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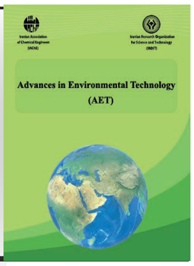

Removal of manganese from an aqueous solution using Micellar-Enhanced Ultrafiltration (MEUF) with SDS surfactants

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ABSTRACT

In the present study, micellar-enhanced ultrafiltration (MEUF) was used to remove manganese (Mn) (II) from synthetic wastewater. The effects of different operational conditions on the filtration performance of MEUF or the membrane were studied. It was found that the transmembrane pressure has a major influence on the permeate flux and an insignificant effect on the rejection coefficient. The permeate flux increased almost linearly with operating pressure, ranging from 0.35 L min^{-1} m^{-2} at 1 bar to 1.79 L min^{-1} m^{-2} at 4 bar. When the pH of the feed solution was changed from 3 to 13, the permeate manganese concentration decreased considerably while the rejection of manganese increased from 75.37% to 99.78%. The results showed that by adding SDS anionic surfactant, the permeate flux and the removal efficiency of manganese increased. The retention of Mn (II) increases from 47% in the absence of SDS to around 97% with 10mM SDS.

In this state, the MEUF system has a rejection of above 97 percent of manganese. The Mn rejection increased slightly with an increase of operating pressure, ranging from 89.658 at 1 bar to 97.971 at 4 bar. Adding NaCl to the solution provokes the complexation of metal cations with chloride ions and the adsorption competition of sodium cations with the metal ions; therefore, the conductivity increment decreases the metal retention. The presence of 60 mM of NaCl reduced the removal efficiency by 50–60%.

1. Introduction

Manganese is the second most plentiful transition metal after iron [1]. In drinking water distribution systems, the sloughing of manganese oxide deposits results in poor aesthetic water quality which has a brown-black color, undesirable taste, and causes the staining of fixtures, equipment, swimming pools, and laundry [2–9]. In addition to the above mentioned problems resulting from the presence of manganese in natural and municipal water systems, it is also a health concern [5]. To control these problems in water distribution systems, recommended levels are enforced. Some references report the allowable recommended levels of manganese in drinking water in the range of 0.01-0.1 mg L^{-1} [3, 10-14]. Also, The World Health Organization has set a limit of 0.4 mg L^{-1} for manganese (II) in drinking water [10].

Manganese occurs in the environment in different physical and chemical forms but primarily as manganese compounds with various valences. Usually, manganese (II) compounds are soluble in water while other manganese compounds (high valence) are not soluble. In aquifer systems, manganese can be transformed from soluble form into insoluble form and vice versa based on the radix status. This characteristic has resulted in an increased interest in the study of the removal of manganese from groundwater resources [15].

Manganese can also be found in surface waters, although typically at lower concentrations than in groundwater.

Traditional methods for the recovery of metal ion from water are adsorption [16,17], ion exchange [18], precipitation [19], and flocculation [20]. The most popular method is adsorption, but the process is slow due to mass transfer limitations.

In order to remove small molecular weight metals from a solution, membranes with relatively lower pore sizes, typically used for reverse osmosis and nanofiltration, were

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introduced. These processes are energy intensive with high capital and operational costs. To overcome the aforementioned problems, a MEUF system was introduced. In this process, small pollutants are bound (for ionic pollutants) or solubilized (for organic pollutants) in large surfactant micelles which can be separated by ultrafiltration membranes with larger pore size. This process was studied for the removal of metal ions from dilute aqueous streams by researchers [21-32]. So far, the use of the MEUF process for removal of Zn [21,25,27], Cd [18– 20], Ni [22], Cu [23], Pb [21,22], Cr [25], di-butyl phosphate (DBP), tri-butyl phosphate (TBP) and uranyl ions [28], adsorbable organic halogens (AOX) [29], methylene blue [30,33], eosin [34], Safranin T [26], naphthenic acids [27], chromate and nitrate [28], phenol/Cd [4], phenol/Cu [29], etc. from aqueous solutions or wastewaters has been investigated by researchers.

Various researchers have demonstrated the significant effect of pH on heavy metal removal. They reported that cationic heavy metal (Mn^{2+}, Co^{2+}, Cu^{2+} , Zn^{2+}, and Cr^{3+}) removal reached over 80% with SDS as the pH increased from 2 to 12. At a lower pH, a reduction in heavy metal removal efficiency was due to the competition between H^+ ions and cationic metal ions to get adsorbed on the anionic micelle surface of SDS [34].

