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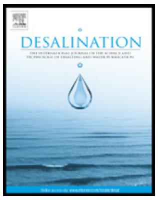

Effects of feed concentration and transmembrane pressure on membrane fouling in Cd^{2+} removal by micellar-enhanced ultrafiltration

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ABSTRACT

In this study, micellar-enhanced ultrafiltration (MEUF) was used to remove Cd^{2+} from synthetic wastewater using a polysulfone spiral wound ultrafiltration membrane and sodium dodecyl sulfate (SDS) as a surfactant. The effects of two important parameters related to membrane fouling in MEUF were investigated: transmembrane pressure (TMP) and feed concentration. The recycling of retentate to the feed tank, which led to continuous growth of SDS concentration, is investigated. The permeate flux and fouling resistance around the critical micelle concentration (CMC) showed that higher SDS concentrations did not always lead to more serious fouling, but fouling became more serious after the SDS concentration reached five times the CMC. With increasing TMP, the flux declined drastically as a result of particle aggregation and gel layer formation. However, the TMP was not always a good criterion of membrane fouling: in different phases, high TMP and low TMP values affected membrane fouling differently; a high TMP such as 0.25 MPa resulted in low membrane fouling at the beginning of the MEUF because of the higher driving power; and a low TMP such as 0.05 MPa caused more serious membrane fouling after stopping because of pore blocking.

1. Introduction

In micellar-enhanced ultrafiltration (MEUF), surfactants are added to wastewater to promote the removal of micropollutants. MEUF shows high efficiency for removal of single metal ions or for simultaneous removal of several types of metal ion(s), such as cadmium and zinc [1–6]. As with most membrane filtration methods, the application of MEUF in water and wastewater treatment requires good control of fouling to prevent significant flux decline, increased operating costs, decreased membrane lifetime, and reduced removal efficiency. Research in this area has focused on the role of several important factors such as water chemistry (pH, ionic environment, and foulant concentration), membrane properties (pore size, hydrophilicity, and charge) and hydrodynamic conditions (TMP and cross-flow velocity). Studies have also been carried out to identify membrane fouling mechanisms (e.g., adsorption, pore blockage, and gel formation) [7].

Fouling in membrane filtration is mainly caused by pore blocking, concentration polarization, adsorption of solids onto the membrane, deposition on the inside membrane surface to form a gel layer, and compression of the gel layer [8,9]. A resistance-in-series model was developed by Tu et al. [10], containing membrane resistance, internal-pore resistance, concentration polarization resistance, and gel-layer resistance. Some authors like Katsoufidou and Jarusutthirak [7–9] have used this model to evaluate flux decline in studies of the treatment of groundwater containing natural organic matter using ultrafiltration (UF) and nanofiltration. This model has proved to be useful in evaluating resistances in membrane filtration. Purkait et al. [11] investigated flux decline during MEUF of an acidic dye with a cationic surfactant using a resistance-in-series model. They found that pore blocking and gel-layer formation caused flux decline. Juang et al. [12] investigated flux decline in a dead-end UF of biosurfactants using this model. They observed that resistance played a more crucial role in flux decline than did gel layer formation and solute adsorption as a result of concentration polarization [13]. Some researchers have divided resistance into reversible resistance and irreversible resistance under conditions in which resistance cannot be clearly distinguished [14]. Various macroregulation and control strategies have been adopted according to the specific conditions in different studies.

Numerous studies of fouling mechanisms have been performed using backwashing to control fouling by reducing the amount of accumulated particles on and in the membrane and increasing membrane flux [7–15]. However, membrane fouling is not always totally reversible by backwashing. The deposited matter on the membrane surface and inside the pores often cannot be totally removed and instead will cause some irreversible fouling, leading to progressive deterioration of membrane performance.

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In previous studies, we reported the effects of pH, temperature, and TMP on removal efficiency and flux decline [16,17]. The objective of the present study was to investigate fouling mechanisms in the treatment of synthetic wastewater, including the effects of feed concentration and TMP on membrane fouling. Recycling of the retention stream led to increasing concentrations of SDS, which made it difficult to analyze the influence of TMP and feed concentrations. To investigate the long-term operational performance of membranes, we stopped the operation of different units for 12 h, without cleaning, to estimate the degree of fouling. In particular, it was confirmed that colloids attached to the membrane surface and in membrane pores could be easily washed out with solutions. The resistance-in-series model was not suitable in these experiments. Different TMPs have different influences on feed concentration, which made it difficult to estimate these parameters independently. Many studies [7–15] have overlooked this point. Membrane fouling and TMP were not always positively correlated. The present study investigated fouling mechanisms without backwashing between adjacent filtration cycles. These results will be helpful in tackling the problems encountered in practical applications of this method.

