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Ahmadali Tabatabai<sup>a</sup>; John F. Scamehorn<sup>a</sup>; Sherril D. Christian<sup>b</sup>

<sup>a</sup> INSTITUTE FOR APPLIED SURFACTANT RESEARCH AND SCHOOL OF CHEMICAL ENGINEERING AND MATERIALS SCIENCE THE UNIVERSITY OF OKLAHOMA, NORMAN, OKLAHOMA <sup>b</sup> INSTITUTE FOR APPLIED SURFACTANT RESEARCH AND DEPARTMENT OF CHEMISTRY, THE UNIVERSITY OF OKLAHOMA, NORMAN, OKLAHOMA

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## Water Softening Using Polyelectrolyte-Enhanced Ultrafiltration

AHMADALI TABATABAI and JOHN F. SCAMEHORN\*

INSTITUTE FOR APPLIED SURFACTANT RESEARCH AND SCHOOL OF CHEMICAL  
ENGINEERING AND MATERIALS SCIENCE  
THE UNIVERSITY OF OKLAHOMA  
NORMAN, OKLAHOMA 73019

SHERRIL D. CHRISTIAN

INSTITUTE FOR APPLIED SURFACTANT RESEARCH AND DEPARTMENT OF CHEMISTRY  
THE UNIVERSITY OF OKLAHOMA  
NORMAN, OKLAHOMA 73019

### ABSTRACT

The use of polyelectrolyte-enhanced ultrafiltration (PEUF) for water softening has been studied at several temperatures in the presence and absence of added salt. It is shown that in the absence of added salt, PEUF is highly effective in the rejection of up to 99.7% of hardness ions from an aqueous stream. At a low concentration of added salt, PEUF is effective in the removal of hardness. As salt concentration increases, however, hardness rejection decreases dramatically. The experimental results have been effectively modeled using an ion-binding model based on Oosawa's two-phase approximation theory.

### INTRODUCTION

Water frequently contains numerous solutes, many of which are not desirable either for residential or industrial purposes. The cations of calcium and magnesium, which are responsible for water hardness, are one category of solutes. Some of the consequences of water hardness are the

\* To whom correspondence should be addressed.

formation of residues in pipes and boilers, resulting in poor energy transfer and corrosion, precipitation of chemicals such as soaps, spots, and stains, and an undesirable taste in drinking water (1). At present, the major processes available for water softening are ion exchange and lime softening. A new class of techniques that may be useful in decreasing hardness are the colloid-enhanced ultrafiltration processes. These methods operate continuously at low pressure and low temperature, providing a very clean stream of water. Previous experimental studies have shown that colloid-enhanced ultrafiltration techniques are effective methods of removing a wide variety of solutes from aqueous streams, but the emphasis in earlier research has been on polluted water cleanup, not improvement of drinking water (2-15).

In the colloid-enhanced ultrafiltration processes described here, a negatively charged colloid is added to a stream containing positively charged calcium and magnesium ions. The stream is allowed to mix so that the dissolved calcium and magnesium ions are bound to the colloid. The stream is then filtered under pressure through an ultrafiltration membrane. The colloid (and ions bound to it) is larger than the average pore size of the membrane and does not pass through the membrane. The filtered stream, the permeate, contains a very small fraction of the solutes in the feed. The solution retained by the membrane, called the retentate, contains calcium and magnesium at high concentrations relative to the feed. It is important to note that the volume of the retentate is much smaller than that of the permeate. This small volume of colloid and the bound ions can therefore be treated or disposed of much more efficiently.

One of the colloid-enhanced ultrafiltration processes is polyelectrolyte-enhanced ultrafiltration (PEUF) (10, 13, 14). In PEUF, the process used in this study shown in Fig. 1, charged solutes are bound to oppositely charged soluble polyelectrolytes (anionic polyelectrolytes in the case of calcium or magnesium). The polyelectrolyte chains are larger than the pore size of the membrane and do not pass through. Commercial filtration membranes are available ranging from 5,000 to 100,000 molecular weight cutoff (MWCO). Previous studies involving PEUF show the effect of membrane pore size and solute and colloid concentration on the effectiveness of the process. The polyelectrolyte ions do not dissociate into smaller species to any noticeable degree (as do surfactant micelles), and therefore they do not pass through the membrane in measurable concentrations.

