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Simultaneous removal of cadmium ions and phenol with MEUF using SDS and mixed surfactants

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ABSTRACT

Micellar-enhanced ultrafiltration (MEUF) is a promising technology to remove metal ions and organic contaminants simultaneously from wastewater. A laboratory experiment was conducted to evaluate the efficiency of a MEUF operation for the removal of Cd^{2+} and phenol using pure SDS and mixed surfactants (Triton X-100/SDS). In pure SDS system, with the increase of the feed SDS concentration, a significant rise in Cd^{2+} rejection was obtained, which peaked at 97.0% with initial SDS concentration being 8.0 mM. Nevertheless, the phenol rejections only kept moderately increasing from 14.5% to 40.0%. In mixed Triton X-100/SDS system, the rejections of Cd^{2+} and phenol were both enhanced by the moderate addition of nonionic surfactant for the same total feed surfactant concentration as the pure SDS system. With the increase of the molar ratios of Triton X-100 to SDS (α), Cd^{2+} rejection increased slightly with α ranging from 0 to 0.8, and peaked at 91.3%. And the phenol rejections kept increasing from 27.7% to 42.4% when α was less than 1.0 followed by slight decrease to 40.2% at $\alpha = 1.5$. Moreover, the SDS dosage and the surfactant(s) concentration in permeate were reduced efficiently. The permeate flux of MEUF with mixed surfactants was lower than that with pure SDS.

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1. Introduction

Micellar-enhanced ultrafiltration (MEUF) is a surfactant-based separation technique which has been employed to remove dissolved organics or multivalent ions from water in the past decade [1–5]. Upon a surfactant being added into the polluted aqueous phase, it aggregates and forms micelles at a concentration higher than its critical micellar concentration (CMC). Micelle can facilitate the solubilization of organic matters and integrates them into its hydrophobic core or/and adsorbs counter metal ions on its surface. The micellar solution is then passed through an ultrafiltration membrane with pore sizes small enough to reject micelles containing the attracted metal ions or/and solubilized organic contaminants [6]. In recent studies, almost all metal ions can be separated via MEUF method, including Cd²⁺ [7], Co²⁺ [8], Ni²⁺ [9], Mn²⁺, Sr²⁺, Cs⁺ [10], Cr^{3+} [11], Zn^{2+} [12], Pd^{2+} [4], Cu^{2+} [13], $AuCl^{4-}$ [14] and $Fe(CN)_{6}^{3-}$ [15]. In those studies, consistently, high removal efficiencies of metal ions with mostly more than 90% have been achieved. Besides, many

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scientists have studied the MEUF of organic matters in aqueous streams [2,16–19].

MEUF is a viable alternative technique which is economical and effective for the cleanup of dissolved contaminants from wastewater compared with conventional techniques since membranes can be added as a retrofit of existing plants. However, previous studies on the removal of dissolved contaminants by MEUF were mainly based on the application in single systems containing either metal ions or organic solutes. In fact, the wastewater from a number of industrial operations (including coal refining, textiles, dyes, and synfuel processing) contains unacceptable concentrations of both dissolved organics and multivalent ions (e.g., heavy metals). Theoretically, as mentioned above, micelles act on metal ions and organics in different locations. It is possible to obtain good removal efficiency of these two types of contaminants by MEUF when they co-exist in wastewater. Simultaneous removal of organic and metal ions with MEUF such as chromate and chlorinated aromatic hydrocarbons, nitrate [20], Cu²⁺ and phenol [21], Cr³⁺ and phenols [22], Cd²⁺ and methylene blue [23], uranyl ions as well as dissolved DBP and TBP [24], were investigated by several authors. Most of them focused on how to obtain high rejection for the removal of dissolved contaminants by MEUF, not including residual surfactant in permeate, which may make the process effluent stream environmentally unacceptable yet [11,25].

