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The effect of initial pH and retention time on boron removal by continuous electrocoagulation process

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ABSTRACT

This work examined boron removal by applying a lab-scale continuous electrocoagulation process. The impact of influent pH (4, 5, 6, 7.45, and 9) and retention time (10, 25, 50, and 100 min) on the treatment process was examined. Plate-type aluminum electrodes were used. The experiments were conducted in continuous mode, and the electric current was kept constant at 5 A. The initial boron concentration was 1,000 mg L^{-1}. The first set of experiments concerning the influence of the influent pH showed that the highest boron removal (67%) was obtained at pH = 6. This pH value was the optimal one for boron precipitation through aluminum borate formation. The increase in the duration of the process from 10 to 100 min resulted in increasing boron removal from 45% to 79%. The longer duration of the electrocoagulation process enabled higher aluminum dissolution, thus allowing the existence of a higher amount of coagulants within the reactor. Moreover, it enhanced boron precipitation because of the longer contact time between the boron ions and the coagulants. By optimizing the key parameters of the process, the continuous electrocoagulation process showed to be an effective alternative for the removal of highly concentrated boron.

Keywords: Boron removal; Continuous electrocoagulation process; Aluminum electrodes; Influent pH; Retention time

1. Introduction

Boron (B) is a trace element widely distributed in the earth's hydrosphere and lithosphere. In the lithosphere, boron can be found in soil or rocks. Its average concentration is 10 mg kg^{-1} in the earth's crust, thus taking up only 0.001% of the earth's elemental composition [1,2]. In terms of boron presence in the hydrosphere, its average concentration is approximately 5 mg L^{-1} in seawater, whereas it

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ranges from 0.3 to 100 mg L^{-1} in groundwater. In wastewater, boron concentration depends on the type of the industrial activity and its consequent treatment [1–5]. Elemental boron is not found in nature; it is mainly traced in the forms of boric acid ($B(OH)_3$), boron trioxide (B_2O_3), and borax (sodium tetraborate (decahydrate): $Na_2B_4O_7{\cdot}(10H_2O)$) [5,6].

The increases and fluctuations observed in boron concentration in surface waters are related to both natural and anthropogenic factors. For example, weathering of rocks and leaching of salt deposits in the seashore can be listed among the natural causes. Subsequently, boron deposits are likely to appear on the shoreline. Being highly volatile, boron can then be traced within the rainfall near coastal areas. Industrial activities and the subsequent discharge of industrial wastewater can additionally trigger an increase of the boron concentration in surface waters. Boric acid and boron salts are widely used as preservatives in various industrial sectors. Moreover, boron compounds have been utilized in the production of high-energy fuels, coolants, and catalysts [7]. Hence, boron concentration in surface waters at industrial and urban areas is constantly increasing. Boron is also detected in the "acid rain" [8–10]. Apart from being widely distributed in soil and water, boron at low concentrations functions as a vital micronutrient for plants and animals. Nevertheless, high boron concentration has been associated with retarded growth, cutaneous disorders in animals and human beings, in addition to negative effects on the male reproductive system and toxicity toward plants [11–13]. The European Commission Directive on Drinking Water (98/83/EC) has set an upper limit of 1 mg L^{-1} regarding the maximum allowable boron concentration in drinking water [14]. The United States Environmental Protection Agency guidelines set a limit of 0.75 mg L^{-1} for reclaimed water reuse because boron is toxic to sensitive plants (e.g., citrus) at concentrations of 1 mg L^{-1} [15]. In this concept, boron removal from water and wastewater is of high importance not only for countries with natural deposits but also for those with high industrial activity.

Different technologies, including evaporation [16], ion exchange [17], chemical precipitation [18], electrodeionization [19], and adsorption [20], have been implemented for the treatment of boron-containing wastewater. Nevertheless, each of them presents disadvantages. For example, evaporation is considered an energy-consuming option with little effectiveness due to the tendency of boric acid to crystallize [11,21]. Moreover, ion exchange requires the regeneration of the selective ion exchange resins at a high frequency. Therefore, small wastewater volumes can be treated each time, while significant amounts of chemicals are required for the regeneration process [11,22]. In addition, chemical precipitation has been linked to high-potential operational costs resulting from the use of chemicals [23]. Finally, adsorption usually performs relatively unsatisfactorily when applied to treat wastewater with high boron concentration [16,24,25].

