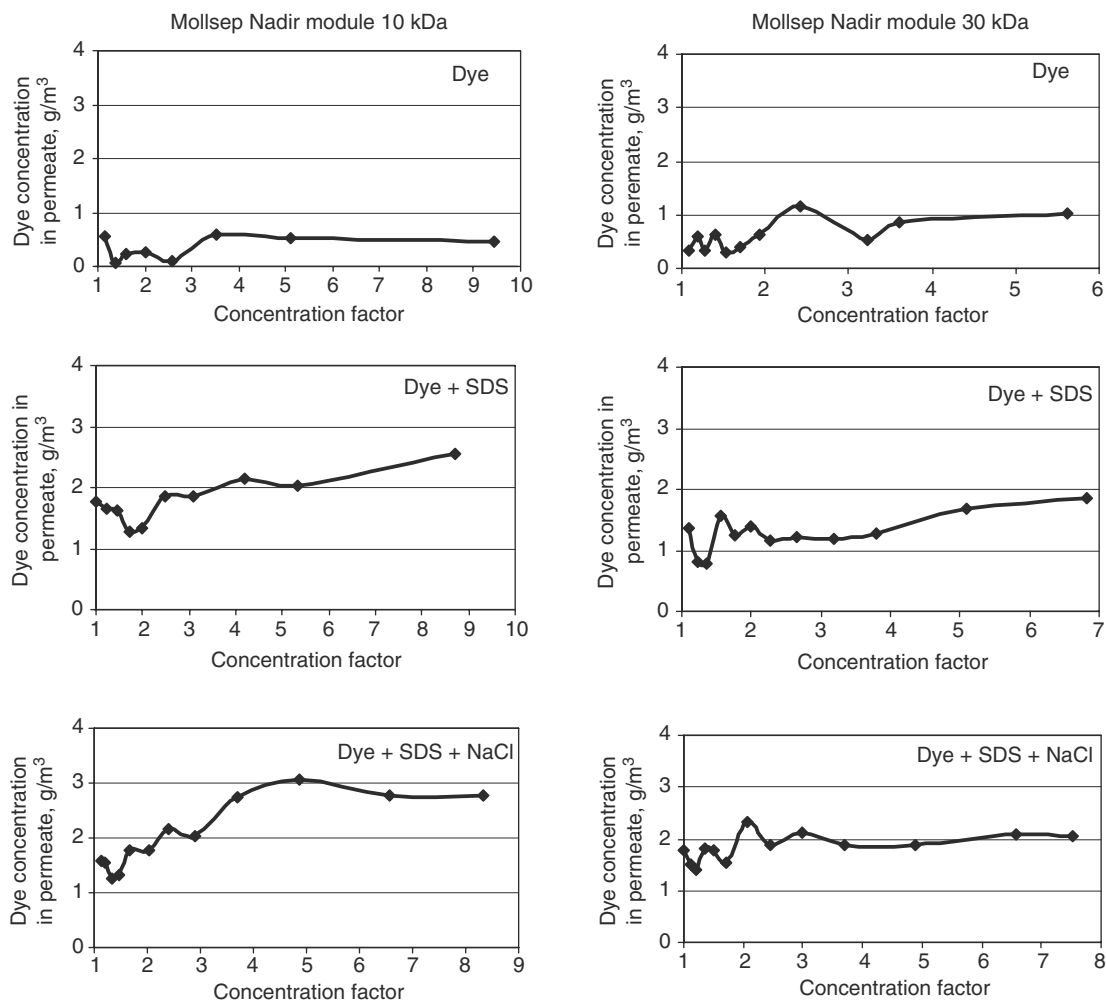


Fig. 5. The effect of concentration factor and dye solution composition on dye concentration in permeate for Mollsep Nadir modules (10 and 30 kDa) ($\Delta P = 0.10$ MPa).