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Accordingly, it is highly desirable to use a surfactant system having a low CMC in order to reduce the surfactant concentration in permeate. Previous literatures [7,11,26] have shown addition of small amount of nonionic surfactant to an anionic surfactant, which usually results in a decrease in the CMC of the anionic-nonionic system compared with the CMC of the pure anionic.

This study aimed at investigating the dependence of rejection and flux on feed SDS concentration and/or the molar ratio of Triton X-100 to SDS, as well as optimizing this specific molar ratio. The differential rejection of contaminations and surfactants as well as flux in pure SDS system and anionic-nonionic surfactants system were also comparatively studied. Our primary goal is to experimentally test the ability of MEUF to simultaneously remove phenol and a divalent metal Cd^{2+} from water with anionic-nonionic surfactant and reduce the residual surfactant in the permeate.

2. Materials and methods

2.1. Chemicals

All chemicals were of analytical agent grade. $Cd(NO_3)_2 \cdot 4H_2O$ was selected as heavy metal ions which was purchased from Shanghai Tingxin chemical factory in China. Phenol was supplied by Tianiin Fuchen chemical reagent factory in China. Sodium dodecyl sulfate (SDS) with a purity of 99%, was obtained from Tianjin Kermel chemical factory. Triton X-100 was supplied by Wako Pure chemical industries company in Japan. Their chemical characteristics were shown in Table 1. Distilled water was used as a solvent in all experiments.

2.2. Membranes

The ultrafiltration experiments were carried out in a cross-flow ultrafiltration unit. The hollow fiber ultrafiltration membrane was used, which was offered from Yidong Membrane Engineering Equipment Ltd., Dalian, China. The membrane material is polysulfone which is hydrophobic in nature. Its characteristic was shown in Table 2.

2.3. Procedure

 $Cd(NO_3)_2 \cdot 4H_2O$ and phenol were added into the deionized water to produce the synthetic wastewater with a Cd^{2+} concentration of 0.45 mM, and a phenol concentration of 1.06 mM, respectively. Then the surfactant with its concentration pre-determined was added into the synthetic wastewater. After adequately mixed, the aqueous solution was subjected to the ultrafiltration process. The ultrafiltration experiments were conducted at room temperature (around 20 °C). All of the experiments were run under neutral pH. The transmembrane pressure (TMP) was invariably maintained at 30 KPa, while the volume of feed solution for ultrafiltration was 3.0 L through all experiments. The experiments were performed with the retentate being recycled back into the feed tank, and the permeate solution was reserved in the permeate tank. When the volume of the permeate stream was 2.6 L, namely the volume of the retentate stream was 0.4 L.

After each run, the membrane had to be thoroughly washed to recover its permeability. First, tap water without pressure was used to rinse out the residual synthetic wastewater for 10 min. Then, it was washed with 0.1 mol L^{-1} NaOH, and 0.1 mol L^{-1} HNO₃ at 30 KPa for 10 min, respectively. Subsequently, the ultrapure water of 45 °C was recycled at 30 KPa for 20 min. Finally, deionized water was filtered to determine the permeate flux in order to check the permeability of membrane. The permeate flux of deionized water was $16 L h^{-1}$ as the membrane was thoroughly washed.

2.4. Mechanisms

Fig. 1 shows the schematic diagram of MEUF technique using SDS which can be used to remove Cd²⁺ and phenol simultaneously. The dominant mechanisms for the rejection of metal ions and organic solutes were different. Due to the different forms of aggregates. organic solutes can be solubilized in different locations in the micelles. The nonionic organic solute will tend to solubilize in a hydrophobic core of micelles by ion-dipole interactions and the divalent cationic metal will bind or adsorb on the surface of the oppositely charged micelle [6,22,24].