In the present study, MEUF was used to remove Mn^{2+} from synthetic wastewater using the ultrafiltration membrane. The UF module was operated in two arrangements, namely linear continuous and cross-flow modes which have vigorously higher flux and membrane effective area than conventional batch cell system. Very few papers have discussed the MEUF process with module for removing metal ions. In this study, the effects of some important parameters on metal removal efficiency and permeate flux were investigated. These parameters include operating time, the concentration of SDS, the operating pressure, and solution pH. These results can be helpful in achieving the practical application of this technique.

A schematic diagram of MEUF for the removal of metal ions is shown in Fig. 1.

2. Materials and methods

2.1. Chemicals

The anionic SDS surfactant (Molecular formula: $C_{12}H_{25}O$-SO_3Na; Molecular weight: 288.38; and the critical micelle concentrations (CMC) of SDS 8.15 mM) was purchased from the Merck company. Manganese (II) ions were prepared by dissolving $MnCl_2.2H_2O$ in distilled water. HCl and NaOH, both having concentrations of 1N, were used for pH adjustment. Polysulfone (PSf) and N-Methyl-2-Pyrrolidone (NMP) were used as the basic polymer and solvent for the preparation of the ultrafiltration membrane. They were provided by the BASF and Merck companies (Germany), respectively. The dimethylformamide (DMF) was purchased from the Akkim Co.

2.2. Apparatus and experimental methods

The applied experimental set-up for this research is shown in Fig. 2. All of the experiments were carried out at room temperature (25±2 °C). This temperature was selected for two reasons, the SDS Kraft point (14 °C) and prevention of any SDS precipitation. The feed tank was initially filled with 10 liters of feed solution. Then, $MnCl_2.2H_2O$ was added into the distillated water to produce the synthetic wastewater with a manganese ions concentration of 100 mg L^{-1}. The SDS, with a predetermined concentration, was added into the synthetic wastewater. The solution was mixed using a stirring bar driven by a magnetic motor at 300 rpm. The solution pH was adjusted by adding HCl or NaOH. Afterward, the solution was continuously feed into the cross-flow ultrafiltration commercial SW30 membrane modules using a diaphragm pump (open flow: 2.2 LPM, pressure: 120 psi). Two pressure gauges were utilized to monitor the pressure in the ends of the membrane module. The experiments were carried out in steady state conditions that were achieved after 10 min of operation. Also, permeate and retentate solution streams were recycled into the system.

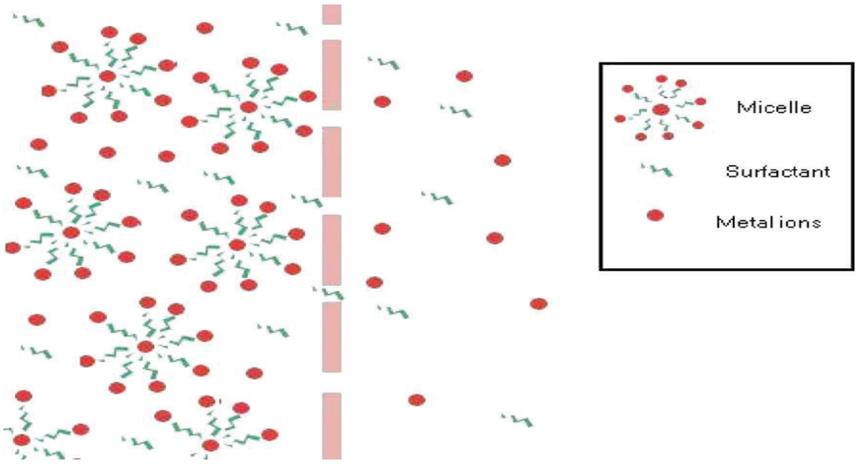

Fig. 1. A schematic diagram illustrating micellar-enhanced ultrafiltration for the removal of metal ions.

