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Removal of Ammonium and Nitrate Ions from Mine Effluents by Membrane Technology

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ABSTRACT

Ammonium and nitrate ions could be removed from synthetic and actual mine effluents by using nanofiltration (NF) and reverse osmosis (RO) membranes. RO membranes were found more effective in removing these ions (>99% for NH_4^+ and about 97% for NO_3^-) than NF membranes (about 66% for NH_4^+ and about 43% for NO_3^-). Removal of ammonia from synthetic solutions containing only ammonia and water by both NF and RO membranes was found to be very low (10–30%), indicating that ammonia in mining effluents existed not as free ammonia but as ammonium salt complexes. The degree of separation depends on the size of the ammonium salt molecule. Removal of ammonia in the form of ammonium iron sulfate from a synthetic solution by NF membranes reached more than 98% while the removal of ammonium sulfate compound from an actual effluent by the same membranes reached about 55%. Ammonia separation by membranes assures the production of quality water suitable for recycling in mining operations since the other metal ions and anions, such as Ca and SO_4, were also retained in the concentrate. It may be feasible to further process the concentrated product containing appreciable amounts of nitrate and sulfate ions to produce ammonium compounds as fertilizer. During the test, no operating problems were observed such as fouling or scaling. The membrane performance was not affected at the end of the test as proved by NaCl separation before and after the ammonia run. These results justify a laboratory-scale test with real effluents in order to evaluate the effects of various parameters including the effect of concentration on the membrane performance.

Key Words. Removal; Ammonium; Nitrate; Mine effluents; Nanofiltration; Reverse osmosis; Membranes; Quality water; Recycle; Concentrate; Fertilizer; Fouling; Scaling; Laboratory scale

INTRODUCTION

The contamination of mine and mill water with nitrogen, in the form of ammonium and nitrate ions, causes some of the more challenging environmental problems. Among these problems are the promotion of corrosion and undesirable algal growths in the receiving bodies of water. Ammonia can be toxic to fish and other aquatic life, and the nitrates, which are the oxidation product of ammonia, can be a health hazard (1). The ammonia and nitrate levels have therefore become a major concern of various mining groups in Canada. The concentrations of ammonium and nitrate ions in these effluents can vary from 10 to 50 mg/L and from 25 to 300 mg/L, respectively. The ministry of environment (MOE) guidelines for the province of Ontario state that nitrogen in the form of ammonia should not exceed 10 mg/L N as NH_3. The discharge of both ammonium and nitrate ions may eventually be regulated, and it may be necessary to remove both ions from mine water before it can be discharged as effluent.

Ammonium and nitrate ions in mine and mill water are generated from the degradation of cyanide in the case of gold mill effluents, from the use of ammonium nitrate-fuel oil (ANFO) blasting agents in mines, and from the use of various nitrogen-containing reagents in the ore processing and extractive metallurgy.

Natural chemical and/or biological degradation of undesirable substances represents the most widely applied method to remove ammonia from mining effluents (2). It involves the transpiration of dissolved ammonia gas from wastewaters to air by retaining the wastewaters in holding ponds. Since a high pH is required to convert ammonium ions to ammonia gas, pH adjustment is also needed. In addition, the pond area should be increased to enhance the evolution of ammonia by maximizing the air/water interfacial area.

Other potential methods, which are in use for removal of ammonium and nitrate ions from aqueous solutions, are reported in the literature (3). However, none of these processes has been applied specifically for the treatment of mine and mill water due to the fact that they are relatively expensive when used on dilute solutions and the anticipated regulatory pressure has not as yet been brought to bear. These processes include ammonia stripping (4, 5), selective ion exchange (clinoptilolite clay) (6, 7), biological nitrification/denitrification (8), breakpoint chlorination (5, 9, 10), chemical and electrochemical oxidation (11), and desalination (12). However, each of these processes suffers from one or more limitations that render them noncompetitive to the natural degradation method where sufficient area is available.

