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# Simultaneous removal and recovery of boron from waste water by multi-step bipolar membrane electrodialysis

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ABSTRACT

We have developed bipolar membrane electrodialysis (BPED) for simultaneous removal and recovery of boron from waste water. BPED is performed with a two-cell electrodialysis unit composed of an anion exchange membrane (AEM) sandwiched between two bipolar membranes (BPM). A semi-batch type operation was performed by circulating solutions through the cells, namely, the removal and concentration cells. The volume ratio of the reserve tanks for the feed stream to the cells was set to 4:1 (removal: concentratation). Tetrahydroxyborate ions were transported from the removal cell to the concentration cell through the AEM. In the ideal case, the boron concentration in the concentrate should be concentrated to 5 times the initial concentration, when the solutions of the same initial concentration were supplied to both cells of concentration and removal. The concentrated solution was then used for second step to further concentrate the solution. This concentrated solution was then used for third step, and so forth. The operation was continued step-by-step until the boron concentration was sufficiently high to easily recover boric acid. The experimental results with a stack of five cells for the sodium borate solutions (initial concentration: 100 mg/L) showed that the boron concentration in the concentrate was about 4, while that in the diluate was less than 10 mg/L, which is below the regulatory value for waste water in Japan. After four steps, the boron concentration in the concentrate reached close to the solubility of boric acid (ca 10,000 mg/L), at which boron can be recovered for recycling.

## 1. Introduction

Long-term excessive intake of boron causes human health effects, such as damage to the reproductive system [1,2]. The World Health Organization recommends that the boron concentration in drinking water should be less than 2.4 mg/L [3]. The guideline boron concentrations in drinking and waste water have been regulated in many countries [4,5]. In Japan, the guideline boron concentration in industrial waste water effluent is 10 mg/L [6]. There are several types of methods to remove boron from waste water [7–9], including membrane separation by reverse osmosis [10,11], electrodialysis [12–19], ion exchange [20,21], adsorption by solids [22–28], and hybrid processes using adsorption and membrane separation [29–32]. Most of the conventional methods are costly, and an efficient low-cost method for removal of boron from waste water needs to be developed. Boron and its compounds are widely used industrial raw materials for glass production, ceramic engineering, metal working industries [7,8]. The boron resources used in Japan are imported, and it is desirable to recover or recycle boron from waste water for national resource security and cost reduction of the waste water treatment process.

In a previous study, we proposed a new separation process for removal of boron from waste water based on bipolar membrane electrodialysis (BPED) [13]. Bunani et al. also applied the BPED process for the recovery of boron [14], [15]. A schematic diagram of the BPED system is shown in Fig. 1. The BPED unit is composed of two bipolar membranes (BPMs) and one anion exchange membrane (AEM), where the AEM is sandwiched between the two BPMs. The BPM is a laminated composite membrane that enables dissociation of water to generate and emit protons and hydroxide ions by applying an electric potential higher than 0.83 V along the membrane [13], The AEM selectively transports anions. The cell formed on the cathode side is the removal cell, while that formed on the anode side is the concentration cell. Feed solutions with the same initial concentration of boron and pH are fed into both cells and circulated into the reserve tanks. Thus, the separation process is performed as a semi-batch operation. The removal efficiency of boron is as high as 90% for an initial boron concentration of 100 mg/L with a wide pH range of 2.3–12.0. The key function of the BPMs is continuous supply of hydroxide ions to the removal cell and

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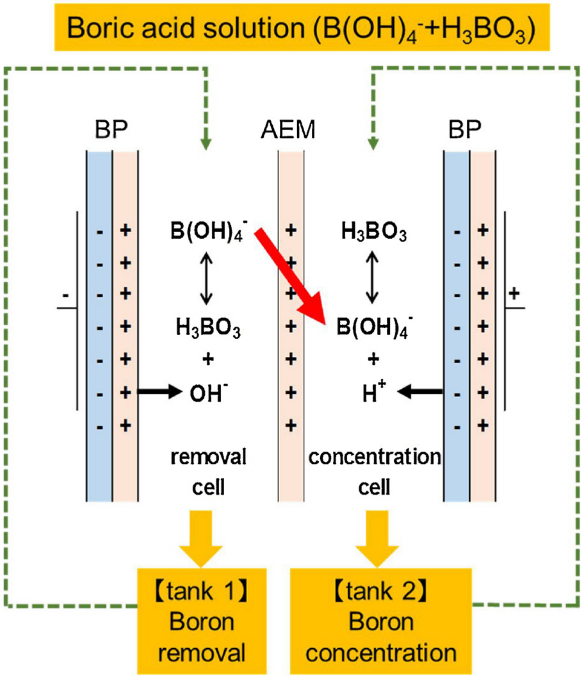