For example, Shih et al. [18] examined chemical (oxo-) precipitation for the lab-scale treatment of wastewater containing boric acid and sodium perborate ($NaBO_3$) for an initial boron concentration of 1,000 mg L^{-1}, at room temperature and pH = 10. Precipitation with calcium chloride ($CaCl_2$) removed 80% of the boron from the sodium perborate solution but was inefficient in terms of boric acid treatment; specifically, less than 5% of boron removal from the boric acid solution occurred. Pretreatment of the boric acid compounds through chemical oxo-precipitation using hydrogen peroxide (H_2O_2) significantly increased the final boron removal from the boric acid solution to 80%. Kipçak and Özdemir [26] investigated the effectiveness of calcined magnesite tailing as an adsorbent for the removal of boron from aqueous solutions at lab scale. They noted that optimum boron removal (95%) occurred for an adsorbent concentration of 2.5 mg L^{-1} under the following conditions: influent pH = 6, contact time = 24 h, temperature = 25°C, and initial boron concentration = 500 mg L^{-1}. All in all, it is underlined that the application of such technologies in similar systems involves high capital/operational costs and is likely to be poorly efficient at full scale [27].

Hence, the emphasis is placed on the implementation of alternative boron removal technologies that combine high efficiency, low waste production, and cost-effective [11]. Electrocoagulation is suggested as an alternative treatment process; it involves the in situ generation of coagulants via the electrical dissolution of metal electrodes. The metal ion generation takes place at the anode; hydrogen gas is released from the cathode. The hydrogen gas would also help the flocculated particles to float out of the water and, therefore, the process is often called electroflocculation [28]. Typically, aluminum, iron, carbon, mild steel, graphite, and titanium plates are used as electrodes. Iron and aluminum in specific have been reported to be very effective for effluents with a high content of colloids and particles [13,29,30]. With aluminum used as electrode material, the following electrocoagulation reactions take place [31]:

$$\text{Cathode: } 3H_2O + 3e^- \rightarrow (3/2)H_2(g) + 3OH(aq)^- \tag{1}$$

$$\text{Anode: } Al(s) \rightarrow Al(aq)^{3+} + 3e^- \tag{2}$$

$$\text{In the solution: } Al(aq)^{3+} + 3H_2O \rightarrow Al(OH)_3(s) + 3H(aq)^+ \tag{3}$$

Several studies have focused on the efficiency of the electrocoagulation process to remove different types of pollutants. For instance, Behloul et al. [32] removed over 90% of malathion pesticide from aqueous solution by applying a lab-scale electrocoagulation unit. This was accomplished after 10 min of electrocoagulation with aluminum electrodes under the following experimental conditions: initial pH = 6, initial pesticide concentration = 40 mg L^{-1}, current density = 10 mA cm^{-2}, salt concentration = 2,500 mg L^{-1}, temperature = 27°C, and distance between the electrodes = 2 cm. Similarly, Choudhary et al. [33] investigated the removal of cyanide, ammonia, and chemical oxygen demand (COD) from coking wastewater in a lab-scale electrocoagulation unit using aluminum electrodes. After optimizing the experimental conditions (i.e., pH = 10.5, current density = 37.2 A m^{-2}, electrolyte concentration = 100 mg L^{-1}, and electrode gap = 17.5 mm), cyanide, ammonia, and COD removals were equal to 82.7%, 52.1%, and 91%, respectively. Furthermore, Gönder et al. [34] examined the treatment of carwash wastewater via electrocoagulation with aluminum electrodes at lab scale. Optimum conditions were determined as pH = 6, current density = 1 mA cm^{-2}, and operating time = 30 min. Under these conditions, COD and oil grease were removed by 88% and 68%, respectively.