2. Experimental

2.1. Materials and analyses

In these experiments, the chemical reagents used were all analytical grade. The synthetic wastewater solutions were prepared using deionized water. The membrane material used in all the experiments was polyether sulfone (PES) with a 6-kDa molecular weight cut-off and 0.4 m^2 effective area. The operating temperature was set at 25 °C all over the experiment (Table 1).

Before being used in the experiments, the membranes were treated by soaking in deionized water and washing with a sterilizing fluid (an aqueous solution of 1 wt.% formaldehyde) to remove impurities left after the manufacturing process or additives used for stabilization, and to ensure that the membrane is sufficiently clean [15]. The SDS concentrations were determined by the methylene blue method.

2.2. Experimental setup

The experimental setup consisted of a cross-flow membrane container with a spiral membrane, a stainless steel feed tank, a high-power diaphragm pump to feed the solution into the membrane container, a manometer for showing the operating pressure, and two rotameters for measuring the permeate flux and the retentate flux. The operating temperature was set at 25 °C and the synthetic wastewater solutions were prepared using deionized water. The retentate stream was recycled into the feed tank throughout the experiment. A schematic diagram of the experimental setup is shown in Fig. 1.

Table 1
Chemical agents and company.

Material (molecular)	Company
SDS ($C_{12}H_{25}NaSO_4$)	Tianjin Kermel Chemical Reagents Development Center, China
Cadmium nitrate tetrahydrate ($Cd(NO_3)_2 \cdot 4H_2O$)	Shanghai Tingxin Chemical Reagent Company, Shanghai, China
Disodium hydrogen phosphate dihydrate ($Na_2HPO_4 \cdot 2H_2O$)	Guoyao Chemical Reagent Plant, China
Hydrochloric acid (HCl)	Guoyao Chemical Reagent Plant, China
Concentrated sulfuric acid (H_2SO_4)	Guoyao Chemical Reagent Plant, China
Water purification system deionized water	Labconco, Kansas City, MO, USA
Spiral wound module	Dalian Yidong Membrane Engineering Equipment Co., Ltd., Dalian, China

First, the solutions were stirred using a magnetic stirrer for about 5 min to provide efficient mixing, and settled for 2 h after agitation to ensure formation of micelles of constant size. In the experiments, a set of permeate volumes from 0 mL to 52,500 mL was called a group, and contained five units. The membrane was cleaned using physical and chemical methods at the end of each group, and there were no backwashes between the units, just a stop for 12 h before the next unit. At the end of each group, the membrane was cleaned with six cleaning steps, which included washing the surface for 1 h with water to remove surface deposits, washing with distilled water for 20 min, washing for 5 min with NaOH solution (0.05–0.1%, v/v) to remove organic fouling, soaking in the same NaOH solution for 10 min, and then running a 1-h cycle. After cleaning, the membrane was washed with deionized water until the pH values of the retentate and permeate were 6–7.

In these experiments, the membrane was kept in deionized water, so we defined the retentate and permeate solutions that were not recycled at the first 1 min of each group during MEUF. The feed solutions and permeate were sampled at frequent intervals, based on the permeate volume. The retentate stream was recycled into the feed tank throughout the experiment. The TMP was maintained at a constant value in each group and monitored with a piezometer. The permeate flux (J) could be measured using rotameters but we recorded the static permeate volume times to ensure accuracy.

2.3. Performance parameters and methods

2.3.1. Permeate flux J

The data J reflect the speed of membrane UF, and the permeate flux is expressed as $J(L\,m^{-2}\,h) = 500\,t^{-1}(s)0.694$, where t is the time taken to collect 500 mL of permeate solution. The pure water flux (J_0) was measured first for each group.

2.3.2. Membrane hydraulic resistance

It was observed in the experiments that gelatinoids formed by SDS dissolved easily, and this influenced the distinctions among different membrane resistances. The mass permeate flux during filtration can be expressed, using Darcy's law [18], in terms of a resistance model as $J = \frac{\Delta P}{\mu(R_m + R_f)}$, where J is the permeate flux ($m^3\,m^{-2}\,s^{-1}$), ΔP is the applied pressure (Pa), R_f is the fouling resistance (m^{-1}), μ (Pa s) is the permeate viscosity at the experimental temperature, and R_m, measured using deionized water, is the clean intrinsic membrane resistance, $R_m = \frac{\Delta P}{J_0}$. $R_t = R_m + R_f$, where R_t is the change in total membrane fouling from UF, osmotic effects, and the effects of polarization by concentration, and the effects of fouling are all included in R_f, which is subdivided into reversible fouling and irreversible fouling.

