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Boron removal from boric acid wastewater by electrocoagulation using aluminum as sacrificial anode

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A B S T R A C T

Electrocoagulation (EC) using metallic aluminum as anode and cathode for boron removal from solution was studied. The electrolytic parameters included pH, current density, and initial boron concentration for optimizing the EC process. Experimental results showed that removal efficiency was increased by elevating pH from 4.0 to 8.0, and then decreased at higher pH. The electrolytic efficacy was increased with increasing current density from 1.25 to 5.0 mA cm^{-2}. With respect of energy consumption, 2.5 mA cm^{-2} of current density was acceptable for an effective EC of boron, while increasing boric acid from 10 to 100 ppm-B did not impair removal efficiency. NaCl as a supporting electrolyte promoted more anodic dissolution of aluminum from the electrode surface than that predicted by the Faraday's law. The optimal conditions under which 95% of boron was removed and less than 5 ppm-B remained in the electrolyte would be pH 8, four pairs of electrodes, and 2.5 mA cm^{-2} in 180 min as treating wastewaters containing 10—100 ppm-B. X-ray powder diffractometer and scanning electron microscope were used and results suggested that the irregular crystallites of hydroxide precipitates were composed of bayerite and boehmite phases simultaneously.

1. Introduction

Boron is available in the environment as calcium and sodium borates or as a result of anthropogenic pollution in the form of boric acid and borate salts [1]. Borate salts in solution exists as highly soluble poly-borate ions of high concentration, but it appears as a monomer with low concentration (< 25 ppm) [2]. Boron is widely used in many industries, such as the manufacture of borosilicate glass, detergents, semiconductor, cosmetics, flame retardants, fertilizers, and dyestuff production [3], and is unintentionally discharged into the environment during the manufacturing processes. The deficiency of boron in plants may result in reduced growth, loss of yield, and death [4]. However, raising levels of boron may be harmful to plants. Boron is toxic to human body with some symptoms, include nausea, vomiting, dermatitis, diarrhea, and lethargy [5]. Recently, World Health Organization has regulated an upper limit of 2.4 ppm-B for drinking water [6].

Several techniques have been developed for reducing boron in wastewaters, including ion exchange, adsorption, chemical precipitation, and reverse osmosis [7—11]. Electrocoagulation (EC) generates coagulants through creating metallic hydroxide from the electrochemically soluble anodes [12,13]. It is an efficient way for treating industrial wastewaters containing organic and inorganic pollutants, such as dye, organic matter, nitrate and arsenate [14—17]. EC is outstanding in some aspects as compared to conventional technologies, including high removal efficiency, compact treatment facility, and less sludge produced. This process involves three stages: (i) coagulants formation due to electrolytic oxidation of sacrificial electrodes, (ii) destabilization of the contaminants, and (iii) flocculation of insoluble particles. Meanwhile, hydrogen gas and oxygen are released from the cathode and the anode, respectively. The gas production is beneficial to rise the flocculated particles to the water surface [18,19].
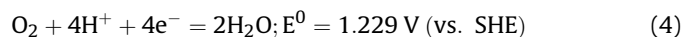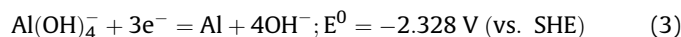
At cathode side:

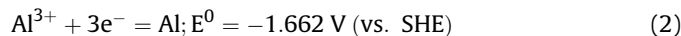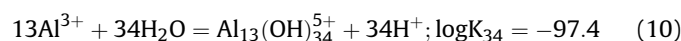$$H_2O + e^- = 1/2H_2 + OH^-; \; E^0 = -0.828 \text{ V (vs. SHE)} \quad (1)$$
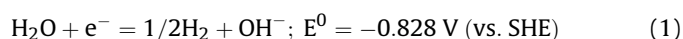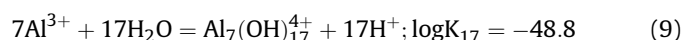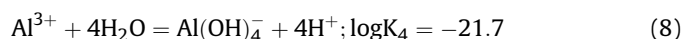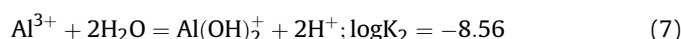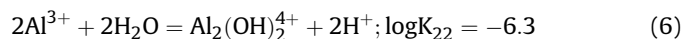
At the anode:

