Research Article

Mineralization of Quinoline by BDD Anodes: Variable Effects and Its Effluent Characteristics

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BDD anodes were selected for quinoline mineralization and influence of operating parameters, such as current density, initial quinoline concentration, supporting electrolyte, and initial pH was investigated. Based on the consideration of quinoline removal efficiency and average current efficiency, at initial quinoline concentration of 50 mg L^{-1} and pH of 7, the optimal condition was confirmed as current density of 75 mA cm^{-2} , electrolysis time of 1.5 h, and Na_2SO_4 concentration of 0.05 mol L^{-1} by orthogonal test. At different electrolysis time, its effluent characteristics were focused on. The biodegradability (the ratio between BOD_5 and COD) was enhanced from initial 0.02 to 0.57 at 90 min. The specific oxygen uptake rate was used to assess effluent toxicity, and the value gradually reduced with decreasing effluent organic concentration with mean value of 5.51, 4.19, and $2.20 \text{ mgO}_2 \text{ g}^{-1} \text{ MLSS}$ at electrolysis time of 15, 30, and 45 min, respectively. Compared with control sample (prepared with glucose), the effluent of quinoline mineralization showed obvious inhibition effect on microorganisms at electrolysis time of 15 min, and then it was significantly faded at 30 min and 45 min.

1. Introduction

Quinoline, as a typical heterocyclic aromatic hydrocarbon, is mainly derived from coal coking and coal gasification. Because of its pollution persistence and endocrine disturbance, it has been listed as priority pollutant by US Environmental Protection Agency (EPA) [1]. Therefore, the quinoline from coking wastewater and coal gasification wastewater should be removed efficiently. However, the practice shows that the coal coking wastewater and coal gasification wastewater containing heterocyclic aromatic hydrocarbons, such as quinoline, are characteristics of poor biodegradability and not helpful for advanced biological treatment [2]. So advanced oxidation processes (AOPs), such as Fenton oxidation, ozonation, wet air oxidation, and electrochemical oxidation, must be considered as possible methods for enhancement of biodegradability. In the above methods, electrochemical oxidation using boron-doped diamond (BDD) appears to be the most suitable technology because of its extremely wide potential window and very low background current [3–5]. Moreover, it has been used for the degradation of different organic pollutants, such as phenol and its

derivatives [6–9], oxalic and oxamic acids [10], salicylic acid [11], sulfanilic acid [12], carmoisine [13], tetrahydrofuran [14], and phenanthrene and cyclodextrin [15]. In conclusion, the above organics degrading by BDD anodes mainly focused on the following four aspects: (1) investigating variable effects; for example, Rabaaoui and Allagui discussed the variable effects of salicylic acid oxidation by BDD anodes in the pH range 3.0–10.0 and different current density and focused on the oxidation products [11]; (2) identifying oxidation pathway; for example, Zhu et al. investigated the oxidation mechanisms of *p*-substituted phenols at BDD anodes [16]; (3) exploring degradation kinetics; for example, Díaz et al. discussed the kinetics of electrooxidation of ammonia, nitrites, and COD from aquaculture saline water system using BDD anodes and found that the ammonia removal was described by a second-order kinetics, while COD and nitrite removal followed zeroth-order kinetics [17]; (4) comparing the organics removal efficiency between BDD anodes and other electrode materials; for example, Bagastyo et al. investigated the electrochemical oxidation of reverse osmosis concentrate on Ti/Pt- IrO_2 , Ti/ SnO_2 -Sb, and BDD anodes and

found that dissolved organic carbon removal was enhanced at BDD anodes in the presence of SO_4^{2-} [18].

In this paper, one of the aims was to determine main variable effects and their optimal conditions for quinoline mineralization using BDD anodes. However, the difference from previous research was that variable effects considered not only the pollutant removal efficiency but also the average current efficiency. More importantly, its effluent characteristics including the biodegradability and microbial toxicity were focused on at different electrolysis time. And the results will help to gain basic reference for selecting advanced treatment technology for quinoline wastewater.

2. Materials and Methods

2.1. Electrolytic System and Chemicals. The electrochemical oxidation of quinoline was carried out in a 3L electrolysis cell equipped with a magnetic stirrer and direct current (DC) power supply. The BDD anodes were purchased from the company of CONDIAS GmbH with electrode size of $195 \text{ mm} \times 26 \text{ mm} \times 2 \text{ mm}$ (length $\times$ width $\times$ thickness), and 312 stainless steel plates were used as cathodes with 1.0 cm of interelectrode gap.