Loosely attached foulants, which cause reversible fouling resistance, are easily removed by “flushing” the membrane with distilled water; a strong shear force corresponding to a high cross-flow velocity is applied

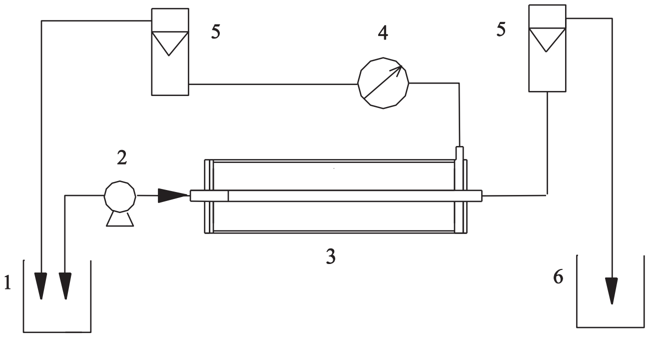

Fig. 1. Schematic diagram of the experimental setup: (1) feed tank, (2) diaphragm pump, (3) membrane, (4) manometer, (5) rotameter, and (6) permeate tank.

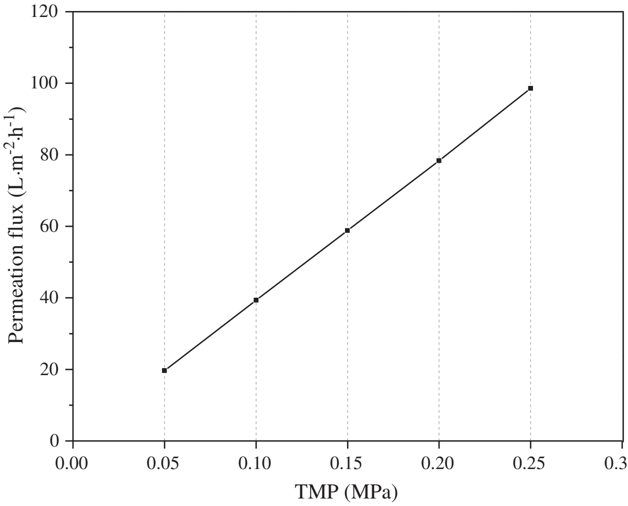

Fig. 2. Relationship between TMP and pure water flux.

to clean the fouled membrane surface. Strongly attached foulants still remain after flushing and are the cause of irreversible fouling [19]. In the experiments, $R_i = R_{fn} - R_{f(n-1)}$ was used to describe changes in accumulated fouling between neighboring units, where n means the number of group.

3. Results and discussion

To describe the trends in the figs easily, we named permeate volumes 0–10,500 mL, 10,500–21,000 mL, 21,000–31,500 mL, 31,500–42,000 mL, and 42,000–52,500 mL for the first, second, third, forth, and fifth unit, respectively. In the figs, the abscissas are divided into five parts corresponding to these units, and this format is used throughout the paper. Each group was divided into five units, and there was a 12-h stop between units in order to evaluate long-term fouling. The feed solutions were all prepared using the same method for each unit of the same group.

3.1. Pure water fluxes and membrane intrinsic resistance

Pure water flux measurements were performed before the start of each fouling experiment. These measurements provided a benchmark for gauging the changes in permeate flux in terms of varying TMP when the clean membrane was fouled.

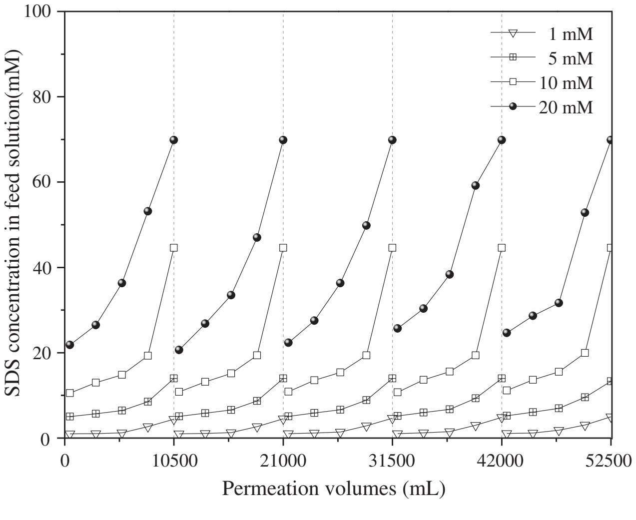

Fig. 3. SDS concentrations at four different initial SDS feed concentrations under a constant operating pressure (0.10 MPa).

