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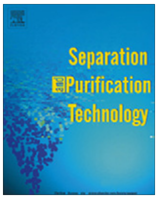

Separation of phenol from various micellar solutions using MEUF

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ABSTRACT

The separation of phenol from micellar solutions using micellar-enhanced ultrafiltration (MEUF) with polyether sulfone membrane was studied. Anionic sodium dodecylulfate (SDS), nonionic triton X-100 (TX-100) and three cationic surfactants, cetyl trimethyl ammonium bromide (CTAB), octadecyldimethylammonium bromide (OTAB) as well as cetylpyridinium chloride (CPC) were used. Several important parameters including distribution coefficient (*D*), concentration of phenol dissolved in the micelles (O_m) and concentration of surfactant in micelle phase (S_m) were determined to evaluate separation efficiency of phenol from various surfactant micelles with different hydrophilic head group and hydrophobic tail length. It was found that the rejection and *D* of phenol examined with CTAB micelles were the highest, while that with TX-100 were lower than SDS at feed surfactant concentration of 10 mM. The rejection and *D* of phenol as well as O_m with three cationic surfactants could be ranked as follows: OTAB > CTAB > CPC, which was contrary to the permeate flux. The rejections of three surfactants were extremely similar with the range of feed surfactant concentration from 1 mM to 30 mM, and S_m examined with OTAB and CPC was higher than that with CTAB.

1. Introduction

The wastewater containing phenol from different chemical and petrochemical industries is discharged into the environment we live in. The great adverse effect of wastewater cannot be ignored. Due to their toxicity, the presence of these compounds in wastewater has directly threatened human health and the ecosystem stabilization. Conventional separation processes, such as distillation, extraction and adsorption, fail to treat wastewater containing organic solutes effectively due to energy–intensity. In addition, traditional ultrafiltration is also ineffective in removing the dissolved low molecular weight organics such as phenol from water [1]. Thus, developing a low energy separation process has been becoming an urgent problem to be studied and solved.

Micellar-enhanced ultrafiltration (MEUF) is a promising separation technique that has been shown to be effective in removing small organic molecules [1–4] and toxic dyes [5–8] as well as heavy metals [9–12] from synthetic produced water. This method has such characters as low-press, better treating efficiency and simple operating, but the main shortcoming is membrane fouling and concentration polarization. The main shortcoming is membrane fouling and concentration polarization which severely limit the MEUF process. A decrease in the membrane permeability results from enhanced membrane fouling effects caused by gel layer formation and plugging of membrane pores. Concentration polarization may promote the convective transport of molecules through the membrane and decrease the filtration efficiency.

In MEUF process, as soon as the surfactant concentration in the aqueous stream exceeds a certain standard, namely critical micellar concentration (CMC), a great number of micelles with aggregate diameters larger than membrane aperture are formed due to aggregation of surfactant monomers. Micelles with solubilizing contaminants are rejected when aqueous stream passes through an ultrafiltration membrane [13]. In consequence, contaminants concentration in permeate stream is much lower than that in initial stream.

Especially, some studies about the MEUF of single phenol and multi-solute systems including phenol in aqueous stream have also been carried out. In the case of MEUF of single phenol system, these researches mainly emphasized the important operating factors of affecting the performance of MEUF in the removal of phenol, such as phenol and surfactant concentrations, operating pressure, temperature, electrolyte, and cross flow rate [1,14,15]. Kim et al. [1] have analyzed the effects of nonionic surfactants with different numbers of methylene groups and oxyethylene groups on the difference of rejection between phenol and benzene. However, the differences of separation of phenol in various surfactants micellar

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solutions (anionic, cationic, nonionic) have not been investigated. In addition, simultaneous removal of phenol and heavy metals (Cr^{3+}, Cu^{2+}) in micellar-enhanced ultrafiltration process have also been reported [16,17]. Witek et al. [16] indicate that the presence of Cr^{3+} in the system does not influence the rejection of phenol.