In terms of boron removal, aluminum electrocoagulation has been reported as an effective method for effluents having relatively low boron concentration. The following studies are cited as indicative examples for synthetic wastewater treatment using lab-scale aluminum electrocoagulation. First, optimal boron removal (i.e., 98%) was noted for a synthetic influent simulating oil industry wastewater with an initial boron concentration of 15 mg L^{-1} under the following conditions: pH = 7, charge loading = 2,400 Ah m^{-3}, and contact time = 90 min [35]. Similarly, 99.7% removal from an initial boron concentration of 10.4 mg L^{-1} was attained for wastewater mimicking oil industry wastewater. The optimum operational conditions that allowed this were pH = 6.3, current density = 17.4 mA cm^{-2}, and contact time = 89 min. [36]. Moreover, Yilmaz et al. [37] concluded that conditions such as pH = 8 and electric current = 5 A optimized the boron removal (i.e., 95%) from a feed solution replicating boron industry wastewater with an initial concentration of 100 mg L^{-1}. Hitherto, the effect of operational parameters via electrocoagulation has been primarily examined for wastewater characterized by rather low boron concentration [16]. For instance, optimal boron removal efficiency (99%) was achieved for synthetic wastewater at lab scale under the following combination of conditions: solution pH = 8, current density = 6 mA cm^{-2}, initial boron concentration = 100 mg L^{-1}, and solution temperature = 20°C [38]. Similarly, Sayiner et al. [39] attained the highest boron removal (70%) for synthetic wastewater at lab scale after combining 50 min of retention time and 30 mA cm^{-2} of current density for an initial boron concentration of 100 mg L^{-1}.

Generally, electrocoagulation is regarded as a developing and energy-consuming process that still calls for optimization and further on-site testing [13,40,41]. More importantly, to the best knowledge of the authors, the effect of operational conditions on removing high boron concentrations via electrocoagulation has not been adequately addressed. A synthetically prepared solution with high boron concentration was, therefore, used to evaluate the effectiveness of the continuous electrocoagulation process for boron removal. Process optimization was attempted through testing the effect of different influent pH values and retention times.

2. Materials and methods

A simulation test bed was prepared to mimic the high boron concentration of industrial wastewater. Synthetic wastewater samples were prepared for the experiments using borax with 99.99% purity from Merck KGaA (Germany). Precisely, a boron solution of 1,000 mg L^{-1} was prepared by dissolving 4,647.28 mg of borax dried at 105°C in 1 L of distilled water.

A lab-scale Plexiglas reactor (16 cm × 8 cm × 8 cm) was used for the experiments. Two groups of aluminum electrodes, alternating between anodes and cathodes by eight plates of each type, were arranged vertically. They were 7 cm × 14 cm in size with an effective surface area of about 1,500 cm^2. The net spacing between them was 5 mm. The electrodes were connected to the terminals of a direct current (DC) power supply with a range of 0–10 A for current and 0–30 V for voltage. Amperemeter and voltmeter were used to measure the current and the applied potential, respectively. Wastewater temperature, conductivity, and pH were also monitored throughout each experimental run (Fig. 2). The moment the DC power supply was switched on was considered as the starting point for each experimental run. At the beginning of each run, the prepared boron solution (i.e., 1,000 mg L^{-1}) was fed into the reactor.

The analytical determination of boron in the samples was conducted by the potentiometric titration of the mannitol/boric acid complex; mannitol forms a complex compound with boric acid. Boron analysis was conducted according to the following protocol: after filtering the samples, solution pH was adjusted to 7.6. 5 g of mannitol were then added to the solution. Following this, the solution was titrated with 0.5 N of potassium hydroxide (KOH) until pH reached 7.6. The boron amount was calculated via the KOH consumption; 1 mL of 0.5 N KOH is equivalent to 17.41 mg of boric acid. [31,38,42]. This method was selected to eliminate any potential aluminum interference in the boron detection; the latter is likely in the case of spectrophotometric methods (e.g., carmine, azomethine-H, and curcumin methods) [43].