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$$Al^{3+} + 3e^- = Al; E^0 = -1.662 \text{ V (vs. SHE)} \tag{2}$$

$$Al(OH)_4^- + 3e^- = Al + 4OH^-; E^0 = -2.328 \text{ V (vs. SHE)} \tag{3}$$

$$O_2 + 4H^+ + 4e^- = 2H_2O; E^0 = 1.229 \text{ V (vs. SHE)} \tag{4}$$

Al(III) oxidized from anode would dissolve as several types of hydrolytic species depending upon pH [20]:

$$Al^{3+} + H_2O = AlOH^{2+} + H^+; \log K_1 = -5.02 \tag{5}$$

$$2Al^{3+} + 2H_2O = Al_2(OH)_2^{4+} + 2H^+; \log K_{22} = -6.3 \tag{6}$$

$$Al^{3+} + 2H_2O = Al(OH)_2^+ + 2H^+; \log K_2 = -8.56 \tag{7}$$

$$Al^{3+} + 4H_2O = Al(OH)_4^- + 4H^+; \log K_4 = -21.7 \tag{8}$$

$$7Al^{3+} + 17H_2O = Al_7(OH)_{17}^{4+} + 17H^+; \log K_{17} = -48.8 \tag{9}$$

$$13Al^{3+} + 34H_2O = Al_{13}(OH)_{34}^{5+} + 34H^+; \log K_{34} = -97.4 \tag{10}$$

During EC process, Al(III) speciation undergoes spontaneous formation of hydroxide precipitates which are supposed to effective adsorbents for boron components. The stability diagram of $Al(OH)_{3(s)}$ as a function of pH is as shown in Fig. 1.

$$Al(OH)_{3(s)} + 3H^+ = Al^{3+} + 3H_2O; \log K_{sp} = 9 \tag{11}$$
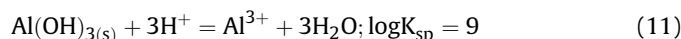
To remove boron efficiently, conventional coagulation and precipitation processes use metal salts, and high quantity is applied at high pH values that normally exceed 12 and high temperature (> 60 °C) [8]. This work aimed to examine the feasibility of EC using aluminum as the sacrificial electrode for boron removal fro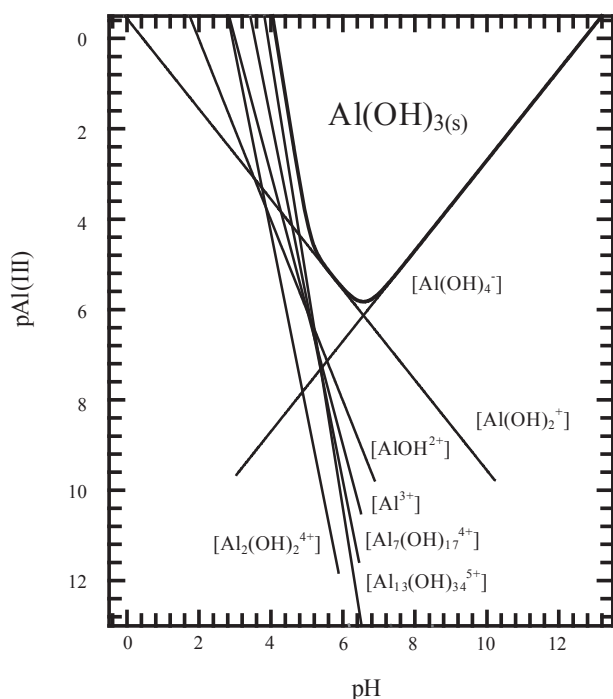m the boric acid solution at controlled temperature (30 ± 0.3 °C). Parameters investigated included the pH, current density, and boron concentration for optimizing EC. Precipitates collected from the electrolytic cell after treating the boric acid were also characterized using X-ray powder diffractometer (XRD) and scanning electron microscope (SEM). Eventually, the efficacy of conventional chemical coagulation (CC) and EC using the same level of precipitants (i.e., Al hydroxide) was compared in parallel.