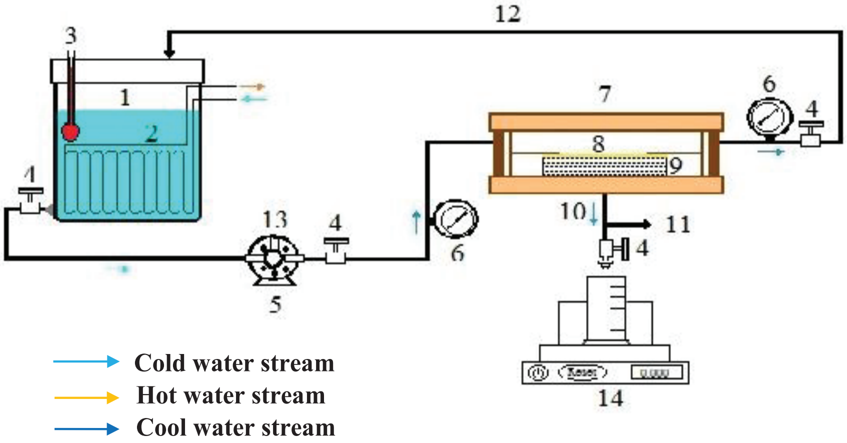

Fig. 2. MEUF experimental setup: (1) feed tank, (2) heat exchanger, (3) thermometer, (4) pressure control valve, (5) diaphragm pump, (6) pressure gauge, (7) cross flow MEUF cell, (8) TFC UF membrane, (9) sintered porous stainless steel disk, (10) permeate flux, (11) return to the feed tank, (12) retentate flow, (13) bypass line, (14) accurate digital electronic precision balance.

The permeation flux of the ultrafiltration membrane is defined as:

$$J_P = \frac{Q}{A.t} \tag{1}$$

Jp is the permeation flux (L m^{-2} min); Q is the permeate volume (L); t is the operating time (min); and A is the area of the membrane (m^2).The rejection coefficient is defined as:

$$R = 1 \frac{C_P}{C_f} \tag{2}$$

C_f is the concentration of manganese (II) ions (mg L^{-1}) in the feed solution and Cp (mg L^{-1}) is the concentration of manganese (II) ions in the permeate (mg L^{-1}).

2.3. Analyses

The permeate flux was continuously measured by a gravimetrical method. By converting cumulative weights to cumulative volumes and using cumulative volume slope versus time curve, the permeate flux, JP (L m^{-2} min^{-1}) was calculated using Eq. (1).

The manganese (II) ions concentration in the permeate was measured by atomic absorption spectrometry (GBC, 908AA Model at 228.8 nm).

Each experiment was repeated for every model solution. Three measurements for metal ion concentrations in permeate and flux rate were made and the results were averaged. The uncertainty in ion concentration measurements were estimated as ±0−2 ppm, ±0.05 L m^{-2} min^{-1} for rejection factor and permeate flux, respectively. A 95% confidence interval was used in both cases.

2.4. Membrane

In this study, commercial SW30 membrane modules were used and the membranes were immersed in Dimethylformamide (DMF) bath. A specific amount of polysulfone was dissolved in N-Methyl-2-pyrrolidone (NMP) and stirred for about 12 hours at room temperature to reach a homogeneous casting solution with 15 %wt concentration. This solution was allowed to degas overnight. To begin casting the polysulfone membrane, the non-woven polyester fabric was attached to a clean glass plate. Subsequently, the well mixed bubble free solution was cast onto the polyester to a thickness of 150 µm with a casting knife. To prevent the formation a of polysulfone layer behind the non-woven polyester fabric, the membrane was immediately immersed into the distilled water bath. This collection was kept one day at an ambient temperature to ensure an adequate exchange.

3. Results and discussion

3.1. Effect of operating time on the permeate flux and the rejection factor

Initially, the distilled water was processed at an operating pressure of 2.5 bar and the permeate flux was measured at about 2.5 L m^{-2} min^{-1}. As shown in Fig. 3, the effect of the operating time on permeate flux was investigated at the 6 mM and 100 ppm of SDS and Mn^{2+} concentration, respectively. It can be found that the SDS concentration in the polarization layer is higher than that of the bulk solution. Therefore, the SDS bulk concentration is considered less than the CMC of SDS (8.15 mM). In this status, the monomers of the surfactant accumulate at the concentration polarization layer and therefore, the SDS concentration reaches above the CMC in this layer and micelles are formed to trap the contaminants.

Fig. 3 represents the variation of the permeate flux with the operating time. As can be seen in Fig. 3, the permeate flux decreases with the increase of the operating time. This phenomenon is attributed to the fact that the surfactant accumulation on the membrane surface increases with the increase of operating time. This behavior is known as concentration polarization. The concentration polarization is caused by the accumulation of retained solutes such as micelles on the membrane surface in a short period of time, where their concentration will gradually increase [25,34].

