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Reverse Osmosis Separation of Sulfate, Nitrate, and Ammonia from Mining Effluents

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Abstract

Cellulose acetate membranes (osmonics) were characterized in terms of pure water permeability constant (A), solute transport parameter ($D_{AM}/K\delta$), and mass transfer coefficient (k) with sodium chloride solution as the reference system. Reverse osmosis behavior of sulfate, nitrate, ammonia, and some metal ions was studied as a function of pressure in the range of 100 to 300 psig. Quirke tailings containing nitrate, sulfate, ammonia, and metal ions such as Ca, Mg, Fe, Cu, and Zn were subjected to reverse osmosis at 300 psig, and product water of suitable quality for use in recycle operations was obtained.

INTRODUCTION

Nitrogen in the form of ammonia and ammonium ions is undesirable because it promotes corrosion and stimulates growth of algae, unwanted vegetation, and offensive slimes (*1*). Nitrogen in the form of nitrates is reported to cause irritation of mucous membranes of the stomach and the bladder. Sulfate in high concentrations is alleged to produce objectionable taste and gastrointestinal irritation and catharsis of the alimentary canal (*1*). Hence it is necessary to reduce the concentration of nitrate and sulfate from contaminated effluents to acceptable levels.

Reverse osmosis is a good method for the removal of dissolved inorganic and organic species from aqueous solutions, and it is characterized by low expenditure of energy and simplicity of processing technology. Because high-pressure reverse osmosis requires high capital investment and gives rise to membrane compaction problems, low-pressure reverse osmosis has been the subject of a number of studies (*2–11*) on the treatment of pollutant-bearing waste effluents in our laboratories. These studies have proved promising in the sense that both valuable metal ions and product water of reasonable quality could be simultaneously recovered for reuse. Hence a systematic study has been undertaken to determine the reverse osmosis separation characteristics of both the individual soluble species present in the mill effluent and the composite solutions containing the various pollutants. The results from such studies are reported in this paper together with the levels of some metal ions present in the product water after reverse osmosis treatment.

EXPERIMENTAL

Reverse Osmosis Cell

The conventional stainless steel static cell consisted of a detachable upper part and a lower part. The membrane was placed on a stainless steel porous plate embedded in the lower part of the cell. The lower part of the cell was provided with an outlet through which the membrane-permeated (product) solution was collected at atmospheric pressure. The upper part of the cell contained the feed solution of interest in contact with the membrane under pressure. The two parts of the cell were fitted and sealed with the aid of two concentric rubber O-rings. The effective surface area of the membrane in the cell was 13.4 cm^2. The required pressure was achieved by compressed nitrogen gas from a gas cylinder. A magnetic stirrer fitted at the top part of the cell, located about 0.64 cm above the membrane, was used to ensure uniform stirring of the feed solution, thereby minimizing concentration polarization effects. The schematic diagram of the whole experimental arrangement is the same as that reported earlier (*5*). Cellulose acetate membranes obtained from Osmonics Inc. were used in these studies, and characterized in the usual manner (*5*).

Procedure

All experiments were conducted at laboratory temperature (23–25°C). The membrane was pressurized with pure water at 400 psig for about 2 h before subsequent use in the experiments at operating pressures of 100 to 300 psig. The pure water permeation rate (PWP), the membrane-permeated product rate (PR) for a given area of the membrane, and the solute separation (f) were determined under present operating conditions. A feed solution containing 1425 mg/L of sodium chloride was used to obtain data on membrane specification and to specify the mass transfer coefficient (k) on the high-pressure side of the membrane. The sodium chloride content of the feed and product solutions was analyzed by conductance measurements. The feed and product solutions containing metal ions Ca, Zn, Fe, Cu, and Mg were analyzed by atomic absorption spectrophotometry at wavelengths of 4227, 2139, 2483, 3248, and 2852 Å, respectively. Sulfate was determined gravimetrically as $BaSO_4$. Ammonia was determined spectrophotometrically using the phenol–hypochlorite method described by Weatherburn (*12*). Nitrate ion was determined by the phenol–disulfonic acid method of Taras (*13*). Product rates were corrected with respect to viscosity and temperature (25°C). Because low concentrations were involved, solute separation (f) was calculated using the relationship

$$f = \frac{\text{solute concentration in feed} - \text{solute concentration in product}}{\text{solute concentration in feed}}$$

RESULTS AND DISCUSSION

Membrane and Specifications

The reverse osmosis membranes can be specified in terms of parameters such as the pure water permeability constant (A) and the solute transport parameter ($D_{AM}/K\delta$) at a particular operating pressure. The solute separation and product rate obtained with a membrane so specified are functions of the mass transfer coefficient (k) on the high-pressure side of the membrane, which is governed by the feed flow rate and the feed concentration used in the experiment. A computer program (*14*) using experimental data on pure water permeation rate, membrane-permeated product rate, and

