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Dyes separation by means of cross-flow ultrafiltration of micellar solutions

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Abstract

Separation of organic dyes from micellar solutions was studied in a cross-flow SEPA CF Osmonics module. Several model dyes (methylene blue, mordant black 11 and mordant black 17) were investigated. Sodium dodecylsulfate (SDS), hexadecyltrimethylammonium bromide (CTAB), oxyethylated coconut fatty acid methyl esters (OMC-10) and binary mixtures of ionic and nonionic surfactants were used as surfactants. Membranes made of cellulose acetate (CQ), polysulfone (PES) and polyvinylidene fluoride (PVDF) (cut-off 15–30 kDa) were used. Ultrafiltration of micellar solutions could be considered as a research method, helpful in determining important parameters such as: (i) the micelle loading and (ii) micelle binding constant used to characterize micellar solutions, and (iii) the distribution coefficient employed in extraction studies. It was found that the type of surfactant is the main factor affecting the rejection. The best separation of the dyes was obtained when the ionic surfactant, charged opposite to dye, was used alone or in the mixture with the nonionic surfactant. Moreover, the introduction of nonionic surfactant into ionic surfactants decreased the CMC of mixed micelles and then the concentration of surfactants in the permeate.

Keywords: Micellar-enhanced ultrafiltration; Methylene blue; Mordant black 11; Mordant black 17

1. Introduction

Production of dyes in the world is estimated to be over 10 000 tonnes per year [1]. About 15% of that amount is lost during the technological processes [2] i.e. dyeing, textile finishing, dye manufacturing, pulp and paper production. Treatment of wastewater containing dyes is one of the most important ecological problems because the effluents containing the dyes are not only highly colored, but also toxic to aquatic life [3]. Textile effluents are highly variable in a composition. They are generally characterized by high concentrations of color, COD, BOD, TOC and dissolved solids. Wool and polyamide are dyed with the acid chrome dyes using the mordant dyeing technique causing the additional contamination of the effluents by high contents of chromium. Acid chrome dyes are the class of dyes that is at the same time most widely used in Eastern Europe and most difficult to eliminate. Due to increasingly stringent environmental legislation, the textile industry is trying to develop effective wastewater treatment technologies. Several techniques used to remove color from wastewater have been examined. Biological processes have been proposed [4]. However, due to the low biodegradability of dyes, conventional biological wastewater treatment is not very efficient [5–7]. Chemical methods such as oxidation and chlorination are more effective [8–11]. Coagulation [12,13] and adsorption onto various supports [13–17] are the most frequently used physical methods. Due to interactions of ionic dyes with oppositely charged ionic surfactants, the extraction of ion pairs can also be used to remove dyes from aqueous streams [18]. However, solvent extraction is not very useful as the concentrations of dye present are usually low and the aqueous stream can be contaminated with diluents. Nowadays the membrane methods of separation are widespread as a method of wastewater treatment [19–23]. The choice of the most suitable membrane process from

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a technical–economic point of view is very important. Having high dye retention, reverse osmosis (RO) and nanofiltration (NF) have been proposed for the treatment of dye waters from the textile industry [23–26]. However, the industries are somewhat reluctant to adopt highly energy-consuming RO and NF processes. Furthermore, NF/RO membranes have a lot more serious issues related to membrane fouling caused by colloids deposition, inorganic precipitation, and biological growth. To overcome these drawbacks, complicated pretreatment and operational techniques are needed [27–29]. In this regard ultrafiltration (UF) appears to be more attractive for wastewater treatment because it gives high fluxes at relatively low pressures. Direct removal of organic substances by ultrafiltration of aqueous solutions is not possible. Such a separation can be achieved using surfactant solutions. Micellar-enhanced ultrafiltration (MEUF) is one of the possible membrane methods to remove traces of organic pollutants from aqueous streams [30–33]. In this process, the surfactant is added to the aqueous stream and it forms micelles of which the diameter is bigger than the membrane pore size. Dissolved organics tend to be soluble in the micelles and usually the more hydrophobic the pollutant is, the more it can be dissolved in the surfactant aggregates [34]. During the ultrafiltration process, micelles containing solubilized organic compounds are rejected by the membrane and a permeate stream passing through the membrane is nearly entirely free from impurities. The main drawback of MEUF is the transfer of surfactant monomers through the membrane being in equilibrium with the micelles and the membrane fouling caused by several factors, including surfactant gelation and adsorption [35]. Thus, the choice of surfactant type is crucial for potential applications. In presented work cationic methylene blue (MB) and two anionic dyes mordant black 11 (MB11) and mordant black 17 (MB17) were used as model dyes. Due to electrostatic interactions increasing the solubilization of dye in micelles of oppositely charged surfactant [36,37] anionic sodium dodecylsulfate (SDS), cationic hexadecyltrimethylammonium bromide (CTAB) and nonionic oxyethylated coconut fatty acid methyl esters (OMC-10) were chosen as model surfactants [34,35,38].