2. Materials and methods

2.1. Chemicals

All reagents were of analytical grade, and used without further purification. The synthetic wastewaters were prepared by dissolving the boric acid ($NaBO_3 \cdot 4H_2O$ powder, 97% purity, Panreac, Spain) into waters which were doubly deionized with a laboratory-grade RO-ultrapure water system (resistance > 18.3 MΩ cm). The pH was adjusted using sodium hydroxide (NaOH, Merck KGaA, Germany) and hydrochloric acid (HCl, SHOWA, Japan). The aluminum metal used as the anode and cathode was provided by Ming-Yuh Sci. Ins., Taiwan.

2.2. Experimental procedure

The batch experiment of EC was carried out in an electrolytic cell that was made of Pyrex glass with dimensions of 12 × 10 × 8 cm (WLH). Metallic aluminum as cathode and anode in pairs (10 × 10 cm) were placed at 1 cm interval and connected to a DC power supplier. Fig. 2 depicts the configuration of electrolytic cell. Given concentrations of boron and sodium chloride (NaCl, 99.5%, Showa) as background electrolyte were fed into reactor. All batch solutions were magnetically stirred at 300 rpm. The power supplier in a constant current mode was switched on to carry out experiments, during which the temperature were maintained at 30 ± 0.3 °C using a water bath. At specific time interval, samples withdrawn were filtered by 0.45 um filter, while precipitates were digested using 3 M HNO_3. The solution pH was measured and conditioned to desired value after every sampling. Boron and aluminum elements in filtrates and digests were analyzed by an inductively coupled-plasma emission spectrometer (ICP-OES, JY 2000-2, HORIDA). A SEM (JEOL JSM-6700F, Japan) and an XRD (Rigaku RX III, Japan) were used to determine the surface morphology and the structural phase of aluminum hydroxide, respectively.
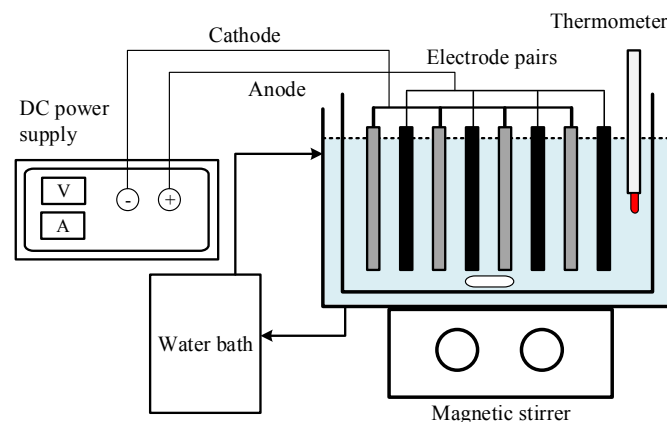

Fig. 1. Solubility diagram of aluminum hydroxide.

Fig. 2. Apparatus of electrolytic cell for electrocoagulation of boric acid using aluminum as electrodes.

3. Results and discussion

3.1. Effect of pH on EC of boric acid

The pH of electrolyte determines not only the overpotential of electrolytic oxidation of aluminum anode but also the efficiency of precipitation of the dissolved Al(III) ions [21]. Hence, the electrolyte pH would influence the coagulation of boron compounds. As shown in Fig. 3, boron removal significantly varies with pH in a range of 4–12 (initial 100 ppm-B) as the current density and the stirring speed are kept at 2.5 mA cm^{-2} and 300 rpm, respectively. Boron level decreases with the increasing pH and reaches a minimum around pH 8 at which the removal efficiency is 84%. Boron removal varied by pH resulting from some factors, such as the formation of aluminum hydroxide, pH$_{pzc}$ (point of zero charge) of precipitates, and electrovalence of boric acid. As predicted by solubility curve of Al(OH)$_{3(s)}$ in Fig. 1, at pH 5–8.5 Al(III) is rather insoluble as the form of Al(OH)$_{3(s)}$ whose surface is positively charged (pH$_{pzc}$ > 9) [22]. The concentration of soluble aluminum approaches to minimum at pH 8 in Fig. 3 meeting the prediction of solubility product constant of Al(OH)$_{3(s)}$. Besides, the acidic constant of boric acid is around 9, which means borate ions predominantly present as B(OH)$_4^-$ in alkaline solution. At high pH values (pH > 10), aluminum flocs are negatively charged and quite soluble as Al(OH)$_4^-$ ions. As a result, pH would strongly dominate the adsorption of boron species in terms of modifying surface properties of aluminum hydroxide.

