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Reverse Osmosis Separation of NaCl Using a Bentonite Membrane

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ABSTRACT

The results of 11 experiments using compacted bentonite membranes in a cross-flow experimental cell equipped with a piston to maintain clay membrane compaction are reported. Due to dispersion in the porous frit, solute concentration buildup adjacent to the membrane was not a problem at the flow rates used in these experiments (6 to 126 mL/hr). The solute rejection efficiency of the bentonite membrane decreased with increasing solution concentration. The rejection efficiency for the 0.5-mm thick membrane ranged from 68% of Cl^- for 100-mM

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(3545 ppm) NaCl solution to 13% of Cl^- for 2300-mM (81,542 ppm) NaCl solution. The membrane exhibited stable solute rejection.

Key Words: Membrane; Clay; Reverse Osmosis; Brine.

INTRODUCTION

A number of laboratory experiments have demonstrated that clays exhibit membrane properties.[1–28] Solute separation mechanisms for clay membranes are described by Fritz[29] and Ishiguro et al.[30] However, investigation of potential uses of clays as membranes has been limited.

Ishiguro et al.[30] performed preliminary reverse osmosis testing on a poorly compacted, 0.5-mm thick, modified montmorillonite membrane (SWy-1 Wyoming bentonite, obtained from the Clay Source Repository, University of Missouri-Columbia) to investigate the reverse osmosis potential of clay. No compaction pressure was reported for the membrane they used; the clay paste was simply smeared into the experimental cell. They concluded that the montmorillonite membrane exhibited characteristics typical for a charged membrane by rejecting NaCl less efficiently with increasing solute concentration. They also concluded that the rejection capability of the montmorillonite membrane for small organic solutes was very low and that the separation of amino acids greatly depended on the charge of the amino acid molecule.

The purpose of this study was to further investigate the potential use of clays as reverse osmotic membranes. Clay membranes should reject solute more efficiently at higher membrane compaction levels.[13,19,29] Therefore, a bentonite membrane compacted at 16.3 MPa (2360 psi) was tested for NaCl rejection capacity.

METHODS

A flat-leaf experimental cell was designed for the reverse osmosis experiments using clays as the membrane (Figure 1). This cell was constructed from 316 stainless steel and is designed to operate at fluid pressures up to 20.7 MPa (3000 psi). The cell uses a piston to compress the clay. This feature is not present in commercially available reverse osmosis (flat leaf) experimental cells, but was necessary to maintain the compaction of the clay in these experiments. The membrane area in the experimental cell is 136.5 cm^2.

The bentonite clay used in these experiments was prepared using methods presented by Fritz and Whitworth.[25] The clay membranes were