The aims of the work were to study the effects of surfactant and membrane types upon the retention of methylene blue, mordant black 11 and mordant black 17 and to determine parameters such as the micelle loading (L_m), the micelle binding constant ($\log K_P$) and the distribution coefficient (D), typical in colloid and extraction studies.

2. Experimental

2.1. Chemicals

Oxyethylated coconut fatty acid methyl esters of an average oxyethylation degree equal to 10 (OMC-10) from the Institute of Heavy Organic Synthesis, Kedzierzyn Kozle, Poland, hexadecyltrimethylammonium bromide (CTAB) and sodium dodecylsulfate (SDS), both from Merck, Germany were used as surfactants. OMC-10 was synthesized in a direct reaction of fatty acid methyl esters with ethylene oxide [38]. Commercial methyl esters, PK-12-18F from Cognis, Germany were used. Their composition are as follows (in %): C_8 – 6.64, C_{10} – 5.59, C_{12} – 47.73, C_{14} – 19.01, C_{16} – 9.7, C_{18} – 8.65, C_{20} – 0.55, unidentified – 1.03, glycerides – 1.1, water below 0.18, iodine value 10–15 g I_2/100 g. Methylene blue from POCh, Gliwice, Poland, Mordant Black 11 and Mordant Black 17 both from Boruta Kolor, Zgierz, Poland were used as model dyes. The chemical structures of the chemicals are presented in Fig. 1. Deionized water from reverse osmosis was used as a solvent.

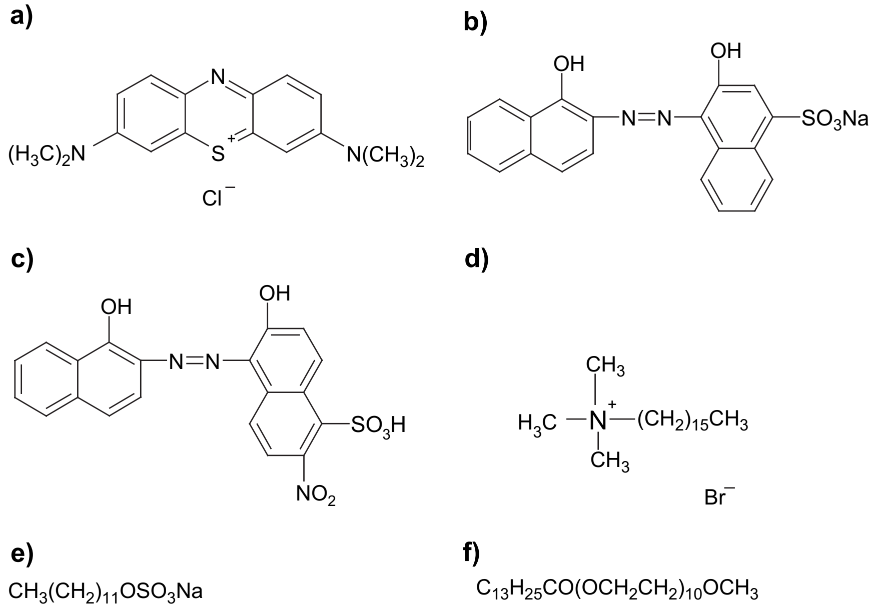

Fig. 1. Chemical structures of (a) methylene blue (MB), (b) mordant black 11 (MB11), (c) mordant black 17 (MB17), (d) hexadecyltrimethylammonium bromide (CTAB), (e) sodium dodecylsulfate (SDS), and (f) oxyethylated coconut fatty acid methyl esters (OMC-10).