2.5. Analysis

The concentration of SDS was measured by the methylene blue spectrophotometric method (ISO-7875-1-1996) with Shimadzu UV-2550 (P/N206-55501-93) spectrophotometer from Japan [5]. The concentration of Triton X-100 was measured by UV absorption at 274 nm with a UV-2550 spectrophotometer (Shimadzu) [27]. The concentration of Cd²⁺ was measured by flame atomic absorption spectrophotometer (PerkinElmer) [28]. The concentration of phenol was determined by HPLC and measured spectrophotometrically at 270 nm [19].

3. Results and discussion

Rejection of Cd^{2+} /phenol and surfactant *R* was defined as:

$$R(\%) = \left(1 - \frac{c_p}{c_f}\right) \times 100\% \tag{1}$$

where c_p is the solute concentration in the permeate and c_f is the solute concentration in the feed solution, respectively.

The fresh membranes were compacted at 30 kPa for 10 min using deionized water before experiments conducted. Their permeabilities were measured using deionized water at various pressures. When deionized water is filtered, the permeate flux follows the equation:

$$J_{\rm w} = \frac{\Delta p}{\mu_{\rm w} R_{\rm m}} \tag{2}$$

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Surfactant	Formula	Molecular structure	Molecular weight (g/mol)	Туре	CMC (mM)
SDS	C ₁₂ H ₂₅ OSO ₃ Na	⁰ 0 ⁰ 0 ⁰ 0 [−] Na ⁺	288.38	Anionic	8.0
Triton X-100	$(C_2H_4O)_nC_{14}H_{22}O$	A Contraction of the second	646.86	Nonionic	0.25

Table 2 Characteristics of the used hollow fiber ultrafiltration membrane module.

Туре	Material	MWCO (Da)	Effective area of membrane (m ²)	Max operating pressure (MPa)	pH operating range	Operating temperature (°C)	Max pump power (W)	Number of fibers	Fiber inner/outside diameter (mm)
ZU503-22	polysulfone	10 K	0.3	0.25	1–14	5–45	40	2200	0.2/0.4

where I_w is the permeate flux of deionized water (L m⁻² h⁻¹); where Δp is transmembrane pressure (Pa); μ_w is the viscosity of distilled water (Pa h); and R_m is the intrinsic membrane resistance (m^{-1}) .

3.1. Simultaneous removal of Cd^{2+} and phenol with SDS

Rejection of Cd²⁺ and phenol with SDS concentration ranging from 0 to 16 mM is shown in Fig. 2. The initial concentrations of Cd^{2+} and phenol were fixed at 0.45 mM and 1.06 mM, respectively, and the initial SDS concentrations were 0, 1.0, 2.0, 4.0, 8.0, 12.0 and 16.0 mM.

As shown in Fig. 2, it depicted an immediate rise in the Cd^{2+} rejection with the increasing feed SDS concentration from 0 to 8.0 mM. Theoretically, there are no micelles formed at the SDS concentrations below the CMC, as a result there is no Cd^{2+} rejection. This unanticipated rejection has been accepted in that surfactant concentration in the layer adjacent to the membrane surface was higher than that in the bulk solution due to concentration polarization where the SDS concentration reached to the CMC and the micelles formed to bind Cd²⁺. Moreover, it was interesting to note that above 40% Cd²⁺ and 14.5% phenol were removed in the absence of SDS. This means partial Cd²⁺ and phenol were adsorbed directly on the membrane surface and in the membrane pores over all the set of experiments. According to this mechanism, the true rejection of the solute was no longer a function of the retentate solute concentration but a function of the solute concentration on the surface of the membrane [11]. However, when the feed SDS concentration was higher than the CMC, the Cd²⁺ rejection reached an asymptotic value beyond which a further increase in the SDS concentration cannot induce further increase in the Cd²⁺ rejection. This phenomenon can be explained by the reason that the shape and aggregation number of micelles changed with the increased SDS concentration, and the efficient binding sites did not increase. The rejection of Cd²⁺ peaked at 97.0% when initial SDS concentration was 8.0 mM, which was about 2.2% lower compared with that in single removal run [23]. According to Dunn et al. [6] the removal of the Ni^{2+} and Zn^{2+} was not influenced by the presence of either phenol or o-cresol. On the other hand, the phenol rejection kept moderately increasing from 14.5% to 40.0% with the initial SDS concentration increased, which was much lower than that of Cd^{2+} . The same conclusion has been reported in other literature [21]. The low phenol rejection may be due to a weak



Permeate

Solubilized Pheno

SDS Micelle

Adsorbed

Cd²⁺

solution by micellar enhanced ultrafiltration (MEUF) using SDS micelle.