Methanol and acetic acid used as mobile phase in HPLC were purchased from Dikma. Quinoline used was purchased from Sigma. All other chemicals used for the preparation of quinoline wastewater and other studies were of analytical grade with more than 99% purity. And water used for solution preparation was ultrapure water with resistivity of $18.2 \text{ M}\Omega\text{cm}$.

2.2. Instruments and Analytical Methods. The pH of solution was determined using a pH meter (HQ30d-PHC101, HACH, USA). COD was measured by a COD meter (ET99722, Hanna Corporation, Italy). Total organic carbon (TOC) was monitored with TOC/TN meter (multi N/C 2100, Analytik Jena AG Corporation, Germany). BOD_5 was determined employing the dilution inoculation method [19]. Quinoline concentration was quantified by HPLC (LC-10Avp Plus, Shimadzu, Japan) with column of Wondasil C18 ($4.6 \text{ mm} \times 150 \text{ mm}$, $5 \mu\text{m}$). The mobile phase was composed of methanol, water, and acetic acid (50 : 49 : 1, v/v) with flow rate of 0.4 mL/min . Quinoline was detected at 280 nm . The concentration of mixed liquid suspended solids (MLSS) was determined by the gravimetric method [16]. Dissolved oxygen (DO) was detected by a DO meter (LDO10103, HACH, USA). The average current efficiency (ACE) was calculated using the following equation [20]:

$$\text{ACE} = \frac{4FV[(\text{COD})_0 - (\text{COD})_t]}{32It}, \quad (1)$$

where F is the Faraday constant (96487 C mol^{-1}), V is volume of treated wastewater (in L), $(\text{COD})_0$ is initial COD concentration (in g L^{-1}), $(\text{COD})_t$ is COD concentration at time t (in g L^{-1}), I is current (in A), t is time (in s), 4 is number of exchanged electrons for the electrochemical oxidation of COD, and 32 is molecular mass of O_2 (in g mol^{-1}).

3. Results and Discussion

3.1. Effect of Operating Parameters on Quinoline Mineralization

3.1.1. Effect of Current Density. Current density is an important factor affecting the electrolysis kinetics and process economics. It corresponds to the ratio between the applied current and the surface of the working electrode [11]. As can be seen in Figure 1, at initial quinoline concentration of 50 mg L^{-1} , Na_2SO_4 concentration of 0.05 mol L^{-1} , and initial pH of 7, when current density was variable with its value of 25, 50, 75, and 100 mA cm^{-2} , respectively, the impacts on quinoline and TOC removal were analyzed. The results showed that, with increasing of current density, removal efficiency of quinoline and TOC significantly increased. At current density of 100 mA cm^{-2} and electrolysis time of two hours, mineralization rate of quinoline was approximately up to 96.2%; however, removal efficiency of TOC was only 80.0%. The reason that, with increasing current density, the higher the concentration of $\cdot\text{OH}$ produced by BDD anodes, the more the quinoline oxidized by $\cdot\text{OH}$ was confirmed, but when current density was high enough, removal efficiency of TOC was more slowly enhanced than degradation rate of quinoline, which resulted from that in the mineralization process of quinoline; it transferred to other intermediate products; as a consequence, the concentration of quinoline decreased, but these intermediate products had not been further mineralized.

3.1.2. Effect of Initial Quinoline Concentration. The influence of initial quinoline concentration on its mineralization process was also investigated. To do this, solutions containing 50 mg L^{-1} , 80 mg L^{-1} , and 100 mg L^{-1} of this compound were comparatively degraded by BDD anodes at Na_2SO_4 of 0.05 mol L^{-1} , initial pH of 7, and current density of 75 mA cm^{-2} . Figure 2 shows that, at initial quinoline concentration of 50 mg L^{-1} and electrolysis time of two hours, removal efficiency of quinoline and TOC was up to 96.6% and 79.2%, respectively. However, when initial quinoline concentration was improved to 100 mg L^{-1} , the above value was 94.2% and 77.5%, respectively. It also could be seen that, with the increasing of initial quinoline concentration, removal efficiency of TOC and quinoline gradually dropped, and when the initial quinoline concentration was higher, electrochemical oxidation for quinoline was significantly inhibited which is because when current density, electrolyte concentration, electrolysis time, and initial pH were constant, the quantity of $\cdot\text{OH}$ produced on the surface of electrode was also constant. Therefore, at lower initial pollutant concentration, $\cdot\text{OH}$ produced on the surface of electrode could completely react with quinoline at shorter electrolysis time. However, at higher quinoline concentration, $\cdot\text{OH}$ was relatively not enough for oxidizing quinoline and its intermediates, resulting in a decrease in mineralization efficiency.

