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# Removal of aromatic compounds in the aqueous solution via micellar enhanced ultrafiltration: Part 1. Behavior of nonionic surfactants

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#### Abstract

The effects of nonionic surfactants having different hydrophilicity and membranes having different hydrophobicity and molecular weight cut-off on the performance of micellar-enhanced ultrafiltration (MEUF) process were examined. A homologous series of polyethyleneglycol (PEG) alkylether having different numbers of methylene groups and ethylene oxide groups was used for nonionic surfactants. Polysulfone membranes and cellulose acetate membranes having different molecular cut-off were used for hydrophobic membranes and hydrophilic membranes, respectively. The concentration of surfactant added to pure water was fixed at the value of 100 times of critical micelle concentration (CMC). The flux through polysulfone membranes decreased remarkably due to adsorption mainly caused by hydrophobic interactions between surfactant and membranes because of hydrophilic properties of cellulose acetate membranes. The surfactant rejections for the cellulose acetate membranes increased with decreasing membrane pore size and with increasing the hydrophobicity of surfactant. On the other hand the surfactant rejections for polysulfone membranes showed totally different rejection trends with those for cellulose acetate membranes. The surfactant rejection trends with those for cellulose acetate membranes. The surfactant and membranes. The surfactant rejections for the polysulfone membranes depend on the strength of hydrophobic interactions between surfactant and membranes. The surfactant rejections for the polysulfone membranes depend on the strength of hydrophobic interactions between surfactant and membrane material and molecular weight of surfactants. © 1998 Elsevier Science B.V. All rights reserved.

Keywords: Micellar enhanced ultrafiltration (MEUF); Nonionic surfactants; Hydrophilic and hydrophobic membranes

## 1. Introduction

The need to separate toxic organic compounds from aqueous solutions is a frequently encountered industrial problem. The process most frequently adopted for this purpose is fixed-bed adsorption but this procedure is neither selective nor energy efficient. Other conventional separation processes such as distillation or extraction often involve a phase change. These techniques are also energy intensive and it is desirable to develop a low-energy separation process. Although a membrane process utilizing traditional ultrafiltration has long been an attractive objective, it has not yet been realized because selective removal of small

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organic molecules can only be attained at very low water flux through the separation membrane.

Scamehorn and coworkers [1,2] recently proposed a micellar-enhanced ultrafiltration (MEUF) method for water clean-up where surfactant is added to the water stream containing organic solutes. When surfactants are present in aqueous solutions above a certain concentration, i.e., the critical micelle concentration (CMC) surfactant monomers begin to assemble in ordered, colloidal aggregates, i.e., micelles having aggregate diameters significantly larger that the largest pore diameter in the separation membrane. One of the most important consequences of micellization is that micelles are capable of solubilizing a certain amount of lipophilic organic compound as solubilizate [3]. The amount of organic compounds that is solubilized depends on the characteristics of surfactant and solubilizate [4]. When the micellar solution is filtered through a membrane having pore diameters smaller than the micelle diameter, most of the surfactant and organic solute remain in micelles in the retentate solution. The MEUF process has been shown to be effective for removing dissolved organics and various cations [1,2,5,6].

From the industrial application viewpoint, however, flow rates through the membrane are important, as well as the rejection of designated species. The most serious limitation of membrane operation is continuous permeation flux decrease that is caused by several factors, such as concentration polarization, adsorption, gel layer formation, and plugging of membrane pores. Even in the absence of solute, a decrease in the membrane permeability has been found for lowrejecting membranes at a certain concentration of surfactant. The flux reduction has in these cases been attributed to adsorption of surfactant molecules in the membrane pores. The adsorption onto membrane surface and pores can be due to various interaction between surfactant and membrane material [7–10].

Even though much work has been done on the MEUF, there is little understanding of the interactions, which affect on the membrane performance. The objective of this work is to obtain the best operation condition for MEUF process in removing organic compounds from the aqueous solution. For this purpose, it is essential to understand interactions between surfactant and membrane materials. In this work, the effects of different types of surfactants on the perfor-

mance of ultrafiltration membranes such as flux and rejection characteristics were studied. Eight nonionic surfactants having different ethylene oxide number and lengths of alkyl chain were examined. Membrane used here were fabricated from the two different polymeric materials, one hydrophobic and one hydrophilic. The hydrophobic membranes were polysulfone and the hydrophilic membranes were cellulose acetate; each having well defined pore size distributions.