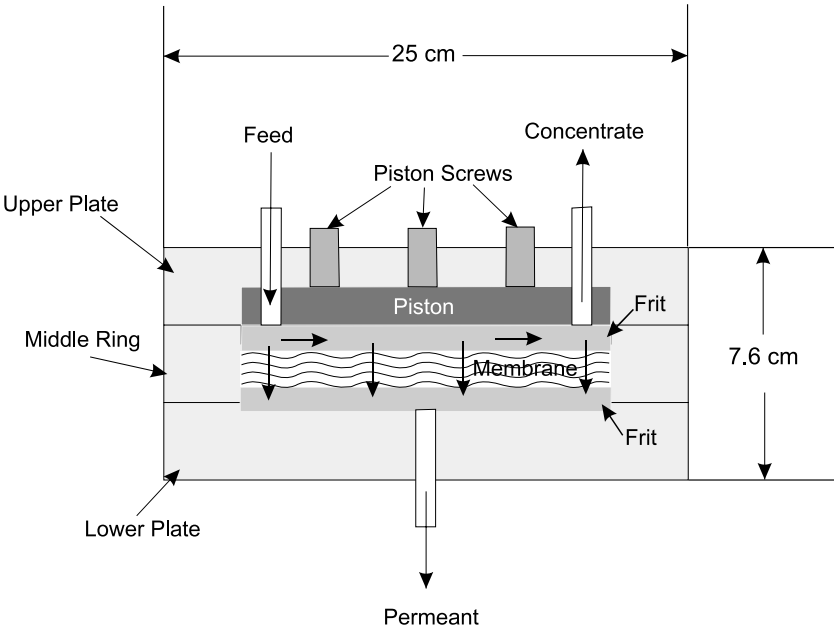

Figure 1. Schematic cross-section of experimental cell used in reverse osmosis experiments. The feed water flows into the feed port and then into the porous frit overlying the membrane. At this point, some of the flow passes through the membrane and some flows parallel to the membrane through the porous frit and exits through the concentrate port. The permeant (the fluid that passes through the membrane) exits through the permeant port. This apparatus is 25-cm long, 20-cm wide, and 7.6-cm thick. Each of the three 316 stainless steel plates that comprise the apparatus are 2.5-cm thick. The membrane area is 136.5 cm^2 and measures approximately 9 by 15 centimeters. The 316 stainless steel frits are each 3.2-mm thick.

prepared by first compacting a thin layer of freeze-dried clay sandwiched between two Millipore and two Whatman #2 filters in a machined and hardened steel die placed in a hydraulic press. The shape of the die exactly matches the membrane recess in the experimental cell. The coarser Whatman filter paper is used to protect the Millipore filter paper from damage during pressing. Exactly 8.00 g of freeze-dried bentonite powder was used to prepare the membrane used in these experiments. The dry clay membrane was first compressed at 16.3 MPa in a hydraulic press for 2 days, then transferred to the cell and compressed while in the partially assembled cell at 16.3 MPa for approximately 1 hour. Next, the cell was assembled and the screws that lock the piston in place were torqued

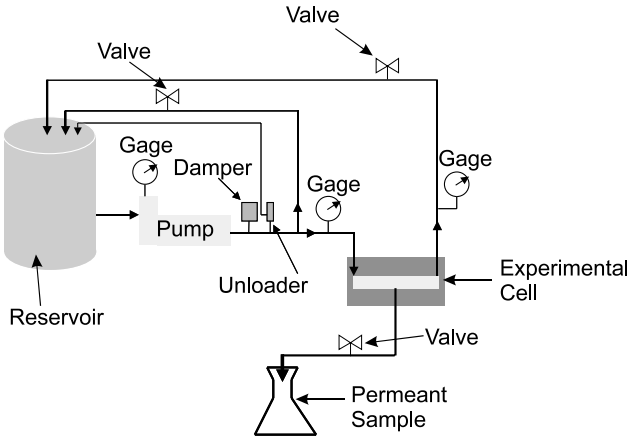

Figure 2. Schematic of experimental setup.

to 0.0031MPa. The cell was then set up as shown in Figure 2 for the experiment.

The resulting membrane was 0.5-mm thick. The density of the freeze-dried clay used was measured using a pycnometer so that membrane porosity could be calculated from

$$\Phi = \left(\frac{1 - M_c}{\rho_c A \Delta x}\right) \cdot 100 \quad (1)$$

where Φ is porosity, M_c is the mass of the clay in grams, ρ_c is clay density (g/cm^3), A is membrane area (cm^2), and Δx is membrane thickness (cm). The bentonite we used had a density of 2.17 g/cm^3 and the membrane porosity was calculated as 46%.

Table 1. Summary of analytical methods, accuracy, and precision for chemical analyses.

Species	Equipment	Method number	Accuracy	Precision
Chloride (< 0.0056M)	IC	EPA300.0	< 1%	< 0.5%
Chloride (> 0.0056M)	FIA	QuikChem 10-117-07-1-J	0.5%	0.5%

Accuracy and precision are stated at 1 standard deviation. Error bars on graphs are at plus or minus 2 standard deviations. IC = ion chromatograph; FIA = flow injection analysis.

