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Dynamic Analysis and Optimization of Surfactant Dosage in Micellar Enhanced Ultrafiltration of Nickel from Aqueous Streams

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Abstract: The micellar enhanced ultrafiltration of Ni(II) ions from the aqueous solution was studied for the dead end system using 20KD Polysulfone membrane. Dynamic behavior of the system was studied with respect to the rejection, yield, and normalized flux. The effect of feed metal ion concentration, surfactant concentration, pH, transmembrane pressure, and S/M ratio was investigated and the optimization of S/M ratio was done. The optimum S/M ratio was 10 while the critical S/M ratio was 5. The effect of monovalent salts was studied on the rejection of metal ions for the salt concentration between 10 mM to 500 mM.

Keywords: Nickel ion removal, micellar-enhanced ultrafiltration, heavy metals, membrane process, wastewater treatment, sodium dodecylsulfate

INTRODUCTION

Metal finishing plants, tanneries, paint and dyes industry, metal mines etc. release huge quantities of wastewater containing hazardous heavy metals like Cu, Cr, Co, Ni etc. These are the metals characterized by US EPA as persistent, bioaccumulative, and toxic (PBT) chemicals. Almost all of these metals are suspected carcinogens. Because of this fact government

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agencies like US EPA had put legislative compliance on released concentrations of these metals; such compliance for release of Ni(II) in wastewater as per 40 CFR 433 for new sources is set at 2.38 ppm for a monthly average and 3.98 ppm for a daily maximum US EPA (1). Micellar enhanced ultrafiltration (MEUF) is a recent technique being studied for the removal of metal ions (2–7) from dilute aqueous streams, originally suggested by Scmehorn et. al., (8).

In MEUF surfactant micelles of ionic surfactant, carrying a charge opposite to that of metal ion are introduced in a solution to enhance the rejection efficiency of the metal ion. The bound micelles being larger in size than the pore size of the UF membrane can be easily retained in UF; the metal ions that are associated to micelles get rejected effectively. The permeate obtained has a very low concentration of both the surfactant and the metal ion. The retentate solution which now has high concentrations of both surfactant and metal ion is much less in volume (approximately 10–30% of feed volume); therefore further treatment is much more cost effective as compared to the direct treatment of the feed solution.

The objective of this work was to study the process of MEUF for the separation of nickel ions Ni(II) from dilute aqueous streams. All studies were carried out by using sodium dodecyl sulfate as a model anionic surfactant. Although Ni^{2+} removal had been studied earlier (7) the study of the effect of surfactant concentration over the process is addressed over here in a different way. The surfactant concentration and the metal ion concentration are two interacting factors and therefore we combined them to a more robust factor as surfactant to metal concentration (S/M) ratio (0.6–100) and tried to find optimal S/M ratio for rejection >99.5%. The other factors covered under the study are the effect of pH (2–10) of feed solution over MEUF and added monovalent salts NaCl, NaBr, and NaI.

MATERIALS AND METHODS

Materials

Sodium dodecylsulfate (SDS), nickel sulfate ($NiSO_4 7H_2O$), cetyltrimethylammonium bromide (CTAB) for the analysis of SDS, sodium hydroxide (NaOH), sulfuric acid (H_2SO_4), carbon tetrachloride (CCl_4) were obtained from Merck and were used as received. Conductivity measurements revealed that the CMC of the obtained SDS was 4.523 mM. An analysis reagent 2-mercapto propionicacid was obtained from Himedia and was used as received. All chemicals were of analytical grade. Stock solutions of $NiSO_4$ and SDS were prepared and were used by diluting appropriately for MEUF experiments. Distilled water was used throughout for making solutions.

Ultrafiltration Setup

Ultrafiltration experiments were carried out in a dead end batch filtration cell; 120 cm^3 in capacity and with an active membrane area of 15.91 cm^2. The cell was fabricated in-house from stainless steel. The membrane for ultrafiltration was obtained from Permionics and was a flat sheet polysulfone membrane with MWCO of 20 kD. The transmembrane pressure gradient (TMP) was maintained by use of N_2 gas at 4×10^5 Pa, unless mentioned otherwise. All experiments were carried out at the temperature of 303 ± 2 K; agitation speed was held constant in all experiments by use of a magnetic motor and was set to 350 rpm throughout.

Analysis

The analysis of SDS as a single component was carried out through conductivity measurements with a conductimeter from EQUIPTRONIX EQ 660. In the presence of Ni(II) the analysis of SDS was carried out by a 2 phase titration method using cationic surfactant CTAB. The analysis of Ni(II) was carried out by UV-VIS spectrophotometry at $\lambda_{max} = 408$ nm according to the method proposed by Lear and Mellon (10). The FP-6200 model of spectroflurometer supplied by JASKO was used for this purpose.