In this paper it is shown that the PEUF process can be used for the effective removal of dissolved calcium and magnesium from water in the presence of a low concentration of sodium chloride at several temperatures. The paper also utilizes a modified Oosawa model (10, 16) which gives an excellent prediction of the hardness concentration in the permeate stream of the ultrafiltration process.

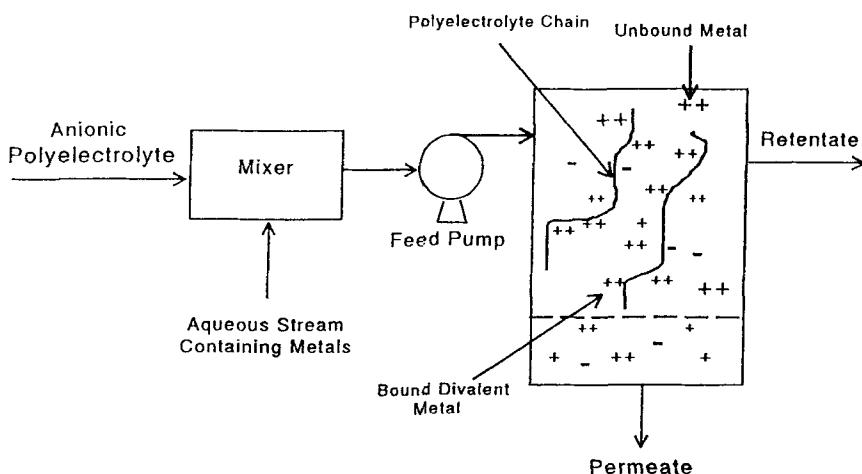


FIG. 1 Schematic of polyelectrolyte-enhanced ultrafiltration (PEUF) for removal of divalent cationic metals.

## EXPERIMENTAL

Crystal calcium chloride dihydrate (FW: 147.02) and crystal magnesium chloride, 6-hydrate (FW: 203.30) were used to simulate calcium and magnesium in hard water. Sodium chloride (FW: 58.44) was used to determine the effect of salinity on the process. Sodium polystyrene sulfonate (PSS) (FW: 206.19), approximate average molecular weight of 85,000 (10), was the polyelectrolyte used in the PEUF process. Elemental analysis showed the PSS monomer to have the empirical formula  $\text{NaC}_8\text{H}_7\text{SO}_3 \cdot 2\text{H}_2\text{O}$ . The PSS was purified using a 10,000 MWCO, spiral-wound membrane in order to remove the lower molecular weight chains to the point where only trace amounts of polyelectrolyte was detected in the permeate. The ultrafiltration membrane was anisotropic cellulose acetate (type C). The purified PSS was then used for subsequent PEUF studies in a 400-mL stirred cell reactor.

A 76-mm diameter membrane used in the 400-mL batch stirred cell reactor, shown in Fig. 2, was soaked overnight in distilled water. A 300-mL solution of the PSS, calcium chloride, and/or magnesium chloride was placed in the stirred cell reactor. The solution temperature was controlled by circulating water from a constant temperature bath through flexible Tygon tubing wrapped around the cell. The experiments were run at 5.5, 15, and 30°C. The solution was stirred at a speed of 845 rpm with a pressure drop of 60 psi across the membrane. Approximately 200 mL of the solution

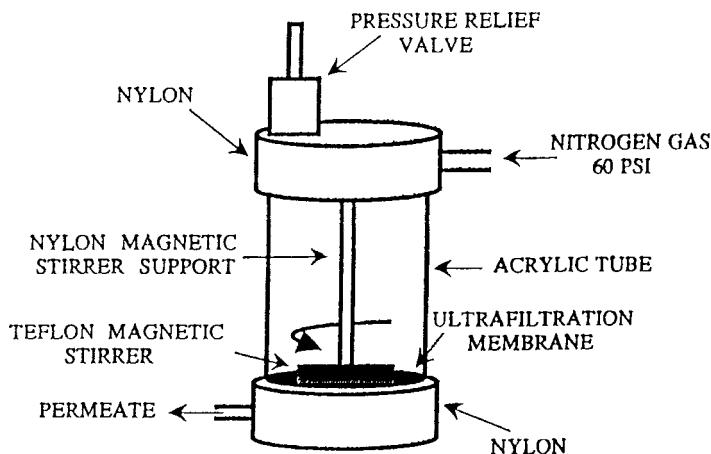


FIG. 2 Stirred cell ultrafiltration unit.