Membrane separation has a potential for the treatment of mining and metallurgical effluents (13). Before the large-scale introduction of thin-film composite membranes, RO cellulose acetate membranes had been investigated for the removal of ammonium and nitrate ions from mining effluents (14). However, the reported separation for these ions did not exceed 30% at an operating pressure of 2.1 MPa (14). The results of investigations on the separation of ammonium and nitrate ions from dilute solutions by hollow fine fiber B-9, polyamide RO membrane, was reported (15). About 80% of the ammonia and 85% of the nitrate ions were rejected from ammonium nitrate and sodium nitrate solutions, respectively, at neutral pH and under standard operating conditions by B-9 Permasep permeator (15).

The inherent advantages of membranes over other processes include the ability of membranes to remove both cationic and anionic species to yield a good quality water permeate which can be recycled in mining operations. Membrane separations require less energy than distillation or freezing-based processing since no phase change is involved. The process is simple and the equipment is compact and modular in nature and capable of continuous operation. However, the metal constituents and other toxic components concentrated in the retentate stream must be further processed or disposed of before discharge.

The purpose of this study is to investigate the feasibility of complete removal of ammonium and nitrate ions from synthetic and actual mine effluents by commercial nanofiltration and reverse osmosis membranes in order to obtain good quality water. The optional treatment of the concentrate and some economic considerations are also included.

MATERIALS AND METHODS

A synthetic solution, feed A, equivalent to the average composition of a number of selected tailing effluents, was prepared for this study and had the composition, in mg/L: 288 Ca, 599 Fe, 1840 SO_4, 63 Zn, and 25 NH_3. Another synthetic solution, feed B, was prepared containing only ammonia and distilled water (31 mg/L NH_3). The following salts of analytical grade were used to prepare the synthetic feed solutions: $FeSO_4(NH_4)_2SO_4 \cdot 6H_2O$, $FeSO_4 \cdot 7H_2O$, $ZnSO_4 \cdot 7H_2O$, $CaSO_4 \cdot 2H_2O$, and 30% NH_4OH solution. An actual mining effluent was obtained from a Canadian mining operation of composition, in mg/L: 266 Ca, 707.1 SO_4, 214 Na, 207 NO_3, and 51.1 NH_3.

Commercial RO and nanofiltration membranes used in this study were obtained from different manufacturers. The characteristics of these membranes as well as their suppliers are listed in Table 1.

The tests were performed in a reverse osmosis system with six cells arranged in series as shown in Fig. 1. The test cells have an active area of 10.5 cm^2. The pressure was maintained at 2.8 MPa with a flow rate of 0.43 L/min. The temperature was not controlled and varied from 24 to 26°C. The system was always operated in a recirculation mode except for sampling periods.

In all tests the initial membrane performance, in terms of percent separation and product rate, was characterized using a 5000 mg/L NaCl test solute. Separation was determined based on the conductivity of feed and permeate samples, and calculated as follows:

$$\%\ \text{Separation} = \frac{\text{conductivity of feed} - \text{conductivity of permeate}}{\text{conductivity of feed}} \times 100$$

This characterization was performed to provide a baseline measure of membrane performance. These characterization tests were repeated after the tests with the various solutions were completed to determine whether any membrane fouling occurred due to the exposure of the membrane coupons to the test solutions.

After the initial characterization, about 4 L of the test solution was introduced into the system. Feed and permeate samples were taken for analysis after the test solution had circulated for 1 hour and again after another 3 hours (total 4 hours).

TABLE 1
Technical Data for Reverse Osmosis and Nanofiltration Membranes Used in This Study

Membrane identification	Supplier manufacturer	Membrane type and material	Separation, % 5000 mg/L NaCl	Operating pH range
DS-3B	Desalination System Inc., Escondido, California	RO, thin-film composite	97–99	1–12
FT	Filmtech Corp., Minneapolis, Minnesota	RO, thin-film composite	98.5	4–11
SU	Toray Ind., Japan	RO, crosslinked thin-film composite	99	3–9
DU	Du Pont	RO, amide	95	4–11
DS-5	Desalination System Inc., Escondido, California	NF thin-film composite	50–70	2–11
HPVD	Hydranautics, San Diego, California	NF polyvinyl alcohol derivative	92	2–8