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Table 2. Summary of experimental parameters for cross-flow experiments.

Experiment number	Experiment duration (days)	Concentrate flow (mL/hr)	Permeant flow (mL/hr)	Average steady-state feed concentration (Cl^-)	Average steady-state permeant concentration (Cl^-)	Cl^- rejection (%)	Inlet pressure (MPa)	Outlet pressure (MPa)
CF-1	14.0	22.7	0.21	3,368 ppm (95 mM)	1,064 ppm (30 mM)	68	3.79	3.45
CF-2	8.58	78.6	0.16	3,403 ppm (96 mM)	1,099 ppm (31 mM)	68	4.14	2.76
CF-3	6.96	108.3	0.13	3,403 ppm (96 mM)	1,170 ppm (33 mM)	66	4.14	2.07
CF-4	6.50	19.2	0.18	3,439 ppm (97 mM)	1,099 ppm (31 mM)	68	4.14	3.10
CF-5	7.00	7.46	0.17	3,368 ppm (95 mM)	1,099 ppm (31 mM)	67	4.14	3.45
CF-6	8.56	44.4	0.12	10,281 ppm (290 mM)	4,963 ppm (140 mM)	52	4.14	2.07
CF-7	7.72	44.7	0.2	21,272 ppm (600 mM)	12,409 ppm (350 mM)	42	4.14	2.07
CF-8	7.96	85.7	0.18	35,807 ppm (1,010 mM)	25,881 ppm (730 mM)	28	5.52	3.10
CF-9	7.96	82	0.19	54,952 ppm (1,550 mM)	42,544 ppm (1,200 mM)	23	5.52	3.10
CF-10	5.75	88.8	0.16	68,779 ppm (1,940 mM)	59,207 ppm (1,670 mM)	14	5.52	3.10
CF-11	4.35	85.5	0.15	81,542 ppm (2,300 mM)	71,261 ppm (2,010 mM)	13	5.52	3.10

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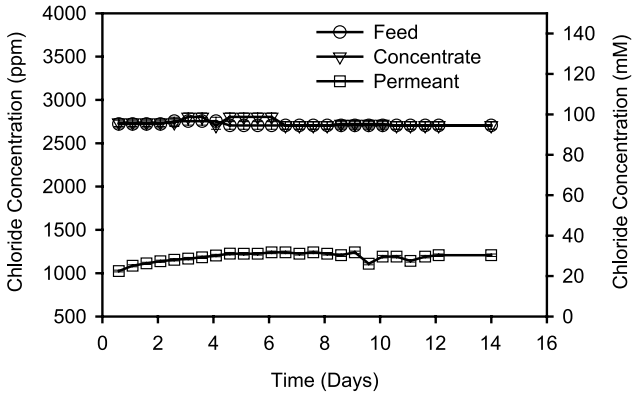

Figure 3. Results of experiment CF-1.

The pump used in these experiments was a Beckman model 110A single-piston, high-pressure liquid chromatograph pump (Palo Alto, CA, USA). It has a variable flow range of from 0 to 594 mL/hr and will operate at pressures up to 41.4 MPa. Before beginning the experimental run, the dry clay membrane was hydrated in place by passing type I deionized water into the cell at rate of 30 mL/hr for 2 days. After 2 days, the outlet needle valve was slowly adjusted until the inlet pressure was about 4.13 MPa (600 psi) and the NaCl solution was flushed through the cell. Samples from the reservoir, outlet, and effluent streams were then taken at almost constant intervals during the experiments. A series of tests using different NaCl concentrations and

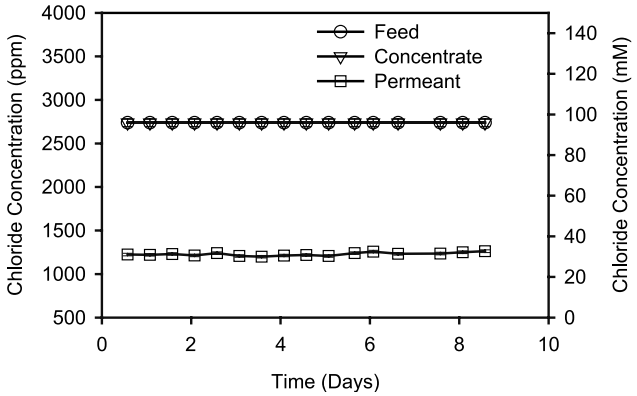

Figure 4. Results for experiment CF-2.