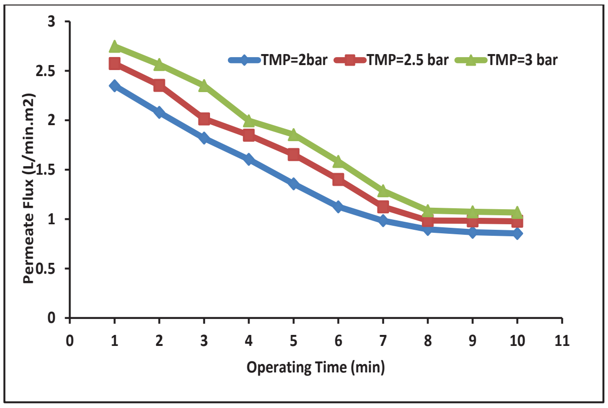

Fig. 3. Effect of operating time on the permeate flux in different trans-membrane pressure.$[Mn^{+2}]$= 100 ppm, [SDS] = 6 mM, at room temperature. TMP is transmembrane pressure.

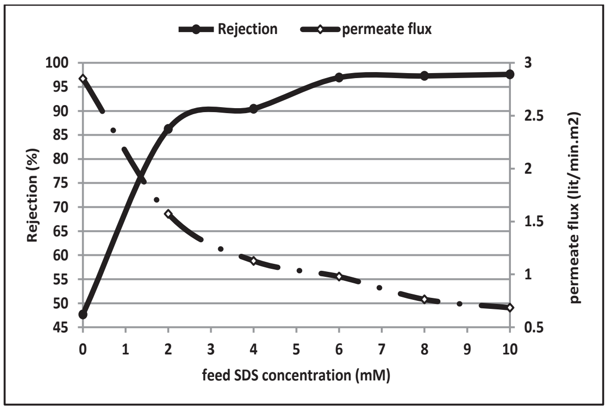

Fig. 4. Effect of feed surfactant concentration on the Mn^{+2} rejections and permeate flux. $[Mn^{+2}]$= 100 ppm, TMP= 2.5 bar, at room temperature.

Fig. 3 represents the variation of the permeate flux with the operating time. As can be seen in Fig. 3, the permeate flux decreases with the increase of the operating time. This phenomenon is attributed to the fact that the surfactant accumulation on the membrane surface increases with the increase of operating time. This behavior is known as concentration polarization. The concentration polarization is caused by the accumulation of retained solutes such as micelles on the membrane surface in a short period of time, where their concentration will gradually increase [25,34].

According to this research and other studies [19,31,35], when the SDS concentration exceeded its CMC, the rejection variations were small (98–99%).

3.2. Effect of SDS concentration on permeation flux and the rejection factor

The variations in the Mn^{2+} rejection are shown in Fig. 4. at 2.5 bar trans-membrane pressure (TMP) using a SDS concentration in the range of 0–10 mM.

Fig. 4 describes the effects of the feed SDS concentration on the retention of Mn (II), SDS, and permeates flux at a fixed Mn (II) concentration of 100 ppm. It depicts an immediate rise in the retention of Mn (II) with the increasing feed SDS concentration, which later attains a plateau at a higher SDS concentration. Although the apparent hydrodynamic diameters were larger, the low SDS concentrations did not benefit the rejection of SDS and Mn because of the small number of micelles. The retention of Mn (II) increases from 47% in the absence of SDS to around 97% with 10 mM SDS [36-38]. Initially, this study employed a SDS concentration of 6 mM due to the reduction of surfactant usage and surfactant loss that may decrease the cost of the separation process. On the other hand, in high SDS concentrations, the surfactant monomers certainly leaked into the permeate through the ultrafiltration membrane and produced secondary pollution.

The advantageous effect is the formation of more micelles and more Mn (II) ions getting entrapped. On the other hand, there is a competition between Na^+ and Mn (II) to bind with the micelles, and the competition depends on both the electrical charge of the ion and their concentration. The bivalent Mn ions were preferentially bound with the micelle. However, the high concentration of Na^+ reversed the tendency. At a low SDS concentration, the first effect is predominant and the retention of Mn increases at a rapid pace; however, at high SDS concentration, the competition between the two counter-ions is more advantageous to Na^+ and the rejection of Mn increases marginal. At a constant Mn (II) concentration, the SDS retention increased with an increase in the feed SDS concentration and showed three stages. When the feed SDS concentration increased to 10 mM or higher, the SDS concentration at the vicinity of the membrane surface reached the CMC of SDS, forming SDS micelles. All these stages corresponded to the variety of Mn retention. A decrease of the micelle size has no influence on the rejection of SDS and Mn^{2+} in the investigated range of conditions. It indicated that the pore sizes of the membrane used were smaller than the micelle size [25].