3.1.3. Effect of Electrolyte Type. Under the conditions of initial quinoline concentration of 50 mg L^{-1} , current density of

FIGURE 1: Effect of current density on quinoline degradation (a) and TOC removal (b).

FIGURE 2: Effect of initial mass concentration on quinoline degradation (a) and TOC removal (b).

75 mA cm⁻², and initial pH of 7, different kinds of electrolytes with same molar concentration of 0.05 mol L⁻¹, including Na₂SO₄, NaCl, Na₂CO₃, and NaHCO₃, were added into BDD system, and the influence was observed from Figure 3.

As shown in the figure, the highest quinoline removal efficiency was obtained at electrolytes of Na₂SO₄ with its value of 96.6%; the reason for this phenomenon is that Na₂SO₄ will not involve oxidizing organic substances in the process of electrolysis. However, NaCl participates in the degrading reaction and produces Cl₂ at anode, which further reacts with H₂O and forms HClO. So NaCl selected as electrolyte is helpful for ammonia removal [21], but no proof showed that

it could accelerate quinoline mineralization. In contrast, the existence of the above matters would decrease the current efficiency. Additionally, compared with Na₂SO₄ as electrolyte, the quinoline removal efficiency was also lower at Na₂CO₃ or NaHCO₃ as electrolytes, which was attributed to the fact that both CO₃²⁻ and HCO₃⁻ could consume ·OH; namely,

Thus Na₂CO₃ and NaHCO₃ were able to cut down concentration of ·OH in wastewater and then declined

FIGURE 3: Effect of electrolyte type on quinoline degradation (a) and TOC removal (b).

quinoline removal efficiency. Therefore, in this paper, Na₂SO₄ was selected as optimal electrolyte.

3.1.4. pH Dependence. Due to the fact that initial pH can exert a significant influence on the generation of hydroxyl radicals, a solution with 50 mg L⁻¹ of quinoline was electrolyzed at initial pH 5.0, 6.0, 7.0, 8.0, and 9.0, with Na₂SO₄ concentration of 0.05 mol L⁻¹, current density of 75 mA cm⁻², and electrolysis time of two hours. As shown in Figure 4, the removal efficiencies of quinoline and TOC were better in the slight alkaline medium; especially, at pH of 9, quinoline and TOC removal efficiency were 99.2% and 80.8%, respectively, which was attributed to that negative charge existed on the surface of the electrode in the slight alkaline medium; OH⁻ is absorbed in the close layer of the electric double layer; as the outer layer of electrodes reacts, active oxygen in the metal crystal lattice will oxidize OH⁻, thus resulting in electron transfer and then producing a large number of $\cdot\text{OH}$; namely,

In addition, at different pH values, the dissociation constant of quinoline also varies [22]. At neutral or alkaline medium, quinoline mainly appears in solution as ion, but at acidic medium, quinoline exists in the form of molecule. As we all know, the electrophilic reaction occurs more easily for ion substance than for molecular substance.

3.1.5. Orthogonal Experiment for Quinoline Removal and Average Current Efficiency. The above factors' experimental results showed that, at specific initial quinoline concentration, electrolysis time, current density, electrolyte type, and initial pH, all factors had influence on quinoline removal,

in which Na₂SO₄ was confirmed as optimal electrolyte and pH showed better quinoline removal in the slight alkaline medium. However, compared with the results at pH of 9 and pH of 7, it was found that pH had slightly influence on quinoline removal at higher pH. Therefore, at initial quinoline concentration of 50 mg L⁻¹ and pH of 7, electrolysis time, current density, and Na₂SO₄ concentration would play major roles for quinoline removal. Thus the orthogonal test L₉(3)³ table was designed to confirm the optimal electrolysis condition (Table 1). Meanwhile average current efficiency, as an important index for evaluating electrode property, was also calculated (based on (1)), and the results are shown in Table 2.