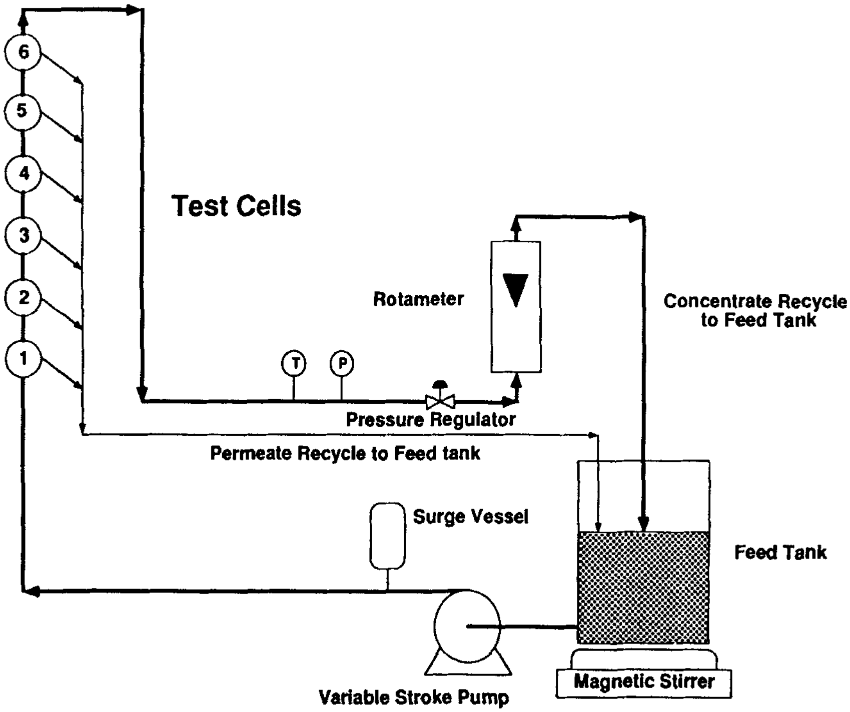

FIG. 1 Schematic diagram of six cell RO/NF test system.

Feed and permeate samples were analyzed for metal ions content using inductively coupled plasma-atomic emission spectroscopy (ICP-AES) while ammonia analysis was performed using both an ion selective electrode (ISE) and spectrophotometric method (Nesslerization method) (16). Nitrate ion analysis was done using high performance liquid chromatography (HPLC).

RESULTS AND DISCUSSION

The form of ammonia in mining effluents depends on the pH of the waste solution. In acidic and neutral pH solutions, ammonia probably exists in the form of ammonium complexes and not as free ammonia. Separation of ammonium complexes by membranes is more efficient than the separation of uncomplexed ammonium hydroxide. Therefore, two feeds of synthetic solutions were prepared, feed A containing ammonia in a complex form and feed B containing only ammonium hydroxide solution as previously described in the Materials and Methods Section.

Removal of Ammonia from Synthetic Feed A (NH_3/SO_4/Fe/Zn/Ca)

The results of the separation of ammonia from feed A by NF and RO after 1 and 4 hours are summarized in Table 2. Both types of membranes (NF and RO) gave good separation for ammonia and other metal ions since ammonia existed in a complexed form (as ammonium iron sulfate complex). The DS5 NF membrane gave better separation for ammonia and other metal ions than did the HPVD NF membrane. Among the four RO membrane coupons, Filmtech Corp. (FT) gave the highest separation for ammonia (97.4%) while Toray Ind. (SU) gave the lowest (78.4%). However, the level of ammonia in all permeates from the tested coupons was found to be under the discharge limits (less than 10 mg/L NH_3). The results after 4 hours of separation, as indicated by the same table, showed slight improvement for separation of ammonia and other metal ions compared to the results at 1 hour except for the nanofiltration membrane, HPVD. Although the permeate recirculated to the feed solution and no concentration tests were done, the analysis of the feed after 4 hours run showed a concentration factor of about 25% in the case of ammonia and vary between 10 and 20% in the case of the other constituents. This concentration factor was due to the removal of permeate aliquots for analysis.