3.3. Effect of the operating pressure on permeation flux and Mn^{2+} rejection factor

The study of the effect of operating pressure was conducted at a constant Mn and SDS concentrations of 100 ppm and 6 mM, respectively, at room temperature. It is evident in 6 mM. that the permeate flux increased almost linearly with operating pressure, within the range from 0.35 $L\ m^{-2}\ min^{-1}$ at 1 bar to 1.79 $L\ m^{-2}\ min^{-1}$ at 4 bar. The permeate flux increased as operating pressure increased because the operating pressure between retentate and permeate was the effective driving force for process [25,28].

Therefore, it can be noted that a very high operating pressure is not necessary for a high permeate flux. Higher operating pressures require more investment in equipment and also higher operational costs. For commercial appli-cations, the recommended operating pressure for this UF membrane separation system is below the 15 bar [34]. Fig. 5 also indicates that a pressure difference of more than 3.5 bar has no meaningful effect on the permeate flux. This finding is in agreement with other studies on UF [34]. The effect of operating pressure on the rejection of manganese was investigated and presented in Fig. 5. shows that the Mn rejection increased slightly with an increase of the operating pressure, ranging from 89.658 at 1 bar to 97.971 at 4 bar. This was due to the following two reasons: 1) At an optimized operating pressure, micelles might be compacted, thereby increasing the micelle solubilization capability and hence, a lower quantity of Mn ions would be solubilized within the micelles [23]; and 2) The increase of an effective driving force caused the increment of the convective transport of solutes filtered through the ultrafiltration membrane to the permeate solutions.

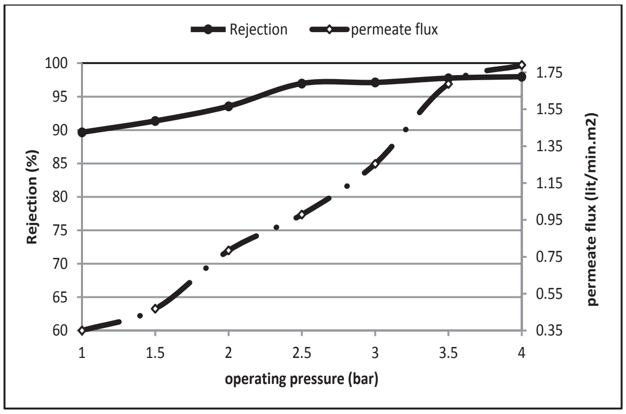

Fig. 5. Effect of operating pressure on the permeate flux and Mn^{+2} rejections.[Mn^{+2}]= 100 ppm, [SDS] = 6 mM, at room temperature.

3.4. Effect of solution pH on permeation flux and Mn^{2+} rejection factor

Fig. 6 shows the pH effect of the feed solution on the permeate manganese concentration and the rejection of manganese. However, when the pH of the feed solution is changed from 3 to 11, the permeate manganese concentration decreased considerably while the rejection of manganese increased from 75.37 to 99.78%. A further increase in the pH of the feed solution from 11 to 13 did not have any significant effect on the permeate manganese concentration or on the rejection of manganese. Anions commonly form species of different valances in water and the concentration of each ion depends on the pH of the solution. Manganese anion is present in different forms depending on the pH. As a result, the maximum binding of manganese [Mn (II)] and its maximum rejection are observed at the pH range mentioned above.

Fig. 6 shows that an increase in the solution pH has little negative effect on permeation fluxes. This phenomenon can be explained by the fact that the shape and the aggregation number of micelles changes with the increase of the feed solution pH and pore blocking can be more intense [17].

3.5. Effect of electrolyte on Mn^{2+} rejection efficiency

The presence of electrolytes can decrease the CMC of ionic surfactants because the electrolytes can weaken the repulsive forces between the head groups, which are normally fighting against the aggregation of surfactant monomers. Therefore, micelles can form comparatively easier in the presence of electrolytes [25]. Fig. 7 shows the effect of electrolytes, which is NaCl in this research, on the Mn^{2+} rejection efficiency at a SDS concentration of 8.06 mM, Mn^{2+} concentration of 100 mg L^{-1}, and a transmembrane pressure of 2.5 bar.