The aim of this work is to study separation efficiency of phenol from various surfactant micelles with different hydrophilic head group and hydrophobic tail length. In present work, several parameters including distribution coefficient (D), concentration of phenol dissolved in the micelles (O_m) and concentration of surfactant in micelle phase (S_m) were determined. Distribution coefficient is an important parameter of MEUF, which reflects relative affinity of the organic for micelles and measures the tendency of organic to solubilize in micelles. A high D value indicates that surfactants can bind a large number of organics leading to the increase in separation efficiency of organics. O_m and S_m, defined as the subtraction of solute concentrations in the retentate and permeate, contribute to understanding the distributions of organic and surfactant between water and micelle phases. In addition, these parameters also reflect the distributions of organic and surfactant in the retentate and permeate, which benefit further treatment of solutes such as recovery of surfactants to avoid secondary pollution.

In this study, phenol was chosen as a model of wastewater with organic contents. Negatively charged SDS, positively charged CTAB and uncharged TX-100, as three representative types of surfactants, were adopted in MEUF experiments. It was found that phenol interacted strongly with oppositely charged CTAB surfactant due to electrostatic interaction. Then, three cationic surfactants (CPC, CTAB, OTAB) with group difference, i.e., the same hydrophobic tail length but different hydrophilic head group and vice versa, were used to investigate effectiveness of removing phenol by MEUF.

2. Experimental

2.1. Chemicals

Anionic surfactant Sodium dodecylulfate (SDS, analysis purity) was purchased by Fuchen Chemical Reagent Factory, Tianjin, China. Nonionic surfactant TritonX-100 (TX-100, chemistry purity) and three cationic surfactants Cetylpyridinium chloride (CPC, analysis purity), cetyl trimethyl ammonium bromide (CTAB, analysis purity) and Octadecyldimethylammonium bromide (OTAB, purity 99%) were obtained from Sinopharm Chemical Reagent Co., Ltd., Shanghai, China. The properties of surfactants were shown in Table 1 [18–22]. Phenol (analysis purity) was purchased from Huihong Reagent Co., Ltd., Hunan, China. The chemicals were used without further purification. Distilled water was used in all experiments.

2.2. Membranes

A hydrophobic membrane made of polyether sulfone with molecular weight cut-off (MWCO) of 5 k, supplied by Yidong Membrane Engineering Equipment Ltd., Dalian, China, was used in all experiments. The effective area of the membrane is 0.4 m^2 and the pH range is 2–12. The maximum permissible temperature is 50 °C.

2.3. Methods

In the preparing process of feed solution, pre-determined amount of phenol and surfactants were weighted and dissolved into 3000 mL distilled water. After stirred fully and settled for 30 min to allow for formation of micelles and solubilization of phenol adequately, then the solution was treated through the ultrafiltration membrane. All of the experiments were carried out at room temperature of 26 ± 2 °C and a constant operating pressure of 0.15 MPa. The retentate was recycled into the feed tank and the permeate samples as well as the retentate samples were collected at certain intervals. In the experiments, the concentration of phenol added to the solution was kept constant at 1 mM while surfactants concentrations were varied from 1 mM to 30 mM. At the end of the ultrafiltration experiments, the concentrations of phenol and surfactants in permeate and retentate samples were determined. The schematic diagram of ultrafiltration installation was shown in Fig. 1.

After each run, the membrane was cleaned with tap water without pressure for about 1 h, and then the distilled water was filtered to wash away most of deposited surfactants and phenol at low pressure for 20 min. Finally, the membrane permeability was checked to ensure that it returned to the initial water flux within 95%.

2.4. Analysis

In aqueous solution of SDS/CTAB/OTAB and phenol, phenol concentration in the permeate and retentate solutions was determined by a UV spectrophotometer (Shimadzu UV-2550) at a wavelength of 270 nm.