In this work, the effect of selected operating parameters (i.e., the influent pH and the retention time) was investigated, by maintaining the same electric current and initial boron concentration in all the experimental runs (Table 1, section 3). The initial pH of the solution is among the most important factors affecting the electrocoagulation process [44,45]. Aluminum hydroxide ($Al(OH)_3$) is amphoteric. Hence, the formation of the $Al(OH)_3$ flocs is highly pH-dependent. In Fig. 1(a), the solubility diagram for $Al(OH)_3$ is provided. The solubility boundary marks the thermodynamic equilibrium that exists between the dominant aluminum species and the $Al(OH)_3$ at a certain pH. Minimum solubility is observed around pH = 6.5; solubility increases as the solution becomes more acidic or alkaline. Within a pH range of 4–9, aluminum complexes with positive charge (e.g., $AlOH^{2+}$, $Al(OH)_2^+$, $Al_2(OH)_2^{4+}$, $Al(OH)_3$, and $Al_{13}(OH)_{32}^{7+}$) and high adsorption capacity are formed. More precisely, the prevailing aluminum form within the pH range 5–8 is $Al(OH)_3$ (Fig. 1(a)). Once the pH goes over 8, the dominant aluminum form is the tetrahydroxyaluminate ion ($Al(OH)_4^-$), which dissolves and does not form flocs [37,45–47]. Furthermore, the boron species depend on the pH value as seen in Fig. 1(b). Above the pH value of 7.5, boron is mainly present in the $B(OH)_4^-$ form. Below the pH value of 7.5, boric acid prevails [37]. In this context, the pH effect on high-concentration boron removal via electrocoagulation was examined by conducting experiments at different influent pH values (i.e., 4, 5, 6, 7.45, and 9). The electric current was kept constant at 5 A, and the retention time was 50 min.

Table 1
The range/values of experimental parameters for this study

Parameter	Range/value
pH	4, 5, 6, 7.45, and 9
Retention time (min)	10, 25, 50, and 100
Electric current (A)	5
Initial boron concentration (mg L^{-1})	1,000
Temperature (°C)	20

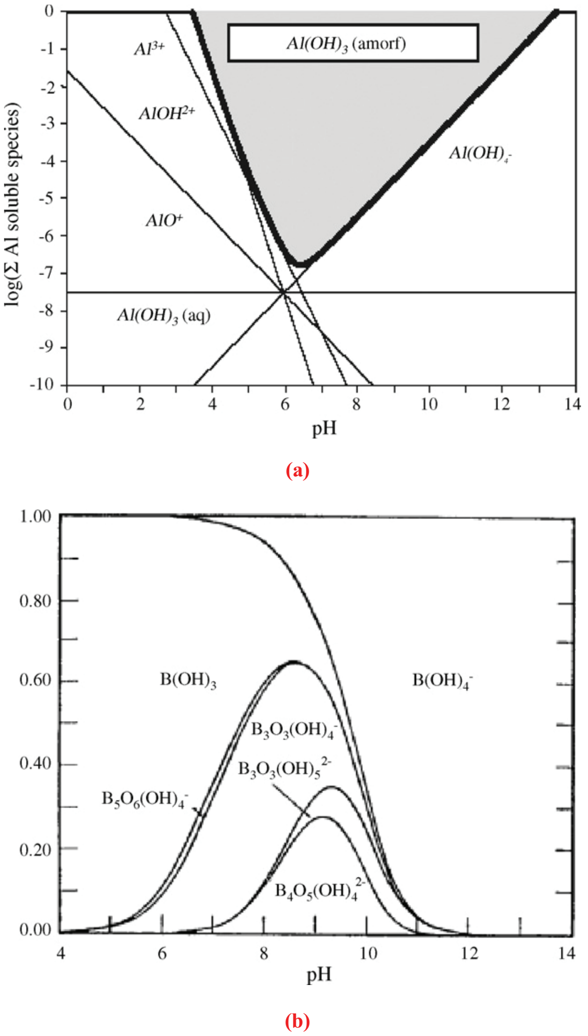

Fig. 1. (a) Dominant aluminum species for different pH values; (b) dominant boron ion species for different pH values [37].

The duration of the electrocoagulation process (i.e., the retention time) is another important factor in determining the level of pollutant removal [45,48]. While exploring the effect of the retention time, the electric current was kept constant at 5 A. The influent pH was adjusted according to the experimental results examining the initial pH effect.

Supporting electrolyte (e.g., calcium chloride [$CaCl_2$]) is used in similar experiments. The electrolyte solution conductivity impacts on the cell resistance. In addition, its properties influence the interaction with the electroactive species, thus affecting the electrode reactions [45,49]. However, investigating the impact of this parameter was out of the scope of this study. Therefore, supporting electrolyte was not used.

