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Three electrode system for *Microcystis aeruginosa* removal on boron-doped diamond and aluminum electrode

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ABSTRACT

Algae in surface water often have an adverse influence in drinking water supplies. Using boron-doped diamond (BDD) and aluminum plate electrodes, the influence factors such as current density, initial pH, environmental temperature, initial algae cell density, Cl^-, SO_4^{2-} and NO_3^- concentration (0.0 mM, 5.0 mM, 10.0 mM, 15.0 mM) on algae removal by electrochemical oxidation and electro-coagulation-flotation were investigated in this paper. The results showed that the algae removal efficiency achieved 100% within 30 min at 2.0 mA/m^2 of current density, initial pH of 7.0, 25 ± 1°C of environmental temperature, and $1.1 \times 10^9 – 1.4 \times 10^9$ cell/L of initial algae density. The removal efficiency of algae increased with the increase of current density, initial algae density and environmental temperature. Neutral and acidic conditions were beneficial to algae removal. Compared with the solution without Cl^-, the Cl^- could promote the removal efficiency of algae. The 10.0 mM and 15.0 mM SO_4^{2-} had a negative effect on the removal of algae. The effect of NO_3^- on the removal of algae was not obvious. The optimal concentration of Cl^-, SO_4^{2-} and NO_3^- were all 5.0 mM.

Keywords: Three electrode system; Boron-doped diamond; Aluminum; *Microcystis aeruginosa*

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1. Introduction

With the contamination by industrial effluents, agricultural activity and domestic wastewater discharge, cyanobacteria (blue green algae) can grow excessively under the high nutrient contents in surface water [1]. There has been an increasing concern on the removal of cyanobacteria in water works. Cyanobacteria can seriously impair surface water quality because of its ability to produce taste and odour compounds [2]. And it can even produce lethal toxic compounds [2,3]. To minimize the threat, different processes have been proposed for algae removal, such as coagulation, sedimentation, flotation, sand filtration [4,5], membrane filtration [3]. Chlorine oxidation, ozonation, oxidation with permanganate and hydrogen peroxide also have been used as pre-oxidation aim to improve the efficiency of next treatment step. But they are expensive, slow and producing harmful by-production [3,6,7].

Electrochemical treatment method had the advantage that there was no need to add any harmful chemicals to the water, appeared to be a promising environmental remediation technology due to its effectiveness, easy operation, and wide application [8]. When active polyvalent metal (e.g. Fe, Al) were used as anodes, flocculating agents flocculate the algae to form algae floc (electro-coagulation). Cathodes generate hydrogen gas micro-bubbles, which float algae to the surfaces of solution (electro-flotation) [9]. Gao et al. [10] obtained excellent removal of algae using aluminum anode. When inactive material (eg Pt, boron-doped diamond (BDD), DSA, Ti/PbO$_2$) were used as anodes, oxidation agents produced by anodes

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might be attacked the algae cells. The algae could be inactivated by direct and indirect oxidation. The inactivated algae cells were easily form algae floc. Mascia et al. [11] obtained effective removal of algae using BDD electrode for pre-oxidation of algae cells. Liang et al. [12] revealed surface damage of algae and leakage of intracellular contents after electrolysis using Ti/RuO_2 anode, and achieved successfully inactivation of algae at 10.0 mA/cm^2 current density. BDD has high performance with many excellent electrochemical features such as wide potential window, high over-potential for oxygen evolution, weak adsorption capability for reactants and oxidative species [13]. Therefore, the three electrode system combining pre-oxidation and coagulation resulted in preferable algae removal within short time.

Thus, the purpose of this study was to evaluate the effectiveness of the three electrode system to remove algae. The anodes were BDD and aluminum plate. The cathode was stainless steel. Using the effect of electro- coagulation-flotation (aluminum anode) and oxidation (BDD anode), the algae were easy to form algal floc after inactivating by oxidation species (produced on BDD anode) in the three electrodes system. In order to determine the effect of operating variables on the removal of algae, current density, the initial pH, the initial algae cells density, the environmental temperature, and the supporting electrolyte were studied.