Method

In each experimental run 100 cm^3 of distilled water was charged in the cell to measure the pure water flux. The membrane was then equilibrated with a very dilute solution of SDS ($\sim$1 mM) for half an hour and fitted in the cell. The cell was then charged with 50 cm^3 of feed solution. The pH of the feed solution was adjusted (if required) between 7 and 7.5 by adding small quantities of 0.01 N NaOH or 0.01 N H_2SO_4. The cell was then pressurized to 4×10^5 Pa. Samples were collected dynamically for each run at times when 25%, 50%, 75%, and 90% of the feed was collected as permeate and were analyzed for Ni (II) content. After the completion of each run the membranes were rinsed thoroughly with distilled water and flux of the distilled water was measured. If this flux was found <95% of the distilled water flux measured before the run the membrane was replaced by a new piece. The % rejection and % yield of Ni(II) were calculated as per following formulae (11).

$$\% \text{ Rejection} = \% \text{ R} = 1 - ([\text{Ni(II)}]_P / [\text{Ni(II)}]_R) \tag{1}$$

$$\% \text{ Yield} = \% \text{ Y} = \frac{[\text{Ni(II)}]_R \times V_R}{[\text{Ni(II)}]_F \times V_F} \times 100 \tag{2}$$

The subscripts F, P, and R indicate corresponding quantity as measured in the feed, the permeate, and the retentate solutions respectively. The membrane being nonuniform in its pore distribution all the flux data were reported in terms of normalized flux calculated as (11).

$$\text{Normalized flux} = \frac{J_P}{J_W} = \frac{\text{Permeate flux}}{\text{Pure water flux}}. \tag{3}$$

RESULTS AND DISCUSSION

Effect of Feed Ni(II) Concentration

Figure 1 shows the effect of feed Ni(II) concentration on rejection and yield of Ni(II) and nomalized permeate flux in the absence of a surfactant. In the applied experimental conditions we did not observe any influence of Ni(II)

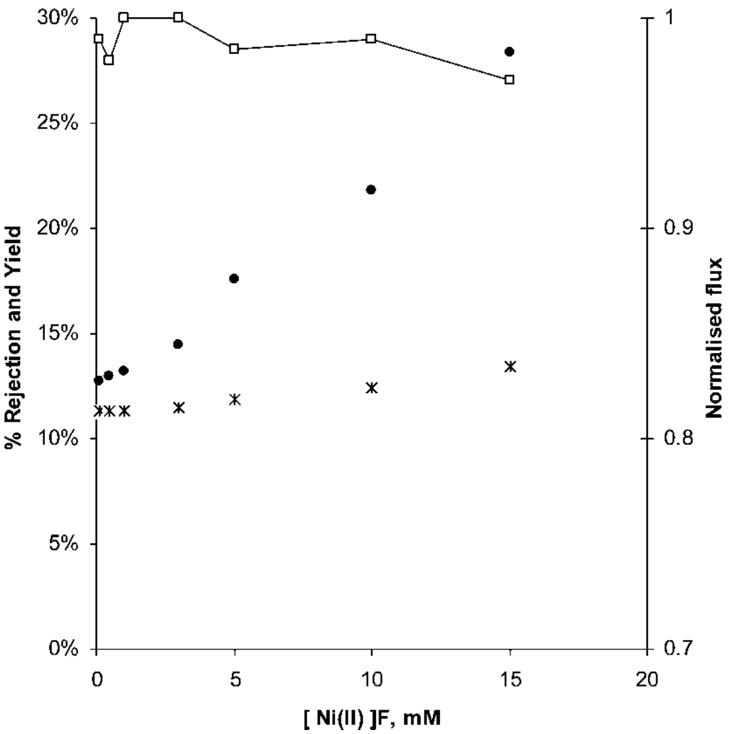

Figure 1. Effect of feed Ni(II) concentration on % R, % Y, and on normalized flux in absence of surfactant: $[SDS]_F = 0$ mM, $[NaCl] = 0$ mM, $[NaBr] = 0$ mM, $[NaI] = 0$ mM, $TMP = 4 \times 10^5$ Pa, T = 301 K, pH = 7, □: Normalized flux, ●: % Rejection, : % Yield.

concentration on the nomalized flux, every thing else being kept constant, the slight variations presented in Fig. 1 remain lower than the experimental error range, estimated at 10%. This can be attributed to the fact that the polysulfone membrane that was used for the study was having molecular weight cut off (MWCO) of 20 kD while the ionic size of Ni(II) is 58.69D; because of such a large difference in the size of Ni(II) ion and membrane pores, this membrane will not offer significant extra resistance for the permeation of Ni(II) ions through it. This also indicates that there will be very less (ideally zero) rejection of Ni(II) in the absence of the surfactant; in fact the observations show that when $[\mathrm{Ni(II)}]_F$ was increased from 0.1 to 15 mM, the % R increased from 12.7% to 28.4% while the % yield remained marginally constant in the range of 11.2 to 13.4%. The observed rejection can be attributed to the hydrophobicity and the residual charge on the membrane. The polysulfone membrane is highly hydrophobic while the Ni(II) ions are highly hydrophilic and this hydrophilic and hydrophobic solute membrane interaction and the residual membrane charge may be considered to be responsible for the observed rejection. The yield coefficient, the prime parameter of the interest from the point of view of recovery of Ni(II) from dilute aqueous streams remained low, indicating that the membrane with MWCO of 20 kD lacks the ability to remove Ni(II) ions from dilute aqueous streams without any added performance enhancer.