3.2. Optimization of electrolytic system for electrocoagulation of boron

Aluminum plates in different arrangements are beneficial to the coagulation efficiency through increasing reactive surface area, as well as the dissolution rate of metal anode. Fig. 4 shows the effect of electrode pairs on the removal of boron; the efficiencies are approximately 75, 77, 81, and 84% for one, two, three, and four electrode pairs, respectively (pH 8, initial B = 100 ppm, current density = 2.5 mA cm^{-2}). In multiple electrodes, the electrodes provide higher surface area favoring more anodic oxidation as compared to single pair of electrodes. Under the same current density, high total cell voltage is required for providing sufficient current in an electrolytic system of multiple electrodes. Consequently, more dissolution of Al(III) ions and hydroxyl ions for the generation of flocs can accommodate more adsorbed boron compounds. In other words, when using the multipolar connection, the
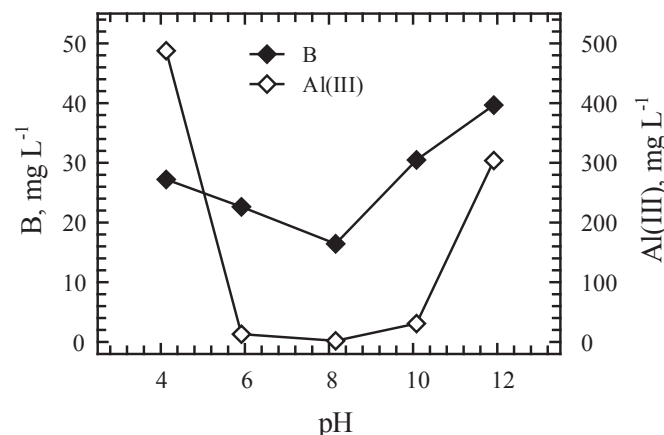

Fig. 4. Effect of electrode pair on boron removal. (pH 8, initial B = 100 ppm, current density = 2.5 mA cm^{-2}).

efficacy of EC of boron is enhanced due to the change in net current used.

The effect of current density being varied between 1.25 and 5 mA cm^{-2} on boron removal was investigated by adjusting the current was from 2.0 to 8.0 A, while boron concentration, pH reaction, and stirring speed remain constant. When the current density is increased from 1.25 to 5 mA cm^{-2}, the removal efficiency increases from 72 to 95% (Fig. 5a); boron level at the end of the experiment can be lower than 5 ppm-B by elevating the energy consumption from 38 to 154 kWh m^{-3} (Fig. 5b). Results show that increasing current density increases the removal of boron due to increasing rate of anodic dissolution. A factor that normalize the removal efficiency (%) with power consumed (kWh m^{-3}) is used to assess the optimal energy input. As shown in Fig. 5c, when current density is increased above 2.5 mA cm^{-2}, values of (%)/(kWh m^{-3}) are not varied too much. It means that the electrolytic system has reached the limit to treat boron using 2.5 mA cm^{-2}. In other words, energy is not effectively used for EC at higher current density than 2.5 mA cm^{-2}.

The current density can be related to an ideal dissolved aluminum by the second Faraday law, which converts the electricity quantity passing through the electrode to the formation of redox species.

$$W = \frac{Q \times MW}{z \times F} \tag{12}$$

where W is the weight of the dissolved component of concerns, Q is total charge involved the electron transfer, which is equal to net current multiplying the reaction time (I × t), MW is the molecule weight of aluminum (27 g mol^{-1}), z is the valency number of the substance and F is the Faradaic constant (96,500 C mol^{-1}). Consequently, the theoretical dissolution of Al(III) from the sacrificial anode that should be precipitated as Al(OH)$_3$ is compared with the weight loss of electrodes after the reaction period. As shown in Fig. 6, all the measured weight losses approximate to the Faradaic values, however the discrepancy among these two values increases with increasing current density. When NaCl is used as the background electrolyte, the anodic strong oxidative chlorine species (Cl$_2$, HOCl) may participate the dissolution of metallic aluminum.