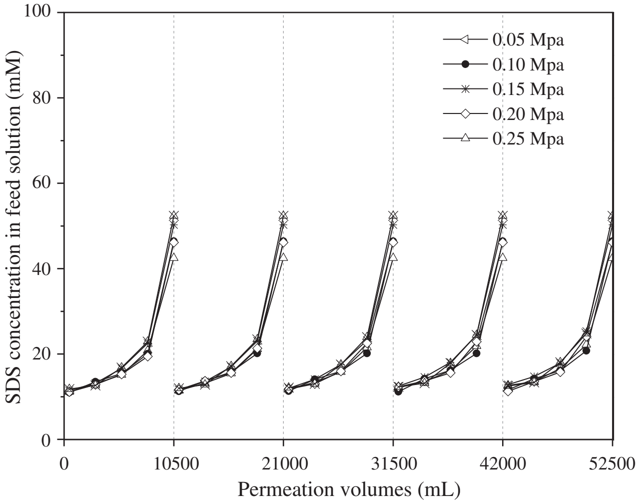

Fig. 4. Changes in SDS concentration in feed solution at different TMPs. Initial SDS feed concentration: 10 mM.

Fig. 2 shows the pure water flux before each group; the pure water flux increased linearly with TMP. The measured membrane intrinsic resistance was $9.15 \times 10^9\ \mathrm{m}^{-1}$, which indicated that the cut-off capacities of the membranes before each group were in substantial agreement and the influence of the membrane itself on fouling could be neglected.

3.2. Changes in SDS concentrations under different conditions

Whether or not fouling is observed in dilute solutions, the permeate flux decreases when the feed concentration increases; this is normal in UF [20]. Purkait [11] found that at higher surfactant concentrations, the number of micelles increases, and consequently, reversible pore blocking is more severe and is the dominant resistance responsible for flux decline. Recycling of retentate to the feed tank leads to continuous growth of the SDS concentration, and this will be considered in this paper.

In the experiments, recycling of the retentate stream to the feed tank led to continuous changes in the SDS concentration. In studies of the effects of feed concentration on membrane fouling, it is important to consider this situation. The critical micelle concentration (CMC) is 7.8 mM.

Fig. 3 shows the SDS concentration increased with accumulating permeate and increasing initial feed concentration in each unit. The ratios of the SDS concentrations, 1:5:10:20 mM, were almost constant in the range 0–7000 mL in each unit. The regularity of the SDS concentration at the end of each unit suggested that higher feed concentrations led to higher SDS concentrations than the initial ratios. Furthermore, the SDS concentration for a feed concentration of 5 mM was found to be close to the CMC throughout the group, although it was lower than the CMC at the beginning and higher at the end.

Fig. 4 shows SDS concentrations at different TMPs. There was little difference for different TMPs or different units. It can be deduced that the influence of SDS concentration could be neglected in investigating the effects of TMP on membrane fouling in the five units.

At low concentrations, surfactants will favor arrangement on the surface. As the surface becomes crowded with surfactant molecules, more molecules will form micelles. At the CMC, the surface becomes completely loaded with surfactant and any additional surfactant must form micelles [21]. However, an additional complication occurs

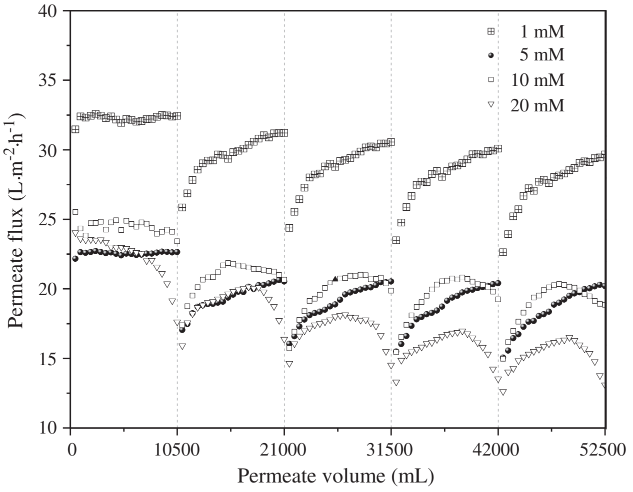

Fig. 5. Variations in permeate flux at different initial SDS feed concentrations. Operating pressure: 0.10 MPa.