Figure 2 shows the effect of feed Ni(II) concentration on performance parameters of MEUF (% R, %Y and nomalized flux). In order to explain the behavior of MEUF in the present situation, consider a micellar system containing micelles surrounded by a completely dissociated electrolyte $NiSO_4$ in the form Ni^{2+} and SO_4^{2-}. The local distribution of these ions surrounding the micelles is determined by the relative magnitude of electrical potential energy (given by the product of the total protonic charge on the ion and electric potential at that point due to the micelle) and the kinetic energy of the molecule (given by the product of Boltzmann's constant and absolute temperature) and the bulk concentration (12). Owing to these driving forces and high charge density of the micellar surface the randomly moving Ni(II) ions in the solution are trapped in the electric field of the micelle and get bound to it. These ions are then bound to the micelles to the extent that they move with the micelles and are no longer osmotically active (13). Now as the magnitude of electrical interaction between micelles and Ni(II) ions is dependent on the charge as a driving force, most of the Ni(II) ions displace Na^+ ions from micellar surface. This effect is quite prominent as can be seen from Fig. 2 that the rejection coefficient over the entire range of the concentration (0.1 to 15 mM) of Ni(II) was greater than 95%. The ion exchange between Na_m^+ and NI(II) takes place as per the reaction given in equation no. (3), and an equilibrium is established between the bound and the unbound Ni(II) ions.

$$2Na_m^+ + Ni_W^{2+} = 2Na_W^+ + Ni_m^{2+}. \tag{4}$$

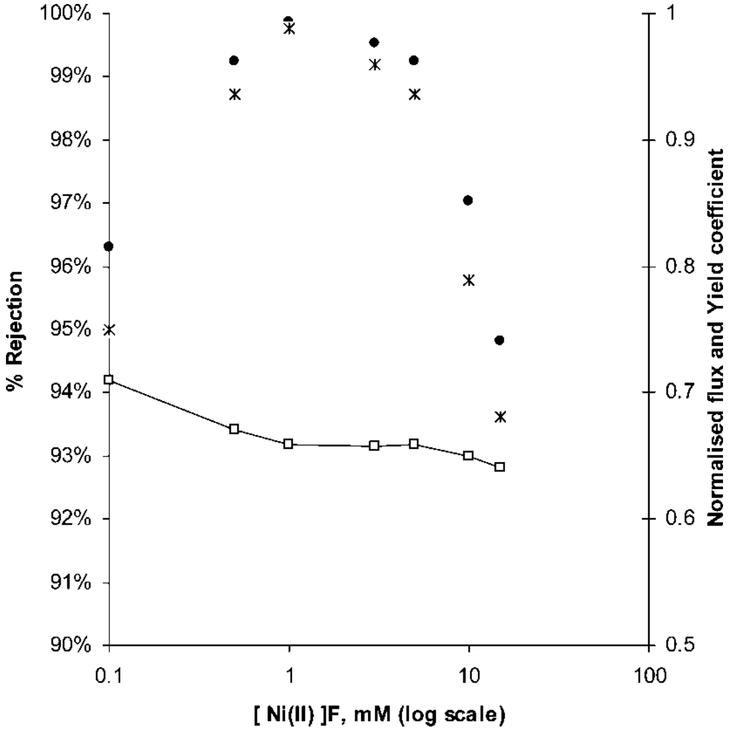

Figure 2. Effect of feed Ni(II) concentration on % R, % Y and normalized flux in presence of surfactant: $[SDS]_F = 10$ mM, [NaCl] = 0 mM, [NaBr] = 0 mM, [NaI] = 0 mM, TMP = 4×10^5 Pa, T = 303 K, pH=7, □: Normalized flux, ●: % Rejection, : Yield coefficient.

Where subscript m denotes ions in a bound state while w denotes ions in an unbound state in bulk solution. Only the bound ions get rejected by the UF membrane, therefore as the feed concentration increases there is a corresponding increase in the concentration of the unbound Ni(II) ions and hence the permeate concentration also increases. At very high concentrations of Ni(II) ions in feed (>5 mM) even if the rejection coefficient is slightly lower, the desired purity of the permeate can be easily obtained by employing a two–stage MEUF. This indicates that a multistage MEUF can be employed to bring down the permeate concentration in the desired range on similar grounds of staged mass transfer operations.

At very low concentration of Ni(II) i.e. at 0.1 mM a drop in the rejection was observed which may be attributed to the fact that at a low concentration of Ni(II) the concentration of Na^+ was relatively very high (nearly 100 times that of Ni(II)) because of which the probability of Ni(II) ion being found in the vicinity of micelles reduced slightly resulting into corresponding decrease in the amount of the bound Ni(II) ions which results into a drop in the rejection; but as soon as the concentration of the Ni(II) was increased to 0.5 mM or higher the probability of the binding increases and the rejection increased to 99% or higher. At a concentration greater than 5 mM reduction in rejection can be attributed to the lack of availability of binding sites.

