



# Batch ion-exchange membrane electrodialysis of mother liquid discharged from a salt-manufacturing process. Experiment and simulation



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## ABSTRACT

Mother liquid discharged from a salt manufacturing plant was electrodialed in the batch process incorporated with  $\text{SO}_4^{2-}$  ion low-permeable anion-exchange membranes for decreasing  $\text{Na}_2\text{SO}_4$  concentration and recovering NaCl. Energy consumption of the electrodialysis was compared with that of the cooling method to precipitate  $\text{Na}_2\text{SO}_4$  as  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ . Energy consumption of cooling method depends largely on cooling energy for decreasing temperature. If the temperature of the discharged mother liquid cannot be cooled under 31 °C in the atmosphere, the cooling method needs much cooling energy, so the electrodialysis is advantageous. If the temperature can be decreased to lower than 31 °C in the atmosphere, cooling method is possible.

Succeeding to the above investigation, mother liquid was supplied to the batch electrodialysis process in the computer simulation program. The performance changes with time were computed with an instantaneous open/shut feed operation system of a salt solution. Computation was carried out to discuss the performance of the electrodialysis experiment of the mother liquid. The computed data are generally the same as the experimental data for ion flux across a membrane pair  $J_S$ , current efficiency  $\eta$ , solution volume circulated in the desalting side  $Q^D$ , energy consumption for desalination  $E_{des}$  and for concentration  $E_{conc}$ . However, the computed solution flux across a membrane pair  $J_V$  and cell voltage  $V_{cell}$  are larger than observed ones. The computed salt concentration at the inlets of desalting cells  $C_{in}$  and the salt concentration at the outlets of concentrating cells  $C'$  are less than observed ones.

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## 1. Introduction

Ion-exchange membranes were developed at first to demineralize saline water to produce drinking water and are now applied to saline water desalination widely for industrial use. Electrodialysis processes are classified to a single-pass, a feed-and-bleed and a batch process. Among these processes, the batch process is applicable to a small- or middle-scale electrodialysis operation.

We can see the batch operation for examples; Rapp and Pfromm [1] removed chloride from the chemical recovery cycle of a Kraft pulp mill. Resbeut et al. [2] applied the batch process to waste water treatment and demineralized phenylalanine solutions. Moresi and Sappino [3] recovered fermentation products from model solutions. Elmidaoui et al. [4] demineralized beet sugar solution

to reduce melassigenic ions. Nataraj et al. [5] applied batch pilot plant for chromium removal.

The performance of the batch process has been discussed from the following viewpoints: Parulekar [6] investigated energy consumption in a constant current and constant voltage batch operation. Demircioglu et al. [7] introduced equations expressing the ionic mass balance around a dilute circulation tank and discussed energy consumption. Moon et al. [8] predicted the performance of continuous and batch electrodialysis process based on the fundamental principles of electrochemistry, transport phenomena and thermodynamics. Oritz et al. [9] developed a mathematical model for a batch process and discussed mass balance, ohmic drop and membrane potential.

Ion-exchange membranes are also applied to concentrate seawater to produce edible salt [10]. However, edible salt is produced in another process being supplied with solar salt as raw materials (Fig. 1). In this process, raw salt (solar salt) is dissolved in water to produce brine at first. Next,  $\text{Na}_2\text{CO}_3$  and NaOH are added into the

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**Nomenclature**

$a$	flow-pass thickness in a cell (cm)	$\Delta H$	enthalpy change (kJ g NaCl <sup>-1</sup> )
$b$	flow-pass width in a cell (cm)	$\varepsilon$	volume ratio of the spacer in a cell
$C$	salt concentration (equiv. cm <sup>-3</sup> )	$\eta$	current efficiency
$E_{conc}$	energy consumption for concentration (kW h equiv <sup>-1</sup> )	$\theta$	crossing angle of spacer rods (radian)
$E_{des}$	energy consumption for desalination (kW h m <sup>-3</sup> )	$\kappa$	specific electric resistance (S cm <sup>-1</sup> )
$E_{NaCl}$	energy consumption to obtain one ton of NaCl (kW h t NaCl <sup>-1</sup> )	$\lambda$	overall transport number of a membrane pair (equiv C <sup>-1</sup> )
$F$	Faraday constant (C equiv <sup>-1</sup> )	$\mu$	overall solute permeability of a membrane pair (cm s <sup>-1</sup> )
$i$	current density (A cm <sup>-2</sup> )	$\rho$	overall volume osmotic permeability of a membrane pair (cm <sup>4</sup> equiv <sup>-1</sup> s <sup>-1</sup> )
$J_S$	ion flux across a membrane pair (equiv cm <sup>-2</sup> s <sup>-1</sup> )	$\phi$	overall electro-osmotic permeability of a membrane pair (cm <sup>3</sup> A <sup>-1</sup> s <sup>-1</sup> )
$J_V$	volume flux across a membrane pair (cm <sup>3</sup> cm <sup>-2</sup> s <sup>-1</sup> )	$\chi$	distance between spacer rods (cm)
$l$	flow-pass length in a cell (cm)		
$N$	number of cells integrated in an electro dialyzer		
$M$	salt quantity (eq)		
$q$	volume velocity of a solution (cm <sup>3</sup> s <sup>-1</sup> )		
$Q$	solution volume (cm <sup>3</sup> , cm <sup>3</sup> /10 min)	<b>Subscripts</b>	
$r$	electric resistance ( $\Omega$ cm <sup>2</sup> )	$K$	cation-exchange membrane
$r_{alter}$	alternating electric resistance of a membrane ( $\Omega$ cm <sup>2</sup> )	$A$	anion-exchange membrane
$r_{dire}$	direct electric resistance of a membrane ( $\Omega$ cm <sup>2</sup> )	$in$	inlet
$R$	gas constant (J K <sup>-1</sup> mol <sup>-1</sup> )	$n$	operating time number
$S$	membrane area (cm <sup>2</sup> )	$out$	outlet
$t$	transport number of ions in an ion-exchange membrane; operation time (min)		
$T$	temperature ( $^{\circ}$ C, K)	<b>Super scripts</b>	
$T_{Cl}^{SO_4}$	permselectivity coefficient of SO <sub>4</sub> <sup>2-</sup> ions against Cl <sup>-</sup> ions	$'$	desalting cell
$u$	linear velocity of a solution (cm s <sup>-1</sup> )	$''$	concentrating cell
$V_{cell}$	cell voltage (V pair <sup>-1</sup> )	$0$	feeding solution circulation tank
		$\#$	concentrated solution tank
		$*$	control key
<b>Greek letters</b>			
$\gamma$	activity coefficient of salt (NaCl) in a solution		