Table 1
Membrane parameters

Material	Symbol	Cut-off (kDa)	Max. ΔP (bar)	Possible pH	Max. ΔT (°C)
Cellulose acetate	CQ	15–30	3.5	2–8	30
Polysulfone	PES	15–30	3.5	2–11	90
Polyvinylidene fluoride	PVDF	15–25	3.5	2–11	90

2.2. Membrane cell and membranes

Cross-flow membrane filtration was carried out in SEPA CF Membrane Cell, from Osmonics, USA, equipped with three types of flat sheet polymeric membranes made of cellulose acetate, polysulfone and polyvinylidene fluoride (Table 1). The effective surface area of the membrane was 0.0155 m^2. The membranes were used repeatedly. Prior to ultrafiltration, the membranes were conditioned in deionized water for 24 h.

2.3. Ultrafiltration process

Ultrafiltration was carried out at room temperature in a continuous manner under a pressure difference (ΔP) of 0.2 MPa, with retentate recirculation to the aqueous feed vessel and permeate collection. The initial feed volume was always the same and equal to 1 dm^3 and the process was stopped when 500 cm^3 was taken as a permeate. After each ultrafiltration of the surfactant solution, the membranes were washed by filtration of deionized water for 15 min. The concentration of methylene blue in the aqueous feed was changed from 0.0005 to 0.005 g dm^{-3}. The concentration of mordant dyes was changed within the range of 0.005–0.05 g dm^{-3}. The differences in the considered concentrations of the dyes result from their various light absorption. The surface tension of the surfactant solutions was calculated from the drop shape, using a Tracker tensiometer from I.T. Concept, France. The critical micelle concentrations (CMC) in deionized water were equal to 0.335, 1.96, 0.35, 0.16 and 0.58 g dm^{-3} for CTAB, SDS OMC-10, and binary mixtures of CTAB with OMC-10 and SDS with OMC-10, respectively. The ratio of ionic to nonionic surfactant in the mixed systems was 2.5:2.5 in CMC scale of the individual surfactant. The total concentration of the surfactants used alone or in a binary mixture was always equal to 5 CMC in deionized water. The concentrations of the dyes were determined by UV spectroscopy using a Specol 1200, Analytic Jena, Germany. Blank samples contained an adjusted surfactant concentration (1 CMC, 5 CMC and 9 CMC for permeate, aqueous feed and retentate, respectively).

3. Results and discussion

Table 2 summarizes the absorption wavelength maxima (λ_{max}) of mordant black 11, mordant black 17 and methylene blue in various micellar solutions. For mordant dyes, differences in λ_{max} were observed for each micellar solution. An exemplary Fig. 2 shows the absorption spectra of mordant black 11 (0.03 g dm^{-3}). The spectrum of the dye in aqueous solution exhibits an absorption maximum (λ_{max}) at 540 nm (for mordant black 17, λ_{max} was 530 nm in aqueous solution). In the presence of CTAB (5 CMC), a spectacular change occurred. The intensity of the band at 540 nm gradually diminished with the appearance of a new band at 660 nm with decreasing intensity, as the CTAB concentrations decreased – the intensity of the band was lower when the binary mixture of CTAB (2.5 CMC) with OMC-10 (2.5 CMC) was used. Spectral investigations with nonionic surfactant above its CMC indicated some different features. A shift of λ_{max} to shorter wavelength (530 nm) together with a weak decrease of the absorbance was obtained in the presence of OMC-10. For the surfactant binary mixtures both band characteristic for cationic and nonionic surfactant were present in the absorption spectra of the dye. However, they showed lower intensity, due to surfactants' lower concentrations.

Table 2
Values of λ_{max} of MB 11, MB 17 and MB in various micellar solutions

Surfactant		λ_{max} MB11 (nm)	λ_{max} MB17 (nm)	λ_{max} MB (nm)	Surfactant
CTAB	Permeate Feed Retentate	660	662	660	SDS
CTAB + OMC-10	Permeate Feed Retentate	530, 660	523, 662		SDS + OMC-10
OMC-10	Permeate Feed Retentate	530	523		OMC-10

Efficiency of separation can be characterized by retention (R) defined as:

$$R = 1 - \frac{[\mathrm{d}]_\mathrm{P}}{[\mathrm{d}]_\mathrm{F}} \qquad (1)$$

where $[\mathrm{d}]_\mathrm{P}$ and $[\mathrm{d}]_\mathrm{F}$ denote the concentration of the dye in the permeate and the retentate, respectively. The concentrations of