TABLE 1

Membrane Specification Data[a]

			Feed, 1425 mg/L NaCl, $k = 14.29 \times 10^{-4}$ cm/s	
Film no.	$A \times 10^6$ g mole H_2O (cm^{-2} s^{-1} atm^{-1})	$D_{AM}/K\delta$ (10^5 cm/s)	Solute separation (%)	Product rate (g/h)
1	1.63	5.33	87.40	19.26
1	1.60	5.69	87.00	18.85

[a] Membrane area 13.4 cm^2; pressure 300 psig.

solute separation at present operating conditions for the aqueous sodium chloride feed solution containing 1425 ppm salt was used in the calculation of A, D_{AM}/K_δ, and k. The data presented in Table 1 on the values of A and D_{AM}/K_δ for sodium chloride at 300 psig specify the characteristics of the membrane used in the present studies. The feed flow conditions used in all the experiments done in the present investigation are specified in terms of the corresponding values of k obtained with the reference system of sodium chloride solution containing 1425 ppm of salt. The data presented (Table 1) show the high productivity of the membrane used.

Separation of Salts

The reverse osmosis separation of specific salts such as sodium sulfate, magnesium perchlorate, magnesium chloride, magnesium sulfate, calcium chloride, calcium nitrate, and ammonium nitrate was studied as a function of pressure in the range of 100 to 300 psig. In every case studied the percent separation reaches a maximum at nearly 300 psig (Table 2). Therefore, all further experiments were carried out at a pressure of 300 psig. For all the salts studied, the product rates were found to increase in a linear fashion in going from a pressure of 100 to 300 psig (Table 2). All solutes studied gave a fairly high separation with the exception of ammonium nitrate.

TABLE 2

Separation of Salts

Salt	Concentration (ppm)	Pressure (psig)	PWP (g/h)	Product rate (g/h)	Percent separation
Na_2SO_4	1034	300	20.9	19.0	93.9
	1034	200	13.7	12.3	94.1
	1034	100	6.6	5.4	93.6
$Mg(ClO_4)_2$	830	300	20.3	16.2	94.7
	830	200	13.5	10.2	93.5
	830	100	6.4	3.7	89.8
$MgCl_2$	1010	300	22.5	17.8	87.1
	1010	200	15.1	11.0	86.1
	1010	100	7.1	4.2	78.2
$MgSO_4$	1020	300	22.5	18.6	88.2
	1020	200	14.8	11.8	87.3
	1020	100	7.2	5.1	86.3
$CaCl_2$	820	300	22.1	19.0	89.5
	820	200	14.5	11.8	87.8
	820	100	6.8	5.1	82.9
$Ca(NO_3)_2$	910	300	20.6	17.2	94.7
	910	200	14.0	10.8	94.5
	910	100	6.4	4.5	87.9
NH_4NO_3	3470	300	22.0	19.3	55.0
	3470	200	14.7	12.4	42.9
	3470	100	7.2	6.5	30.3

TABLE 3

Analysis of Quirke Tailings after R.O. Treatment[a]

Constituent	Concentration (ppm)	
	Feed water	Product water
Ca	100.0	5.0
Zn	2.0	0.09
Fe	10.2	0.10
Cu	2.0	0.09
Mg	18.0	1.00
NO_3	110.0	47.0
SO_4	1170	88.5
NH_3	22.6	9.1

[a] Pressure 300 psig; membrane area 13.4 cm^2.

A sample of Quirke tailings effluent was subjected to reverse osmosis treatment at 300 psig, and the product water was analyzed for the metal ions Ca, Zn, Fe, Cu, Mg, and other pollutants such as nitrate, sulfate, and ammonia. The analytical data of the product water from reverse osmosis treatment are given in Table 3. The metal ion concentrations are within acceptable levels as far as environmental limits are concerned.

A comparison of the average concentrations of sulfate and nitrate (1400 ppm SO_4 and 120 ppm NO_3) in the tailing (*15*), and the levels found in the product water (88.5 ppm SO_4 and 47 ppm NO_3) indicate that there is a respective reduction of about 16 and 2.6 times of these species. Further, the sulfate concentration in the product water is also below the average level (142 ppm) (*16*) found in the Quirke Lake whence the water was drawn for mining operations. However, the nitrate concentration in the permeate is definitely higher than the average level (7.4 ppm) (*16*) observed in the lake. The product water was subjected to a second stage reverse osmosis treatment. The product water from the second stage reverse osmosis treatment analyzed 19 ppm sulfate, 20 ppm of nitrate, and 4 ppm of ammonia. This sample of product water is satisfactory for recycling purposes.

It can be concluded from these studies that the reverse osmosis treatment of mill effluents of Quirke-type composition can yield product water of suitable quality which can be used in water recycle operations.

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