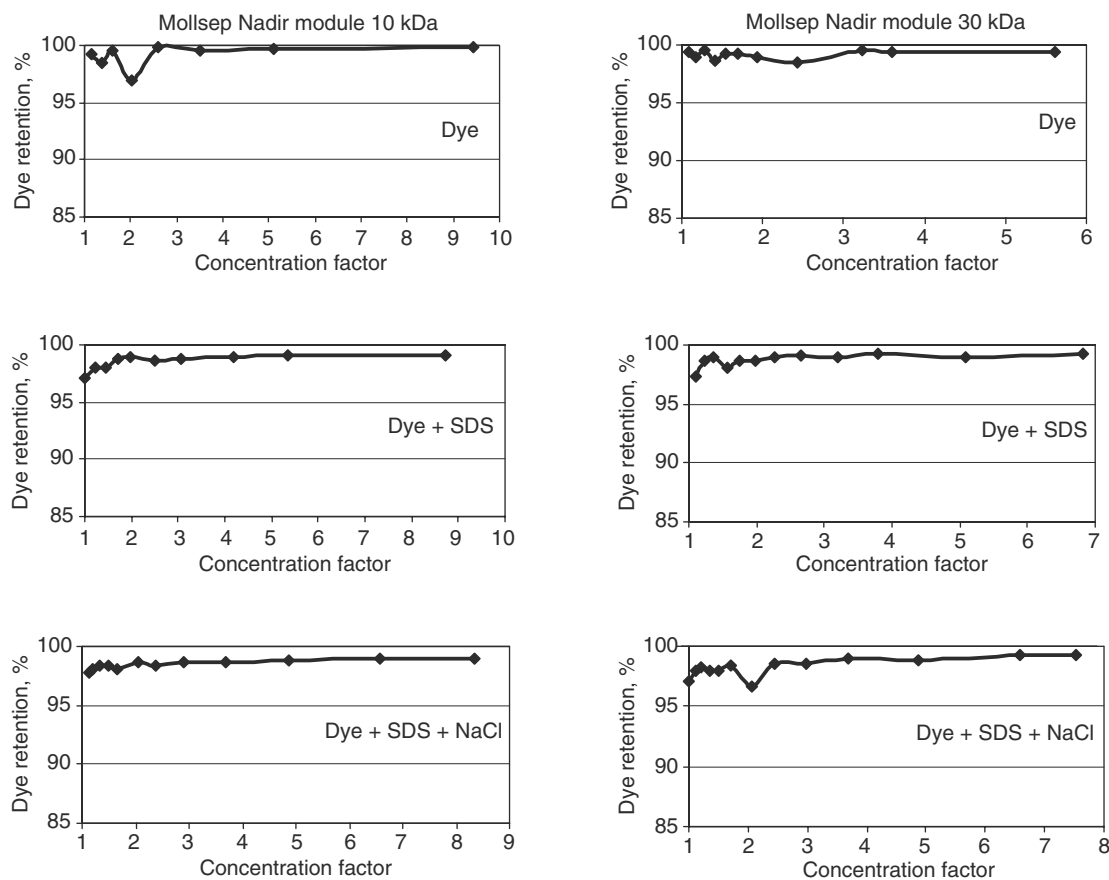


Fig. 6. The effect of concentration factor and dye solution composition on dye retention for Mollsep Nadir modules (10 and 30 kDa) ($\Delta P = 0.10$ MPa).

Based on the results obtained in the concentration process, it was concluded that the both applied Mollsep Nadir modules were characterized by excellent separation properties towards high-molecular-weight dye (Direct Black) – dye concentration in the permeate was below 3 g/m^3 and retention coefficient exceeded 97%, irrespective of the value of concentration factor and solution composition. However, a gradual worsening of permeate quality was observed with the increasing concentration factor. The increase of dye concentration in the permeate during concentration process was especially pronounced for dye mixtures containing both anionic surfactant and

mineral salt, and in a less extent — when SDS was present in the dye solutions. It should be stress out that dye rejection coefficients remained almost constant in the course of concentration procedure.

The observed influence of anionic surfactant on anionic dye removal efficiency can be explained by possible molecular interactions. Owing to the same charge of the experimental dye and SDS, it can be anticipated they do not interact with one another and repulsive electrostatic interactions prevent the formation of the complex between negatively-charged dye particle and an anionic SDS molecule. Furthermore, a conversion of dye pre-aggregates to single dye particles, as a result

of electrostatic repulsion between dye and surfactant particles, can take place, thus increasing the dye concentration in the permeate. These findings were consistent with our former investigations performed with flat hydrophilic membranes [23]. We confirmed a significant drop in anionic dye retention caused by SDS addition to treated dye solution. The report by Simončič and Špan [24] included a similar conclusion — when the same-charged azo dye (Acid Orange 7) and surfactant SDS were used, the noncoulombic interactions were not strong enough to dominate over the electrostatic repulsion between the same-charged ions.