2. Materials and methods

2.1. Algae culture

Microcystis aeruginosa was purchased from Wuhan Institute of Hydrobiology of Chinese Academy of Sciences (FACHB-315). They were incubated with BG-11 medium in a chemostat at 26 ± 1°C under illumination (3000Lux) on a 14 h light/10 h dark cycle [14]. The growth curve was drawn by monitoring the algae density.

2.2. Apparatus

As shown in Fig. 1, the experimental apparatus consisted of a DC power supplier, a magnetic stirrer, and an electrochemical reactor. The columniform reactor (height = 10.2 cm, diameter = 12.0 cm) is a polypropylene column with an effective column of 1000 mL. And the sampling place is located 6 cm above the bottom of the reactor. The three-electrode device has a cathode (stainless steel AISI201) and two anodes (BDD, lined with tantalum; and aluminum 1060, which approximate purity was 99.6%.). The cathode is placed in the middle of two anodes, with 1.0 cm inter-electrode gap. And the effective area of the three electrodes (length =, height =) are 29.25 cm^2. A constant current provided by a M8872 DC power supplier (Maynuo Electronics, China).

2.3. Experimental procedure

Batch static experiments were performed at 25 ± 1°C. When the parameters as current density, initial pH, environmental temperature, initial algae cell density was investigated, the *M. aeruginosa* was diluted with deionized water to 1.1×10^9–1.4×10^9 cell/L after cultivating for 7 days (at the logarithmic growth period). The initial pH of raw water was adjusted to desirable values using 0.1M NaOH, or 0.1 M H_2SO_4. When the Cl^-, SO_4^{2-} and NO_3^- concentration (0.0 mM, 5.0 mM, 10.0 mM, 15.0 mM) was investigated, the M. aeruginosa was filtered through 0.45 μm membrane after cultivating for 7 days. The algae cells were then transferred to fresh BG-11 medium without Cl^- or SO_4^{2-} and NO_3^- as raw water. Different concentration of NaCl or Na_2SO_4 or $NaNO_3$ added to the raw water as testing water, respectively. The initial pH of testing water was adjusted to desirable values using 0.1 M NaOH, 0.1 M H_2SO_4 or 0.1 M HCl. After each batch experiment, the solution was allowed to settle for 15 min for sedimentation or flotation. The method used by Brookes et al. [15], *M. aeruginosa* counted with a microscope (BA310, MOTIC CHINA GROUP CO.LTD). The pH was determined by a portable pH meter (HACH, America).