The concentrations of CTAB and OTAB were analyzed by colorimetric method with methyl orange. The samples were measured by UV spectrophotometer at the wavelength of 470 nm, 509 nm to analyze the concentrations of CTAB and OTAB, respectively. In aqueous solution of TX-100/CPC and phenol, the concentrations of phenol and CPC were determined by HPLC having a 25 cm × 0.46 cm column Spherisorb ODS2 (particle size 5 μm). Operating conditions were as follows: mobile phase acetonitrile/water (75/25 by volume), injection volume 20 μL, flow rate 1 mL/min, temperature 30 °C, the UV wavelength 250, 270 nm for CPC and phenol, respectively.

2.5. Calculations

The rejections of phenol and surfactant as well as permeate flux are used to assess the filtration efficiency. Permeate flux as one of

Table 1
Characteristics of surfactants.

Name	MW [a] (g/mol)	CMC (mM)	HLB [b]	Aggregation number [c]	MW of micelle [d] (g/mol)	Solubility [e]
SDS	288	8.2	40	80	23040	s
TX-100	625	0.24	13.5	140	87500	s
CPC	358	0.88	26	95	34010	s
CTAB	364	0.9	15.8	–	–	s
OTAB	392	0.65	–	–	–	s

[a] Mean molecular mass according to the product declaration.
[b] HLB means hydrophile–lipophile balance.
[c] Aggregation number of micelle.
[d] Mean molecular mass of micelle.
[E] s means readily soluble in water.

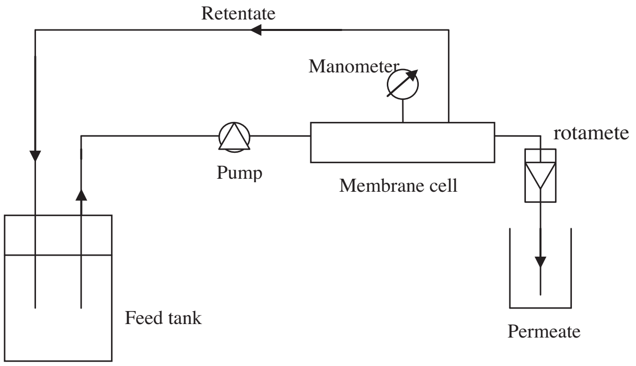

Fig. 1. The schematic diagram of ultrafiltration installation.

effective indicators is defined as the quantity of flow that passes through the membrane per unit area and per unit time. They are calculated using the following equation:

$$R = (1 - C_P/C_i) \times 100\% \tag{1}$$

$$J = V/(\Delta t \times S) \tag{2}$$

where C_P and C_i are the solute concentration in permeate solution and feed solution (mM), respectively. J is the permeate flux (L/m^{2}h). V denotes the permeate volume (mL). Δt and S refer to the time difference (s), the membrane area (m^2), respectively.

It is supposed that only free phenol and surfactant molecules can pass through membrane and their concentrations in the permeate are the same as that in the retentate [19,23]. The distribution coefficient (D) represents the binding force between phenol and surfactant and two other important parameters (O_m, S_m) are closely related to distributions of solutes between water and micelle phases. They are defined as:

$$D = O_R/O_P \tag{3}$$

$$O_m = O_R - O_P \tag{4}$$

$$S_m = S_R - S_P \tag{5}$$

where the subscripts R, P and m denote retentate, permeate and micelle phases, respectively. O represents phenol concentration and S represents surfactant concentration (mM).

3. Results and discussion

3.1. Micellar enhanced ultrafiltration of CTAB, SDS and TX-100

3.1.1. Variations of the rejection and distribution coefficient of phenol with permeate volume

Variations of the rejection and distribution coefficient (D) of phenol with permeate volume in the presence of SDS, CTAB and TX-100 at an operating pressure of 0.15 MPa are shown in Fig. 2. The feed phenol and surfactant concentrations are kept at 1 mM, 10 mM, respectively.