in the case of high concentrations of SDS. Above the CMC (7.8 mM) the dodecyl sulfate anions aggregate and anionic micelles, which are roughly spherical, appear. At SDS concentrations above 75 mM, the spherical micelles associate to form rod-like aggregates, and these aggregates can pass through the membrane; this second transition is called the second CMC [22].

The SDS concentrations in both Figs. 3 and 4 were lower than 75 mM, so the increasing surfactant concentrations produced larger numbers of micelles.

3.3. Effects of feed concentrations and TMP on permeate flux

Flux and TMP are the best indicators of membrane fouling. Under constant pressure operation, flux declines as a result of membrane fouling.

Experiments with different feed concentrations (Fig. 5) were carried out to investigate the influence of feed concentration and CMC on flux. The initial feed concentrations (1 mM, 5 mM, 10 mM, and 20 mM) represented three types of CMC, classified as below CMC (1 mM), around CMC (5 mM and 10 mM), and higher than CMC (20 mM).

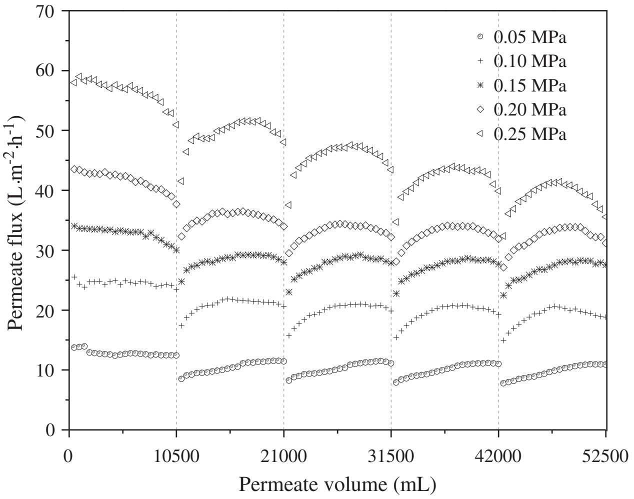

Fig. 6. Variations in permeate flux for fixed initial SDS feed concentration at different TMPs. Fixed initial SDS concentration: 10 mM.

According to Fig. 5, the sequential order of the membrane permeate fluxes can be described as 20 mM > 5 mM > 10 mM > 1 mM after 7000 mL in every unit. In addition, the flux for 5 mM was lower than that for the other two samples (10 mM and 20 mM) up to 7000 mL, where the SDS concentration was about 5 CMC. Fig. 3 shows the SDS concentration for 5 mM around the CMC throughout the group. Mizoguchi et al. and Patist et al. [23,24] found that as the surface became crowded with surfactant molecules, there were not enough molecules to form micelles, and some pre-micelles were thought to convert to micelles on the membrane surface as a result of concentration polarization. For concentrations near to but higher than the CMC, micelles formed and could not block the pores and therefore did not influence the permeation flux, leading to the permeate flux order 5 mM > 10 mM.

These flux results showed at the same pressure, the permeate flux was lower for higher feed concentrations. This is because at higher feed surfactant concentrations, the number of micelles is greater, leading to an increase in gel-type resistance over the membrane surface [15]. Also, the multiple effects of low feed concentration and the driving force of water. At the beginning of a unit, the membrane was fouled by a gel layer and pore blocking, the low-concentration feed solutions dissolved some of the micelles and the driving force unblocked the pores, so the flux increased. However, as the feed concentration increased, the ability to dissolve micelles decreased, until a point was reached at which the recovery rate of the flux was lower than the rate of fouling by gel layer formation and pore blocking. This would be shown in Fig. 6.