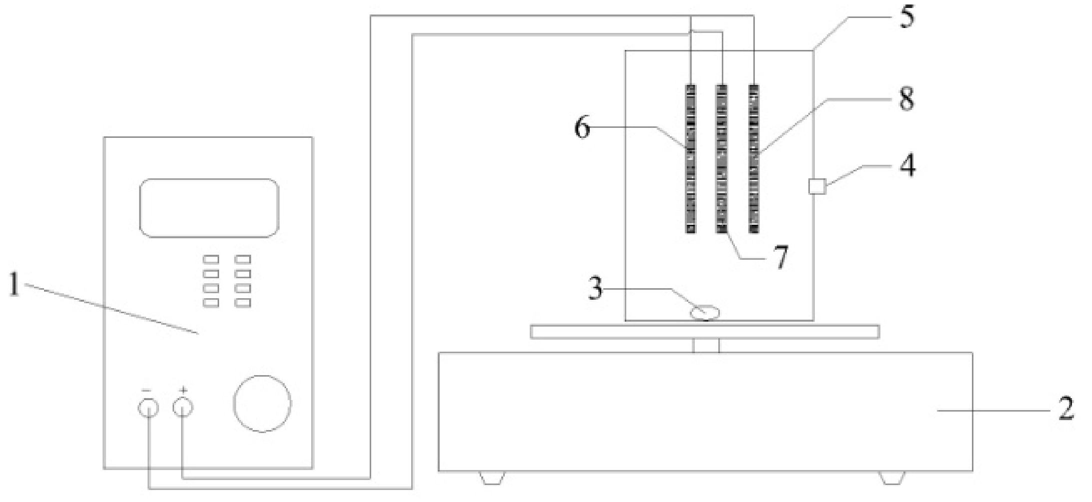

Fig. 1. An illustration of the experimental system. (1) DC power supplier; (2) magnetic stirrer; (3) magnetic bar-stirrer; (4) Sampling place; (5) cell; (6) BDD anode ; (7) stainless steel cathode; (8) aluminum anode.

The removal efficiency of *M. aeruginosa* was expressed as follows [10]:

$$\text{Algae removal efficiency } (\%) = \frac{C_0 - C_t}{C_0} \times 100 \qquad (1)$$

where C_0 was the initial cell density of *M. aeruginosa* and C_t was the final cell density of *M. aeruginosa* (cell/L).

3. Results and discussion

3.1. Effect of applied current density

Current density and power-on time not only determined the production of flocculants and active material [10,12], but also affected the mass transfer of solution between the three electrodes and the mixing of solution [16,17]. Thus, the algae removal efficiency under different current density of 0.5 mA/cm², 1.0 mA/cm², 1.5 mA/cm², 2.0 mA/cm², 3.0 mA/cm² and 4.0 mA/cm² in the three electrodes system was evaluated.

As shown in Fig. 2, the algae removal efficiency increased while the current density increased at a certain time. The algae removal efficiency reached up to 100% after 10 min at a current density of 3.0 mA/cm². When the current density was 2.0 mA/cm², 90% and 99.1% removal efficiency can be achieved at 20 min and 30 min. Algae can be completely removed at 30 min. However 40 min would be required to achieve 90% removal efficiency of algae at a current density of 1.5 mA/cm². It might be explained that more hydroxyl radical and Al^{3+} were produced in three electrodes system at a higher current density, which could promote the flocculation of algae cells. The hydroxyl radical could attacked the cell membrane and wall, disrupted the integrity of cell membrane, or electrolyzed the molecules in the cell surface, which resulted in a large number of cell death and lysis [18]. The Al^{3+} polymerized into polynuclear hydroxyl complexes, resulted in flocculation reaction. Moreover, the higher current density could generate more bubbles by electrolysis. Thus algae floc floated on the solution surface along with those bubbles. However, when the current density and the reaction rate increased, the energy consumption increased correspondingly. And the polarization and passivation phenomena of the electrode were also accelerated. The rapid release of Al^{3+} could easily lead to an excess of Al^{3+} in solution [19]. Considering the energy consumption at different current density, current density of 2.0 mA/cm² should be appropriate for the three electrodes system for algae removal.

3.2. Effect of initial pH

In previous studies, the initial pH was one of the key parameters influencing the performance of electrochemistry process [20,21]. Thus, the algae removal effective under different initial pH of 5.0, 6.0, 7.0, 8.0, 9.0 was evaluated.

As shown in Fig. 3, low initial pH was beneficial to algae removal. Especially when the initial pH was 6.0, the removal of algae achieved 70% at 10 min and 100% at 20 min. However, when the initial pH was in alkaline range, the removal efficiency of algae declined with the increase of initial pH. It takes at least 60 min to remove the algae completely. This might be explained in the following aspects: Firstly, the changing of pH could not only affect the morphology of Al^{3+} in solution, but also affect the formation of colloid on anode surface [22]. When the pH was between 4.5 and 6.0, the main form of Al^{3+} was positively charged polynuclear complexes. Therefore, negatively charged algae would be easily absorbed onto the positively charged polynuclear complexes, and floated on the solution surface along with bubbles. When the pH was in the range of 7.0–7.5, the coagulation mechanism was charge neutralization and adsorption bridging. In alkaline conditions, the main form of Al^{3+} was aluminum hydroxide precipitates. The removal mechanism of algae was enmeshment action. Besides, the main active chlorine species was HClO under the pH of 2.0–8.0, which promoted the oxidation of algae cells because of its electronegativity. Secondly, the Zeta potential of solution decreased