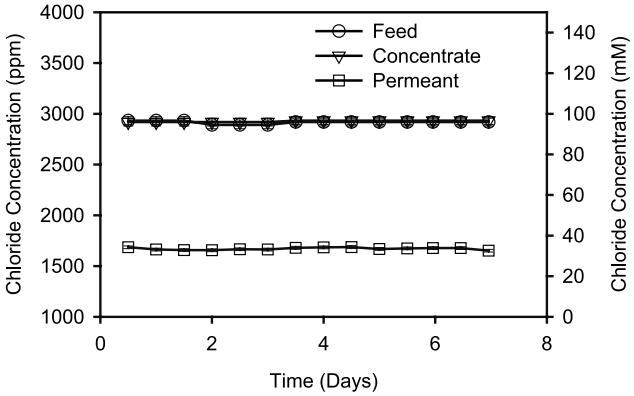

Figure 5. Results for experiment CF-3.

different flow rates were carried out to investigate the effect of differing solute concentration and flow rate on salt rejection efficiency.

Chemical Analyses

Ion chromatography (IC) was used to measure low concentrations of chloride below 5.6 mM (200 ppm) after dilution. The ion chromatograph was a Dionex 600 with an AS50 autosampler and chromatography compartment, CD25 conductivity detector, and a GP50 gradient pump

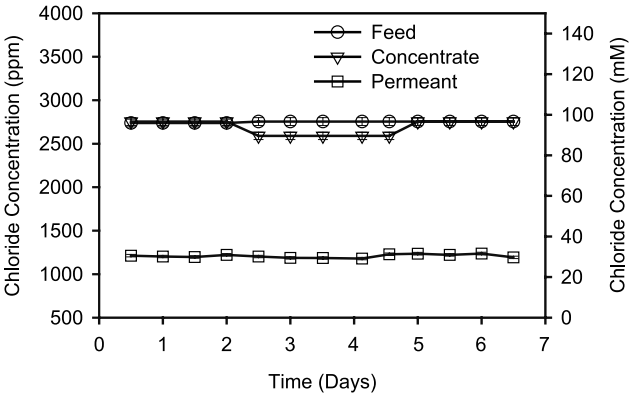

Figure 6. Results for experiment CF-4.

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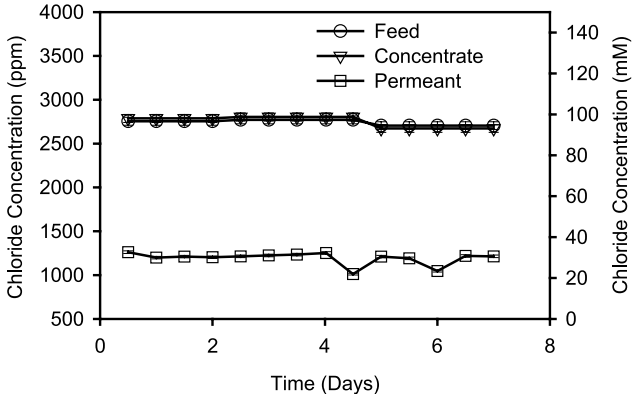

Figure 7. Results for experiment CF-5.