$$Cl_2 + 2e^- = 2Cl^-; E^0 = 1.36 \text{ V (vs. SHE)} \tag{13}$$

$$HOCl + H^+ + 2e^- = Cl^- + H_2O; E^0 = 1.48 \text{ V (vs. SHE)} \tag{14}$$

$$Cl_2 + H_2O = HOCl + H^+ + Cl^- \tag{15}$$

Fig. 3. Effects of pH on electrocoagulation of boric acid and the total aluminum in solutions after 180 min (initial B = 100 ppm, current density = 2.5 mA cm^{-2}).
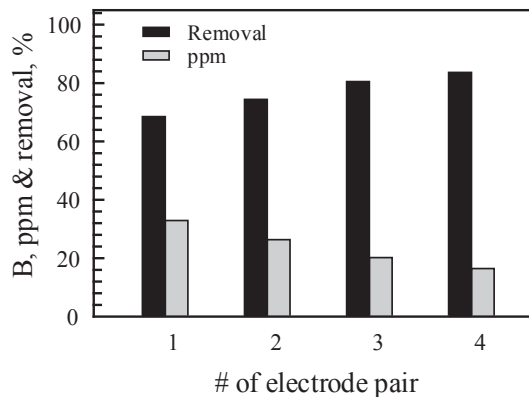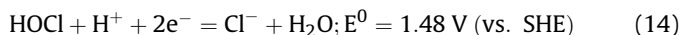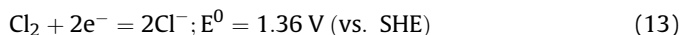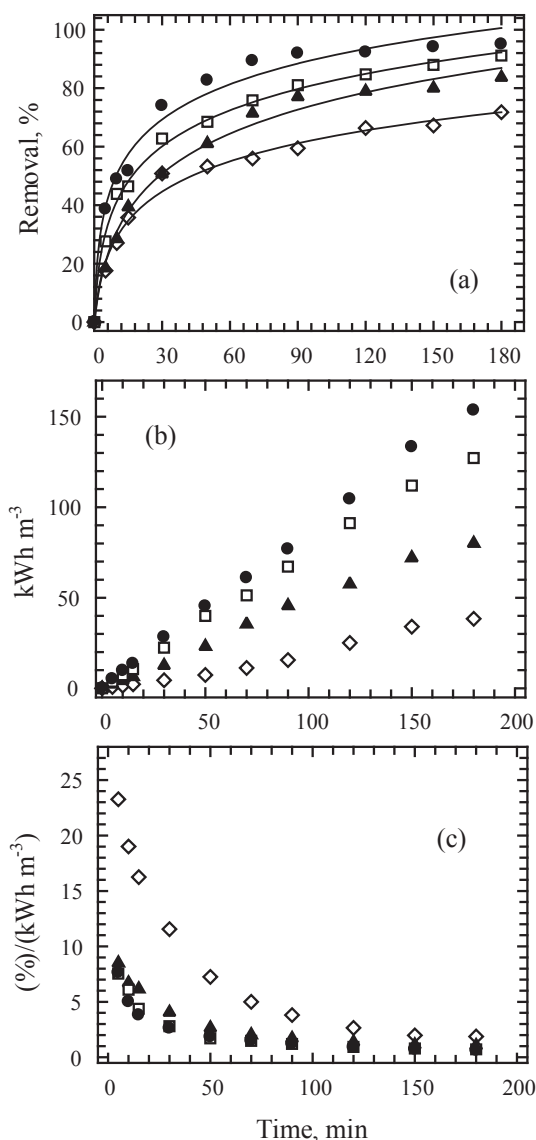
Fig. 5. Effect of current density on (a) removal efficiency of boron, (b) energy consumption as a function of electrolytic time, and (c) boron removal normalized with power input. ($\diamond$ 1.25 mA cm^{-2}, $\blacktriangle$ 2.5 mA cm^{-2}, $\square$ 3.75 mA cm^{-2}, $\bullet$ 5 mA cm^{-2}) (initial B = 100 ppm, pH 8, 4 electrode pairs).
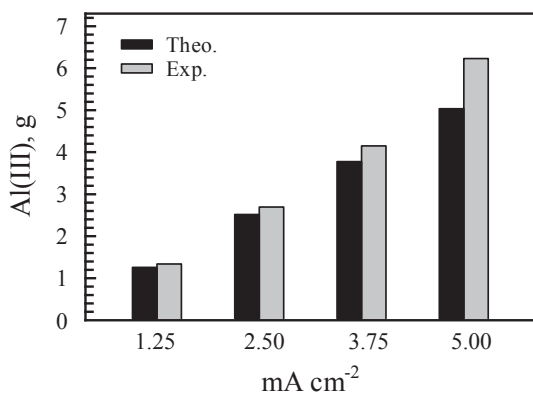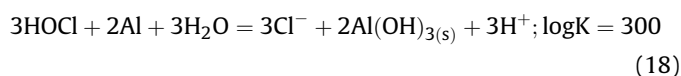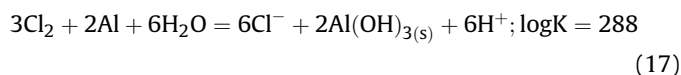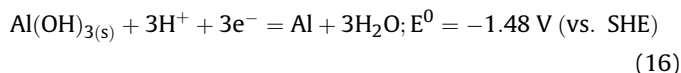