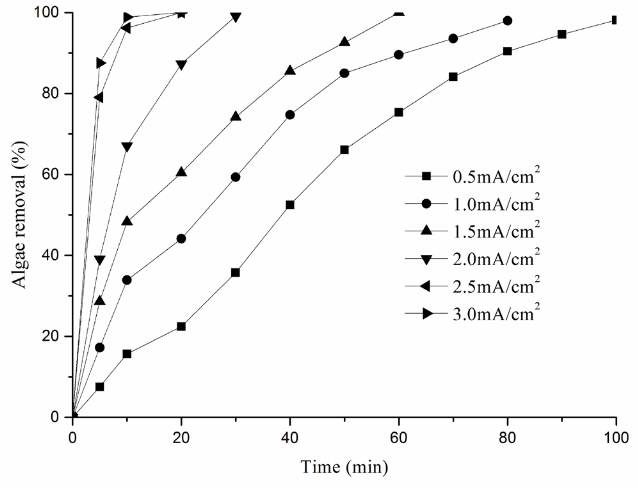

Fig. 2. Algae removal rate as a function of time under different current density. Initial pH: 7.0; the plate distance: 1.0 cm; volume: 1.0 L; initial cell density: 1.1×10^9–1.4×10^9 cell/L.

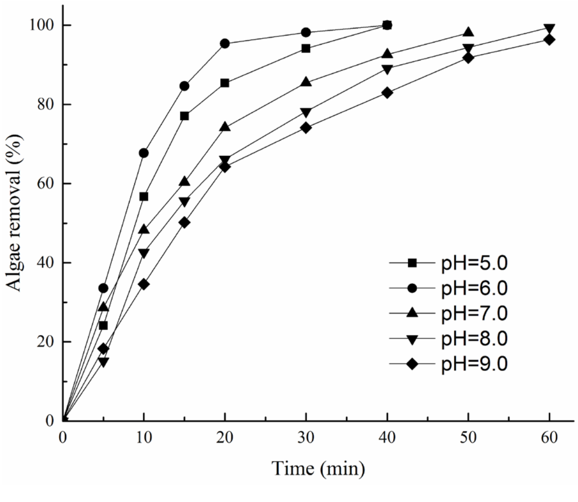

Fig. 3. Algae removal rate as a function of time under different initial pH. Current density: 1.5 mA/cm²; the plate distance: 1.0 cm; volume: 1.0 L; initial cell density: 1.1×10^9–1.4×10^9 cell/L.

with the increased of pH, which will increase the flocculation difficulty of algal cells. Thirdly, under acidic conditions, the stronger chemical dissolution of the aluminum electrode could generate more Al^{3+} to promote the reaction rate and the removal efficiency. Lastly, the particle sizes of bubbles (hydrogen and oxygen) were affected by pH. Under alkaline conditions, these gas bubbles will become larger, which is not conducive to flotation separation process, resulting in the lower removal efficiency of algae [23].

3.3. Effect of initial algae cells density

With the different seasons and various hyper-eutrophication of water, algae cell density is constantly changing. Thus, the algae removal efficiency with different algae cell density (0.5×10^9–2.0×10^9 cell/L) in the three electrodes system was evaluated.