was filtered through the membrane to produce eight samples of 25 mL each, obtained at known times. The permeate samples were weighed and the fluxes calculated. The permeate samples were analyzed for concentrations of PSS, calcium, and/or magnesium. The rejection was determined by analyzing the sample at the midpoint of each run, that is, the point at which 100 mL of the solution had passed through the membrane. The PSS was analyzed using a diode array UV spectrophotometer (wavelength of 226 nm). The calcium and magnesium were determined by atomic absorption spectroscopy (both flame and graphite furnace analysis, depending on concentration).

### ION-BINDING MODEL

It is desirable to model the experimental results so that the permeate metal concentration can be predicted when the retentate metal and colloid concentrations are known (10). This is achieved by using the two-phase approximation theory by Oosawa (16) to determine the fraction of each ion that is bound to the polyelectrolyte or is free in the bulk by relating the counterion binding to the surface potential of the polyelectrolyte. Such a model requires use of material and charge balances for the electrolyte species in the solution. It is assumed that the thermodynamic activity of each electrolyte passing through the ultrafiltration membrane is the same in the permeate as in the retentate: for example, for a compound such as  $\text{CaCl}_2$ , the activity product  $a_{\text{Ca}^{2+}} a_{\text{Cl}^-}^2$  will be the same in the permeate

and the retentate. This assumption has been experimentally confirmed in studies (10, 13) of heavy metal removal using PEUF. For a solution containing PSS,  $\text{CaCl}_2$ , and,  $\text{MgCl}_2$ , the equations describing activity equilibrium reduce to

$$a_{\text{CaCl}_2,\text{ret}} = a_{\text{CaCl}_2,\text{per}} \quad (1)$$

$$a_{\text{MgCl}_2,\text{ret}} = a_{\text{MgCl}_2,\text{per}} \quad (2)$$

where  $a$  is the thermodynamic activity, and per and ret denote permeate and retentate, respectively. In the absence of sodium chloride, the ionic strength is low enough so that the activity coefficient terms may be taken to be unity. Therefore, Eqs. (1) and (2) can be expressed in terms of species concentration as follows:

$$[\text{Ca}^{2+}]_{\text{ret}}[\text{Cl}^-]_{\text{ret}}^2 = [\text{Ca}^{2+}]_{\text{per}}[\text{Cl}^-]_{\text{per}}^2 \quad (3)$$

$$[\text{Mg}^{2+}]_{\text{ret}}[\text{Cl}^-]_{\text{ret}}^2 = [\text{Mg}^{2+}]_{\text{per}}[\text{Cl}^-]_{\text{per}}^2 \quad (4)$$

where the concentrations are those of the unbound ions. The charge balance for both the retentate and the permeate streams must also be satisfied according to

$$[\text{PSS}^-] + [\text{Cl}^-] = [\text{Na}^+] + 2[\text{Mg}^{2+}] + 2[\text{Ca}^{2+}] \quad (5)$$

where  $[\text{PSS}^-]$  is zero in the permeate. To predict the permeate concentration, the concentration of the ions bound to the polyelectrolyte in the retentate must be determined. Assuming that PSS has an extended, rodlike configuration (17), the Oosawa approximation treats counterions as either bound to the polyelectrolyte or free in the bulk aqueous solution. The extent of the counterion binding of the monovalent and divalent ions is related to the equilibrium surface potential of the polyelectrolyte. In the case of the rodlike PSS, the Oosawa model leads to the following expressions for the logarithms of the concentration ratios (bound/free) for the monovalent and the divalent ions:

$$\ln[(1 - \beta)/\beta] = \ln[\phi/(1 - \phi)] + (\beta q + \beta' q')zQ \ln(1/\phi) \quad (6)$$