The % yield also shows a similar trend as % rejection. Normalized flux on the other hand shows a steady decrease, which may be attributed to pore blockage caused by SDS micelles entering in the membrane pores. The SDS micelles have a micellar size of approximately 17.8 kD (14). Micelles of this size can easily enter in the pore of 20 kD MWCO membrane but may not come out on the other side on account of steric hindrance and tortuasity offered by an asymmetric membrane pore structure.

Dynamic Study of MEUF of Ni(II) Ions

Figure 3 shows a dynamic behavior of MEUF. From the figure it can be seen that the normalized flux decreased rapidly initially and remained nearly

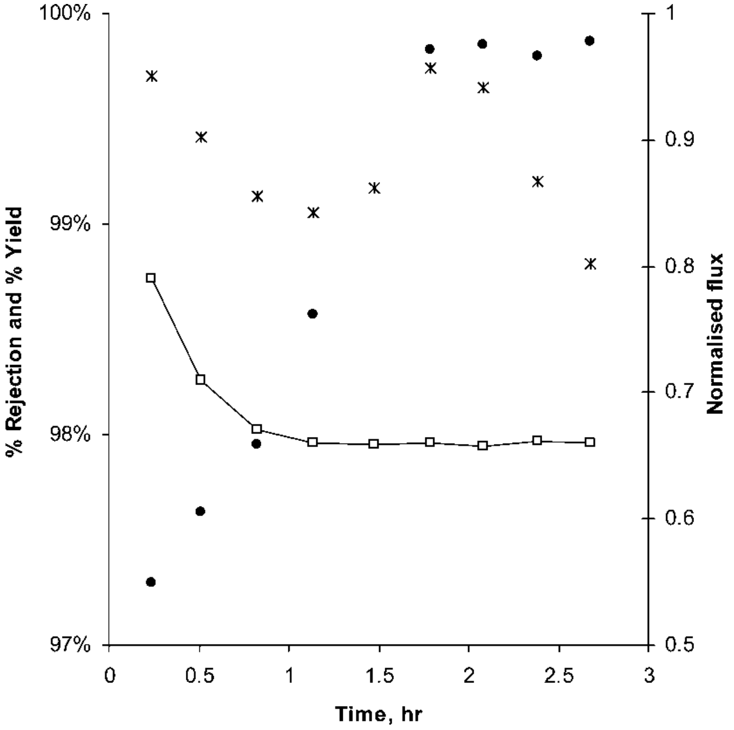

Figure 3. Dynamic behavior of MEUF: $[Ni(II)]_F = 1$ mM, $[SDS]_F = 10$ mM, $[NaCl] = 0$ mM, $[NaBr] = 0$ mM, $[NaI] = 0$ mM, $TMP = 4 \times 10^5$ Pa, $T = 303$ K, pH = 7, □: Normalized flux, ●: % Rejection, : % Yield.

constant throughout (like a typical dead end ultrafiltration). The concentration polarization was negligible under the experimental conditions, from the fact that the flux decline over the time was only 16%. However as the process goes on, the retentate becomes more and more concentrated in Ni(II) and the rejection coefficient goes on increasing. This can be easily correlated to the increase in the surfactant concentration with a corresponding increase in the micellar fraction in the retentate which in turn is responsible for the increasing rejection; however, after a certain time the increase in the micellar fraction is negligible and therefore the rejection attains a constant value. The trend in the % yield does not show any specific variation (% Y was >99% almost throughout).

Effect of Feed Surfactant Concentration

Figure 4 presents the effect of volume fraction on permeate concentration at different SDS concentrations in feed. It is observed that at all concentrations of SDS the permeate concentration remains almost constant throughout the run and varies only in its magnitude with respect to SDS concentration. These observations support the assumption of the presence of an equilibrium between bound and unbound Ni(II) ions. Minor variations in the permeate concentrations may be regarded as mass action shifts in the concentrations that take place dynamically in order to keep the ion exchange equilibrium Eq. (3) and conditions of electrical neutrality on the permeate and retentate side Eq. (4) and (5) intact. The conditions of electrical neutrality in the retentate and the permeate take the form:

$$2[\mathrm{Ni(II)}]_R + [\mathrm{Na}^+]_R = 2[\mathrm{SO}_4^{2-}]_R + [\mathrm{DS}^-]_R \tag{5}$$

$$2[\mathrm{Ni(II)}]_P + [\mathrm{Na}^+]_P = 2[\mathrm{SO}_4^{2-}]_P + [\mathrm{DS}^-]_P \tag{6}$$

From Fig. 4 it can also be noticed that with regard to metal concentration in the permeate stabilization of concentration occurs after a marked decrease. The extent of decrease of concentration of the permeate with an incremental addition of surfactant was larger for 4 mM to 5 mM than for 1 mM to 2 mM. This is because of the shift in the operating regime from the premicellar regime to the postmicellar regime as the CMC of SDS that was used for experimentation was 4.523 mM. No significant extra purification of the permeate was observed (stabilization of $[\mathrm{Ni(II)}]_P$) for addition of any extra amount of SDS in feed beyond $[\mathrm{SDS}]_F = 5$ mM. With the addition of SDS at a concentration of 8 mM or higher, by keeping the feed Ni(II) concentration constant at 1 mM the permeate was almost pure and was in the concentration range of 0.02 to 0.06 mM or 1.18 ppm to 3.52 ppm which is below the norms specified by the US EPA (1).