As shown in Fig. 4, the algae removal efficiency decreased with the increase of initial algal cell density. When the initial density of algae increased from 1.5×10^9 cell/L to 2.0×10^9 cell/L, the time needed to completely remove the algal cells increased from 30 min to 50 min. This was due to the electrochemical treatment load and the algal cell density was positively correlated at the same current density. Besides, the flocculation and active substance generated by electro-coagulation and electrochemical oxidation was relatively stable at a certain current density and power-on time [10,24]. Therefore, higher algal cells density resulted in a relative shortage of the active substance, which required a longer time to generate a sufficient amount of the flocculated material for completely removal.

This indicated that complete removal of algal cells could be achieved over longer time or higher current density at higher initial algae density.

3.4. Effect of environmental temperature

Generally, the algae blooms break out in spring, summer and autumn. The temperature difference between different seasons can be up to 20°C. Thus, the environmental temperature is an important parameter affecting the removal of algae. In previous studies, the different environmental temperature showed different effects on electro-coagulation for the removal of different pollutants [25,26]. Thus, the algae removal efficiencies of the three electrode systems at different ambient temperatures (15°C, 25°C and 35°C) were evaluated.

As shown in Fig. 5, under the same reaction conditions, the algae removal efficiency increased with increasing temperature. Particularly at the initial stage, the algae removal efficiency obtained 21% and 61% at temperature of 15°C and 35°C after 5 min. This is mainly due to the hydration of colloidal particles increased with the decrease of water temperature, resulting in the water viscosity in hydration film became larger, which is not conducive to mutual adhesion between colloidal particles. Besides, Brownian motion of the particles was also closely related to the temperature, that is, at higher temperature showed more activity, thereby increasing the possibility of collision between substances [27]. In addition, Vepsalainen et al. [28] found that the current efficiency of aluminum anode dramatically improved with the increasing temperature, leading to the enlargement of the dissolving rate of aluminum anode and the releasing rate of Al^{3+}, which increased the production of flocculants.

3.5. Effect of the supporting electrolyte type

The electrolyte in solution could influence the removal effects as it modifies the conductivity of the effluent, facilitates the passage of the electrical current, and allows the production of some oxidant agents [29]. Thus, the effect of anions, such as Cl^-, SO_4^{2-} and NO_3^- on the removal of algae in the three electrodes system was evaluated.

3.5.1. Effect of Cl^- on the removal of algae

Fig. 6 shows that the adding of Cl^- to solution could increase the removal efficiency of algae. When the concen-

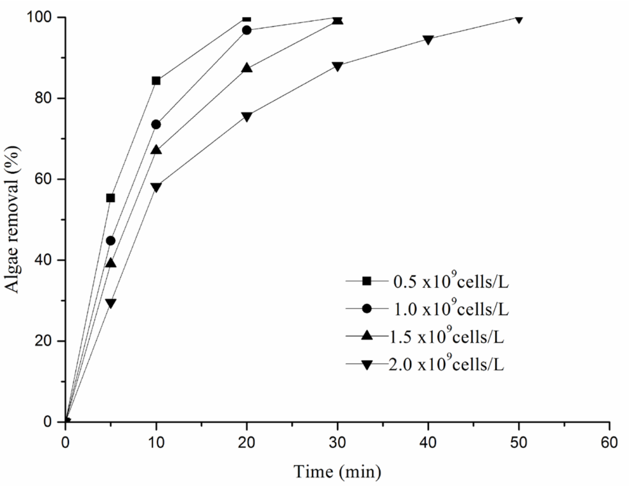

Fig. 4. Algae removal rate as a function of time under different initial algae cell density. Initial pH: 7.0; current density: 2.0 mA/cm^2; the plate distance: 1.0 cm; volume: 1.0 L.