### 2. Materials and procedure

The membranes used for MEUF were fabricated from two different types of polymers. The hydrophobic membranes were made in this laboratory from polysulfone (Udel, P-1700) that was supplied by Amoco and the hydrophilic membranes were made from cellulose acetates (PLBC, PLCC, PLGC, and PLTK) that were provided by Millipore. Polysulfone, Udel P-1700, is an amorphous polymer with a glass transition temperature of 185°C. According to the information provided by supplier, molecular weight cut-off (MWCO) for this set cellulose acetate membrane is 3000, 5000, 10000 and 30000, respectively. Flat ultrafiltration membranes of polysulfone, supported by polyester fabrics, were prepared in this laboratory by the wet phase inversion method [11,12]. Polymer solution containing various amounts of polysulfone and dimethylformamide, DMF, was cast onto the nonwoven polyester fabrics using a doctor blade with the thickness of 0.15 mm. The casting solution was immediately precipitated by immersion in a water bath.

Nonionic surfactants of polyoxyethyleneglycol alkylether,  $H(CH_2)_nO(CH_2CH_2O)_mH$ , [hereinafter referred to as  $C_nE_m$ ] were provided by Nikko. The numbers (*n*) of the hydrophobic alkyl group and those (*m*) of the hydrophilic ethylene oxide group in homogeneous series of polyoxyethyleneglycol alkylether,  $C_nE_m$ , were changed systematically for MEUF experiments. Some important characteristics of those surfactants are listed in Table 1.

The ultrafiltration experiments were performed in an Amicon 8050 batch-stirred cell. The feed volume of aqueous solution and the effective membrane area were 200 ml and  $13.4 \text{ cm}^2$ , respectively. Pressure in the batch cell was maintained by nitrogen gas. The

Table 1 Characteristics of nonionic surfactants used in this study

	Molecular weight (g/g mol)	CMC <sup>a</sup> (mol/l)	Micelle diameter <sup>b</sup> (nm)
$C_{10}E_{8}$	510	$1.0 \times 10^{-3}$	5.0
$C_{12}E_{8}$	538	$7.1 \times 10^{-5}$	7.0
$C_{12}E_{7}$	494	$6.9 \times 10^{-5}$	_
$C_{12}E_{6}$	450	$6.8 \times 10^{-5}$	_
$C_{12}E_{5}$	406	$6.5 \times 10^{-5}$	25.0
$C_{14}E_{8}$	566	$9.0 \times 10^{-6}$	_
$C_{16}E_8$	594	$1.0 \times 10^{-6}$	12.0

<sup>a</sup>Data were obtained from [17].

<sup>b</sup>Micelle sizes were measured by dynamic light scattering method. Note that solution concentrations were fixed at the value of 100 times of CMC.

aqueous mixture was stirred at a constant speed. The membranes used in these experiments were soaked for one day in an aqueous solution, containing 50 vol% of ethanol, to saturate the pores before the ultrafiltration runs were started. The measurement of permeate flux and surfactant rejection was performed at 25°C and 3 bar, unless otherwise specified. The concentration of nonionic surfactant was determined colorimetricly [13]. Briefly the blue complex of nonionic surfactant and ammonium cobaltthiocyanate reagent formed in this method is extracted into benzene from a saturated salt solution and then measured with a ultravioletvisible spectrophotometer (Shimadzu model: UV-240) at 320 µm. The concentration of permeate containing higher than 10 mM surfactant was measured using differential refractometer.

### 3. Results and discussion

In practical applications of MEUF process, the surfactant would be added to the feed stream containing the dissolved organic compounds. In applying MEUF process to remove organic compounds from the aqueous solution, several factors have to be considered. These are the membrane properties such as MWCO and hydrophilicity, the characteristics of surfactants ( $C_nE_m$ ) such as *m* and *n*, and the characteristics of the organic compound dissolved in the aqueous solution. The factors can be varied systematically to obtain the best operation conditions for a MEUF process for selective removal of an organic