In terms of product rate, nanofiltration membrane DS5 exhibited the highest value among the tested membranes while the reverse osmosis membrane FT exhibited the lowest product rate (Table 3). The product

TABLE 2
Removal of Ammonia and Other Ions from Feed A, Analysis of Feed and Permeate Samples (pressure = 2.8 MPa, flow rate = 0.43 L/min, room temperature)

		Ca		Fe		NH_3		SO_4		Zn	
		mg/L	Separation (%)	mg/L	Separation (%)	mg/L	Separation (%)	mg/L	Separation (%)	mg/L	Separation (%)
Feed:	1 h	288		599		25		1840		63	
	4 h	318		672		30		2110		69	
DS-3B:	1 h	2.0	99.3	3.0	99.5	0.98	96.1	17.8	99.0	0.5	99.2
	4 h	2.0	99.4	3.0	99.6	0.86	97.1	14.8	99.3	0.4	99.4
FT:	1 h	2.0	99.3	3.0	99.5	0.65	97.4	13.9	99.2	0.4	99.4
	4 h	2.0	99.4	3.0	99.6	0.68	97.7	12.5	99.4	0.3	99.6
SU:	1 h	14.0	95.1	36.0	94.0	5.4	78.4	114.7	93.8	4.0	93.7
	4 h	21.0	93.4	48.0	92.9	5.3	82.4	161.6	92.3	5.0	92.8
DU:	1 h	0.6	99.8	5.0	99.2	1.3	94.7	17.3	99.1	0.5	99.2
	4 h	0.5	99.8	1.0	99.9	1.3	95.5	10.4	99.5	0.2	99.7
DS-5:	1 h	5.0	98.3	14.0	97.7	1.8	93.0	49.4	97.3	2.0	96.8
	4 h	5.0	98.4	12.0	98.2	1.6	94.7	39.2	98.1	1.0	98.6
HPVD:	1 h	16.0	94.4	35.0	94.2	4.2	83.2	117.3	93.6	4.0	93.7
	4 h	36.0	88.7	74.0	89.0	7.2	75.9	276.2	86.9	8.0	88.4

TABLE 3
The Flow Rate and pHs of Permeates Feed A (pH 4.8 for initial feed and 4.4 for final feed)

Coupon		DS3-B	FT	SU	DU	DS5	HPVD
Rate ($m^3/m^2/day$):	1 h	1.18	0.68	1.57	1.03	2.65	2.15
	4 h	1.13	0.68	1.56	1.03	2.04	1.09
pH:	1 h	6.0	6.2	7.1	6.3	7.0	5.5
	4 h	5.6	4.8	5.8	5.9	5.3	5.1

rate after 4 hours did not differ from that at 1 hour for the RO membranes, while for the NF membranes, DS5 decreased slightly and HPVD was significantly lower (Table 3).

The pHs of both feed and permeate were measured and are listed in Table 3. A slight increase in permeate pH was observed for both tests compared with the initial feed pH. This was due to the removal of most of the metal ions and cations in the concentrate, indicating that the quality of the permeate improved as the pH approached neutrality.

In this test, nitrate removal was not studied as this particular feed solution had no nitrate constituent.

Removal of Ammonia from Synthetic Feed B (NH_3/H_2O)

When the same procedure was repeated for the feed solution containing only dissolved NH_3 in water (NH_4OH solution), it was found that the percent separation for ammonia was in the range of 10 to 30% (Table 4).

TABLE 4
Removal of Ammonia from Feed B by NF and RO Membranes. Analysis of Feed and Permeate Samples

	NH_3 (mg/L)		Separation (%)		Rate ($m^3/m^2/day$)		pH	
	1 h	4 h	1 h	4 h	1 h	4 h	1 h	4 h
Feed	30.7	31.0					9.7	10.5
DS-3B	24.8	23.0	20.0	25.8	1.21	1.27	9.8	10.3
FT	23.2	22.8	25.0	26.5	0.96	1.01	10.0	10.4
SU	24.9	23.2	19.7	25.0	1.39	1.47	9.9	10.4
DU	23.4	20.8	24.5	32.9	1.14	1.16	10.0	10.3
DS-5	27.6	23.2	11.0	25.0	2.70	2.81	10.2	10.5
HPVD	32.0	25.4	0.0	18.1	4.35	4.32	10.4	10.5

This indicated that most of the NH_4OH solution passed through the NF and RO membranes since the size of NH_4OH was small (about double that of water). The flow rates after 1 and 4 hours were found to be equal. The pHs measured in the permeates were found to be in the alkaline region (Table 4), confirming the permeation of ammonium hydroxide through the tested membranes.