(Sunnyvale, CA, USA). The column was a Dionex AS14. A series of standard solutions was used to calibrate the instrument. Internal reference standards were run near the beginning and end of each IC run. Calibration checks were run every 20 to 30 samples and a duplicate sample was run every 10 to 12 samples. Internal reference standards of $Cl^- = 40.4$ ppm (1.14 mM) were periodically run. The results of these analyses were $Cl^- = 40.4$ ppm (1.14 mM), 40.8 ppm (1.15 mM), and 40.1 ppm (1.13 mM).

High chloride concentrations above 200 ppm (5.6 mM) were analyzed with a Lachat QuikChem 8000 flow injection analysis automated ion analyzer (FIA) using QuikChem method 10-117-07-1-J. This method covers the determination of chloride in drinking, ground, and surface waters, and

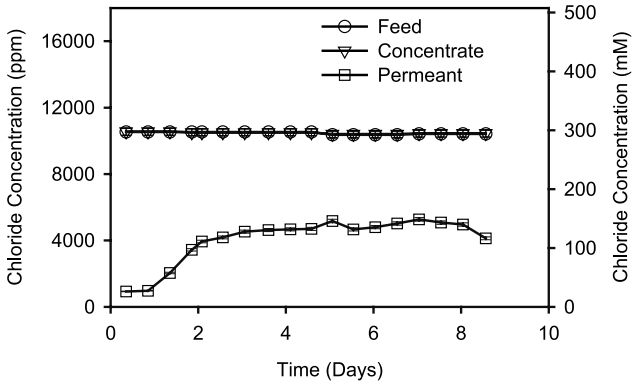

Figure 8. Results for experiment CF-6.

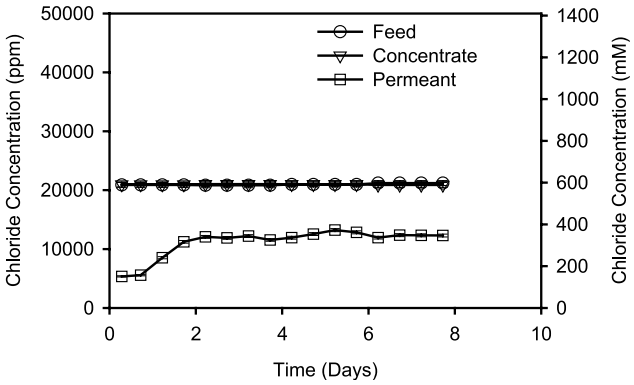

Figure 9. Results for experiment CF-7.

domestic and industrial wastes. The applicable range is 200 ppm (5.6 mM) to 24,995 ppm (705 mM) Cl. Dilutions were made when concentrations were above this range. The method detection limit is 7.45 ppm (0.21 mM). Triplicate analysis were conducted for each calibration standard and a 0.5% RSD was set as replicate criteria. Analytical accuracy and precision of chemical measurements are presented in Table 1.

RESULTS

We performed 11 experimental runs with NaCl solutions using commercially available bentonite clay as a membrane in a cross-flow

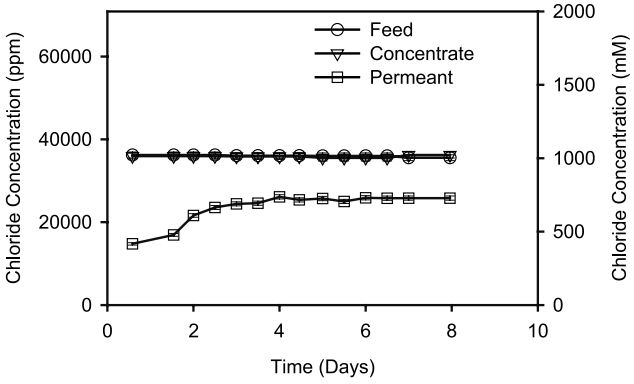

Figure 10. Results for experiment CF-8.