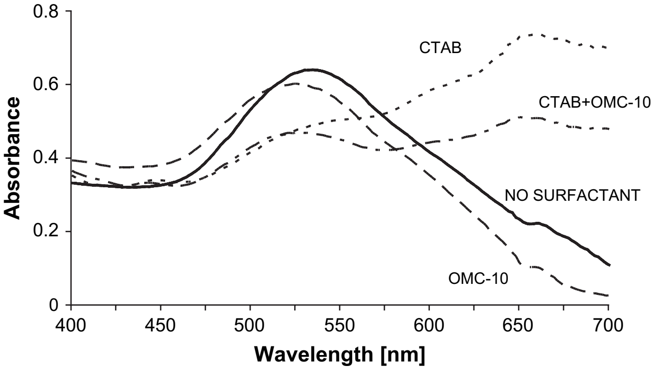

Fig. 2. Visible absorption spectra of mordant black 11 [0.03 g dm^{-3}] in the presence of CTAB, OMC-10, binary mixtures of CTAB and OMC-10 and without an addition of any surfactant. Concentration of the surfactants used alone or in a mixture was equal to 5 CMC.

the dyes did not change during the ultrafiltration time e.g., the concentration of MB11 and MB17 in the permeate were equal to 0.64 and 0.74 mg dm^{-3}, respectively (CQ membrane, dye concentration in the feed solution equal to 0.05 g dm^{-3}). The same was observed for methylene blue – permeate concentration was equal to 0.49 mg dm^{-3} during the ultrafiltration of SDS solution using CQ membrane (initial dye concentration equal to 0.005 g dm^{-3}). An increase of dyes' feed concentration in the considered ranges (0.0005–0.005 and 0.005–0.05 g dm^{-3} for methylene blue and mordant dyes, respectively) did not change the retention. As a result, average values could be calculated from six independent measurements (Table 3). The results obtained indicated that the retention depended on the type of membrane and surfactant used in the experiments. For methylene blue the best retention (above 94%) was obtained for the hydrophilic membrane made of cellulose and SDS solution. The worst recovery ($R = 36.2\%$) was obtained for OMC-10 used alone which could not form the ion-pair with the dye. The same phenomenon was observed for the membranes made of polysulfone and polyvinylidene fluoride.

For both mordant dyes similar values of the retention were obtained when CTAB or its mixture with OMC-10 were used. An advantage of using the binary mixture was a lower concentration of the surfactants in the permeate i.e., 0.160 g dm^{-3} for the binary mixture instead of 0.335 g dm^{-3} for CTAB without any essential reduction in the retention. Due to similar chemical structure of mordant black 11 and mordant black 17 the differences in the solubilization and subsequent retention were not observed. Retention depended significantly on the hydrophobicity of the membranes (Table 3). Similar values of retention were obtained for both hydrophilic membranes (CQ and PES). For the hydrophobic membrane made of polyvinylidene fluoride, retention was smaller for separation of all the dyes. An exemplary Fig. 3 shows the retention of mordant black 11 for all considered surfactant types and for three membranes used in the experiments.

Micelles of surfactants are dynamic aggregates and they are in equilibrium with individual surfactant molecules, passing through the pores of a membrane. The residence time and micelle lifetime, which characterize an exchange of one surfactant molecule between the micelle and the bulk, and the micelle breakdown are very low, i.e. in the order of μs and ms, respectively, although it is impossible to give general values as these characteristic times depend significantly upon the surfactant type and its hydrophobicity. Ultrafiltration occurred continuously and 15–30 min were required to filtrate a volume of 500 cm^3. Thus, the distribution of the dye in both of the pseudophases (micellar and aqueous) and then in the permeate and retentate was in equilibrium. As a result, the ultrafiltration could be used to estimate the distribution coefficients (D) of the dyes between the retentate and permeate, defined as the ratio of dye concentrations in the retentate $[\mathrm{d}]_\mathrm{R}$ and the permeate $[\mathrm{d}]_\mathrm{P}$. Straight-line relationships of

Table 3
Average retention R, micellar binding constant log K_P, micelle loading L_m at the higher dye concentration and distribution coefficient D of mordant black 11, mordant black 17 and methylene blue