Removal of Ammonium and Nitrate Ions from Feed C (Real Effluent)

The analysis of the real effluent showed that it contained appreciable amounts of NH_3 and NO_3^- (51.1 and 207 mg/L, respectively). The pH of this feed was nearly neutral (pH 6.4), and therefore heavy metal ions content in the test solution was negligible. The feed was then subjected to membrane separation without any pretreatment.

The results after 4 hours are shown in Table 5. Good separation was obtained for both NH_4 and NO_3 ions by RO membranes while NF membranes were less effective. The permeate obtained from the SU coupon attained <0.2 mg/L for ammonia and about 7 mg/L for nitrate, representing >99.0% separation of NH_3 and about 97% separation of NO_3^-.

The reason for the low separation of ammonia by NF membranes (55% separation for DS5 coupon) was possibly due to ammonia in the real effluent being present not as ammonium iron sulfate (Feed A) but as the relatively smaller ammonium sulfate complex since no iron was present in the mining effluent.

For all RO membranes tested, more than 97% separation was achieved for both Ca and SO_4 ions (Table 5), assuring the production of a good quality permeate water.

TABLE 5
Removal of NH_3, NO_3, Ca, and SO_4 Ions from Feed C (actual mining effluent). Analysis of Feed and Permeate Samples after 4 Hours

	Ca		SO_4		NH_3		NO_3	
	mg/L	Separation (%)	mg/L	Separation (%)	mg/L	Separation (%)	mg/L	Separation (%)
Feed	265	0.0	705	0.0	51.1	0.0	207	0.0
DS-3B	2.2	99.2	<9	>99	2.5	95.1	18.5	91.1
FT	4.7	98.2	11.1	98.4	3.1	93.9	17.0	91.8
SU	<1	>99	<9	>99	>0.2	>99	7.0	96.6
DU	<1	>99	<9	>99	1.8	96.5	23.1	88.8
DS-5	36	86.4	9.0	98.7	23.1	54.8	124	40.0
HPVD	32	88.0	35.1	95.0	17.5	65.8	119	42.5

TABLE 6
The Flow Rate and pHs of Permeates Feed C (pH 6.4 for initial feed)

Coupon		DS-3B	FT	SU	DU	DS5	HPVD
Rate (m^3/m^2/day):	1 h	1.24	1.19	1.22	0.83	2.73	3.00
	4 h	1.24	1.19	1.27	0.96	2.65	2.71
pH:	1 h	—	—	—	—	—	—
	4 h	7.0	7.1	7.4	7.0	6.7	6.7

The flow rate and pHs are listed in Table 6. The flow rate after 1 and 4 hours was about the same, indicating no fouling or scaling problems. The pH of the permeate solutions after the 4-hour test were slightly increased compared with the pH of the feed solution (pH was about 7). Figure 2 summarizes the results on the effect of exposure time to mining effluent on the flux rates of the tested membranes.

The percent separation of 5000 mg/L NaCl and the flux rates before and after running ammonia test solutions compared with the flux rates of pure water permeability (PWP) are summarized in Fig. 3 for all three tests. As the figure shows, the flux rates for all the RO membranes tested were found to be nearly the same before and after each test, except for the FT membrane where the flux rate increased after the first test, then stabilized