As shown in Fig. 2, it is observed from the rejection and D of phenol decrease with the permeate volume. It is worth noting that the D value examined with CTAB declines dramatically with permeate volume from 400 to 600 mL, and thereafter this downward trend becomes gently, while that with TX-100 and SDS a slight decrease is observed throughout the experiment. For the case of MEUF with CTAB, because phenol gets easily solubilized on the outer periphery of the micelles with positively charges due to electrostatic interaction [24,25], only few phenol molecules occur in water phase and go through the membrane pores smoothly leading to a higher D at the initial stage of the experiment, and then with increasing the permeate volume, concentration polarization occurs leading to the increase in the convective transport of the phenol to the permeate side [14], and thereby increasing the permeate concentration sharply and subsequently decreasing D value rapidly. However, at the end of experiment the permeate and retentate concentrations of phenol reach the balance and D values examined with CTAB, SDS and TX-100 get stable at about 3.5, 1.6 and 1.4, respectively, as shown in Fig. 2.

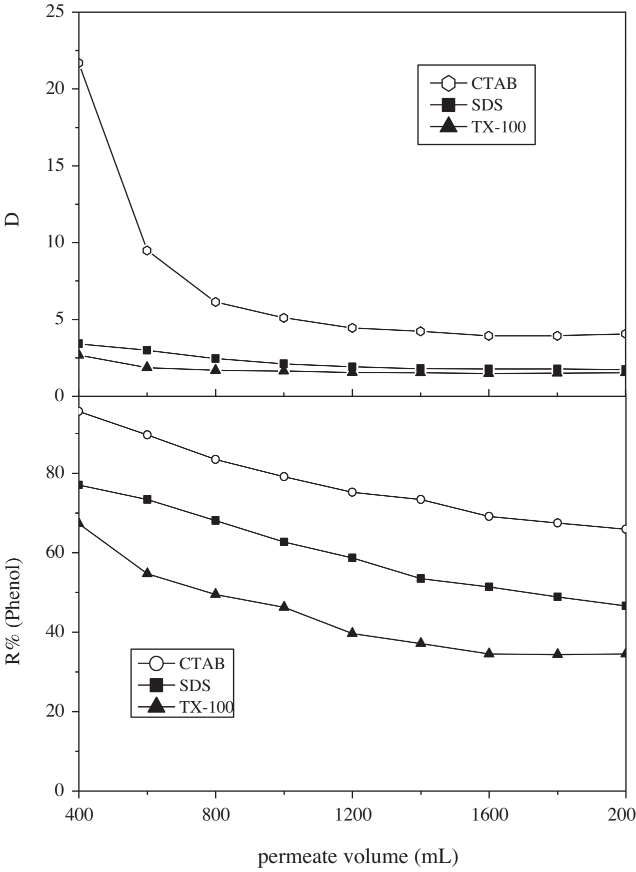

Fig. 2. Variations of rejection and distribution coefficient of phenol with permeate volume in the presence of SDS, CTAB and TX-100. Operating pressure: 0.15 MPa; feed surfactant concentration: 10 mM; phenol concentration: 1 mM.

It is observed that the rejection and D value of phenol examined with CTAB are the highest, which may be explained by the fact that the number of binding sites and relative affinity of anionic phenol for oppositely charged CTAB surfactant increase due to electrostatic interaction as mentioned earlier, leading to a large number of phenol to solubilize in CTAB micelles. On the contrary, the lowest rejection and D value of phenol examined with nonionic surfactant TX-100 are observed, which indicates that the binding force between phenol and TX-100 is minimum due to only extremely weak hydrophobic interaction. These results are accord with the study reported by Zaghbani et al. [7] that methylene blue interacts strongly with oppositely charged SDS surfactant. In addition, because of electrostatic repulsion between phenol and SDS with the same negative charges, the trend of phenol to solubilize in SDS micelles declines leading to the decrease in the rejection and D value of phenol as shown in Fig. 2. These indicate that electrostatic interaction between anionic phenol and ionic surfactants plays a more important role compared with hydrophobic interaction. However, it is worthwhile to note that even though TX-100 has lower CMC and larger aggregation number, the rejection and D of phenol in