According to Table 2, in terms of quinoline removal, electrolysis time (B) extends the most significant effect and the order of significant effect on quinoline removal efficiency was electrolysis time (B) > current density (A) > Na₂SO₄ concentration (C). However, in terms of average current efficiency, current density (A) extends the most significant effect and the order of significant effect on average current efficiency was current density (A) > Na₂SO₄ concentration (C) > electrolysis time (B). Taking the above-mentioned two facts into consideration, a compromised optimal condition was selected as A₂B₂C₃, namely, current density of 75 mA cm⁻², electrolysis time of 1.5 h, and Na₂SO₄ concentration of 0.05 mol L⁻¹ with quinoline removal efficiency of 85.9% and average current efficiency of 4.17 × 10⁻².

3.2. Effluent Characteristics of Quinoline Mineralization by BDD Anodes

3.2.1. Variation of BOD₅/COD. The COD and BOD₅ of quinoline mineralization by BDD anodes were measured every 15 minutes under the above optimal conditions (Figure 5). As the electrolysis time increased, BOD₅ of effluent gradually

FIGURE 4: Effect of pH on quinoline degradation (a) and TOC removal (b).

TABLE 1: Factors and levels of orthogonal test.

Levels	Variables		
	A (current density) (mA cm ⁻²)	B (electrolysis time) (h)	C (Na ₂ SO ₄ concentration) (mol L ⁻¹)
1	50	1.0	0.03
2	75	1.5	0.04
3	100	2.0	0.05

FIGURE 5: COD, BOD₅, and BOD₅/COD of quinoline effluent at different electrolysis time.

increased from an initial value of 2.02 mg L⁻¹ to 16.61 mg L⁻¹ at 1.5 h, and the COD of effluent gradually declined from an initial value of 117.1 mg L⁻¹ to 28.8 mg L⁻¹ at 1.5 h. Meanwhile, the BOD₅/COD ratio increased from an initial value of 0.02 to 0.57 at 90 min, which indicates that the biodegradability of quinoline effluent increased with electrolysis time.

3.2.2. Variation of SOUR. The specific oxygen uptake rate (SOUR) can be used in a simple static test to assess the composition stability and toxicity of wastewater [23, 24]. Therefore, the effluent SOUR values of quinoline mineralization by BDD anodes were measured at 15, 30, and 45 min under the above optimal conditions. Based on the methods in [25], the activated sludge from an in-service sequencing batch reactor for domestic wastewater treatment was inoculated in the above effluent and control sample with same COD concentration (prepared with glucose), and the SOUR value of activated sludge was calculated (shown in Table 3 and Figures S1, S2, and S3 in Supplementary Material available online at <http://dx.doi.org/10.1155/2015/617850>). The value gradually reduced with decreasing effluent organic concentration. At 15 min, microorganisms were obviously inhibited by quinoline effluent organic pollutants with the average SOUR being 5.51 mgO₂ g⁻¹ MLSS. At the same time, a control sample had an SOUR of 7.01 mgO₂ g⁻¹ MLSS. At 30 min, the inhibition effect had decreased, with the average SOUR being 4.19 mgO₂ g⁻¹ MLSS for the quinoline effluent sample and 5.10 mgO₂ g⁻¹ MLSS for the control sample. At 45 min, the inhibition effect was only slight, with the average SOURs being 2.20 and 2.58 mgO₂ g⁻¹ MLSS for the quinoline effluent sample and control sample, respectively. The above results again indicate that the biodegradability of quinoline effluent increased and the toxicity reduced with increasing electrolysis time.

TABLE 2: $L_9(3)^3$ orthogonal test results.