$$\ln[(1 - \beta')/\beta'] = \ln[\phi/(1 - \phi)] + (\beta q + \beta' q')z'Q \ln(1/\phi) \quad (7)$$

where  $\beta$  and  $\beta'$  are the apparent degrees of dissociation for the monovalent and the divalent ions, respectively,  $\phi$  is the fraction of the total solution volume in which the bound ions are located,  $q$  and  $q'$  are the fractions of the free ion charge carried by the two types of counterions,  $z$  and  $z'$  are the absolute values of the counterion charges (1 for  $\text{Na}^+$ , 2 for  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$ ), and  $Q$  is the dimensionless potential parameter, important in determining the extent of binding of counterions. For PSS,  $\phi$  is taken to be

equal to 0.2 L/mol (10), the apparent molar volume of the styrenesulfonate, multiplied by the total molarity of styrenesulfonate units in solution. The variables  $\beta$ ,  $\beta'$ ,  $z$ ,  $z'$ ,  $q$ , and  $q'$  in Eqs. (6) and (7) can be represented in terms of known concentrations of sodium and PSS to yield

$$\ln\left(\frac{[\text{Na}]_{\text{br}}}{[\text{Na}]_{\text{fr}}}\right) = \ln\left(\frac{\phi}{1 - \phi}\right) + \frac{[\text{PSS}] - [\text{Na}]_{\text{br}} - 2[\text{metal}]_{\text{br}}}{[\text{PSS}]} Q \ln\left(\frac{1}{\phi}\right) \quad (8)$$

$$\ln\left(\frac{[\text{metal}]_{\text{br}}}{[\text{metal}]_{\text{fr}}}\right) + \ln\left(\frac{\phi}{1 - \phi}\right) = 2\ln\left(\frac{[\text{Na}]_{\text{br}}}{[\text{Na}]_{\text{fr}}}\right) \quad (9)$$

where  $[\text{Na}$  or  $\text{metal}]_{\text{br}}$  and  $[\text{Na}$  or  $\text{metal}]_{\text{fr}}$  denote the sodium or divalent metal ions bound and free in the retentate, respectively, and metal indicates total calcium and magnesium.

It is the binding of the divalent cations responsible for hardness to the highly charged polyelectrolyte anions which immobilizes these cations, preventing them from passing through the membrane.

## RESULTS AND DISCUSSION

The results of the PEUF runs are shown in Table 1. The removal efficiency of the calcium and magnesium is represented by rejection,  $R$  (%), as defined by

$$R = 1 - \frac{[\text{total metal}]_{\text{per}}}{[\text{total metal}]_{\text{ret}}} (100\%) \quad (10)$$

### Effectiveness of PEUF for Water Softening

The PEUF runs performed at [PSS] to [total metal] ratios of 3.5 and 6.7 in the absence of added salt are shown in Fig. 3. The percent rejection of total metal is plotted against the total metal concentration in the retentate. Total metal represents calcium plus magnesium. As the ratio of [PSS] to [metal] increases, the rejection increases. This is because an increase in the availability of the negatively charged sites on the PSS chains increases the magnitude of the PSS surface electrical potential and therefore enhances binding of the positively charged ions. It is also noted that at a constant ratio of [PSS] to [total metal], as the concentration of PSS and metal are reduced simultaneously, the concentration of metal in the permeate decreases, resulting in increased rejections, as predicted by the model (10). This effect is especially useful in a situation where a low concentration of hardness is present in water, and ultrapure water is desired.

TABLE I  
Polyelectrolyte-Enhanced Ultrafiltration of Calcium and Magnesium: Concentrations in Molarity<sup>a</sup>