compound from the aqueous solution. To achieve this objective, hydrophobic polysulfone membranes and hydrophilic cellulose acetate membranes, having various MWCO, were examined. The number (m) of ethylene oxide groups, which affects hydrophilic properties of surfactant, and the length (n) of alkyl chain, which affects hydrophobic properties of surfactant, were also varied systematically. First of all in order to understand the rejection characteristics of organic compounds in MEUF process, the separation mechanisms of nonionic surfactants from the aqueous solution in the absence of organic compounds have to be examined. For this purpose, the mixture of water and nonionic surfactant was examined in this paper; the results of studies involving aqueous solution containing organic compound and nonionic surfactant will be reported later. Figs. 1 and 2 show the permeate flux and surfactant rejection examined with polysulfone membranes at the elapsed time of 100 min. The permeate flux and the surfactant rejection decreased rapidly during the first 30 min and then leveled off asymptotically because of surfactant adsorption at the solid-liquid interface [8-10]. The permeate flux pattern exhibited by cellulose acetate membranes were similar to those exhibited by the polysulfone membranes, but the decrease of surfactant rejection with elapsed time was much smaller in the case of the cellulose acetate membranes relative those noted for polysulfone membranes. Data reported hereafter were determined with the samples collected after the first 30 min.

# 4. Removal of nonionic surfactants with cellulose acetate membranes

It is known that the flux through the membrane cause a convective flow of both permeate and retantate materials towards the membrane surface. Solute concentration will be greater near the membrane surface than in bulk solution because of concentration polarization. The layer formed by concentration polarization reduces flux rates and solute rejection [14–16]. Consequently a stirring bar spinning next to the membrane surface was used to minimize concentration polarization. The relative flux, i.e., the ratio of permeate flux of pure water to that of solution, was examined as a function of stirring speed for each



Fig. 1. Flow rate of water through polysulfone membrane as a function of operating time. Note that polysulfone membrane used here was cast from the solution containing 22 wt% of polysulfone.



Fig. 2. Rejection of  $C_{16}E_8$  surfactant through polysulfone membrane as a function of time.

solution examined here. In such cases the relative flux increased with stirring speed and then leveled off to an asymptotic limit at 600 rpm. Consequently every experiment described here was performed at a stirrer speed of 700 rpm. Note that the relative fluxes at a

plateau and stirring speed were varied with surfactant concentration.

To explore the effects of the length of alkyl chain on the membrane performance, the number of methylene groups (n) of the surfactant,  $C_n E_m$ , was changed systematically from 10 to 16 in increment of 2 while the number of ethylene oxide groups (m) which represents hydrophilic properties of the surfactants  $[H(CH_2)_nO(CH_2CH_2O)_mH]$  was fixed at 8. As listed in Table 1, the CMC values of surfactants increase about 10 fold per decrease of two methylene groups in the hydrophobic part of a surfactant, whereas a change from 8 to 5 ethylene oxide units in the hydrophilic part of a surfactant caused only a slight increase in CMC. For the analysis of experimental results, the concentration of surfactant added to pure water was fixed at the value of 100 times of CMC, unless otherwise specified. As shown in Fig. 3, the permeate flux of pure water through the cellulose acetate membranes increases as the values of MWCO increase. The same trend was observed when the aqueous solution containing surfactant was used as the feed solution. At a fixed MWCO the decline in the flux occurs concomitantly with an increase of surfactant concentration in



Fig. 3. Fluxes of pure water and surfactant solutions through the various cellulose acetate membranes at the operation pressure of 3 bar.



Fig. 4. Surfactant rejections examined with cellulose acetate membranes having various molecular weight cut-off at the operating pressure of 3 bar.

the retentate. Thus Fig. 4 shows that for all of the ultrafiltration membranes used here, the concentration of surfactant appearing in the permeate is less than 10% of surfactant concentration of feed solution. The percent rejection in the cases of  $C_{16}E_8$  and  $C_{14}E_8$  were especially effective, being higher than 98%. The rejection of surfactants ( $C_nE_m$ ) increased with increasing number (*n*) of methylene groups and decreasing

the MWCO value. The average micelle size increases with increase in the alkyl chain length (n) of a surfactant, while it decreases with increase in the ethylene oxide chain length (m) of a surfactant. The average diameter of micelles formed from  $C_{10}E_8$  is the smallest set of those observed using other surfactants. As reported elsewhere [18], the average pore size of ultrafiltration membrane having 30 000 MWCO is smaller than 5 nm. Since the average diameters of micelles formed are always larger than pore diameter of membrane as listed in Table 1, It is logical to conclude that this difference is indeed responsible for the observed increase in retentivity. The numbers (m) of ethylene oxide group in the surfactant were changed from 5 to 8 while the number of methylene (n) group was fixed at the value of 12. Fig. 5 records the surfactant rejection examined with various cellulose acetate membranes, which show that percent rejection of surfactant decreases somewhat with m, but it is greater than 92% even when the surfactant was C<sub>12</sub>E<sub>8</sub>. The surfactant rejection decreased with increasing both the number of ethylene oxide and the value of MWCO of a membrane as shown in Fig. 5. The CMC values of surfactants slightly decrease with decreasing the number (m) of ethylene oxide group while the average size of micelles increases. Those results also indicated that the surfac-