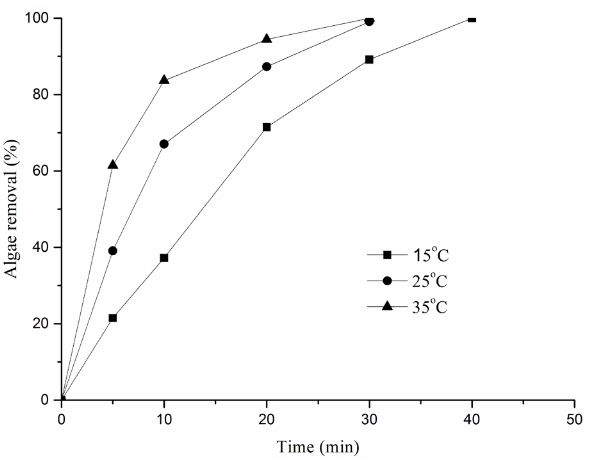

Fig. 5. Algae removal rate as a function of time under different environmental temperature. Initial pH: 7.0; current density: 2.0 mA/cm^2; the plate distance: 1.0 cm; volume: 1.0 L; initial cell density: 1.1×10^9–1.4×10^9 cell/L.

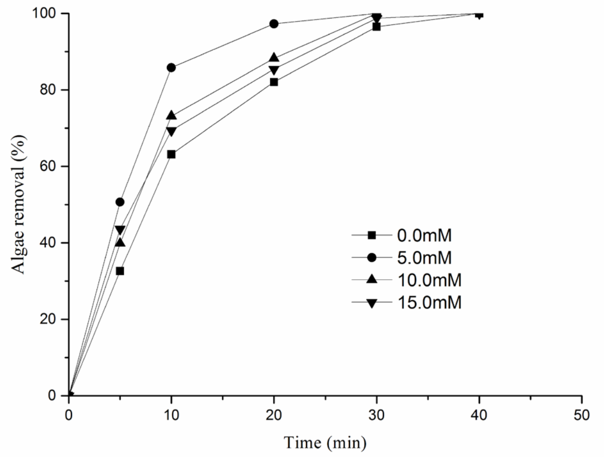

Fig. 6. Algae removal rate as a function of time under different concentrations of Cl^-. Initial pH: 7.0; current density: 2.0 mA/cm^2; the plate distance: 1.0 cm; volume: 1.0 L; initial cell density: 1.1 × 10^9–1.4 × 10^9 cell/L. The initial pH was adjusted by 0.1 M H_2SO_4.

tration of Cl^- was 5.0 mM, the removal efficiency of algae was 85.8% at 10 min, which was higher than that of the solution without Cl^- (about 22.6%). However, when the concentration of Cl^- was 10.0 mM, 15.0 mM, the removal efficiency of algae was lower than the concentration of 5.0 mM. Through the above analysis showed that Cl^- promoted the removal of algae in three electrodes system. Firstly, the Cl^- could generated oxidizing species (Cl_2, and HClO) on the anode surface in the electrolysis process, which can be expressed as [13]:

$$2Cl^- \rightarrow Cl_2 + 2e^- \tag{2}$$

$$BDD + H_2O \rightarrow BDD(\cdot OH) + H^+ + e^- \tag{3}$$

$$BDD(\cdot OH) + Cl^- \rightarrow BDD + 1/2\ Cl_2 + OH^- \tag{4}$$

$$Cl_2 + H_2O \rightarrow HClO + H^+ + Cl^- \tag{5}$$

These oxidizing species could inactivate algae cells. Besides, the micro-bubbles formed by Cl_2 have a flotation effect on algae cells. Secondly, Cl^- could promote the surface activation and delay the passivation of aluminum electrode, and increase the release of Al^{3+} by pitting corrosion. As shown in Fig. 7a and 7b, the surface of the aluminum electrode formed such deep pits, which verified the effect of pitting corrosion of Cl^- on aluminum anode [30].

However, excessive Cl^- could not be completely electrolyzed. These remaining Cl^- could attached to the surface of Al $(OH)_3$ precipitate, increasing the negative charged of Al $(OH)_3$ precipitate or decreasing the positively charged of hydrolyzate of aluminum polymerization [31]. That was not beneficial to the removal of algae because of the reduction of flocculants.