PSS/metal ratio	Temp (°C)	Metal,ret. experimental	PSS,ret. experimental	NaCl, feed	Metal,per. experimental	Metal,per. model	% Rejection, experimental	% Rejection, model
8.98	5.5	1.548E-03	1.390E-02		7.820E-06	4.959E-06	99.49	99.68
6.74	5.5	2.111E-03	1.422E-02		1.280E-05	1.361E-05	99.39	99.36
7.86	5.5	1.796E-03	1.412E-02		6.520E-06	7.860E-06	99.64	99.56
8.35	15.0	1.704E-03	1.423E-02		6.000E-06	6.543E-06	99.65	99.62
9.11	15.0	1.510E-03	1.376E-02		8.230E-06	4.644E-06	99.45	99.69
6.67	15.0	2.071E-03	1.381E-02		1.180E-05	1.336E-05	99.43	99.35
2.25	30.0	6.180E-03	1.388E-02		9.170E-04	1.337E-03	85.16	78.37
3.88	30.0	3.676E-03	1.426E-02		1.320E-04	1.414E-04	96.41	96.15
4.26	30.0	3.480E-03	1.481E-02		9.300E-05	9.726E-05	97.33	97.21
8.26	30.0	1.778E-03	1.469E-02		1.210E-05	7.204E-06	99.32	99.59
0.65	30.0	2.385E-03	1.551E-03		1.687E-03	1.695E-03	29.27	28.95
28.59	30.0	4.411E-03	1.261E-01		1.300E-05	1.819E-05	99.71	99.59
5.37	30.0	1.461E-02	7.837E-02		5.600E-04	6.531E-04	96.17	95.53
6.82	30.0	1.144E-02	7.810E-02		2.490E-04	3.003E-04	97.82	97.38
6.48	30.0	2.770E-04	1.796E-03		7.490E-07	2.955E-07	99.73	99.89
6.15	30.0	1.244E-03	7.648E-03		1.120E-05	5.937E-06	99.10	99.52
4.36	30.0	3.330E-04	1.453E-03		8.180E-06	1.367E-06	97.54	99.59
7.77	30.0	2.076E-03	1.612E-02		1.300E-05	1.055E-05	99.37	99.49
5.15	30.0	2.845E-03	1.464E-02		6.550E-05	4.113E-05	97.70	98.55
5.24	30.0	2.510E-03	1.513E-02		8.759E-05	2.313E-05	96.51	99.08
3.62	30.0	4.910E-04	1.777E-03		7.325E-06	6.936E-06	98.51	98.59
7.54	30.0	1.872E-03	1.411E-02		1.910E-05	9.048E-06	98.98	99.52
3.26	30.0	4.757E-03	1.552E-02		3.310E-04	3.615E-04	93.04	92.40
12.83	30.0	6.154E-03	7.933E-02		3.266E-05	5.055E-05	99.47	99.18
12.30	30.0	6.689E-03	8.225E-02	0.00034	4.600E-05	6.306E-05	99.31	99.06
12.57	30.0	6.074E-03	7.636E-02	0.00342	5.120E-05	6.634E-05	99.16	98.91
12.18	30.0	6.604E-03	8.044E-02	0.00855	8.990E-05	1.063E-04	98.64	98.39
4.12	30.0	4.680E-04	1.927E-03	0.00878	1.540E-04	5.458E-05	67.09	88.34
6.01	30.0	2.353E-03	1.413E-02	0.00881	9.400E-05	7.695E-05	96.01	96.73
5.06	30.0	2.814E-03	1.424E-02	0.00089	1.520E-04	4.985E-05	94.60	98.23
4.87	30.0	3.012E-03	1.466E-02	0.00855	3.470E-04	1.468E-04	88.48	95.13
4.45	30.0	3.261E-03	1.450E-02	0.01690	4.900E-04	3.153E-04	84.97	90.33
3.52	30.0	4.074E-03	1.434E-02	0.03422	1.370E-03	9.242E-04	66.37	77.31

<sup>a</sup> ret = retentate, per = permeate. Metal: calcium + magnesium. Model values based on  $Q = 2.10$ .

Figure 4 shows the effect of [PSS] to [total metal] ratio on rejection. The theoretical stoichiometric ratio of PSS to either calcium or magnesium is 2 to 1. The actual ratio needed for effective ultrafiltration may be higher than the stoichiometric ratio. In this study a PSS to metal ratio of at least 6 is required to obtain rejections higher than 99%. The calcium and magnesium rejections obtained are as high as 99.71% for a PSS to metal ratio of 28.6 and as low as 29.27% for a PSS to metal ratio of 0.65. Also, under similar conditions calcium and magnesium are removed with the