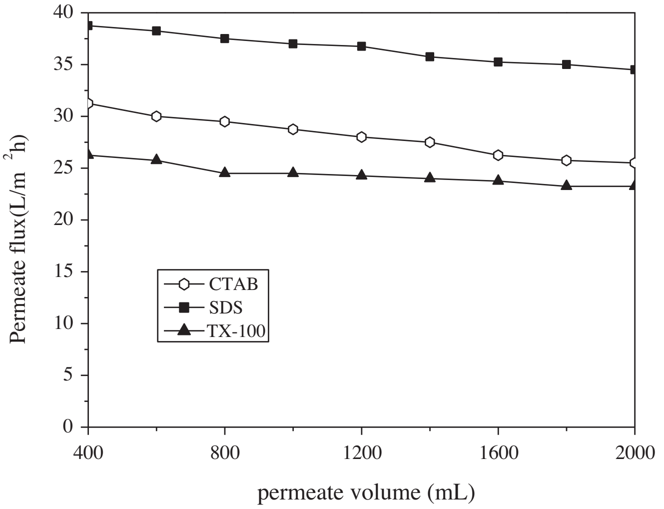

Fig. 3. Variation of permeate flux with permeate volume in the presence of SDS, CTAB and TX-100.

the presence of TX-100 are still lower than that of SDS. Since 10 mM (40CMC) of the feed TX-100 concentration exceeds CMC III (30CMC) [26], i.e. a abrupt point where the size and shape of micelle show abrupt changes with increasing surfactant concentration, the transformation of surfactant micelles occurs at high concentration, thereby reducing the solubilization capability of single micelle [15,26,27].

3.1.2. Variation of the permeate flux with permeate volume

Fig. 3 shows variation of the permeate flux with permeate volume in the presence of SDS, CTAB and TX-100 at an operating pressure of 0.15 MPa. The feed phenol and surfactant concentrations are kept at 1 mM, 10 mM, respectively.

From Fig. 3, it is shown that the permeate flux with the most hydrophilic SDS is higher in comparison to that with the less hydrophilic CTAB and the most hydrophobic TX-100. The decrease in flux is due to the adsorption of surfactants on the membrane surface determined mainly by the hydrophobic interaction between membrane material and surfactants [28,29]. Since polyether sulfone is hydrophobic in nature, the interaction between membrane material and surfactants with more hydrophobic property increases, resulting in adsorption of more surfactants micelles on the membrane surface. Consequently the permeate flux increases with the increase of hydrophilicity (HLB value) of surfactant as following order: SDS > CTAB > TX-100. What's more, the permeate flux is maximum during MEUF with SDS because it may be more difficult to form micelles as higher CMC and lower aggregation number of single SDS micelle as listed in Table 1, leading to fewer micelles in large size deposited over the membrane surface.

3.2. Micellar enhanced ultrafiltration of CTAB, OTAB, CPC

3.2.1. Variations of the rejection, distribution coefficient and concentration dissolved in the micelles of phenol with feed surfactant concentration

Variations of the rejection, distribution coefficient (D) and concentration dissolved in the micelles of phenol (O_m) in the presence of three cationic surfactants are presented in Fig. 4. Phenol is added at a concentration of 1 mM and operating pressure is kept constant at 0.15 MPa.

As shown in Fig. 4, the rejection and D of phenol examined with three cationic surfactants rise continuously with increasing feed surfactant concentration from 1 to 30 mM, while O_m almost grows linearly. Obviously, aggregation number and size of micelles increase with increasing feed surfactant concentration [15], namely, more surfactants are presented in micelle form, and the number of binding sites between surfactant micelles and phenol increases which can be seen from higher D value in the figure. These results lead to more phenol can be solubilized in the micelles (higher O_m), and thus increasing the rejection of phenol.