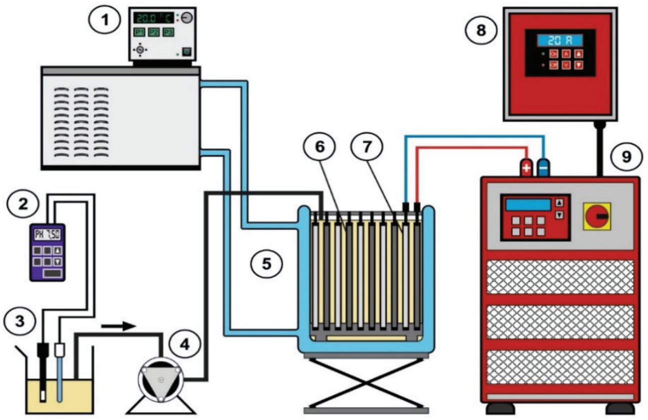

Fig. 2. Process diagram of the experimental setup implemented in this study for the treatment of solutions with high boron content under different influent pH values and retention times. 1: Temperature control; 2: pH & conductivity meter; 3: pH control unit; 4: pump; 5: electrocoagulation cell; 6: anode; 7: cathode; 8: amperemeter, voltmeter; 9: direct current (DC) power supply.

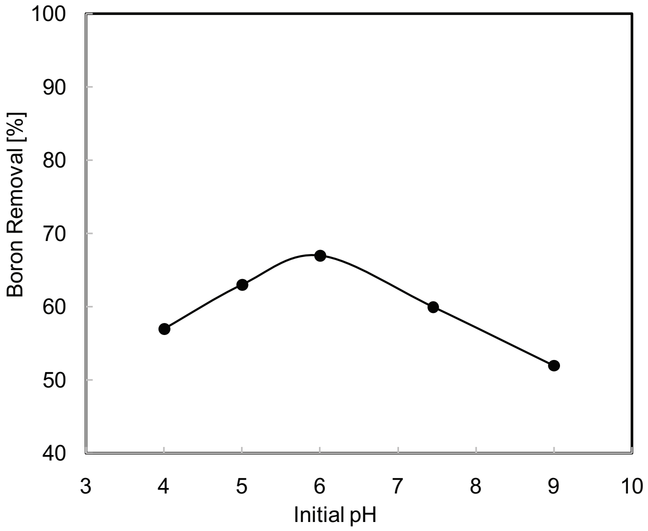

Fig. 3. Effect of different influent pH values on boron removal. The electric current was kept constant at 5 A. Retention time was 50 min.

3. Results

3.1. Initial pH

The results concerning the initial pH effect showed that boron removal increased from 57% to 67% following the pH increase from 4 to 6. On the contrary, further increase in initial pH to 9 resulted in gradually decreasing the boron removal to 52% (Fig. 3). Hence, it can be deduced that the optimum boron removal was achieved at pH = 6. Solid $Al(OH)_3$ is the dominant form especially when the pH is between 6 and 7 [47,50]. Moreover, boron mainly appears in the form of boric acid ($B(OH)_3$) within this pH range [37]. It is likely that an environment with a pH around 6 provides the best condition for boron precipitation through the formation of aluminum borate ($AlBO_3$).

Similarly, Yilmaz et al. [37] examined the efficiency of the electrocoagulation process for an initial boron concentration of 1,000 mg L^{-1} at different pH values (i.e., 4, 6, 8, and 10). The highest removals of 75% and 94% were noted at the pH values of 6 and 8, respectively. Likewise, removals of 80% and 84% were achieved at pH values of 6 and 7, respectively, for an initial boron concentration of 500 mg L^{-1}; the tested pH values were in the range of 4–9 [38]. Analogously, boron removal up to 90% was attained for an initial boron concentration of

100 mgL^{-1} with the pH ranging from 6 to 8; the authors had tested a wide pH interval (i.e., 4–12) [51]. Moreover, Yilmaz et al. [31] treated a solution with an initial boron concentration of 500 mg L^{-1} and tested various pH values (i.e., 4, 5, 6, 7, 8, and 9). Optimum removal (i.e., approximately 90%) was attained under pH = 8. Dolati et al. [27] also concluded that the highest boron removal (i.e., ≈ 70%) was achieved at pH = 8 among the different tested pH values (i.e., 4, 6, 8, and 10) for a boron solution of 100 mg L^{-1}. In all cases, a pH environment ranging from 6 to 8 was favorable to both the $Al(OH)_3$ and the $B(OH)_3$ presence. Under such conditions, boron precipitation via the $AlBO_3$ formation improves. Consequently, it can be concluded that maintaining a pH range that favors the pollutant precipitation with the coagulants will significantly improve the efficiency of the electrocoagulation process. This interval can be between 6 and 8 for the removal of high boron concentration via aluminum electrocoagulation.