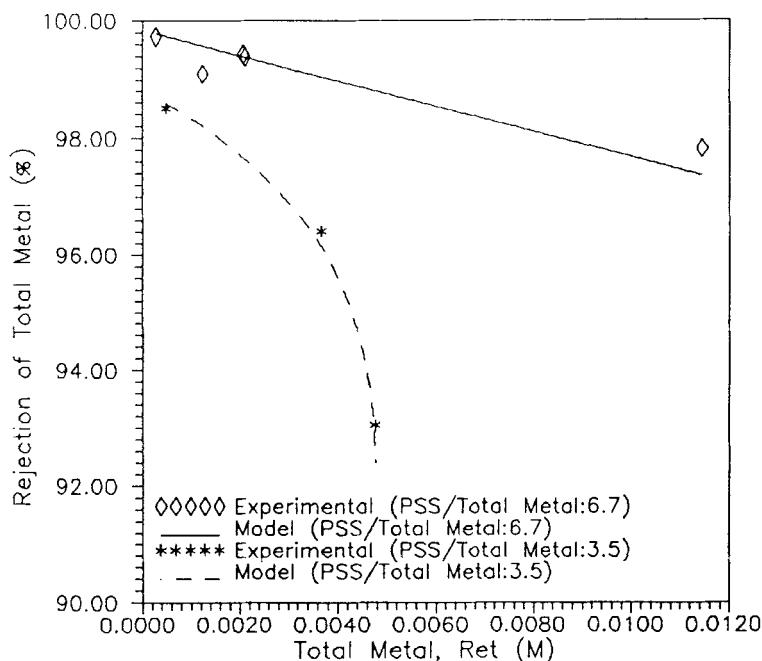


FIG. 3 Rejection of total metal versus total metal concentration present in the retentate in the absence of added salt. PSS/total metal ratios of 3.5 and 6.7.

same rejection. Rejections predicted by the model agree quite well with the observed rejections.

#### Effect of Added Salt

The ionic strength of the aqueous stream greatly affects the effectiveness of the PEUF process. Increasing the salt concentration and therefore the ionic strength of the stream leads to compression of the electric double layer and therefore the electrostatic attraction between the positively charged calcium and magnesium ions and the negatively charged PSS ions is greatly reduced. As a result, the unbound calcium and magnesium in the stream pass through the ultrafiltration membrane leading to poor rejection of these ions. Figure 5 shows the effect of salt concentration on the permeate calcium concentration. As the salt concentration is increased, the concentration of metal in the permeate increases. In the presence of 2000 ppm (0.0342 M) of sodium chloride, calcium rejection is only 66%, while in the presence of only 20 ppm (0.00034 M) of sodium chloride, a

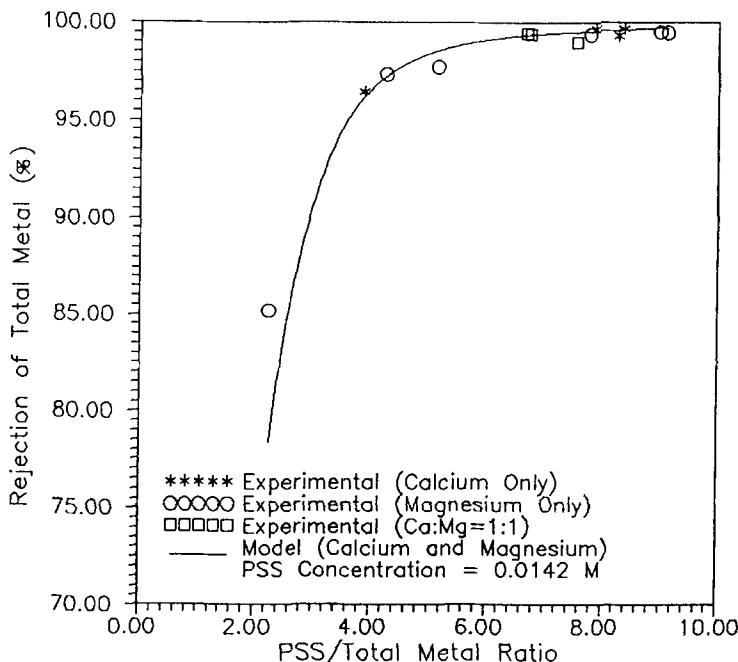


FIG. 4 Rejection of total metal as a function of PSS/total metal ratio at a constant PSS concentration.

rejection of nearly 99% is possible. At a higher concentration of PSS, however, better rejections are possible. The presence of salt also causes deviation between the model predictions and the experimental results. This is especially true at higher salt concentrations and lower PSS to metal ratio. Deviations from the model in the presence of salt could be somewhat decreased by introducing a parameter explicitly accounting for added monovalent salt concentration (10). Activity coefficient correlations might also be made to improve the correlation of data.