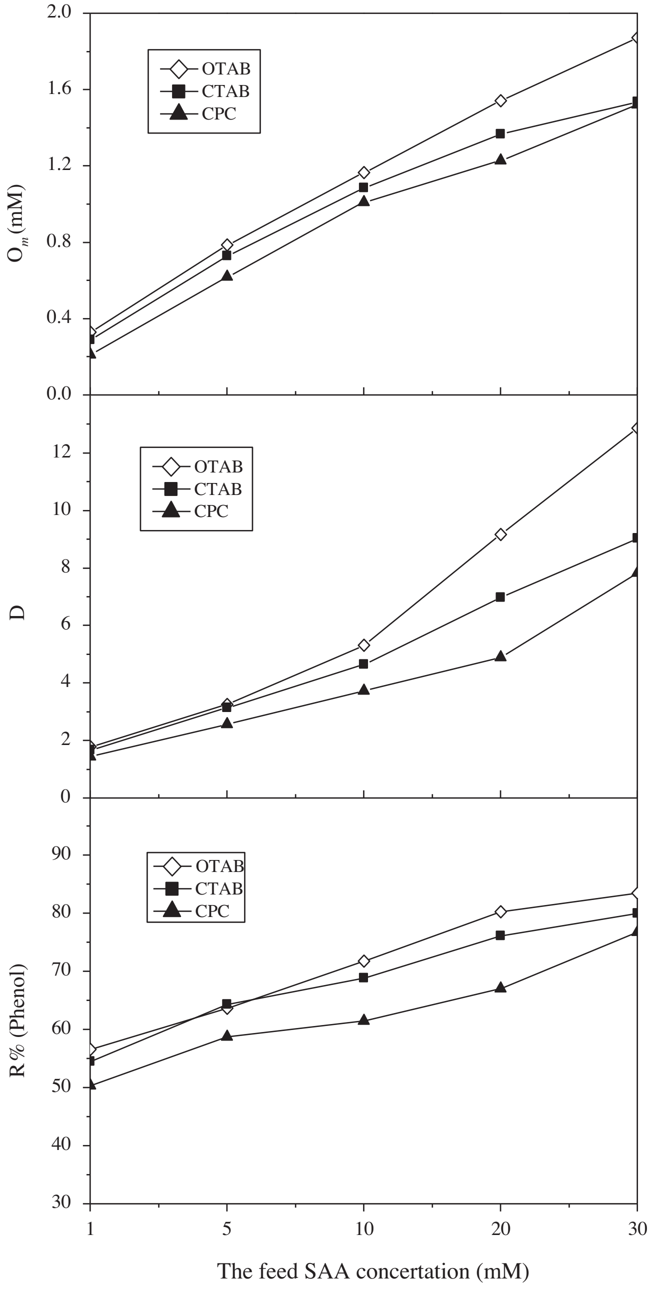

Fig. 4. Variations of the rejection, distribution coefficient and concentration dissolved in the micelles of phenol at various feed surfactant concentrations in the presence of OTAB, CTAB and CPC.

It is observed that the rejection of phenol with OTAB is slightly higher than that with CTAB and CPC. When the concentration of surfactant added to the solution is 10 mM, the rejection of phenol in the presence of OTAB, CTAB and CPC is 71.8%, 68.9%, 61.5%, respectively. The increase in D and O_m with OTAB in comparison to that with CTAB and CPC is also obtained, which indicates that relative affinity of phenol for OTAB increases and phenol molecules are located more deeply in the OTAB micelles with a longer hydrophobic alkyl chain. Because the CMC of OTAB is lower and the aggregation number of micelles is greater (Table 1), larger micelles in average size formed by OTAB more easily occur at lower concentration [1], leading to the increase in phenol concentration solubilized in OTAB micelles. However, even though the value of CMC for CTAB (0.9 mM) is pretty close to that of CPC (0.88 mM) as listed in Table 1, the more hydrophobic CTAB is proved more effective than the less hydrophobic CPC. As for three surfactants, it can be noticed that with the decrease of hydrophilicity, the rejection, D and O_m values of phenol increase as follows by order: OTAB > CTAB > CPC. Structural difference in three surfactants leads to different hydrophilicity as listed in Table 1, and an increase in aggregation number of surfactant molecules in a micelle caused by the decrease in hydrophilicity [30,31] results in increasing the number of binding sites and the tendency of phenol to solubilize in micelles.