Dye	Membrane	Surfactant	R (%)	log K_P[-]	L_m (mol mol^{-1})	D[-]
Mordant black 11 [0.005–0.05 g dm^{-3}]	CQ	CTAB	99.3	4.45	0.045	210.4
		CTAB + OMC-10	98.6	4.69	0.249	93.9
		OMC-10	64.1	2.82	0.076	4.21
	PES	CTAB	99.2	4.43	0.047	199.8
		CTAB + OMC-10	98.5	5.04	0.222	156.7
		OMC-10	25.5	2.08	0.034	1.52
	PVDF	CTAB	82.6	3.03	0.046	9.91
		CTAB + OMC-10	77.1	4.03	0.217	35.6
		OMC-10	14.8	1.78	0.038	1.26
Mordant black 17 [0.005–0.05 g dm^{-3}]	CQ	CTAB	98.9	4.29	0.044	147.6
		CTAB + OMC-10	97.9	4.85	0.251	107.2
		OMC-10	46.5	2.48	0.045	2.35
	PES	CTAB	98.9	4.28	0.044	131.8
		CTAB + OMC-10	97.1	4.61	0.296	52.5
		OMC-10	23.3	2.03	0.036	1.47
	PVDF	CTAB	74.6	2.79	0.042	5.67
		CTAB + OMC-10	69.6	3.44	0.228	4.52
		OMC-10	12.8	1.69	0.037	1.21
Methylene blue [0.0005–0.005 g dm^{-3}]	CQ	SDS	94.3	2.70	6.21×10^{-4}	49.0
		SDS + OMC-10	72.3	2.39	9.83×10^{-4}	6.09
		OMC-10	36.2	2.34	4.93×10^{-3}	2.02
	PES	SDS	91.4	2.51	5.90×10^{-4}	18.5
		SDS + OMC-10	75.5	2.35	9.66×10^{-4}	5.67
		OMC-10	41.3	2.38	5.91×10^{-3}	2.04
	PVDF	SDS	88.8	2.26	5.80×10^{-4}	12.9
		SDS + OMC-10	73.6	2.31	9.48×10^{-4}	5.23
		OMC-10	47.3	2.50	7.25×10^{-3}	2.44

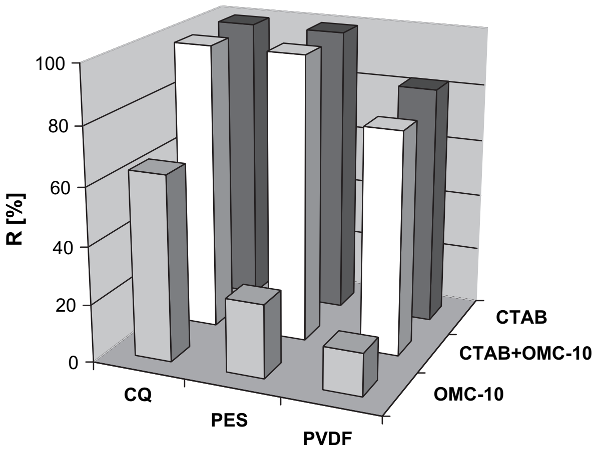

Fig. 3. Average retention of mordant black 11 for three membranes and three surfactants systems.

$[d]_R$ vs. $[d]_P$ with the free term near zero were observed for all cases considered in the research. A typical relationship observed for the ultrafiltration of mordant black 17 in CTAB solution through the PES membrane is expressed as follows: $[MB17]_R = 130.78 \times [MB17]_P - 0.0056$, $R^2 = 0.99$, where R^2 denotes the determination coefficient that confirms the statistical validity of the equation. Thus, the slopes give the distribution coefficients (Table 3).

Retention is well correlated with the distribution coefficient. An exemplary Fig. 4 shows the relationship of retention versus distribution coefficient for mordant black 17. An identical relationship was obtained for mordant black 11 and methylene blue, regardless of the membrane and the surfactant. The distribution coefficient should be above 15 to achieve high efficiency of separation.

The ultrafiltration experiments permitted estimation of loading of micelles (L_m) with solutes, defined by the following equation:

$$L_m = \frac{[d]_R - [d]_P}{[S]_R - CMC}\left[\frac{mol}{mol}\right] \tag{2}$$

derived for the considered case when $V_R = V_P = ½V_F$, where d, S and V stands for a dye, surfactant and the volume of the stream, and the subscripts R, P and F denote the retentate, permeate and feed, respectively. In the conditions of ultrafiltration applied in this work loading depended upon the initial concentration of each dye in the feed approximately linearly in the range of concentrations evaluated. Exemplary Fig. 5 shows this dependence for methylene blue. The linearity suggested that micelles were still able to solubilize more dyes. The membrane type did not affect loading, confirming the reproducibility of ultrafiltration experiments and of the analytical procedure. For methylene blue the use of a nonionic surfactant caused a sharp increase in loading, due to the decreased CMC value. For the mordant dyes the increase in micelle loading was observed when the surfactant mixture was used. This could be explained by an increase in the micelle diameter and decrease in CMC value due to a synergistic effect in the surface tension reduction observed for that mixture. The loadings for the mordant dyes concentration equal to 0.05 g dm^{-3} and for methylene blue concentration equal to 0.005 g dm^{-3} are given in Table 3.