As shown in Fig. 6, the results for various TMPs showed that the sequential order of the membrane permeate fluxes is 0.25 MPa > 0.20 MPa > 0.15 MPa > 0.10 MPa > 0.05 MPa, but the flux did not increase linearly as it did for pure water flux (shown in Fig. 2). Furthermore, a higher TMP caused a faster initial flux decline in the first unit, and a faster increase in the initial flux in the other four units. The flux decline caused by the accumulation of molecules on the nearby membrane surface, known as concentration polarization, may lead to membrane fouling. It is also known that an increase in the TMP leads to concentration polarization and increases the concentration at the membrane surface. Higher TMP caused increased gel layer fouling for all solutions as a result of the increasing feed concentration. However, a similar trend in flux decline to that observed in Fig. 5 was seen, i.e., increasing the initial feed concentration caused more fouling. This situation assists convective flux through the membrane as a result of the enhanced driving force [13,25,26]. In general, this indicates that the resistances of the gel layer and the concentration polarization layer will increase (see Figs. 7 and 8).

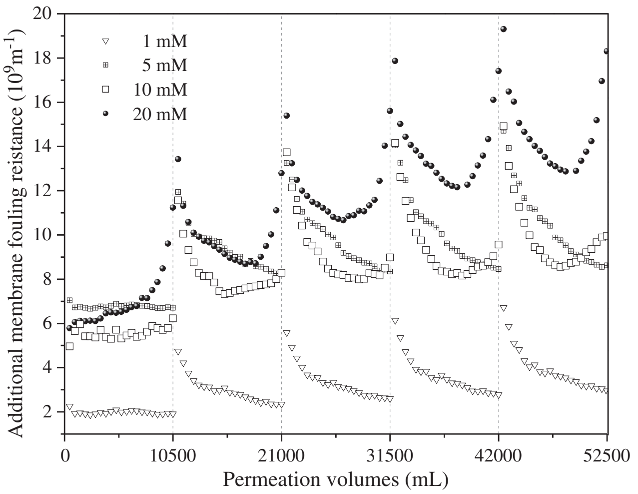

Fig. 7. Variations in additional membrane fouling resistance at different SDS feed concentrations. Operating pressure: 0.10 MPa.

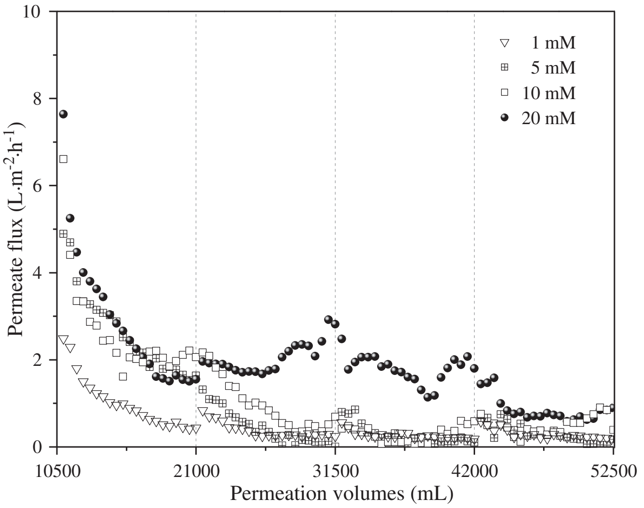

Fig. 8. Variations in increasing rate of fouling resistance at different initial SDS feed concentrations. Operating pressure: 0.10 MPa.

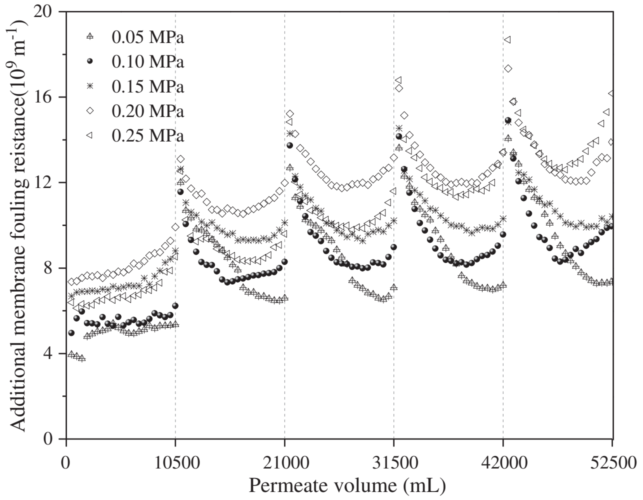

Fig. 9. Variations in additional membrane fouling resistance at different TMPs. Initial SDS feed concentration: 10 mM.

Figs. 5 and 6 also show that the flux started at a higher level at the beginning of each unit, Jermann [14] found that in filtration/backwashing experiments with surfactants, a significant (albeit incomplete) recovery of membrane flux was clearly observed immediately after each backwash. So it is easy to deduce that the initial feed solution plays a similar role to that of backwashing, and that lower concentrations led to higher flux recovery.