brine to remove Ca<sup>2+</sup> and Mg<sup>2+</sup> ions. Finally, the brine is evaporated in a multiple-effect evaporation process and NaCl is crystallized in evaporators.

In this process, SO<sub>4</sub><sup>2-</sup> ions are accumulated simultaneously and its concentration is increased. If Na<sub>2</sub>SO<sub>4</sub> concentration in the concentrated brine exceeds 40 g/l, Na<sub>2</sub>SO<sub>4</sub> is contaminated into NaCl crystals and deteriorates the product quality. Accordingly, the evaporation is suspended before Na<sub>2</sub>SO<sub>4</sub> contamination. Succeedingly, NaCl crystals are extracted from the evaporator, dehydrated in a dehydrating unit and dried in a drying machine to produce edible salt. In the mother liquid discharged from the final crystallizer, SO<sub>4</sub><sup>2-</sup> ions are accumulated.

One of the methods to recover SO<sub>4</sub><sup>2-</sup> ions in the concentrated brine in the above salt manufacturing process is precipitation of SO<sub>4</sub><sup>2-</sup> ions as Na<sub>2</sub>SO<sub>4</sub>·10H<sub>2</sub>O by cooling the brine to 0  $^{\circ}$ C (cooling method). However, the cooling method consumes energy when the liquid is mechanically cooled from 70  $^{\circ}$ C to 0  $^{\circ}$ C, so the brine is usually discharged to the outside of the process. Thus, the discharged mother liquid in the process dissolves a considerable amount of NaCl crystals and it deteriorates NaCl recovering ratio of the process.

Another method to recover SO<sub>4</sub><sup>2-</sup> ions in the concentrated brine is electro dialysis with ion-exchange membranes. In electro dialysis seawater concentration with ion-exchange membranes, it is necessary to reduce the permeation of divalent ions across the membranes to prevent CaSO<sub>4</sub> precipitation in concentrating cells. For reducing Ca<sup>2+</sup> permeation, a polycation layer is formed on a cation-exchange membrane [11–13]. An anion-exchange membrane is given SO<sub>4</sub><sup>2-</sup> ion low-permeability by forming a polyanion layer or developing cross-linking structure on the membrane [14,15]. The above technology was applied not only for prevention

of CaSO<sub>4</sub> scale precipitation but also for purification of saline water [16–18].

In the previous investigation, we operated a single-pass electro dialysis unit to treat the mother liquid and discussed permselectivity for SO<sub>4</sub><sup>2-</sup> ions of the membrane [19]. This investigation separates SO<sub>4</sub><sup>2-</sup> ions from NaCl in mother liquid using a batch electro dialysis unit incorporated with SO<sub>4</sub><sup>2-</sup> ion low-permeable anion-exchange membranes and discusses energy consumption in the process. Further, in the previous investigation [20], an instantaneous open/shut feed operation system of salt solution was developed in the program to compute the performance of batch desalination. In this investigation, the same program is applied to the batch electro dialysis operation of the mother liquid. The computed data are compared to the experimental data to confirm the reasonability of the program.

## 2. Ca<sup>2+</sup>, Mg<sup>2+</sup> and SO<sub>4</sub><sup>2-</sup> ion concentrations in brine in a salt manufacturing process

Fig. 1 illustrates the salt manufacturing process incorporated with seven stage multiple-effect vacuum evaporators [21]. At first, Ca<sup>2+</sup> and Mg<sup>2+</sup> ions in the brine are removed (pretreated) by adding Na<sub>2</sub>CO<sub>3</sub> and NaOH in the thickener. The pretreated brine is supplied to the No. 1 evaporator, further to the evaporators from the No. 2 through No. 7 in series. Finally 70  $^{\circ}$ C mother liquid saturated with NaCl and dissolving Na<sub>2</sub>SO<sub>4</sub> is discharged from the No. 7 evaporator. Boiler steam is also introduced to the evaporators from the No. 1 through No. 7 in series, discharged to the condenser and extracted to the vacuum pump. Table 1 shows Ca<sup>2+</sup>, Mg<sup>2+</sup> and SO<sub>4</sub><sup>2-</sup> ion concentrations in the raw salt brine, purified brine supplied to the No. 1 evaporator and the mother liquid discharged

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