#### Effect of Temperature

The PEUF process was run at PSS to total metal ratio of 6.7 and 9.1 at 5.5, 15, and 30°C. As shown in Fig. 6, temperature has a negligible effect on the metal concentration in the permeate at these ratios. The flux of the aqueous stream of the permeate, however, was reduced from 89.8 L/h·m<sup>2</sup> at 30°C to 49.6 L/h·m<sup>2</sup> at 5.5°C as shown in Fig. 7. The decrease in flux at low temperatures may be attributed to the increased viscosity of

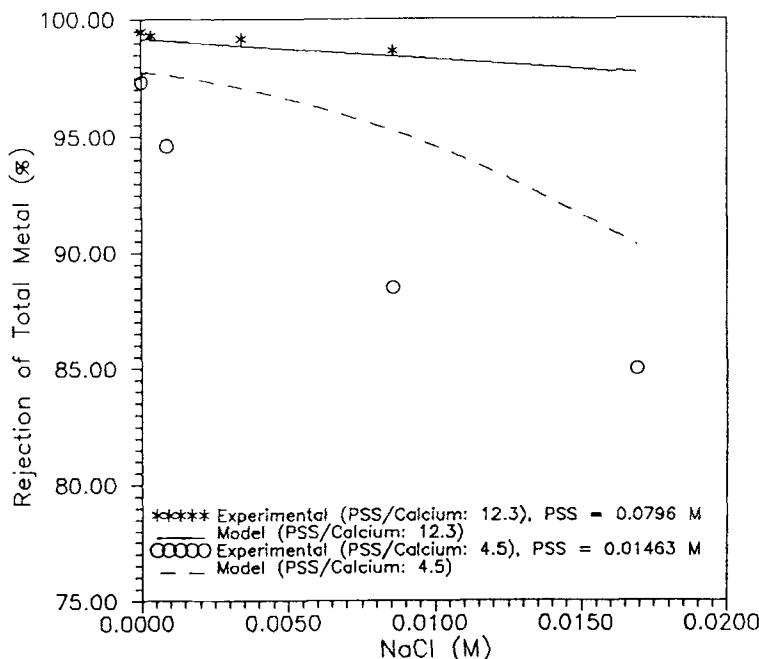


FIG. 5. Effect of added NaCl concentration on permeate metal concentration.

the aqueous stream as well as contraction of the ultrafiltration membrane pores.

### Effectiveness of Model

A single adjustable parameter,  $Q$ , is required to predict permeate concentrations of the divalent metal ions from the known values of the metal and polyelectrolyte ion concentrations in the retentate. By means of a nonlinear least-squares regression analysis (8, 10), it is possible to obtain the optimum or best fit value of  $Q$  for fitting the entire collection of data in Table 1. The value of  $Q$  is determined to be  $2.10 \pm 0.09$ . This value corresponds to a mean relative error of 42.8% in the predictions. This error is partly due to the wide range of PSS, metal, and salt concentrations employed in the experimental runs. The inclusion of data at high salt concentrations in the analysis also contributes greatly to the overall error as is indicated by the relatively large deviation between experimental results and model predictions in Fig. 5.