Fig. 6. Comparison of experimental and Faradaic aluminum dissolution (Al(III) in gram) as a function of current density (pH 8, electrode pair = 4, initial boron = 100 ppm).

Many researches have reported that when using NaCl to support the electrolysis the iron or aluminum anodes offered faster removal of organic pollutants with much lower energy consumption for EC [23,24]. Considering the negative reduction potential of half-cell aluminum corrosion, the immediate oxidation of aluminum by active chlorine is a critical side-reaction that contributes the weight loss of the sacrificial anode.

$$Al(OH)_{3(s)} + 3H^+ + 3e^- = Al + 3H_2O; E^0 = -1.48 \text{ V (vs. SHE)} \quad (16)$$

$$3Cl_2 + 2Al + 6H_2O = 6Cl^- + 2Al(OH)_{3(s)} + 6H^+; logK = 288 \quad (17)$$

$$3HOCl + 2Al + 3H_2O = 3Cl^- + 2Al(OH)_{3(s)} + 3H^+; logK = 300 \quad (18)$$

On the other hand, Cl$^-$ ions could alleviate the passivation of anode through incorporation of Al(OH)$_2$Cl, Al(OH)Cl$_2$ and AlCl$_3$ into the surface oxide film, which was eventually dissolved by the formation of AlCl$_4^-$ in the solution [25,26].

In order to examine the treatment capacity of EC of boric acid using aluminum as the sacrificial anode, boron removal was conducted with initial levels of 10, 50, and 100 ppm under current density of 2.5 mA cm^{-2} and pH 8.0. Fig. 7 indicates the removal efficiency as a function of time for different initial boron concentrations. Increasing initial boron concentration from 10 to 100 ppm slightly decreased removal efficiency of boron from 86 to 83% after 180 min of EC. The amount of aluminum hydroxide produced by the sacrificial anode should remain constant under a fixed current density for all boron levels. Therefore, boron in a range of the chosen concentrations did not remarkably affect the efficiency of EC removal of boron. It implies that the amount of aluminum hydroxide under given electrolytic conditions can be capable of adsorbing at least 100 ppm, even though the initial rate of removal is different among these initial boron concentrations. Boron removal of lower boron level indeed goes rapidly than dose higher boron concentrations.