**MWCO of Cellulose Acetate Membrane** 

Fig. 5. Surfactant rejections examined with cellulose acetate membranes having various molecular weight cut-off at the operating pressure of 3 bar.

tant aggregated in the micelle remained in the retentate solution during the MEUF process.

# 5. Removal of nonionic surfactant with polysulfone membrane

Fig. 6 shows the pure water fluxes and rejection of PEG (weight average molecular weight=18 500, polydisperse index=1.03) exhibited by polysulfone membranes. The rejections of PEG examined with the membranes made from the casting solution containing 22 wt% and 23 wt% of polysulfone were higher than 95%. Fig. 7 shows the corresponding solution fluxes through these polysulfone membranes. The decrease of surfactant solution fluxes through the polysulfone membranes (Fig. 7) relative to that for pure water were found to be more pronounced than the corresponding flux decrease observed cellulose acetate membranes (Fig. 3). The decrease of flux observed for the polysulfone membranes using  $C_{16}E_8$  surfactant was especially remarkable. The decline in the solution flux occurred with an increase of surfactant concentration in the case of the cellulose acetate membranes. while a reverse trend was observed for the polysulfone



Weight Percent of Polysulfone

Fig. 6. Fluxes and PEG (weight average molecular weight= 18500) rejections examined with polysulfone membranes at the operating pressure of 3 bar.



Fig. 7. Fluxes of pure water and surfactant solutions through the polysulfone membranes at the operation pressure of 3 bar.



Fig. 8. Surfactant rejections examined with polysulfone membranes at the operating pressure of 3 bar.

membranes. As shown in Fig. 8, the surfactant ( $C_n E_8$ ) rejections increased remarkably with increase in *n*, i.e., the hydrophobic chain length. In general, the surfactant rejections observed for polysulfone membranes were lower than those (Fig. 4) observed for cellulose acetate membranes. Both the MWCO exhibited by PEG (monodisperse and its number average



Fig. 9. Surfactant rejections examined with polysulfone membranes at the operating pressure of 3 bar.

molecular weight=18 500) and the average pore size of cellulose acetate membrane, designated as PLTK (MWCO=30 000), were larger than the corresponding values exhibited by polysulfone membranes. The surfactant rejections observed for the PLTK membranes (Fig. 4) were always higher than those for polysulfone membranes (Fig. 8).

The results observed in correlation of percent rejection of  $C_n E_m$  surfactants by polysulfone membranes with the wt% of polymer in the casting solution, used to make those membranes, show that the efficiency of  $C_n E_m$  rejection increases with increase in *n* at constant m (Fig. 8), and also with m at constant n (Fig. 9). This is contrary to expectations based on the observations recorded in Figs. 6 and 7, which support the point of view that pore size in the polysulfone membrane decreases with increase in the wt% in the casting solution used to make that membrane. The results observed with cellulose acetate membranes (Figs. 3-5) support the point of view that the efficiency of  $C_n E_m$  surfactants should increase with increase in the ratio micelle diameter produced by the surfactant to the diameter of the pore size in the membrane used for separation. The results reported for polysulfone membranes appear to be inconsistent with this hypothesis.