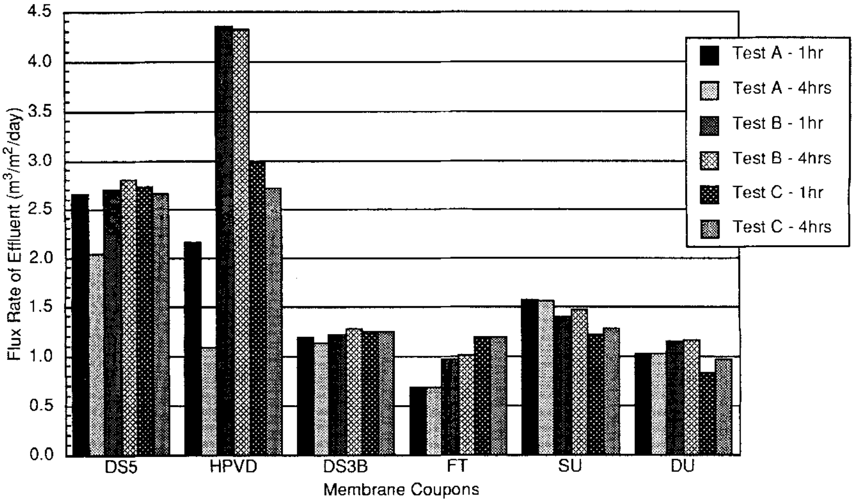

FIG. 2 Effect of exposure time to mining effluent on the flux rate of various membranes tested at 400 psig.

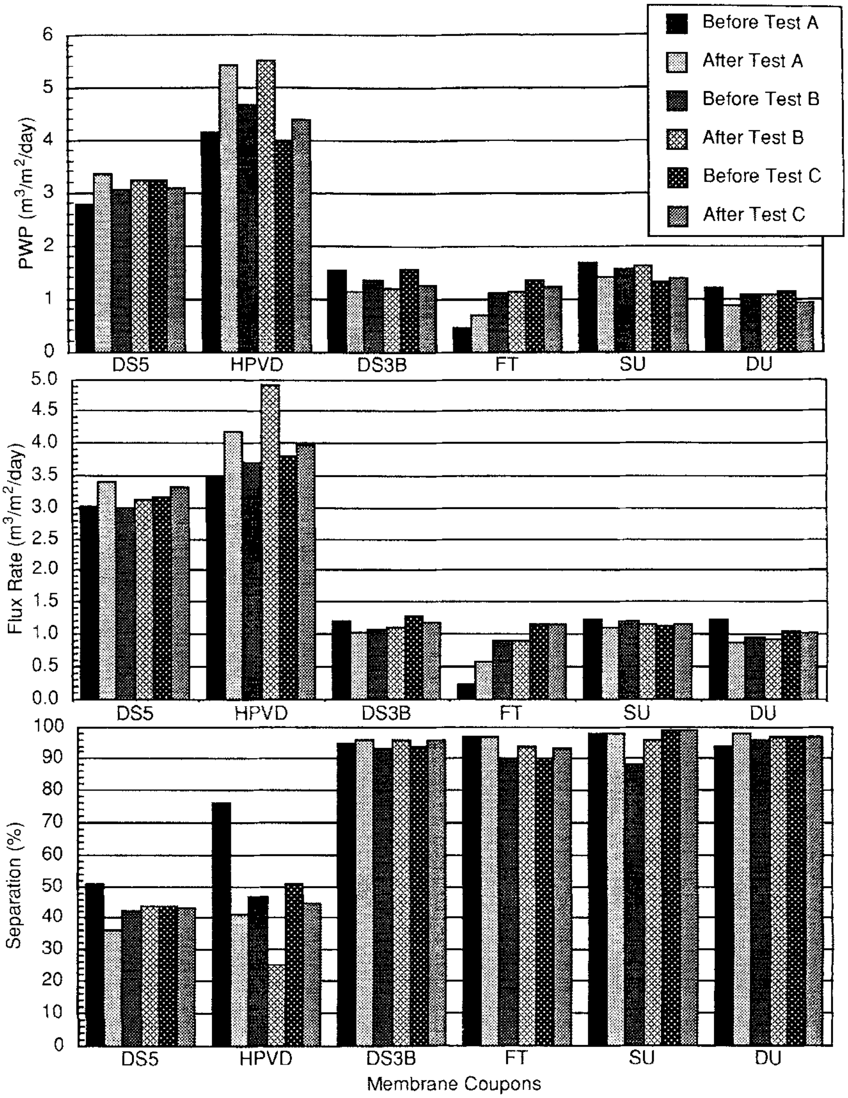

FIG. 3 Effect of mining effluent on the performance of various membranes tested with 5000 mg/L NaCl and at 400 psig.