SDS Monomer

Unsolubilized

Phenol

n E

Dissociative

Cd²⁺ Ow

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Feed

Solution

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tendency for phenol to be solubilized in SDS micelles, which was inferred from the relatively hydrophilic characteristic of phenol molecules with a solubility of 8.2% at 15 °C. There may be an ioninduced dipole interaction between cationic surfactant hydrophilic groups and the π -electrons of aromatic solutes, tending to increase the extent of solubilization. However, this type of specific solutesurfactant interaction may be less important for the anionic surfactant. Hence, whether divalent or monovalent counterions neutralize the micellar charge in the Stern layer and electrical diffuse double layer, there should be no significant effect on phenol solubilization unless the counterions could cause a substantial change in micelle size or shape (e.g., a sphere to rod transition). In the previous experiments, the maximum loading of the micelle with phenol was less than 7%, and calculations have shown that this degree of solubilization will not greatly decrease the extent of counterion binding.

As shown in Fig. 3, with the increase of the initial SDS concentration, the SDS rejection increased firstly and then decreased. As more surfactant micelles formed decrement of the quantity of residual SDS monomers. The augmented micelles may be easily retrained by using an ultrafiltration membrane with appropriate pore sizes. When the feed SDS concentration increased to 8.0 mM or higher, the SDS concentration at the adjacent layer of the membrane surface reached the CMC of SDS already, forming SDS micelles. However, high concentration of SDS could induce the deformation of micelles near the membrane surface and facilitate micelles with metal ions and phenol molecules passing through the membrane.

Fig. 4 shows the typical results for the MEUF of Cd²⁺ and phenol as a plot of the permeate flux versus the time at various initial SDS concentrations. As shown in Fig.4, the permeation flux decreased during the operating. The hydraulic resistance *R*_f of the membrane caused by fouling materials (e.g. gel layer formation, concentration polarization, pore block and pore adsorption) was calculated using permeate flux data and the compressible cake resistance in series model shown below [29]

$$J_{\rm s} = \frac{\Delta p}{\mu_{\rm s}(R_{\rm m} + R_{\rm f})} \tag{3}$$



Fig. 2. Rejections of Cd²⁺ and phenol as a function of SDS concentration in feed.



Fig. 3. Rejection of SDS as a function of SDS concentration in feed.

where J_s is the permeate flux of solution $(Lm^{-2}h^{-1})$; μ_s is the viscosity of solution (Pa h); where Δp is transmembrane pressure (Pa); and R_m is the intrinsic membrane resistance (m^{-1}) . In membrane processes, the retentate stream was continuously recycled into the feed tank. The feed volume decreased and initial concentration of solutes increased continuously, resulting in increased μ_s . Consequently, permeate flux decreased.

In the first 1.0 min, the permeate flux decreased quickly. This behavior was attributed to the concentration polarization, namely SDS micelles deposited quickly on the membrane surface and blocked in the membrane pores in a short time. In Fig. 4, the higher the initial concentration of SDS was, the faster permeate flux decreased. When the feed SDS concentration was below the CMC, most of the SDS molecules existed as free monomers with much smaller size than the pore diameter. Under these conditions, monomers could easily pass the membrane leading to high permeate flux. As SDS concentration increased, the monolayer surfactant coverage of the membrane was completed and the concentration polarization caused the permeate flux to decline.