Figure 5 shows the effect of feed SDS concentration on % R, % Y and normalized flux. The % R as well as % Y increases initially and then

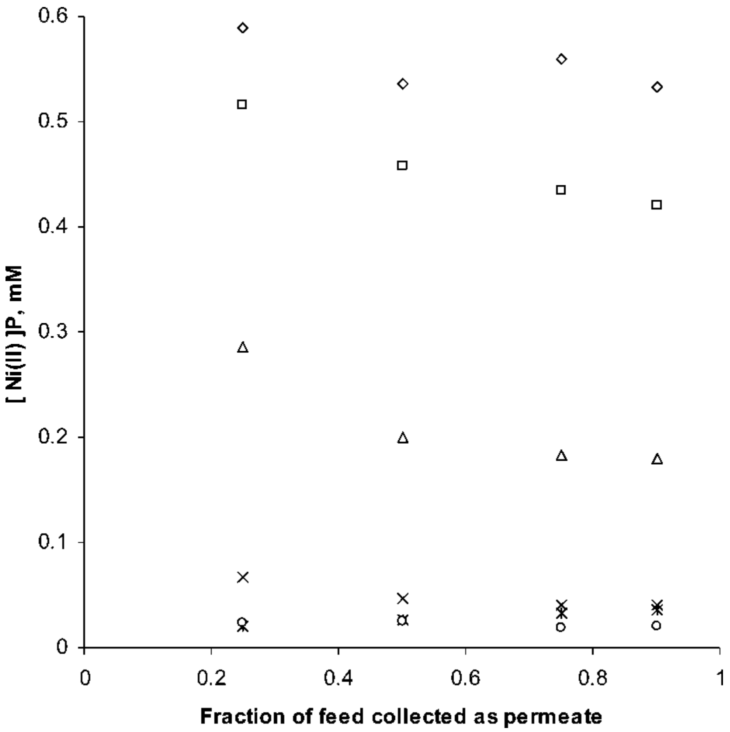

Figure 4. Effect of (permeate volume/feed volume) on permeate concentration at different surfactant concentration: $[Ni(II)]_F = 1$ mM, $[NaCl] = 0$ mM, $[NaBr] = 0$ mM, $[NaI] = 0$ mM, $TMP = 4 \times 10^5$ Pa, $T = 302$ K, $pH = 7$ ◇: $[SDS]_F = 1$ mM, $[SDS]_F = 2$ mM, △: $[SDS]_F = 4$ mM, ×: $[SDS]_F = 5$ mM, : $[SDS]_F = 6$ mM, o: $[SDS] = 8$ mM.

reaches a constant value. This may be explained, by the fact that the addition of the surfactant has two consequences acting in two opposite ways (14). The first effect of SDS addition (in postmicellar region) is the increase in the micelles concentration in the solution. As a consequence a number of likely sites for the attachment of the metallic ion is also increased and hence the % rejection. On the other hand there exists a competition between Na^+ and Ni(II) to bind themselves to the polar heads of micelles and the result of competition, as discussed earlier depends on both the electrical charge of the ion and on their bulk concentration. Qualitatively because of bivalence the Ni(II) ion is preferentially attached to the micelle; however, the high concentration of Na^+ may invert this tendency. The first effect is predominant in the low concentration of the surfactant and consequently the Ni(II) retention increases initially. At moderately high concentrations of surfactants these two effects complement each other and the rejection coefficient does not vary any more.