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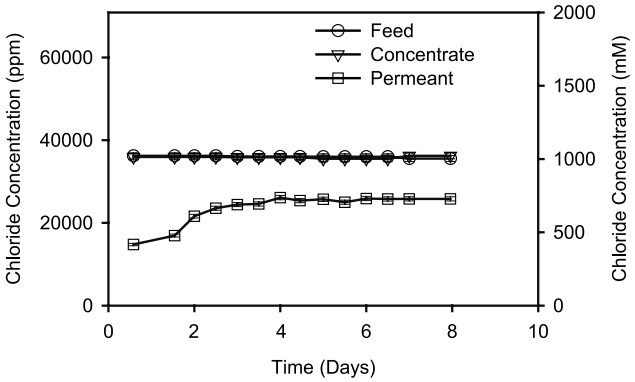

Figure 11. Results for experiment CF-9.

reverse osmosis cell (Table 2). A single clay membrane was used for experiments CF-1 through CF-11 (Figures 3–13).

The Cl^- rejection results are plotted in Figures 3–13 and ranged from 68% for 100-mM (3545 ppm) solution to 13% for 2300-mM (81,542 ppm) solution. Rejection, R, was calculated via

$$R = \frac{C_f - C_p}{C_f}(100) \tag{2}$$

where C_f is the steady-state feed Cl^- concentration and C_p is the steady-state permeant concentration. The graphs indicate that steady-state solute

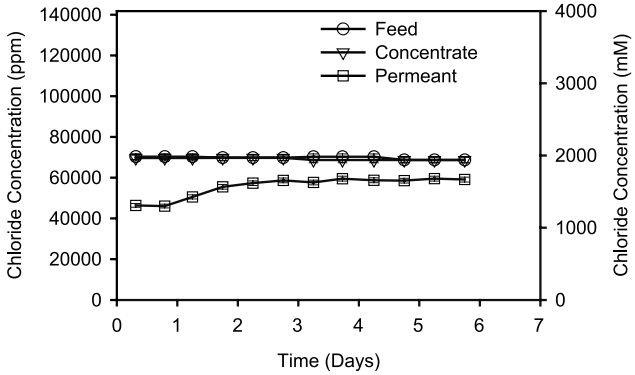

Figure 12. Results for experiment CF-10.

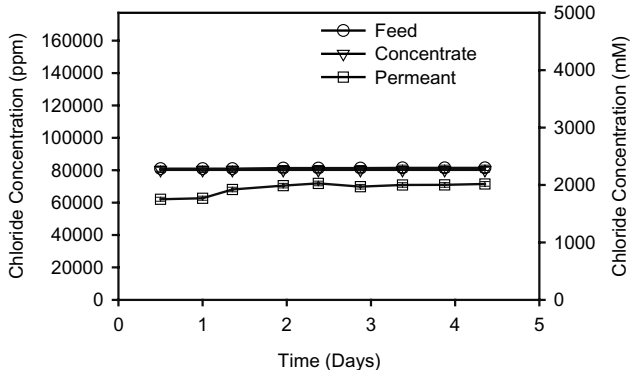

Figure 13. Results for experiment CF-11.

rejection was obtained. In experiments CF-6 through CF-11, the solution concentration was increased after each experimental run and the early permeant concentrations reflect the displacement of the previous solution from the cell and membrane before steady state was reached.

DISCUSSION

The 11 experiments were designed to test chloride rejection as a function of solution feed rate, and test chloride rejection as a function of concentration. Determination of chloride rejection as a function of flow rate included experiments CF-1 through CF-5, and determination of chloride rejection as a function of solute concentration included experiments CF-1, CF-2, and CF-6 through CF-11.