3.2. Simultaneous removal of Cd^{2+} and phenol with mixed surfactants

Fig. 5 shows Cd^{2+} and phenol rejections in the mixed surfactants system at fixed 4.0 mM feed SDS concentration, 0.45 mM feed Cd^{2+} concentration and 1.06 mM feed phenol concentration with various Triton X-100/SDS molar ratios α . When α was 0.1, the CMC decreased



Fig. 4. Effects of initial SDS concentration on permeate flux.



Fig. 5. Rejections of Cd^{2+} and phenol as a function of Triton X-100/SDS molar ratio α .

from 8.0 mM for pure SDS to 2.04 mM because of synergistic effect [27], which implied that insignificant amounts of micelles formed at such a low surfactant concentration. Generally, in the pure SDS solution, the electrostatic repulsive forces between the hydrophilic head groups of monomeric SDS molecules can hinder micelle formation. When a nonionic surfactant was participated in the micelle formation, parts of nonionic surfactants counterbalance the charge of ionic hydrophilic groups, and thus reduced the electrical repulsion in the Stern layer of the micelles. The decrease in the charge density at the surface of micelle led to a diminish of the electrical potential. As a result, the lowered electrical potential stimulated the formation of micelles with lower CMC, and more SDS became available in the micellar formed to bind with Cd²⁺ [30]. However, an increase in the nonionic surfactant dosage caused a decrease in the degree of counterion binding as the bonding interaction between surfactant micelles and Cd²⁺ being weakened, resulting in a reduced contribution to separation efficiency per SDS molecule in micellar form. The Cd²⁺ rejection increased slightly when α was ranging from 0 to 0.8, and peaked at 91.3%, which implied that the nonionic surfactant tends to lower the CMC more than the degree of counterion binding. When α was above 0.8 the nonionic surfactant lowered the degree of counterion binding more than it lowered the CMC. The Cd²⁺ rejection thus decreased with further addition of Triton X-100. As shown in Fig. 5, the phenol rejection kept increasing from 27.7% to 42.4% as the α increased when α was less than 1.0, and then decreased slightly to 40.2% at $\alpha = 1.5$, which was higher than the phenol rejection with pure SDS. The result is similar to



Fig. 6. Rejections of surfactants as a function of Triton X-100/SDS molar ratio α .



Fig. 7. Surfactant concentration in permeate as a function of total surfactant(s) concentration.

previous studies [1,6]. As indicated by Mukerjee [31], a solubilizate on organic may provide greater stability on larger micelles than smaller ones because the former incorporates a relatively greater number of the solubilizate molecules. When Triton X-100 was added in SDS solution, attachment of ethylene oxygen group (C_2H_4O) to SDS micelles increased the size of the micelles. This effect may increase the average aggregation number of the surfactant. Therefore, the separation performance could be expected to be better in MEUF using Triton-SDS rather than SDS due to the greater stability and capacity of Triton X-100 micelles.

As shown in Fig. 6, the rejection of Triton X-100 increased from 77.9% to 89.8% when Triton X-100/SDS molar ratio α increased from 0 to 1.5 at a fixed SDS concentration of 4.0 mM. However, rejection of Triton X-100 kept invariable approximately when α was higher than 0.3. It indicated that most of the nonionic surfactant molecules were present in the micellar form, even if the mole fractions of nonionic surfactant concentration were very low. As expected, the rejection of SDS increased monotonously from 50.5%–77.3% with the increasing α at a fixed SDS concentration of 4.0 mM, which was much higher than that in pure SDS system. This was probably because a synergistic effect involving the nonionic surfactants promoted the formation of SDS micelles and increased the mean size of the micelles, which may be more easily retrained by ultrafiltration membrane [21].