The apparent anomaly may be rationalized on the basis of adsorption of the hydrophobic portion of the surfactant on the membrane surface and along the "cells" of the microporous "passageways", which serves to construct the pore size diameters thereby reducing the flux therethrough. This results in an accumulation of a gel layer on the high pressure side of the membrane thereby impeding flux [8–10,19,20]. Among the various interactions, it is known that hydrophobic interactions play the most important role in the formation of a gel layer and in the adsorption onto the membrane pores [7,21–23]. The hydrophobic interactions are mainly determined by hydrophobic properties of membrane materials and by the properties of both the hydrophilic and hydrophobic parts of the surfactant. In the case of a homologous series of nonionic surfactants, the extent of adsorption decreases with an increase of the numbers (m) of ethylene oxide group and increases with an increase of the alkyl chain length (n). Interaction forces increase with an increase of the numbers of alkyl chains while interaction forces slightly decrease with an increase of the numbers of ethylene oxide group. Molecular weight of surfactant molecule also plays an important role, i.e., the longer chain length the higher is shear force needed to go through the membrane pores. The absolute amounts of water and surfactant to go through the membrane pores decrease with increase of interaction forces and the chain length. Since molecular weight of C<sub>16</sub>E<sub>8</sub> is larger than the other surfactants and hydrophobic interactions of C16E8 with polysulfone membrane are stronger than any other cases, the highest surfactant rejection and the most remarkable flux decrease are observed with C<sub>16</sub>E<sub>8</sub> at the same operating pressure. The rejection of  $C_{12}E_5$  surfactant is the lowest at constant *n* even though the surfactant concentration of  $C_{12}E_5$  is the lowest and the average micelle size is the largest among them. It seems that the chain length plays the most important role when the strength of hydrophobic interaction is slightly changed by the variation of the chain length of ethylene oxide group. The decline of surfactant rejection with an increase of polymer contents in the casting solution also would be explained by the surfactant adsorption. The solution flux decrease with a decrease of pore size. The absolute amounts of surfactant to go through membrane pores may increase because the shear force



Fig. 10. Surfactant rejections examined with cellulose acetate membranes at the various operating pressures.

delivering surfactant increases with a decrease of pore size at the same applied pressure.

For more thorough examination, the membrane performance was tested at various operating pressures. As shown in Fig. 10, surfactant rejections examined with cellulose acetate membrane increased with oper-



Fig. 11. Surfactant rejections examined with polysulfone membranes at the various operating pressures.

ating pressure. The increase of rejection with operating pressure is usually observed in the ultrafiltration and reverse osmosis process mainly due to the water flux increase. On the other hand, Fig. 11 showed that surfactant rejections with polysulfone membranes decreased with operating pressure. The decline of solution flux with polysulfone membranes was also more prominent than with cellulose acetate membranes. As reported elsewhere [20], those results are observed in the systems where the strong attractive forces exist between membrane material and a solute. As shown in Fig. 11, the decline of surfactant rejection with  $C_{16}E_8$  with an increase of operating pressure was the most remarkable. The absolute amounts of surfactant to go through the membrane pores rapidly increase when the operating pressure is high enough to break interactions between membrane material and surfactant.

### 6. Summary

MEUF process could be adopted for obtaining pure water from the aqueous solution containing small amount of toxic organics. The separation mechanism of nonionic surfactant in the absence of organics has been studied in order to adopt MEUF for the rejection of organics. The performance of MEUF process in removing nonionic surfactants was shown to depend on the membrane characteristics, surfactant characteristics, and operating conditions. The concentration of surfactant was fixed at the value of 100 times of CMC. Thus, the absolute concentrations of surfactants were varied with number of methylene group and length of ethylene oxide chain of surfactants. The flux decline for the polysulfone membranes was remarkable while that for the cellulose acetate membranes was not serious. The trend of the permeate flux and surfactant rejection examined with cellulose acetate membranes indicates that surfactant aggregates (i.e., micelles) can be eliminated by filtration using a membrane having pore diameter smaller than the micelle diameter. The solution flux and rejection behavior were similar to those observed in the traditional ultrafiltration and reverse osmosis processes. The surfactant rejections increased with decreasing membrane pore size and with increasing operating pressure. The hydrophobic properties of surfactant affected the membrane performance, i.e., surfactant rejections increased with increase in hydrophobicity of surfactants, which can be controlled by varying the numbers of methylene group and ethylene oxide group. The surfactant rejection behavior for the polysulfone membrane was totally different from that for the cellulose acetate membrane. Surfactant rejections decreased with increasing operating pressure and pore size of a membrane while those increased with increasing molecular weight and hydrophobicity of surfactants. The results obtained with polysolfone membranes indicate that the performance of MEUF process mainly depends on the adsorption characteristics of surfactant on membrane surface and membrane pores and molecular weight of surfactants. Thus, hydrophobic interactions between membrane material and surfactant play an important role in determining the performance of MEUF process.

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