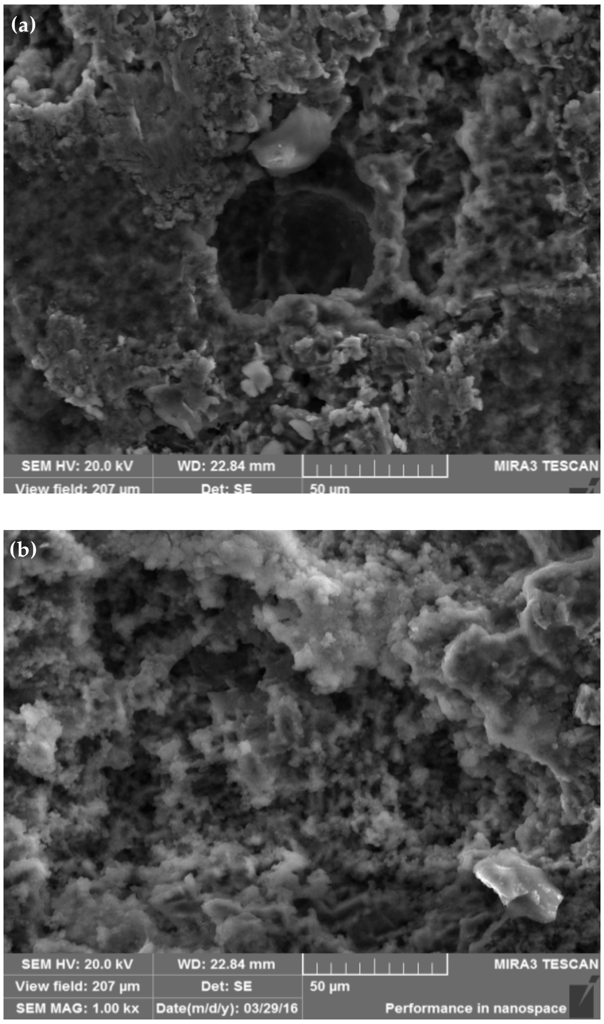

Fig. 7. (a) The scanning electron microscopy image of aluminum electrode with Cl^-. (b) The scanning electron microscopy image of aluminum electrode without Cl^-.

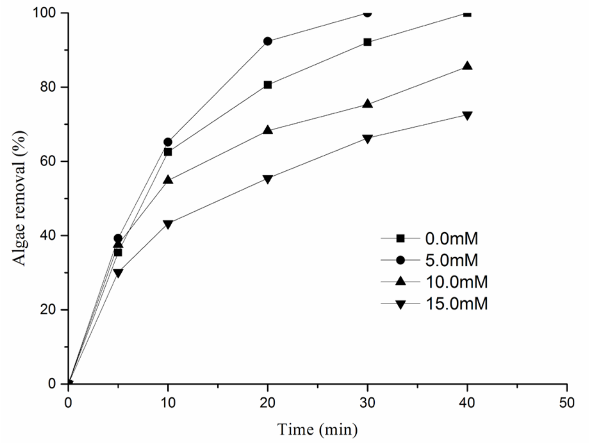

Fig. 8. Algae removal rate as a function of time under different concentrations of SO_4^{2-}. Initial pH: 7.0; current density: 2.0 mA/cm^2; the plate distance: 1.0 cm; volume: 1.0 L; initial cell density: 1.1 × 10^9–1.4 × 10^9 cell/L. The initial pH was adjusted by 0.1 M HCl.