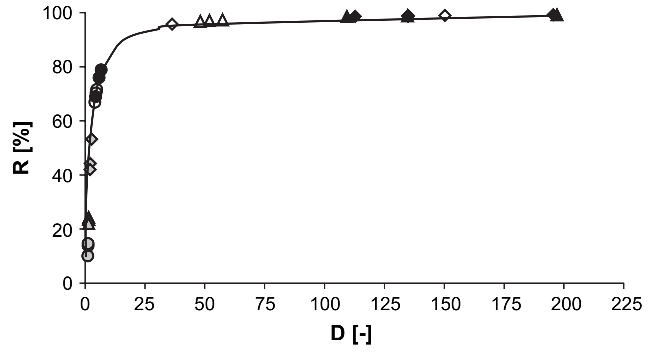

Fig. 4. Relationship of retention versus distribution coefficient for mordant black 11 for three membranes and three surfactants systems (CTAB (5 CMC): CQ (◆), PES (◇), PVDF (◆); CTAB (2.5 CMC) + OMC-10 (2.5 CMC): CQ (▲), PES (△), PVDF (▲); OMC-10 (5 CMC): CQ (●), PES (○), PVDF (●)).

The ultrafiltration data enabled the micellar binding constant (K_P) to be estimated:

$$K_P = \frac{[d]_M}{[d]_W S}\left[\frac{1}{mol}\right] \tag{3}$$

where the subscripts M and W denote the micellar and aqueous pseudophases, respectively, and S is the concentration of the surfactant which contributes to the surfactant pseudophase. Actually $[d]_M = [d]_R - [d]_P$, $[d]_W = [d]_P$ and $S = [S]_R - CMC$. As the retentate consists of a micellar pseudophase and an aqueous pseudophase, the value of C after ultrafiltration of 50% feed volume is equal to $2[S]_F - 2$ CMC. Thus, depending upon the considered system the critical micelle concentration of individual surfactant or of the binary mixture was used. $[d]_M$ and $[d]_W$ are the concentrations expressed in the total volume of the solution. Approximately similar values of the binding constants were obtained for the considered initial

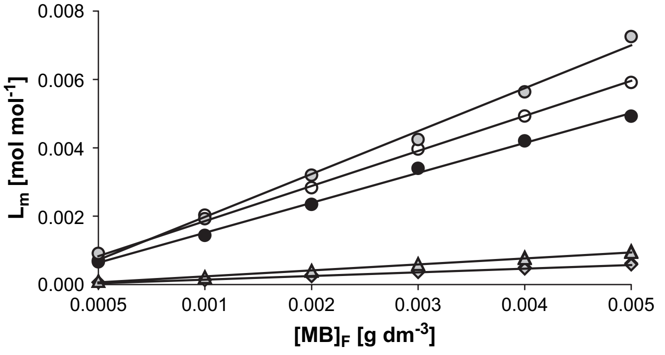

Fig. 5. Loading of micelles with methylene blue for three membranes and three surfactants systems (SDS (5 CMC): CQ (◆), PES (◇), PVDF (◆); SDS (2.5 CMC) + OMC-10 (2.5 CMC): CQ (▲), PES (△), PVDF (▲); OMC-10 (5 CMC): CQ (●), PES (○), PVDF (●)).

concentration range of all the dyes. The average $\log K_P$ values are given in Table 3.

4. Conclusions

Cross-flow ultrafiltration experiments showed the usefulness of membranes made of cellulose acetate and polysulfone for repeated separation of dyes from micellar solutions. The best separation of dyes was obtained when the ionic surfactant charged opposite to dye was used alone or in the mixture with the nonionic one. However, the addition of nonionic surfactant caused substantial decrease in retention of methylene blue. In systems with nonionic surfactant used alone the retention of dyes was dramatically lower. The introduction of nonionic surfactant into anionic SDS or cationic CTAB decreased the CMC of mixed micelles and then the concentration of surfactants in the permeate. Ultrafiltration of micellar solutions can also be considered as a research method, helpful in determining important parameters such as (i) the micelle loading and (ii) micelle binding constant used to characterize micellar solutions, and (iii) the distribution coefficient employed in extraction studies as was shown.

Acknowledgement

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