However, as shown in Fig. 4, the rejections of phenol in the presence of three surfactants are pretty close to each other with the range of 50–85% when the feed surfactant concentration increases from 1 mM to 30 mM. This may be due to the same rejection mechanism exhibited by three cationic surfactants, i.e. electrostatic interaction plays a key role in separation of phenol.

3.2.2. Variations of the rejection and concentration in micelle phrase of surfactant with feed surfactant concentration

Variations of the rejection and concentration in micelle phrase of surfactant (S_m) at various feed surfactant concentrations in the presence of OTAB, CTAB and CPC are described in Fig. 5. Phenol is added at a concentration of 1 mM and operating pressure is kept constant at 0.15 MPa.

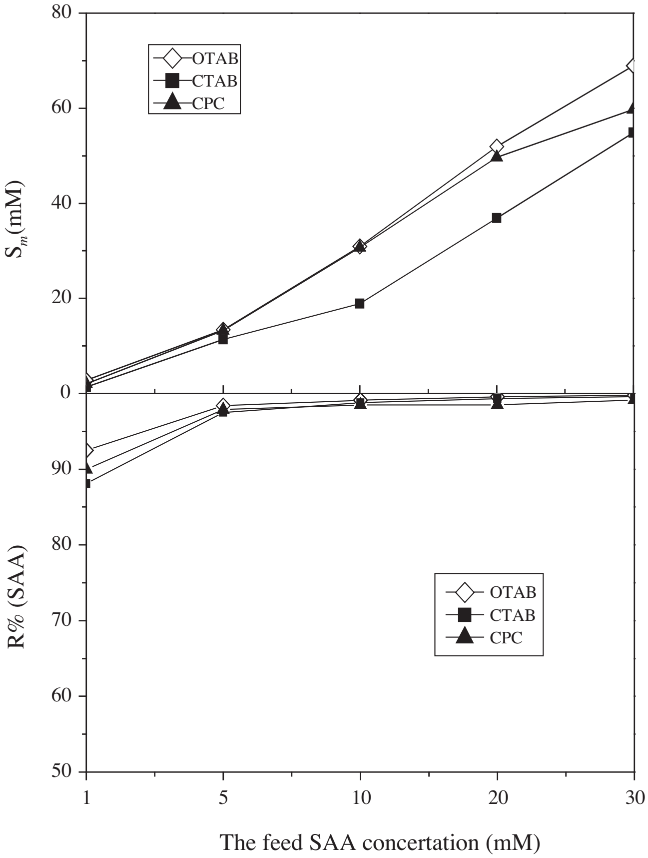

Fig. 5. Variations of the rejection and concentration in micelle phrase of surfactant at various feed surfactant concentrations in the presence of OTAB, CTAB and CPC.

As shown in Fig. 5, it can be seen that the rejections of three surfactants increase rapidly, and then beyond 10 mM the curves flatten out at around 99%, while S_m values increase slowly and are approximate to each other when the feed surfactant concentration varies from 1 mM to 5 mM, and then maintain rapid growth. With the increase in the feed surfactant concentration, the number and concentration of micelles in the solution increase [32], namely, more surfactant molecules go into the state of micelle (higher S_m), so the number of free surfactant molecules in aqueous solution decreases accordingly. Furthermore, only free surfactant molecules can permeate into the permeate tank as previous hypotheses and surfactant concentration in the permeate solution remains around CMC, so according to Eq. (1), the increase in observed rejection of surfactant with the feed surfactant concentration (C_i) increasing is observed. This also explains the fact that the rejections of three surfactants are extremely similar, even up to 99% at higher feed surfactant concentration.