Chloride Rejection as a Function of Flow Rate Parallel to the Membrane

The purpose of these experiments was to determine if a concentration polarization layer (a zone of increased concentration), or CPL, was forming adjacent to the membrane during solute rejection. If concentration polarization occurs, the membrane is exposed to higher solute concentrations and membrane rejection efficiency will decrease.[31] In this series of experiments, feed solution flow rates were varied between 0.1 mL/min and 1.7 mL/min. There was no significant difference in Cl^- rejection for the lower flow rates and the rejection was slightly, though significantly, lower

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for the highest flow rate (Figure 14). The results suggest that when a porous frit is used with a clay membrane, instead of the plastic waffle-shaped grid typically used with synthetic membranes, high cross-flow rates may not be needed to achieve optimum solute rejection. This is thought to occur because the frit material is a porous media and dispersion effects in the pores destroy the CPL at lower flow rates than might otherwise be expected.

Chloride Rejection as a Function of Solute Concentration

These experiments (CF-1, CF-2, and CF-6 through CF-11) had similar flow rates and varied the feed solution concentration between 355 ppm (10 mM) and 81,542 ppm (2300 mM) Cl^-. The average Cl^- rejection is plotted as a function of concentration (Figure 15). Chloride rejection decreases as the chloride concentration increases in the feed water.

Ishurguro et al.[30] reported on two runs using montmorillonite membranes in a cross-flow cell with NaCl solutions. As interpreted from their figure, their NaCl separation results are: 88% and 30% for 35.5 ppm (1.0 mM) NaCl, 83% and 25% for 355 ppm (10 mM) NaCl, and 50% and 3% for 3545 ppm (100 mM) NaCl. We did not use a 35.5 ppm (1.0 mM) or a 355 ppm (10 mM) NaCl solution, however, our runs with approximately 3550 ppm (100 mM) NaCl had Cl^- rejections between 66% and 68%. Our Cl^- solute rejection with approximately 3550 ppm (100 mM) NaCl was consistently higher than the results achieved by Ishiguro et al.[30] The higher solute rejection may be attributable to the higher compaction (16.3 MPa) used in our membrane. Ishiguro et al.[30] did not physically compact the clay in their experiments; it was simply smeared into the cell. Thus,

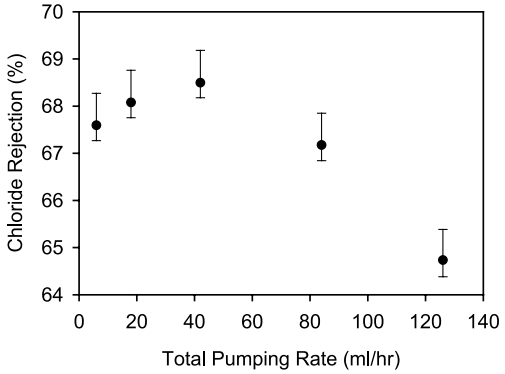

Figure 14. Chloride rejection vs. total pumping rate.

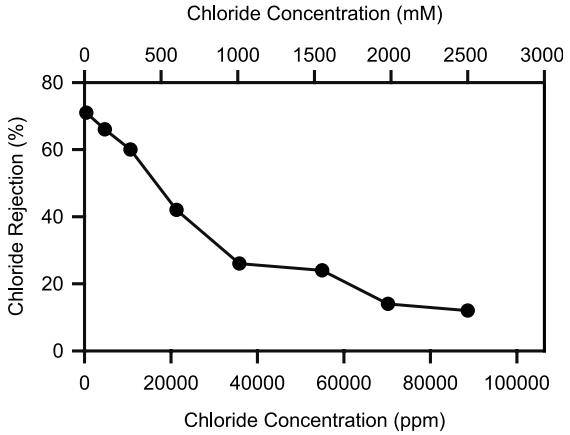

Figure 15. Chloride rejection as a function of chloride concentration.

quantitative comparison of the compaction level used in our experiment with that used in theirs is difficult.