3.4. Effects of SDS concentrations on membrane fouling resistance

Fig. 7 shows a series of additional membrane resistance curves for various initial feed concentrations.

In the first unit, the curve of R_f in 20 mM SDS increased with permeate volume, while the curves remained flat for the other three concentrations. The flat trend before the increase indicated that the membrane can tolerate water quality changes to a certain degree. Analysis of Figs. 3 and 7 shows that membrane fouling was more serious with SDS concentrations in the range 14–15 mg L^{-1}, i.e., almost equal to 5 CMC, and concentrations lower than 5 CMC resulted in little change to R_f. A possible explanation for this phenomenon is that surfactant molecules aggregated and formed spherical micelles when the surfactant concentration in the wastewater was high enough or the filtration runtime was long enough [14].

In the other four units, the curves were affected by the lack of cleaning between the units of a group: R_f decreased at the beginning, but then increased after reaching its peak. A contributing factor to this phenomenon may be the existence of balance: the increasing SDS concentration led to more serious particle aggregation and formation of a colloidal gel [26], both of which were intensified by the absence of backwashing: a low initial feed concentration at the beginning of the next unit will contribute to high waves, and flushing of the colloid gel attached to the membrane surface and membrane pores. Particle aggregation in MEUF was found to play an important role in membrane filtration. As the SDS concentration increased, the surfactant would form micelles of a characteristic size after reaching the CMC. Foulants were more effectively removed by adsorption on the SDS colloids, while particle aggregation would form larger micelles, leading to larger values of R_f. This observation is in agreement with the reports in the literature that polymers are potentially the most significant fouling species in a sample [27].

Fig. 8 shows the increasing rate of resistance, called R_i, which was calculated by one-to-one correspondence of the difference of two neighboring terms. This index could help us to reduce the effects of stopping operation for 12 h. In the second unit, i.e., from 10,500 mL to 21,000 mL, R_i declined for three concentrations (1 mM, 5 mM, and 10 mM). The flat trend from 31,500 mL to 52,500 mL indicated saturation of the sorption capacity of the membrane, but the values for 20 mM were higher than those for the other three concentrations, and it was the last to reach sorption saturation. There may be two reasons for the decreasing resistance in the second unit: the membrane has a high capacity at the beginning, or the 12-h stop, leading to large resistance, but the resistance declines at the beginning of the next unit because of the feed waves. Comparing Figs. 3, 7, and 8, it can be concluded that membrane fouling resistance does not show a direct relationship with SDS concentration, and it rises faster if the initial concentration is higher.

3.5. Effects of TMP on membrane fouling resistance

Experiments with different TMPs were carried out to investigate how TMP contributes to concentration polarization or fouling. The effects of the applied TMPs are shown in Fig. 9.

Comparing Figs. 6 with 9, it can be seen that the rate of the decline of R_f did not agree with the sequential order of changes in permeate

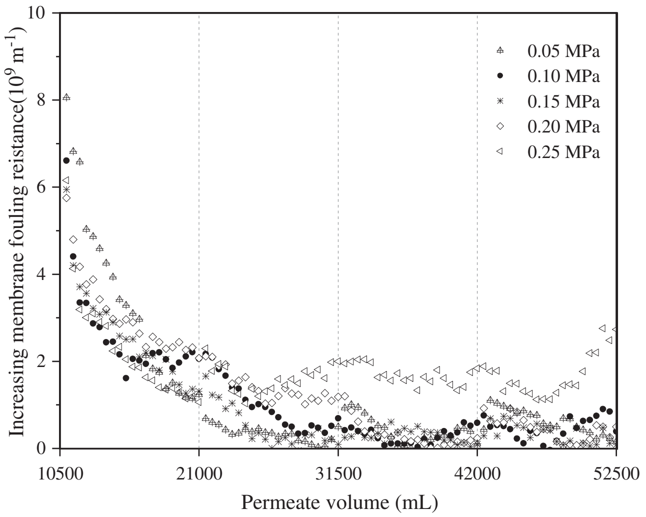

Fig. 10. Variations in increasing membrane fouling resistance at different TMPs. Initial SDS concentration: 10 mM.

flux with TMP. Furthermore, Fig. 4 showed that TMP had little influence on SDS concentration, which indicated the main factor influencing R_f in the permeate volume was the TMP.