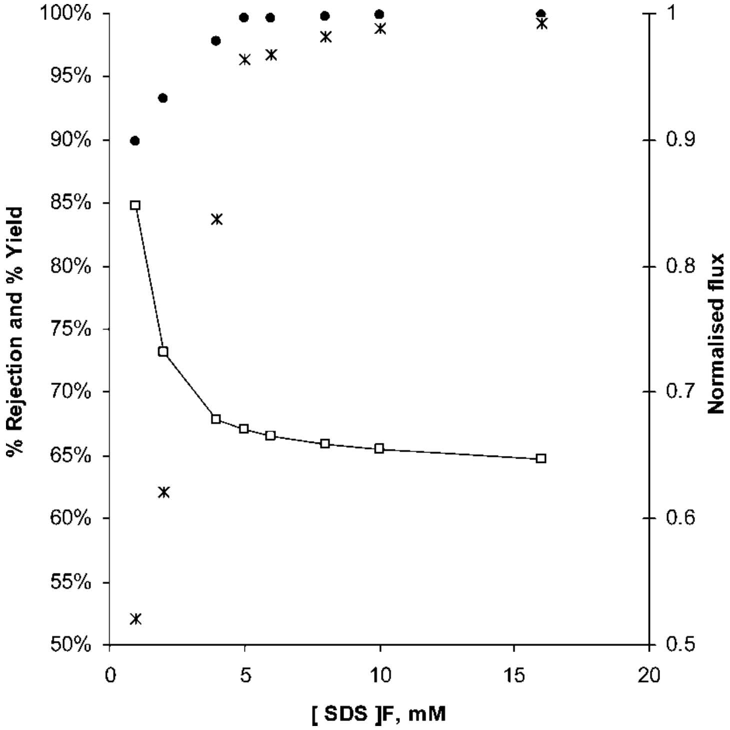

Figure 5. Effect of feed SDS concentration on % R, % Y and normalized flux: $[Ni(II)]_F = 1$ mM, [NaCl] = 0 mM, [NaBr] = 0 mM, [NaI] = 0 mM, TMP = 4×10^5 Pa, T = 302 K, pH = 7, □: Normalized flux, ●: % Rejection, : % Yield.

From Fig. 5 it can be seen that the rejection at very low surfactant concentration was approximately 89% which may be explained on the basis that, SDS being a long chain molecule even in monomeric form, it gets rejected by the membrane to a certain extent. This results in increase in the local concentration of SDS near membrane surface. Such an increase may be beyond the CMC of SDS, resulting in the formation of micellar subregion near the membrane surface. This secondary membrane of micelles formed near the membrane may contribute to the rejection in the premicellar regime. This effect is usually termed as a presieveing effect (15). The preseiveing effect may also be considered to be responsible for a large flux decline that is observed specifically in the premicellar region.

Effect of S/M Ratio

Figure 6 shows the effect of variation of surfactant to the metal ion ratio (S/M) on the rejection of Ni(II) ions. It is observed that the rejection of Ni(II) was

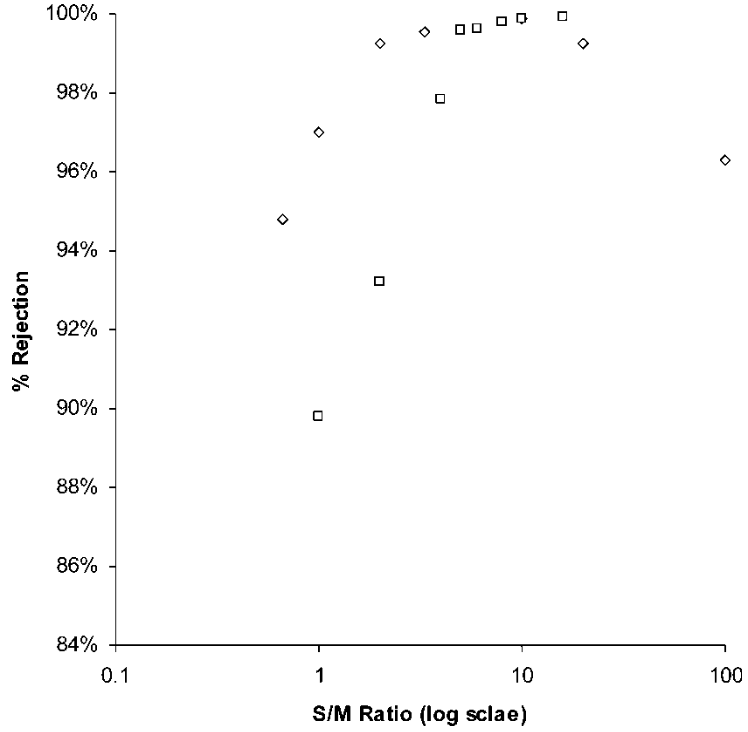

Figure 6. Effect of feed S/M ratio on % R: [NaCl] = 0 mM, [NaBr] = 0 mM, [NaI] = 0 mM, TMP = 4 × 10^5 Pa, T = 302 K, pH = 7: $[Ni(II)]_F = 1$ mM, ◇: $[SDS]_F = 10$ mM.

slightly lower at very high (S/M > 50) and at very low (S/M < 2) S/M ratios. This may be because of the competition between the counter ions as explained in the previous section.

The experimental results show that the rejection of the Ni(II) ions beyond S/M value of 5 does not very much which is in agreement with Huang et al. (3). From the figure it can also be seen that the highest rejection (R > 99.5%) is obtained somewhere near S/M of 10. We thus can conclude that critical S/M ratio, the ratio beyond which the variation of rejection coefficient is sluggish, is found to be 5 while the optimal value of S/M is observed to be 10.

For the data in Fig. 6 we have performed two sets of experiments. One set keeping the Ni(II) concentration constant and varying surfactant concentration to get desired S/M ratio and the other set keeping the surfactant concentration constant and varying metal concentration. It can be noticed that while working at the same S/M ratios but at different Ni(II) concentrations the rejection coefficients are quite different for example at S/M=1 and [SDS] = 1 mM, $[Ni(II)]_F = 1$ mM R = 89.8% while at S/M = 1 but

$[SDS] = [Ni(II)]_F = 10$ mM $R = 97.02\%$. Such a difference in the performance parameter at the same S/M ratio but at a different metal ion concentration indicates that these two variables i.e. the surfactant concentration as well as the metal ion concentration are highly interacting variables.