**Fig. 1.** Schematic of the BPED system, which is a unit cell composed of two BPMs and one AEM.

protons to the concentration cell. The dominant form of boric acid depends on the ambient pH conditions. Under alkaline conditions ($pH > pK_a = 9.14$), the tetrahydroxyborate ion ($B(OH)_4^-$) is dominant, while under acidic condition ($pH < 9.14$), boric acid ($H_3BO_3$, a non-electrolyte) is dominant [33]:

$$H_3BO_3 + H_2O \Leftrightarrow H^+ + B(OH)_4^-;\ pK_a = 9.14 \qquad (1)$$

The boric acid supplied to the removal cell combines with hydroxide ions to form tetrahydroxyborate ions. Because tetrahydroxyborate ions are anions, they are transported through the AEM to the concentration cell, where they combine with protons supplied by the other BPM located on the anode side to form boric acid (a non-electrolyte). Thus, boron is continuously transported from the removal cell to the concentration cell. Theoretically, all of the boric acid in the diluate should move to the concentrate. However, it would take an infinite amount of time for the concentration of boron in the removal cell to decrease to zero. In contrast, boron is concentrated in the concentrate. When the volumes of the circulation reservoir tanks of the diluate and the concentrate are the same, the concentration of boron in the concentrate would double. For example, the concentration of boron in the concentrate would be 200 mg/L when the initial feed concentration of boron was 100 mg/L. This means that after the separation process, the effluent with a higher concentration of boron needs to be treated. If the BPED process is repeated, the concentration of boron in the concentrate would increase step-by-step. Finally, the concentration of boron in the concentrate become close to the solubility of boric acid. In this study, the above concept of simultaneous removal and recovery of boric acid by multi-step BPED was investigated using a laboratory-scale experimental apparatus. There are several advantages of the BPED process; BPED is essentially a no-waste generating process, and power-saving compared with other separation processes. Because scaling-up generally does not affect the performance of the BPED process, the obtained results can be used to investigate the feasibility of practical separation of boron from waste water.

## 2. Experimental

A number of operational and environmental parameters affect the separation performance of BPED. In this study, we investigated the effect of the volume ratio of the reservoirs on circulation of the feed solutions into the removal and concentration cells. Electrodialysis was performed with a laboratory-scale apparatus (AGC Engineering, Tokyo, Japan). The bipolar membrane (BPM) used was Neosepta®, BP-1B, supplied by ASTOM, Co., Tokyo, Japan. The membrane thickness was 0.22 mm. The anion exchange membrane (AEM) used was Selemion® AMV, supplied by AGC Engineering, Co., Tokyo. The membrane thickness is 0.15 mm, and the resistance is 2.0–3.5 Ωcm$^2$, the ion exchange capacity is 2.0–2.3 meq/g [34]. The electrode solution was 0.1 M sodium sulfate solution. The electrodialysis apparatus was composed of a stack of five BPM–AEM–BPM units. The effective diffusion area of the membranes in the stack was 117.5 cm$^2$, and the gap between the membranes was 1.5 mm. The total potential along the membrane stack was kept constant at 12.50 V. The electrodialysis was conducted as a semi-batch type operation, as shown in schematically in Fig. 2. The feed solutions were circulated through the BPED system by using two reservoir tanks: one for circulation of the feed solution through the

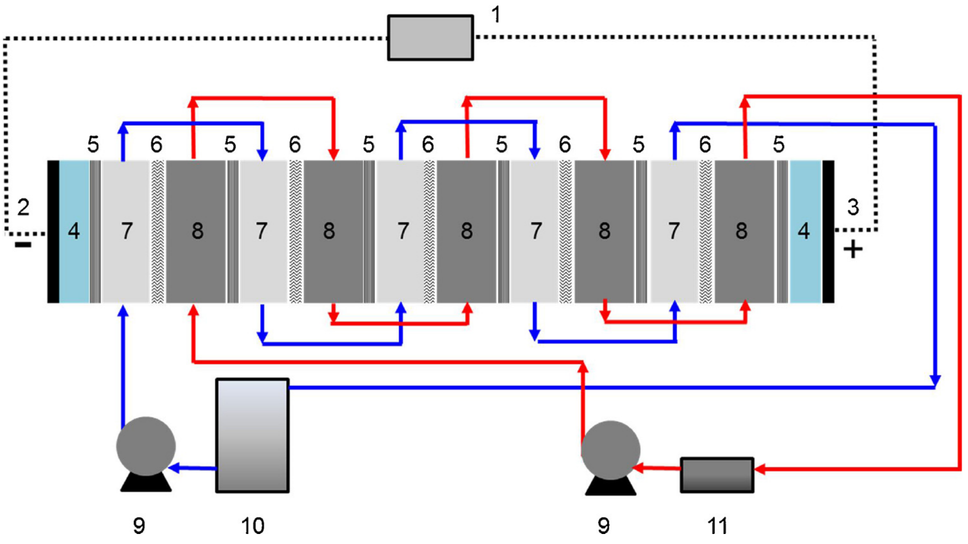

**Fig. 2.** Flow diagram of the semi-batch separation process.
1. DC power supply, 2. Electrode (anode), 3. Electrode (cathode), 4. Electrode cell, 5. Bipolar membrane, 6. Anion exchange membrane, 7. Removal cell, 8. Concentration cell, 9. Liquid pump, 10. Reservoir tank for removal cells, 11. Reservoir tank for concentration cells.

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