3.5.2. *Effect of SO_4^{2-} on the removal of algae*

From Fig 8, when the concentration of SO_4^{2-} were 0.0 mM, 5.0 mM, 10.0 mM, 15.0 mM, the removal efficiency of

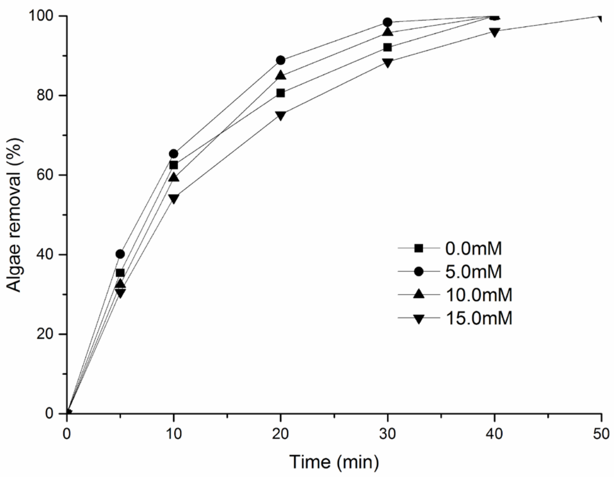

Fig. 9. Algae removal rate as a function of time under different concentrations of NO_3^-. Initial pH: 7.0; current density: 2.0 mA/cm^2; the plate distance: 1.0 cm; volume: 1.0 L; initial cell density: 1.1 × 10^9–1.4 × 10^9 cell/L. The initial pH was adjusted by 0.1 M HCl.

algae were 80%, 92%, 68%, 56% at 20 min. It was concluded that the appropriate concentration of SO_4^{2-} could promote the removal of algae, but would reduce the algae removal efficiency at a higher concentration SO_4^{2-}.

This was mainly due to the electrolyte could promoted floc colloid aggregation and settlement, which improving the efficiency of algae removal [31]. However when the concentration of Na_2SO_4 was too high, SO_4^{2-} could be adsorbed on the surface of the aluminum electrode and formed a layer of insoluble sulfates film, leading to the augment of anode surface passivation. That was not conducive to the anode dissolution and the release of Al^{3+}. In addition, SO_4^{2-} would help to generate $Al(OH)_3$ precipitate and prevent the formation of polymeric aluminum. Meanwhile, SO_4^{2-} could be attached to the surface of $Al(OH)_3$, which increased negative charge of SO_4^{2-} or declined positively charge of polymeric aluminum. All of these increased the difficulty of flocculation for negatively charged algal cells [19].

3.5.3. Effect of NO_3^- *on the removal of algae*

From Fig 9, the effect of NO_3^- on the removal of algae was not obvious. The highest removal efficiency of algae was recorded at the concentration of 5.0 mM. Firstly, NO_3^- was not possible to produce an oxidant agent in solution [32], that is, it could not promoted the inactivation of algae. Secondly, electro-flotation was affected by the concentration of NO_3^-. Chen et al. [33] find that with the increase of NO_3^- concentration, the removal efficiency decreased. In short, it can be obtained that the concentration of NO_3^- have little effect on the removal of algae.

4. Conclusions

The above results suggested that the three electrodes system was a method for the treatment of algae. The following conclusions were drawn:

1. The removal efficiency of algae increased with the increase of current density. Considering the energy consumption at different current density, current density of 2.0 mA/cm^2 should be appropriate for the three electrodes system for algae removal.
2. The algae removal efficiency was increased under acid condition. Especially when the initial pH was 6.0, the removal of algae achieved 70% at 10 min, 100% at 20 min. However, the removal efficiency of algae would decrease with the increasing initial pH under alkaline condition.
3. It needed longer time to achieve completely removal of algae with higher initial cell density. The higher environmental temperature could promote the removal efficiency of algae and shorten the electrolysis time.
4. Cl^- could promote the removal efficiency of algae. 10.0 mM and 15.0 mM SO_4^{2-} had a negative effect on the removal of algae. The effect of NO_3^- on the removal of algae was not obvious. The optimal concentration of Cl^-, SO_4^{2-} and NO_3^- were all 5.0 mM.

Acknowledgements

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