Number	A (current density) (mA cm ⁻²)	B (electrolysis time) (h)		C (Na ₂ SO ₄ concentration) (mol L ⁻¹)	Quinoline removal (%)	ACE (×10 ⁻²)
1	1	1		1	40.2	4.02
2	1	2		2	61.8	4.22
3	1	3		3	76.8	4.16
4	2	1		2	46.3	3.16
5	2	2		3	85.9	4.17
6	2	3		1	91.7	3.36
7	3	1		3	74.2	4.00
8	3	2		1	92.7	3.40
9	3	3		2	99.4	2.74
Quinoline removal	K1	178.8	160.6	224.5		
	K2	223.8	240.3	207.4		
	K3	266.2	267.8	236.9		
	$\overline{K1}$	59.6	53.5	74.8		
	$\overline{K2}$	74.6	80.1	69.1		
	$\overline{K3}$	88.7	89.3	78.9		
	R	29.1	35.7	9.8		
ACE	Factors			BAC		
	Primary → secondary					
	Optimal condition			$A_3B_3C_3$		
	K1	12.40×10^{-2}	11.18×10^{-2}	10.78×10^{-2}		
	K2	10.69×10^{-2}	11.79×10^{-2}	10.12×10^{-2}		
	K3	10.14×10^{-2}	10.26×10^{-2}	12.33×10^{-2}		
	$\overline{K1}$	4.13×10^{-2}	3.73×10^{-2}	3.59×10^{-2}		
	$\overline{K2}$	3.56×10^{-2}	3.93×10^{-2}	3.37×10^{-2}		
	$\overline{K3}$	3.38×10^{-2}	3.42×10^{-2}	4.11×10^{-2}		
	R	0.75×10^{-2}	0.51×10^{-2}	0.74×10^{-2}		
	Factors			ACB		
	Primary → secondary					
	Optimal condition			$A_1B_2C_3$		

Note: Ki ($i = 1, 2, 3$) is the sum of tested index corresponding to the levels 1, 2, and 3 under the same factor; ki ($i = 1, 2, 3$) is $Ki/3$ ($i = 1, 2, 3$); R is the difference of $ki(\max)$ and $ki(\min)$ under the same factor.

TABLE 3: SOUR of effluent at electrolysis time of 15 min, 30 min, and 45 min.

Samples	OUR (mg L ⁻¹ min ⁻¹)	MLSS (g L ⁻¹)	SOUR (mgO ₂ g ⁻¹ MLSS h ⁻¹)
15 min			
Sample 1	0.1739	1.986	5.25
Sample 2	0.1908	1.988	5.76
Control sample	0.2307	1.975	7.01
30 min			
Sample 1	0.1008	1.396	4.33
Sample 2	0.0991	1.470	4.04
Control sample	0.1322	1.554	5.10
45 min			
Sample 1	0.0865	2.642	1.96
Sample 2	0.1028	2.528	2.44
Control sample	0.0946	2.202	2.58

Note: OUR is oxygen uptake rate.

4. Conclusions

BDD anode represented an efficient method for mineralization of quinoline, and the effect of electrolyte type showed that the excellent quinoline and TOC removal were achieved at $0.05 \text{ mol L}^{-1} \text{ Na}_2\text{SO}_4$ as electrolyte; the effect of pH showed that removal efficiencies of quinoline and TOC were better in the slight alkaline medium. Moreover, at initial quinoline concentration of 50 mg L^{-1} and initial pH of 7, orthogonal experiment for quinoline removal and average current efficiency showed that the optimal electrolysis condition was current density of 75 mA cm^{-2} , electrolysis time of 1.5 h, and Na_2SO_4 concentration of 0.05 mol L^{-1} with quinoline removal efficiency of 85.9% and average current efficiency of 4.17×10^{-2} .

At different electrolysis time, the characteristic of quinoline effluent treated by BDD anodes was evaluated from variation of BOD_5/COD and toxicity to microorganism. BOD_5/COD was enhanced from initial 0.02 to 0.57 at 90 min. The SOUR was used to assess compost stability and toxicity of wastewater, and the value gradually reduced with decreasing effluent organic concentration with mean value of 5.51, 4.19, and $2.20 \text{ mgO}_2 \text{ g}^{-1} \text{ MLSS}$ at electrolysis time of 15, 30, and 45 min, respectively. Meanwhile, at the same COD concentration, the SOUR of control sample (prepared with glucose) was 7.01, 5.10, and $2.58 \text{ mgO}_2 \text{ g}^{-1} \text{ MLSS}$, respectively. It can be comparatively found that the effluent of quinoline mineralization showed obvious inhibition effect on microorganisms at electrolysis time of 15 min, and then it was significantly faded at 30 min and 45 min. The above results will help to gain basic reference for selecting advanced quinoline wastewater treatment technology.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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