Fig. 7 shows the permeate surfactant concentration in pure SDS system and mixed surfactants with various feed surfactant(s)



Fig. 8. Effects of initial Triton X-100/SDS molar ratio α on permeate flux.

concentrations at 0.45 mM feed Cd²⁺ concentration and 1.06 mM feed phenol concentration. In the pure SDS solution, with the increase of the feed SDS concentration, the permeate Cd^{2+} or phenol concentration decreased. Oppositely, the permeate SDS concentration increased approximately linearly, but it was below the CMC of SDS. Theoretically, the permeate SDS concentration should be CMC because the membrane with 10 kDa cannot reject free SDS monomers, whose concentration is CMC. This result indicated that actually some permeation of micelles and some rejection of monomers could go on simultaneously [19]. In Triton X-100/SDS solution, at a fixed initial SDS concentration of 4.0 mM, with the increase of the feed Triton X-100 concentration, total surfactant concentrations in permeate decreased gently, which was much lower than the permeate SDS concentration in the pure SDS system under the same conditions. According to the above discussion, the nonionic surfactants could not only improve the retention of Cd²⁺, phenol and SDS, but also reduce the dosage of the surfactants.

Fig. 8 shows the typical results for the MEUF of Cd^{2+} and phenol as a plot of the permeate flux versus the time at various Triton X-100/ SDS molar ratio. Apparently, increasing Triton X-100 concentration exerted a bigger impact on the membrane fouling than increasing feed SDS concentration. Compared to the pure SDS system, at the same total feed surfactant concentration, the mixture of the Triton X-100/ SDS showed a severe flux decline as the concentration of surfactant in the retentate increased. It could be explained as follows: the change in viscosities of mixed surfactants of Triton X-100 and SDS were more severe than that of pure SDS. The solution viscosity was affected by the addition of nonionic surfactant, so according to Eq. (3), the increase of hydraulic resistance against the flux [32]. Besides, it was found that the flux values of Triton X-100/SDS mixed systems were always lower than those in pure SDS systems. This may be due to the higher molecular weight of Triton X-100 compared with SDS. Surfactant molecules with higher molecular weight and longer chain can hardly pass through the membrane [27]. In addition, when Triton X-100 was added into SDS solution, attachment of ethylene oxygen group (C_2H_4O) to SDS micelles increased the size of the micelles leading to the enhancement of gel layer resistance over the membrane surface causing a flux decline. This effect was more pronounced as α increased. According to what is mentioned above, MEUF with SDS was thus better than that with mixed surfactants in terms of permeate flux and membrane pollution.

4. Conclusions

Simultaneous removal of Cd²⁺ and phenol was investigated using SDS and mixed surfactants by MEUF. In pure SDS system, with the increase of the feed SDS concentration, a significant rise in the Cd²⁺ rejections was obtained. Nevertheless, the phenol rejections were comparatively low, keeping a moderate increase from 14.5% to 40.0%, which may be caused by its relatively hydrophilic characteristic. When SDS concentration was fixed at 4.0 mM, the optimal range of the molar ratios of Triton X-100 to SDS for the rejection of Cd²⁺ and phenol was 0.5–0.8. For the same total feed surfactant concentration, the removal of Cd²⁺ and phenol were both enhanced by the moderate addition of nonionic surfactant because the lowered electrical potential stimulated the formation of micelles with lower CMC. With the increase of the molar ratios of Triton X-100 to SDS, the rejection of Cd²⁺ increased first and then decreased, which implied that the nonionic surfactant lowers the CMC more than the degree of counterion binding. And the rejection of phenol increased continuously when the molar ratios of Triton X-100 to SDS was lower than 1.5. Moreover, the SDS dosage and the surfactant (s) concentration in permeate were reduced efficiently in mixed surfactants. The permeate flux of MEUF with mixed surfactants was lower than that with pure SDS, that the former leading more serious membrane fouling, due to the higher viscosity of Triton X-100/SDS mixture solution. As mentioned above, MEUF is feasible to remove Cd²⁺

and phenol simultaneously by the anion–nonionic surfactants, Triton X-100/SDS, with the negligible permeate flux of surfactants.

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