3.3. Characterization of precipitates after EC of boric acid

The morphology of precipitates collected from the electrolytic reactor is shown in Fig. 8. SEM observation indicates that precipitates are primarily composed of irregular-shape crystallites stacking on flake solids. The rough surface of electrodes after
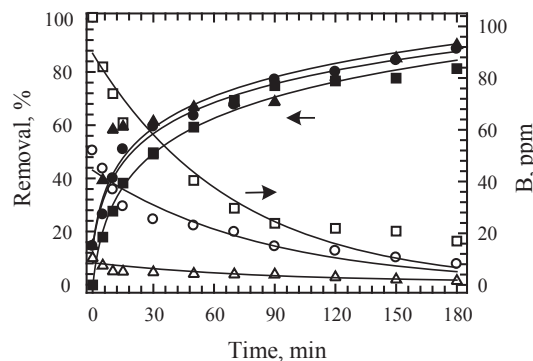

Fig. 7. Effect of initial concentration of boric acid on (ppm-B) boron removal ($\triangle$ $\blacktriangle$ 10 ppm, $\circ$ $\bullet$ 50 ppm, $\square$ $\blacksquare$ 100 ppm) (current density = 2.5 mA cm^{-2}, pH 8, 4 electrode pairs).
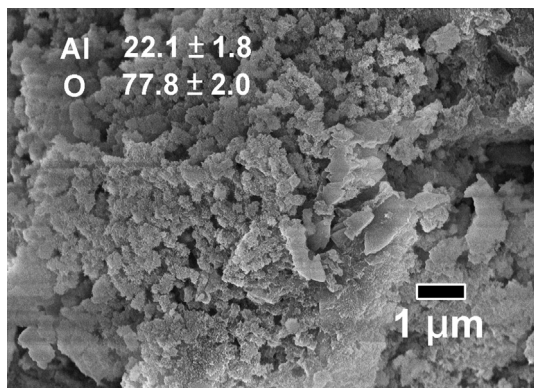
Fig. 8. SEM microscopic and EDS elemental analysis of aluminum hydroxide coagulates from electrolytic dissolution using aluminum as electrodes.

several cycles of EC may be related to the passivation at active sites of anodes due to either the dissolution of aluminum or the directly oxidized film formed on electrodes. Energy Dispersive Spectrometer reveals that a molar ratio of aluminum to oxygen elements is around 2.2–7.7, which is slightly less than the stoichiometric ratio of $Al(OH)_3$. Nevertheless, boron compounds are absent probably due to the relatively low level of boron species adsorbed in the sludge of aluminum hydroxide. According to ICP analysis of HNO_3 digested precipitates, the weight ratio of aluminum hydroxide and boric acid were $91.8 \pm 1.4\%$ and $0.8 \pm 0.1\%$, respectively.

Fig. 9 presents XRD patterns of recycled precipitates after EC of boric acid under current densities of 1.25–5 mA cm^{-2}. The main crystalline phase of $Al(OH)_3$ is bayerite (JCPDF 20-0011) at low current densities applied (1.25 and 2.5 mA cm^{-2}). With increasing current higher than 3.75 mA cm^{-2}, however, an additional phase of
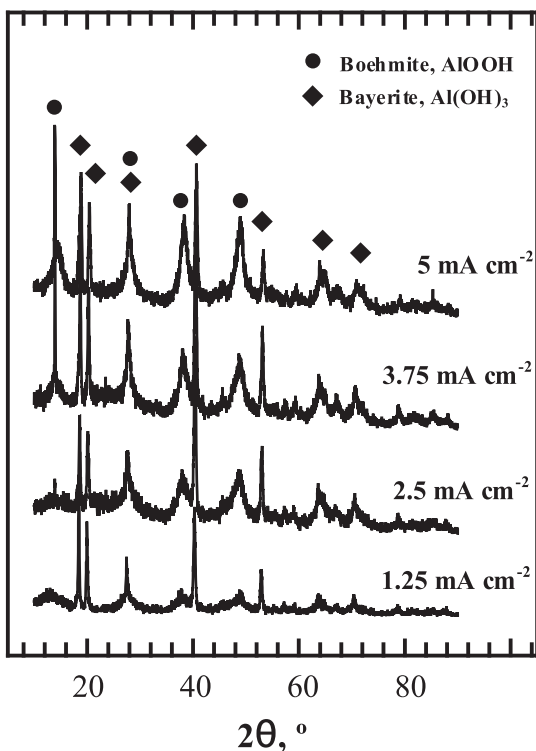
boehmite (AlOOH, JCPDF 21-1307) appears. The diffractive signal of boron species is absent due to its rather low content in crystallites, leading to being masked by that of hydroxides. On the other hand, it is interesting that boehmite phase exhibits in the precipitate at high current density; the anodic oxidation of metallic aluminum might include a half-cell reaction of AlOOH formation whose standard reduction potential is quite close to the dissolved aluminum ions (Al(III)) ($E^0 = -1.662$ V) and aluminum hydroxides ($E^0 = -1.48$ V) [27].