As it has been mentioned, the effect of mineral salts on the separation efficiency of ionic macroparticles has been investigated by many research [7,12,20,21], but the results reported are rather inconsistent. Probably, when the feeds of high ionic strength are treated, the positive ions (i.e. Na^+) in the solution can shield the negative membrane groups, as well as neutralize the negative charges of anionic compounds. Hence,

the electrostatic repulsion between anionic dye particle, anionic surfactant molecule, and membrane ionic groups is markedly weakened. As a result the molecular sieve effect can be dominant and membrane selectivity decreases.

3.2.3. Efficiency of SDS removal in the course of concentration process

The effect of concentration factor on the efficiency of SDS removal is given in Figs. 7 and 8. The permeate quality (Fig. 7) as well as the percentage removal of surfactant (Fig. 8) in the course of ultrafiltration concentration was evaluated.

Based on the results obtained it was concluded that the applied Mollsep Nadir modules were characterized by rather poor separation properties towards anionic surfactant (SDS) in the course of concentration process. No obvious relationships were found between concentration factor, solution composition and efficiency of SDS removal. Some incomprehensible fluctuations

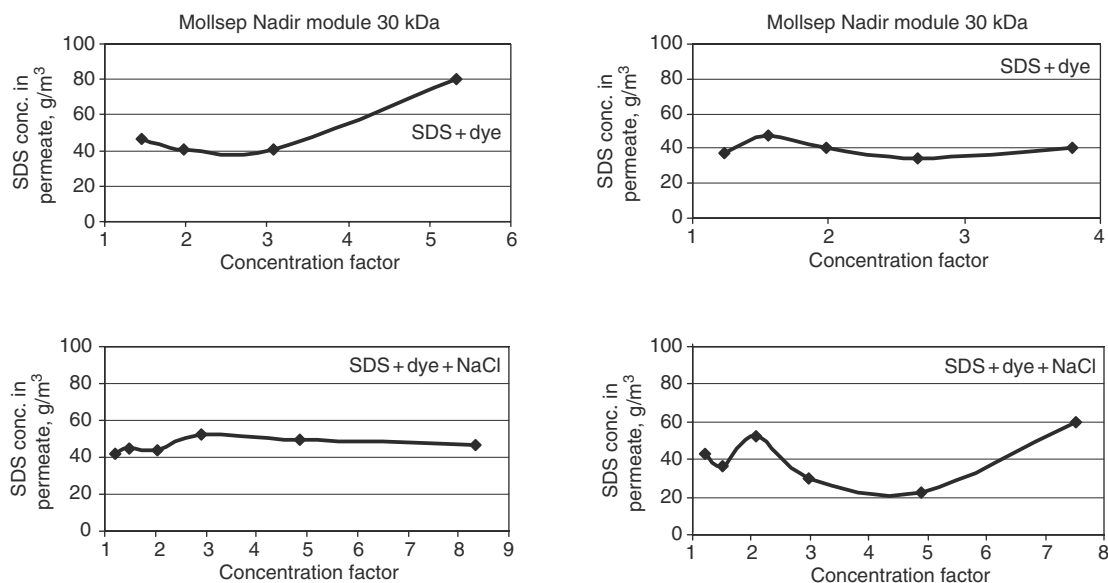


Fig. 7. The effect of concentration factor and dye solution composition on SDS concentration in permeate for Mollsep Nadir modules (10 and 30 kDa) ($\Delta P = 0.10$ MPa).