3.2. Retention time

While exploring the effect of the retention time, the influent pH was adjusted to 6 which was the optimal pH value in terms of boron removal as discussed in section 3.1. As shown in Fig. 4, comparable changes in the boron removal were observed as the retention time increased. Boron removal equal to 45%, 53%, 65%, and 79% was measured for durations equal to 10, 25, 50, and 100 min, respectively. After 10 min of electrocoagulation, the removal efficiency was quite low (45%). Therefore, an operation time of 10 min was found insufficient to achieve satisfying boron removal. Increasing the retention time caused a considerable rise in the removal rates with the highest one (i.e., 79%) occurring at the highest retention time of 100 min.

Similarly, in the study by Sayiner et al. [39], the boron removal increased from approximately 15% to 70% as the retention time increased from 5 to 60 min for an initial boron concentration of 250 mg L^{-1}. Isa et al. [36] reported that prolonging the retention time from 30 to 90 min resulted in increasing the boron removal from 60% to 85% for an initial boron concentration of 30 mg L^{-1}. While applying aluminum electrocoagulation for the treatment of an initial boron concentration of 100 mg L^{-1}, Dolati et al. [27] noted a significant raise in the boron removal (from 25% to 75%) following the retention time extension from 10 to 120 min. Bektaş et al. [52] treated a high initial boron concentration (i.e., 1,000 mg L^{-1}) via electrocoagulation with aluminum electrodes. They observed that the gradual increase of the retention time (i.e., 5→10→20→30→50 min) significantly enhanced the boron removal (i.e., 30%→60%→75%→87%→96%, respectively). Providing more time for the electrocoagulation process results in increasing the amount of the electro-generated Al^{3+}, thus leading to the production of a higher number of flocs comprising of insoluble monomeric and polymeric aluminum hydroxides. Moreover, this translates into a longer contact time between the heavy metal ions and the flocs. Therefore, more heavy metal ions are removed through adsorption and co-precipitation with the flocs [45,48].

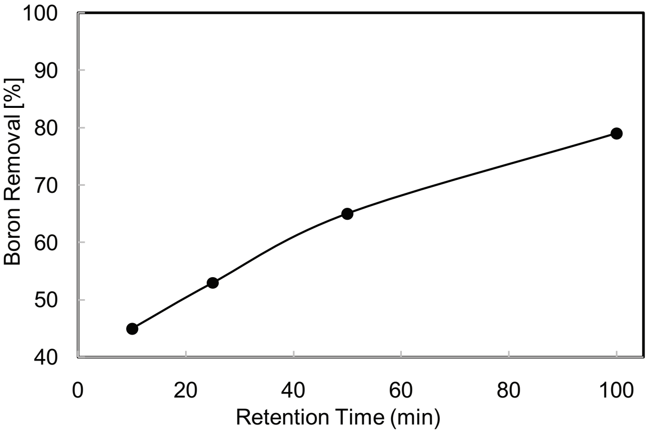

Fig. 4. The effect of retention time on boron removal. The electric current was kept constant at 5 A. Influent pH was adjusted to 6.

4. Conclusions

In this work, the efficiency of the continuous electrocoagulation process with plate-type aluminum electrodes was investigated for the removal of boron from effluents characterized by high initial boron concentrations (1,000 mg L^{-1}). The effect of changing the operational variables of influent pH and retention time was examined. The electric current was kept constant at 5 A throughout the study. The following major conclusions were reached:

- The optimal boron removal (67%) occurred at pH = 6. This result was supported by the pH-related activity for aluminum hydroxides. At pH = 6, solid $Al(OH)_3$ is the dominant aluminum form, thus enhancing boron precipitation via the $AlBO_3$ formation.
- Increasing the electrocoagulation duration from 10 to 100 min increased the removal of boron from 45% to 79%. Extending the time of the electrocoagulation process enabled higher anodic dissolution and, thus, a higher amount of coagulants was released. The existence of more coagulants per unit of pollutants within the reactor led to increased boron removal.

By optimizing the initial pH and the process duration, this work showed that the continuous electrocoagulation process with plate-type aluminum electrodes can be an effective treatment for wastewaters with high boron concentration. Future work can focus on the optimization of other parameters such as current density and temperature.

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