$$AlOOH_{(s)} + 3H^+ + 3e^- = Al + 2H_2O; E^0 = -1.542 \text{ V (vs. SHE)} \tag{19}$$
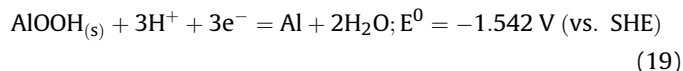
3.4. Comparison of CC and EC for boron removal

CC is conventionally adopted for treating heavy metal contaminated wastewaters, and also for the recycling of suspended materials. CC process was also carried out in an electrolytic system for boron removal using the RuO_2/IrO_2-Ti electrode, dimensionally stable anodes, and aluminum salt which was identical to those dissolved from aluminum electrodes in EC process in the reaction period. The current density of 2.5 mA cm^{-2}, pH 8.0, and 100 ppm-B were kept for both CC and EC processes. Experimental results are as presented in Fig. 10. Accordingly, EC is more versatile in boron removal as compared to CC. Boron level is reduced with increasing the contact time by EC (efficiency of boron removal is 56% in 180 min), while CC removes boron in a very short period, but the removal efficiency maintains at only 38% after 10 min. In CC process, the supersaturation of Al(III) drove the sudden blooming of nuclei of aluminum hydroxides; the mass transport of boric acid into flocs could be retarded by the acute precipitation. By contrast, aluminum hydroxides from anode dissolution of EC are continuously generated during electrolysis, and are more favorable to immobilize boron species.

4. Conclusions

This work investigated the EC of synthetic wastewater containing 100 ppm-B. Metallic aluminum as sacrificial electrodes eliminated more than 95% of boron from NaCl electrolyte at around pH 8. The aqueous boron at the end of electrolysis could be lower than 5 ppm-B. Removal efficiencies increased with increasing current density by improving the rate of anode dissolution. However,

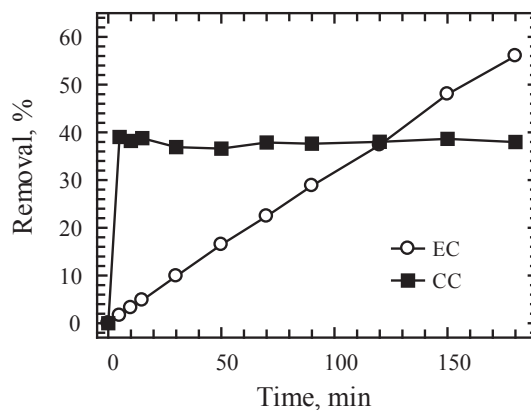**Fig. 9.** XRD patterns of aluminum hydroxide from electrocoagulation experiments under various current densities.

Fig. 10. Comparison of boron removals by electrocoagulation (EC) using aluminum electrodes and chemical coagulation (CC) using RuO_2/IrO_2-Ti electrodes in 0.1 M Al(III) electrolyte. (pH 8, boric acid = 100 ppm-B, one electrode pair).

based on the consideration of energy-saving, a current density of $2.5 \, mA \, cm^{-2}$ applied by four pairs of electrodes is most appropriate for boron removal. The increment in initial boron concentration (from 10 to 100 ppm-B) insignificantly influenced the removal efficiency, but slightly reduced the initial rate of EC of boron. NaCl as the supporting electrolyte enhanced the passivation of electrodes, leading to more aluminum dissolution than the anticipated value of Faraday's law. SEM images revealed that the precipitates from EC were of irregular crystallites which were of bayerite and boehmite phases according to XRD analysis. EC was proven to be more reliable than the conventional CC in the decontamination of boron-containing wastewaters.

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