after the second and the third test. Separation percents, however, were found to be the same before and after each test for all RO and NF membranes except for the HPVD membrane which showed a decrease in percent separation after each test. These characterization tests, however, indicated the good performance of membranes during the ammonia run over a period of 4 hours. No signs of coupon deterioration were observed due to the exposure of these coupons to the mining effluent (Fig. 2).

The Optional Treatment of the Concentrate

In membrane separation, one stream is introduced to the membrane module and split into two streams, the permeate and the concentrate. The larger volume of permeate is always a good quality water which can be recycled to the operation or discharged with minimal additional treatment. The smaller volume of concentrate contains most of the feed solutes. In this application, the concentrate contains appreciable amounts of NH_4, NO_3, SO_4, and Ca ions. All of these ions are considered nutrient ingredients for soil. Therefore, it can be expected that when phosphate ion is added to the concentrate, a fertilizer can be obtained containing ammonium phosphate, ammonium nitrate, calcium ammonium phosphate, and/or ammonium sulfate (17). The phosphate product might add a by-product revenue to the process to offset the cost of the membrane system.

Process Economics

An estimate of the treatment cost (±30%) associated with the proposed membrane process can be developed. If we assume that membranes used in the industry typically cost about \$100 US/m^2, and membrane cost usually represents about 20 to 30% of the system cost for aqueous separation system. It can be expected that treatment of neutral pH effluent containing negligible amounts of iron and other heavy metals will not require special equipment. The capital cost of the system will therefore be approximately \$500 US/m^2. At operating conditions of 2.8 MPa and 25°C, the permeate flux is expected to be about 1165 kg/m^2/day, leading to an estimated capital cost of \$0.36 US/kg/day or \$360 US/m^3/day. Four major factors will contribute to production costs: energy, labor, membrane replacement, and capital cost recovery. Based on typical values for operating costs from the literature (18), energy, labor, and membrane replacement costs will be \$0.11, \$0.08, and \$0.10 US/m^3, respectively. The membrane life in this application is assumed to be 3 years. Assuming an 85% on-stream factor, amortizing the estimated capital cost for periods of 5 and 10 years at an interest rate of 10% leads to capital recovery costs of \$0.57 and \$0.46 US/m^3, respectively. Based on these calculations, the total estimated cost of treatment will be in the range of $1.80 to $2.20 US/1000 US gallons. These production cost estimates are less than the typical values cited (18) for brackish water treatment ($2.30 US/1000 US gallons). Any fertilizer by-product recovery will add a dollar value to the process, leading to further decreases in the production costs.

CONCLUSIONS AND RECOMMENDATIONS

All the commercial RO membranes tested in this study demonstrated an acceptable performance for the removal of ammonium and nitrate ions from mining effluents. Separation of ammonium and nitrate ions was >99% and about 97%, respectively. Ammonium and nitrate levels in the permeates were therefore within the acceptable limits specified by current regulations. However, NF membranes exhibited a lower performance than RO membranes in terms of ammonium and nitrate separation from effluents of near neutral pH, and therefore could not be considered for further tests in such solutions.

Uncomplexed ammonium hydroxide was not effectively separated by RO and NF membranes while larger ammonium complexes could be separated by the same membranes. However, NF membranes were found to be effective for the removal of larger ammonium complexes such as might be formed in effluent solutions at lower pH, but they were not effective for the type of complexes formed at near neutral pH.

The permeate could be recycled to the mining operation since it constituted a good quality water while the concentrate contained most of the ions originally present in the feed solution.

No major operational problems have been identified in this test. Therefore, an extended laboratory-scale test should be done in order to investigate the effect of various operating parameters, including the concentration effect, on the performance of membranes over a longer period of time. A suitable volume of concentrate may be collected and tested to see whether it can be further processed to produce a fertilizer product.

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