It is observed from Fig. 5 that S_m in the presence of OTAB is higher than that of CTAB, which can be superficially explained as larger amount of micelle aggregation due to lower CMC, leading to the increase of OTAB concentration in micelle phase. In addition, a mess of OTAB micelles are retained and recycled into the feed tank, thus increasing OTAB concentration in the retentate, which is helpful to recovery of OTAB surfactant. However, it is noted that S_m in the presence of CPC is higher than that of CTAB, despite the similar CMC. Because CTAB micelles are easily deposited on the membrane surface due to strong hydrophobic interaction as mentioned in Section 3.1.2, the CTAB concentration in the retentate becomes lower than CPC, while similar concentrations of both surfactants in the permeate are observed at about CMC as discussed earlier. Then according to Eq. (5), the decrease in S_m value is observed in CTAB micelles solution.

3.2.3. Variation of the permeate flux with feed surfactant concentration

Variation on permeate flux in the presence of OTAB, CTAB and CPC is described in Fig. 6. Phenol is added at a concentration of 1 mM and operating pressure is kept constant at 0.15 MPa.

As shown in Fig. 6, it is observed that permeate flux decreases with the feed surfactant concentration, which may be due to accumulation of surfactant monomers adjacent to the membrane surface, leading to the increase in adsorbed layer and concentration polarization resistance [33]. In the case of MEUF with CTAB, when the feed surfactant concentration increases from 1 mM to 30 mM, permeate flux decreases from 32.9 to 23.6 L/m^{2}h. Besides,

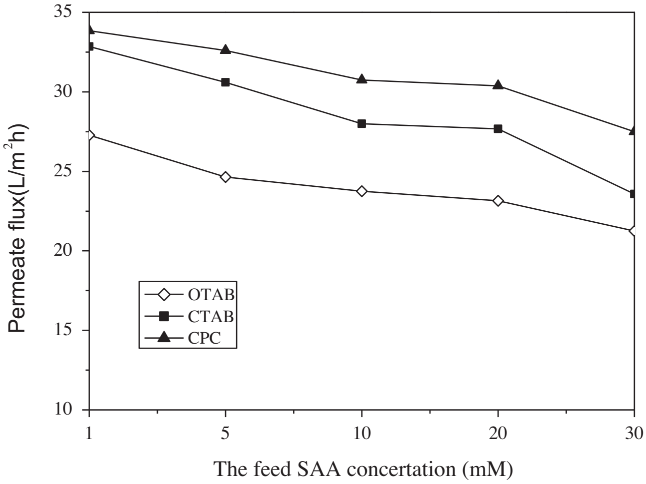

Fig. 6. Variation of permeate flux at various feed surfactant concentrations in the presence of OTAB, CTAB and CPC.

at the same feed surfactant concentration the permeate flux examined with CPC is maximum, whereas that with OTAB is minimum. Because of increasing in hydrophobicity and decreasing in CMC, the increase in the number of micelles deposited over the membrane surface or on the pore walls results in the membrane fouling and pore plugging, and thereby a corresponding decrease of the flux [15,33].

4. Conclusions

The MEUF process of phenol has been investigated in various micellar solutions using polyether sulfone membrane. Anion SDS, cationic CTAB and nonionic TX-100 surfactants were adopted as models of micellar solutions in this study. It is found that the rejection and distribution coefficient (D) with CTAB are higher than that with SDS and TX-100 at feed surfactant concentration of 10 mM due to strong electrostatic interaction, while separation of phenol with TX-100 is proved less effective than SDS owing to the transformation of surfactant micelles. The permeate flux examined with SDS is the highest due to higher hydrophilicity and CMC as well as lower aggregation number of micelle. Three cationic surfactants with different hydrophilic head group and hydrophobic tail length (OTAB, CTAB, CPC) were selected. It is found that the rejection, D and O_m of phenol determined with three cationic surfactants could be ranked in the following order: OTAB > CTAB > CPC, which is attributed to different CMC and hydrophilicity caused by structural difference, whereas the permeate flux shows opposite results. In addition, the rejections of phenol with three surfactants are pretty close to each other with the range of 50–85% when the feed surfactant concentration increases from 1 mM to 30 mM because of the same rejection mechanism. The rejections of three surfactants are extremely similar, even go up to 99% beyond 10 mM of feed surfactant concentration. S_m examined with OTAB and CPC is higher than that with CTAB.

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