Relation Between Pumping Pressure and Solution Concentration

The theoretical maximum osmotic pressure increases with increasing solute concentration. Therefore, the hydraulic pressure needed to offset osmotic backflow should increase as feed water concentrations increase. This was the case with the bentonite membranes tested. As the feed

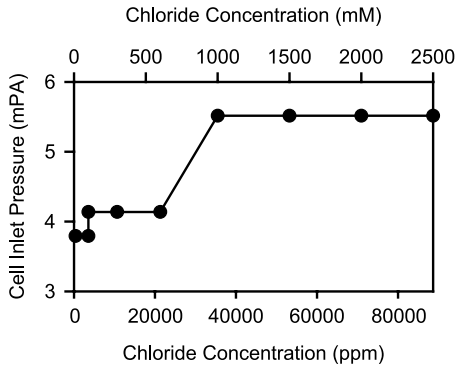

Figure 16. Feed solution pressure as a function of chloride concentration.

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solution concentration increased, the required pumping pressure generally increased as well (Figure 16).

Synthetic membranes designed for seawater desalination require feed pressures of 5.5 MPa (800 psi) to 8.14 MPa (1180 psi).[32] Seawater has a dissolved solute concentration of about 35,000 ppm (1000 mM) NaCl equivalent. The bentonite membranes used in these experiments required a feed pressure of 8.14 MPa (800 psi) for solution concentrations between 35,000 ppm (1000 mM) and 88,630 ppm (2500 mM).

Permeant Flow Rate

The permeant flow through the 0.5-mm thick bentonite membranes ranged between 0.2 to 5% of the total flow delivered by the pump. The clay membranes we used did not exhibit high permeant flow. For example, Mariñas and Selleck[31] ran bench-scale cross-flow tests with two synthetic membranes (FT-30, FilmTec Corp., Minneapolis, MN and TFC, UOP Fluid systems, San Diego, CA) and reported permeant fluxes between 2.8×10^{-4} and 1.07×10^{-3} $cm \cdot s^{-1}$. Our permeant fluxes average three orders of magnitude less (1.14×10^{-6} to 2.44×10^{-7} $cm \cdot s^{-1}$) than those reported by Mariñas and Selleck.[31] Since the relationship between flow rate and pressure is generally considered to be linear, when all other factors such as solute type and concentration, and membrane properties, such as porosity and compaction, remain constant, thinner clay membranes should result in significantly increased permeant flux. As an example, consider experiment CF-2 where the permeant flux (4.27×10^{-7} $cm \cdot s^{-1}$) was 0.5% that of the total flow. If the membrane thickness were decreased to 0.005 mm and 0.0005 mm, the permeant fluxes would then be 4.27×10^{-5} and 4.27×10^{-4} $cm \cdot s^{-1}$, respectively. Thus, very thin clay membranes may exhibit permeant flow rates similar to synthetic membranes.

SUMMARY AND CONCLUSION

The total flow rate was not critical in achieving optimum solute rejection although it appears that the highest flow rates used in our experiments (above 100 mL/hr) may have had a slight deleterious effect on solute rejection efficiency. Due to dispersion in the porous frit used adjacent to the membrane, the observed concentration polarization layer (CPL) seems to be destroyed at low flow rates. This observation suggests that membranes used with porous frits adjacent to the membrane may reach optimum solute rejection at lower flow rates than required when spacer grids are used.

Solute rejection efficiency decreases with increasing solute concentration. For the membranes and experiments reported here, the Cl^- rejection efficiency ranged from 68% for 100-mM (3545 ppm) NaCl solution down to 13% for 2300-mM (81,542 ppm) NaCl solution. For more compacted clay membranes, solute rejection efficiencies should be higher.

The clay membrane we used was 0.5-mm thick. The active layer of most synthetic membranes is only 0.04 μm (0.00004 mm), approximately 12,500 times thinner than the clay membranes used in these experiments. Since Darcy's law states that the flow through a material of constant permeability is inversely proportional to its thickness, then a very thin clay membrane with identical properties would be expected to have much higher permeant flow rates than the ones used in these experiments. The membranes exhibited stable Cl^- rejection for a range of concentrations between 3545 ppm (100 mM) and 81,542 ppm (2300 mM).

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