In units 2–4, each of the five lines has a trough, but they appear at different permeate volumes. The order of the decline in the R_f, 0.10 MPa < 0.15 MPa < 0.20 MPa, was constant during the whole period, but the other two TMPs gave different trends. The R_f s in the TMPs (0.05 MPa and 0.25 MPa) showed different trends: R_f for 0.05 MPa was lowest in the first unit, with no obvious changes, but it declined from a high value to a low value close to that in the first unit in the subsequent four units. while the position of R_f for 0.25 MPa was always changing relative to the values for other TMPs. the R_f for 0.25 MPa did not follow the same sequential relationships as that for the other four TMPs: the higher the TMP, the lager the R_f. Obviously, it can be concluded that the five troughs in units 2–5 reflect the R_f speeds at different TMPs.

The figure shows that TMP is not always a good criterion for membrane fouling, especially at the beginning of MEUF. A possible explanation is that the capacity for fouling accumulation is much stronger than the capacity for removal of fouling in the TMP range of 0.05–0.20 MPa. For 0.25 MPa, membrane fouling did not continue to increase with increasing TMP, this indicated that the effects of different TMPs on R_f differed. Other possible underlying mechanisms were both based on the 12-h stop between units and the flushing, which resulted in various degrees of particle fouling. The stop for 12 h would increase blockage of membrane pores and result in a denser gel layer. The permeate and retentate wash the membrane surface and pores as a result of crossflow UF, which also causes different accumulation of membrane fouling.

Resistance curves for different TMPs are shown in Fig. 10. It can be seen that all five lines decline between 10,500 mL and 21,000 mL, and show similar trends throughout the experiments. The rate of R_i declined from the second unit to the fifth unit, and all cases finally converged to almost the same steady-state. It was obvious that R_i for 0.25 MPa remained higher, which indicated that a high TMP led to more serious fouling for long runtimes. This is because the shapes and aggregation numbers of the micelles changes with increasing SDS feed concentration, and the number of efficient binding sites does not increase with increasing SDS concentration [7].

Increasing feed solution concentrations and TMPs resulted in increased gel layer fouling with time, but concentration polarization and internal pore fouling did not change much. Concentration polarization is caused by adsorption inside the pores or at the membrane surfaces of materials, but internal pore fouling is only observed within the membrane pores. Internal pore fouling can be removed by backwashing, for example, with low-concentration SDS. According to our results, concentration polarization caused temporary fouling. In addition, the formation of gel layers, which were not removed by water washing, caused a greater flux decline than that caused by internal pore fouling [28].

4. Conclusions

The effects of some important parameters related to membrane fouling in MEUF were investigated, including TMPs and feed concentrations. During filtration, relatively rapid fouling takes place initially as a result of internal pore adsorption; this persists for a long time. In parallel, SDS molecules are rejected by the membrane, pore blocking becomes important, and a fouling cake develops on the membrane. For a fouled membrane, the initial feed solutions play a similar role to backwashing, and lower concentrations lead to higher flux recovery, but the effects of TMP are more complicated. SDS molecules are deposited on the membrane surface and the gel layer formed near the membrane surface is flushed by the initial feed solutions.

Different initial feed concentrations had different effects on membrane fouling: higher concentrations did not always lead to more serious fouling, and there was a relationship with the CMC. For the two cases in which the initial feed concentrations were both near the CMC, the higher concentration gave a higher flux and lower resistance. However, above the CMC, the higher the feed concentration was, the more serious the fouling. At the CMC, the surface becomes completely loaded with surfactant and any further additions must form micelles. Recycling of retentate to the feed tank led to continuous growth of the SDS concentration, and the flux declined sharply when the SDS concentration was higher than 5 CMC.

The permeate flux increased with TMP, but with a different relationship to that obtained for pure water flux. Furthermore, higher TMPs caused faster initial flux declines as a result of the large driving force. Membrane fouling did not always increase with TMP. At the beginning of MEUF, a high TMP such as 0.25 MPa gave a lower resistance, but a low TMP such as 0.05 MPa caused more serious fouling. The possible underlying mechanisms were both related to stopping the operation for 12 h, leading to different degrees of pore blocking and gel layer formation before the beginning of the next unit, and flushing caused by the different driving forces of the solution for different TMPs.

This research has practical applications. Since low-concentration solutions can recover flux as effectively as pure water, and the large driving force caused by a high TMP can remove the gel layer attached to the membrane surface and pore blockages, this provides a possible method for washing membranes using alternating low-concentration and high-concentration solutions or by using different TMPs.

Acknowledgments

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