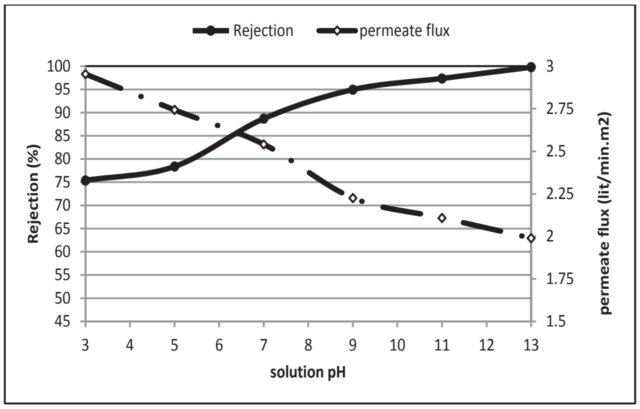

Fig. 6. Effect of solution pH on the Mn^{+2} rejections and permeate flux. [Mn^{+}]= 100 ppm, [SDS] = 6 mM, TMP= 2.5 bar, at room temperature.

As shown in Fig. 7, the Mn^{2+} rejection efficiency decreases with the increase of the NaCl concentration (from 15 to 60 mM) and reaches an unvarying low value. The result does not comply with what is pointed out in the literature concerning this topic [33,35], namely the presence of electrolytes cannot increase the Mn^{2+} rejection efficiency. This behavior may be attributed to the following two factors: (1) the cations of electrolytes, namely Na^{+} in this experiment, can be bound to the micelles and occupies the binding sites. Accordingly, the Mn^{2+} rejection efficiency decreases with the increase of the NaCl concentration; and (2) The anions of electrolyte, namely Cl^{-}, can form the complexes with metal ions, namely Mn^{2+} [33,39]. In this experiment, the two negative effects of the presence of NaCl may exceed the positive effect of the decrease of CMC due to the presence of NaCl.

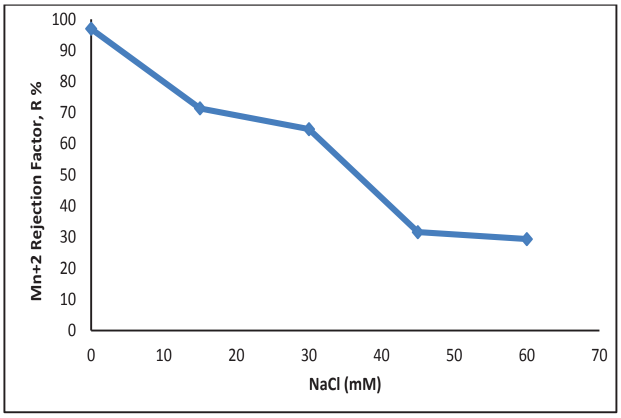

Fig. 7. Effect of electrolyte concentration on the Mn^{+2} rejections. [Mn^{+2}]= 100 ppm, [SDS] = 6 mM, TMP= 2.5 bar, at room temperature.

4. Conclusions

The use of MEUF to remove manganese ions from an aqueous solution using anionic surfactants and sodium dodecyl sulfate (SDS) was investigated with a polysulfone membrane.

The effect of some important parameters including the operating time, feed SDS concentration, operating pressure, concentration of feed electrolyte, and solution pH were investigated.

The results indicated that the permeate flux increased with increasing, to optimal levels, operating pressure and cross-flow velocity.

When the initial SDS concentration was below the CMC (6 mM), unexpectedly high Mn^{2+} rejection (97%) was obtained due to concentration polarization occurring near the membrane–solution interface.

Manganese rejection was found to be highly dependent on the pH condition of the feed solution and approaches maximum at a pH of 13. Manganese rejection increased with an increase in feed surfactant concentration, but found to be almost constant at 2 to 10 mM feed surfactant concentrations. This means higher surfactant concentration in the feed does not lead to a significant improvement in the metal separation.

MEUF by ultrafiltration membrane has a good rejection of Mn^{2+} under different operating conditions, as the rejections were higher than 97.0%.

Manganese rejection decreased with an increase in feed NaCl concentration. Furthermore, the presence of electrolytes can diminish the Mn^{2+} rejection efficiency by SDS.

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