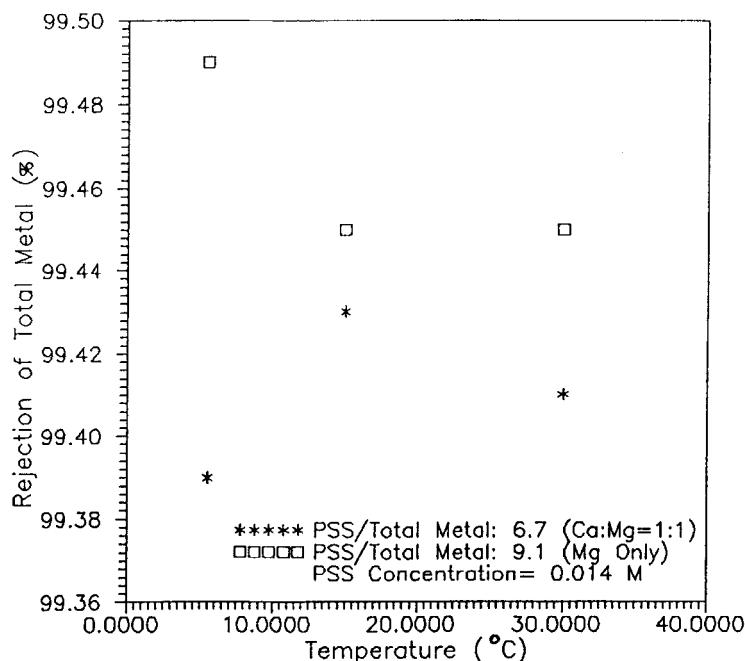


FIG. 6 Effect of temperature on total metal rejection.

The model is highly effective in predicting the permeate concentration in the absence of added salt and in the presence of low concentrations of added salt.

## CONCLUSIONS

1. The polyelectrolyte-enhanced ultrafiltration process, operating at a relatively low pressure and temperature, is highly effective in the removal of hardness from aqueous streams, resulting in rejection of up to 99.7%.
2. Increasing the PSS to metal ratio leads to higher rejections.
3. Calcium and magnesium are removed with the same rejection under similar conditions.
4. At constant ratio of PSS to total metal, decreasing the PSS and metal concentration simultaneously leads to lower metal concentration in the permeate and therefore higher rejection.

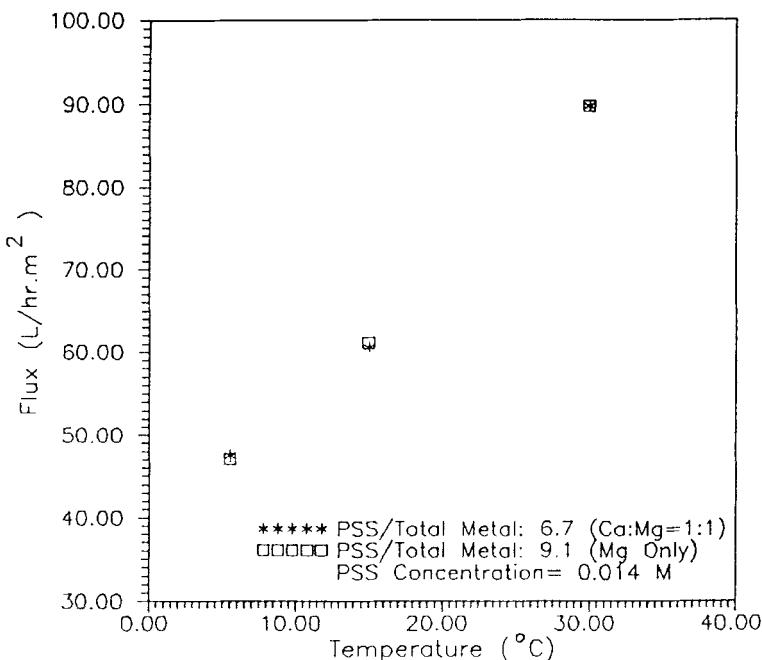


FIG. 7 Effect of temperature on flux.

5. PEUF is also effective in hardness removal in the presence of very low concentrations of added salt. At higher salt concentrations, however, the rejection decreases dramatically.

6. Moderate temperature changes have a negligible effect on the rejection of calcium and magnesium. The permeate flux, however, is reduced at a lower temperature.

7. The ion binding model provides an excellent prediction of metal ion concentrations in the permeate in the absence of added salt or in the presence of a low concentration of salt. The presence of a high salt concentration, however, results in moderate to extreme deviations of the model predictions from observed data.

This study has demonstrated the technical feasibility of PEUF to soften water. Currently, the recovery and regeneration of the polyelectrolyte is being studied to make the process more economically viable.

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