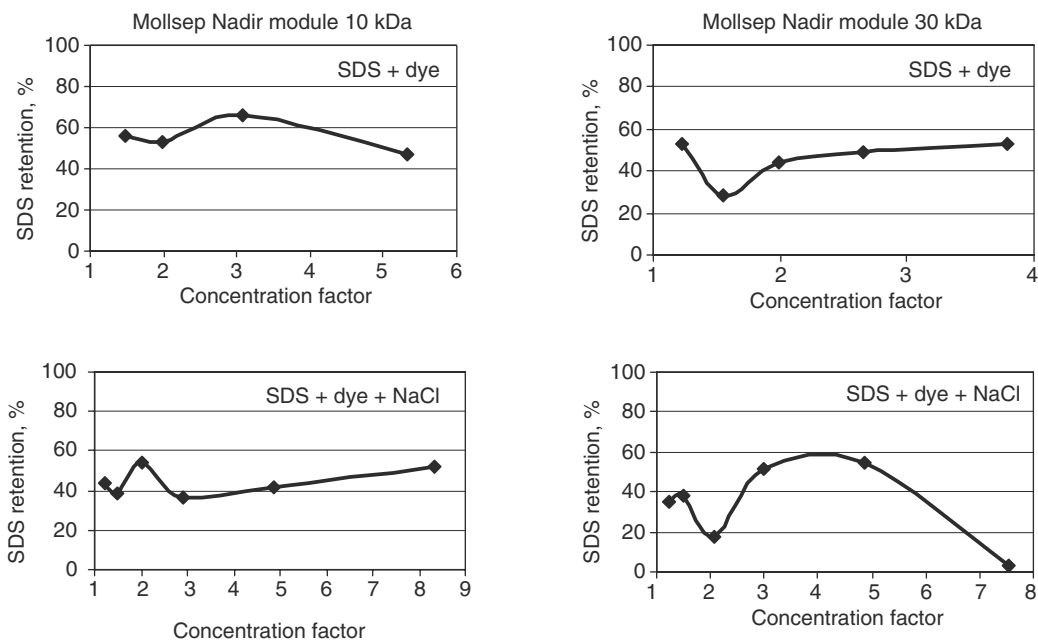


Fig. 8. The effect of concentration factor and dye solution composition on SDS retention for Mollsep Nadir modules (10 and 30 kDa) ($\Delta P = 0.10$ MPa).

in SDS permeate concentrations, as well as in SDS rejection coefficients were observed. The worst results were obtained in the course of concentration process for 30 kDa Mollsep Nadir module and three-component solution — a considerable increase of SDS concentration in permeate and a dramatic drop in SDS rejection was noted.

The effect of anionic dye and mineral salt on SDS separation can be explained similarly to clarification given in paragraph 3.2.2. Due to the same charge of solutes (anionic surfactant and anionic dye), the repulsive electrostatic interactions prevent the formation of high-molecular-weight complexes between dye and surfactant molecules. Even, the appearance of smaller molecules, due to conversion of dye pre-aggregates and surfactant pre-micells, is possible, thus worsening the SDS retention. On the other hand, it can be anticipated that particles of anionic surfactant can play a role similar to that of mineral salts, as in aqueous

solutions they probably weaken the membrane negative charge, but to a lesser extend due to their low ionic strength.

As it was stated, a rather small SDS rejections were obtained. At the beginning of the process the SDS retention coefficients ranged from 35 to 55%. These values were a little smaller than those previously determined for flat membranes made of polyethersulfone [25], however it should be kept in mind, that current SDS retentions were determined for solutions containing dye or dye and NaCl. In consequence, SDS concentrations in permeate were rather high, approaching approximately 20 to 60 g/m³, or even 80 g/m³. However, this seemingly negative finding, can be an advantage of concentration process when dye-surfactant mixtures are treated. Due to low SDS retention with simultaneously very good dye separation, it was possible to fractionate the dye-surfactant solution into two streams: concentrate — mostly rich in organic dye, containing

a certain amount of SDS, and permeate — rich only in surfactant. In order to improve the efficiency of concentration process and receive two useful streams, more opened membranes (characterized by cut-off values higher than 30 kDa) should be tested. The UF membrane should be selected with the aim of ensuring almost complete dye retention and the lowest surfactant rejection.

It seems that the mechanism governing the anionic dye and anionic surfactant separation from their aqueous mixtures by ultrafiltration membranes can be explained mainly by electrostatic repulsion between solutes, adsorption of surfactant monomers and dye particles in membrane pore interior, as well as by molecular sieve effect and interactions between membrane material and solutes present in the treated solutions.

4. Conclusions

- (1) The Mollsep Fiber (Nadir) modules (10 and 30 kDa) yielded over 97% retention of organic dye (Direct Black) irrespective of the value of concentration factor and solution composition. Anionic surfactant (SDS) and mineral salt (NaCl) had a slight negative effect on permeate quality in the course on concentration process.
- (2) The module permeability was kept almost constant during concentration of aqueous mixtures of dye, surfactant and mineral salt.
- (3) The Mollsep Fiber (Nadir) modules (10 and 30 kDa) exhibited poor surfactant (SDS) retention — less than 60%. The content of SDS in permeate was highly influenced by solution composition and subjected to great variations in the course of concentration process.
- (4) It was possible to fractionate the dye-surfactant solution into two streams: concentrate — mostly rich in organic dye, containing a certain amount of SDS, and permeate — rich only in surfactant.

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