Effect of pH

Figure 7 shows the effect of pH on the % rejection of Ni(II) ions in MEUF. The pH of the feed solution was varied from 2 to 10. The rejection of Ni(II) remained nearly constant in a broad pH range of 3 to 10. Compared to the results obtained at a high pH Ni(II) rejection was found to be decreased by around 4% at $pH < 3$ which is in agreement with Juang et. al. (15). This is because at low pH ($pH = 2$ say) the concentration of H^+ ions becomes greater than the feed Ni(II) concentration (=1 mM); therefore the Ni(II) ions now have to compete with Na^+ as well as H^+ ions for their binding with SDS micelles.

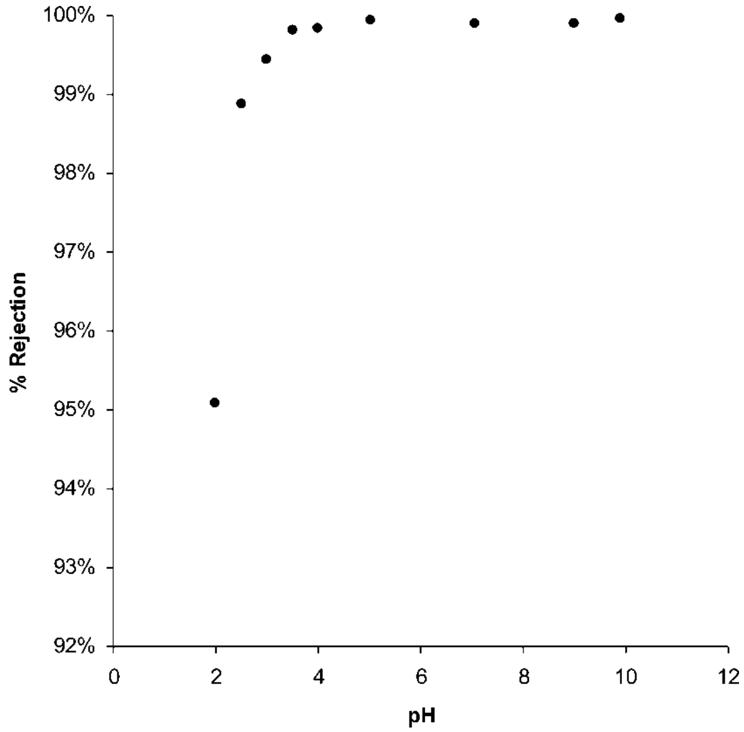

Figure 7. Effect of pH of feed solution on % R: $[Ni(II)]_F = 1$ mM, $[SDS]_F = 10$ mM, $[NaCl] = 0$ mM, $[NaBr] = 0$ mM, $[NaI] = 0$ mM, $TMP = 4 \times 10^5$ Pa, $T = 303$ K ●: % R.

Effect of Added Monovalent Salt

Figures 8 and 9 show the effect of added monovalent salt on the % R and %Y of MEUF respectively. We studied the performance of MEUF by the addition of three salts namely NaCl, NaBr, and NaI. These salts are used in the electroplating and the metal finishing industry for making buffer solutions, and hence can be easily found in the wastewater streams coming from these units. The basic effect of addition of the salt is the decrease in the rejection of metal ions. The presence of the salt results in a drop in CMC of SDS and therefore results in a higher micellar concentration. However the second effect cancels out the advantage of the higher micellar concentration. As per the second effect Ni(II) ions now have to compete with the Na^+ ion which are in excess because of the addition of salt resulting in a drop in the rejection. In fact the observations support this hypothesis; and % rejection (Fig. 9) reduced for each salt when the salt concentration was increased from 10 to 500 mM. (SDS precipitates out at Nacl/NaBr/NaI concentration >1 M during the run because of the common ion effect). The decrease in the rejection as shown by different salts is different and the extent of the

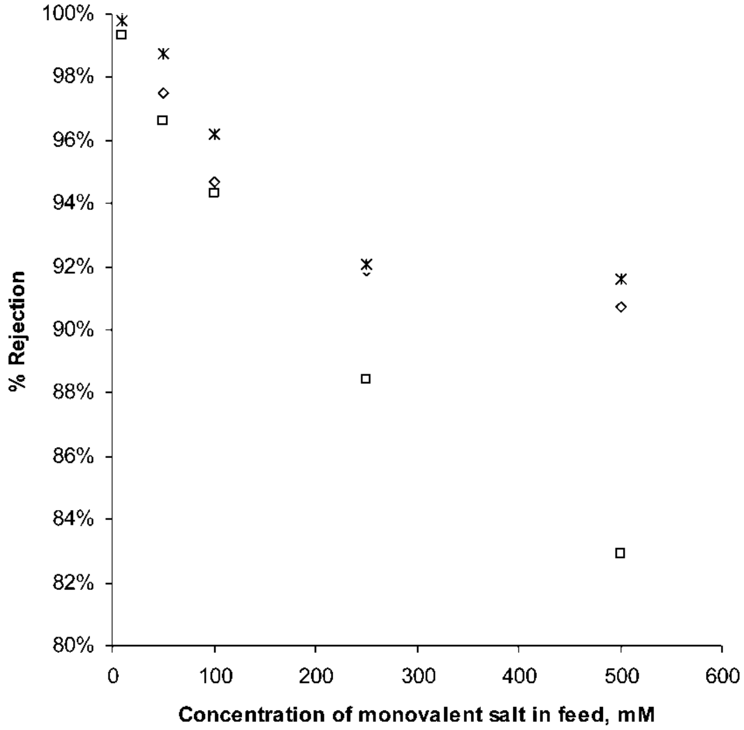

Figure 8. Effect of added monovalent salt on % R: $[Ni(II)]_F = 1$ mM, $[SDS]_F = 10$ mM, TMP $= 4 \times 10^5$ Pa, T $= 303$ K: NaCl, ◇: NaBr, : NaI.

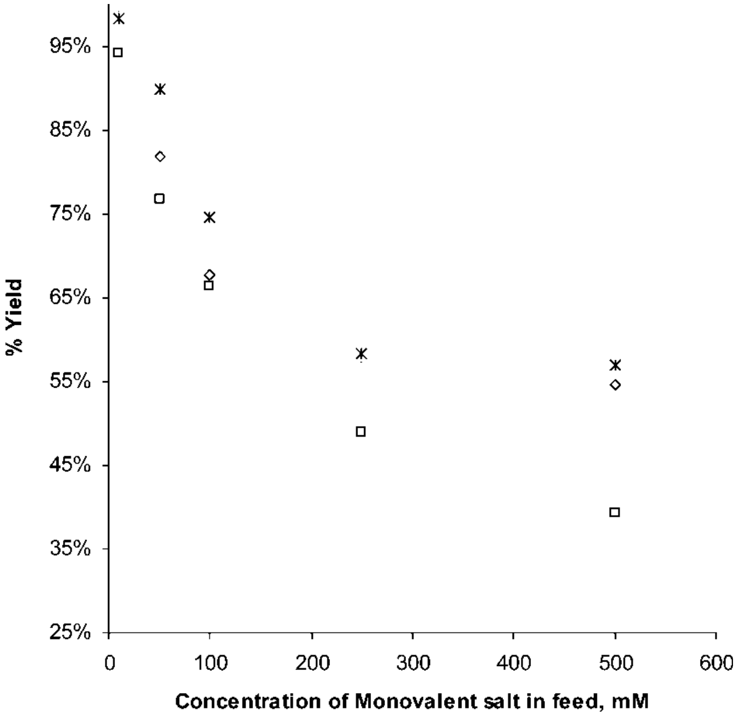

Figure 9. Effect of added monovalent salt on % Y: $[\mathrm{Ni(II)}]_F = 1$ mM, $[\mathrm{SDS}]_F = 10$ mM, TMP $= 4 \times 10^5$ Pa, T $= 303$ K: NaCl, ◇: NaBr, : NaI.

decrease in rejection increases in the order: NaI < NaBr < NaCl. This behavior can be attributed to the electronegativity effect of the co-ions I^-, Br^- and Cl^-. The Cl^- ion being the most electronegative among all can displace the micelle from a bound Ni(II) more strongly than the other two, or in other words will compete more strongly with the micelle to form ion pair with Ni(II) than I^- and Br^-. This effect worsens the rejection behavior of the process. The decrease in the yield coefficient is as shown in Fig. 9; a similar reasoning may be applied to the observed decline in the % Y of the system. However it is to be noted that the decrease in the % Y is greater in magnitude than that for the % R.

CONCLUSION

From the results of the present study the following conclusions may be drawn:

In the presence of a surfactant, the rejection of Ni(II) ions may be of the order of 99.5% when the pH is between 3–10 while it decreases at pH less than three. The presence of monovalent salt decreases the rejection and the effect of NaCl is more pronounced than NaBr or NaI.

The gel layer formation and the membrane fouling is insignificant in the experimental range of concentration. A dynamic study shows that the concentration polarization is negligible because the flux decline over the process time is only 16%, and rejection is the function of the process time and permeate volume. For the present system the critical S/M ratio was 5 and the optimum S/M ratio was 10.

NOMENCLATURE

% R	Percentage rejection
% Y	Percentage yield
J_P	Permeate flux [m.s^{-1}]
J_W	Pure water flux [m.s^{-1}]
TMP	Transmembrane pressure [Pa]
T	Temperature [K]
S/M	Surfactant to metal ion ratio
V	Volume [cm^3]
[Ni(II)]	Concentration of Ni^{2+} [mM]
[Na^+]	Concentration of Na^+ [mM]
[So_4^{2-}]	Concentration of So_4^{2-} [mM]
[DS^-]	Concentration of dodecyl sulfate ion [mM]
[SDS]	Concentration of Sodium dodecyl sulfate [mM]
λ_{max}	Wavelength [nm]

Subscripts

F	Quantity measured in feed stream
P	Quantity measured in permeate stream
R	Quantity measured in retentate stream

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