

Scale-up of BDD anode system for electrochemical oxidation of phenol simulated wastewater in continuous mode

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ABSTRACT

Scale-up of boron-doped diamond (BDD) anode system is significant to the practical application of electrochemical oxidation in bio-refractory wastewater treatment. In this study, the performance of a smaller BDD anode (24 cm²) system in continuous mode electrochemical oxidation of phenol simulated wastewater was first investigated and well described by the response surface methodology (RSM). Furthermore, the RSM was extended to examine the scale-up feasibility of BDD anode systems with similar configurations. It was demonstrated that both COD degradation efficiency and specific energy consumption could be expected at the same level even as the system was enlarged over 100 times, which implied that BDD anode system could be successfully scaled up through controlling the same retention time, current density, initial COD, and conductivity conditions. Based on this study, a larger BDD anode (2904 cm²) system was constructed and systematic measurements were made on its performance in electrochemical oxidation of phenol simulated wastewater. Very good agreement was found between measured and predicted results by RSM. At the optimized conditions, the larger BDD anode system could easily reduce the COD of phenol simulated wastewater from 633 mg L⁻¹ to 145 mg L⁻¹ (<150 mg L⁻¹, National Discharge Standard of China) during 80 min with specific energy consumption only 31 kWh kgCOD⁻¹.

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1. Introduction

Many industries generate bio-refractory organic wastewaters, such as coking plant, oil refinery, dye factory, and paper manufactory. Usually, after biological treatment, the effluent COD values of these wastewaters were still higher than 150 mg L⁻¹ (National Discharge Standard of China) [1–5]. Thus, efforts on advanced treatment will be necessary. Electrochemical oxidation is of great potential in the treatment of bio-refractory organic wastewaters, since it has been proven to be very effective to various refractory organic pollutants, i.e., phenols [6], dyes [7], surfactants [8], and herbicides [9], and many real wastewaters, e.g., textile dye effluent [10], landfill leachate [11], coking wastewater [12], and tannery waste liquor [13].

As one of the important factors for electrochemical technology, electrode materials have significant effects on the effectiveness, efficiency, and mechanisms of electrochemical processes. Lots of electrode materials have been investigated in previous studies [14–19], including graphite, platinum, IrO₂, RuO₂, SnO₂, PbO₂, and boron-doped diamond (BDD) electrodes. The results demonstrated that BDD electrode has many advantages, such as strong oxidation

ability, high current efficiency, weak electrode fouling, and long service life [19–23], and it was of great potential in electrochemical treatment of bio-refractory organic wastewaters. Therefore, the scale-up of BDD anode system is the next critical step that would make electrochemical oxidation to be a practical technology for wastewater treatment.

However, few researches on the scale-up of BDD anode system have been reported. Hitherto, investigations were usually performed at BDD anodes with a working area of 1–100 cm² [6,7,23–27]. Only Tröster et al. [28] constructed a flow cell using a stack of BDD electrodes with total active area of 1 m², but detailed comparisons between laboratory and pilot plant results were not reported. Urriaga and co-workers [29–31] investigated the electrochemical oxidation of landfill leachate at pilot scale (total BDD anode area = 1.05 m²), but the system consisted of 150 cells (anode–cathode pair), i.e. each BDD anode area was only 70 cm². Moreover, their results demonstrated that the COD removal efficiency in pilot plant system was slightly lower than that in laboratory system due to the different hydrodynamics.

In this study, a smaller BDD anode system was first constructed using 24 cm² BDD electrode. The performance of this system in continuous mode electrochemical oxidation of phenol simulated wastewater were soundly investigated by the response surface methodology (RSM), because RSM is a powerful tool to describe and optimize complex systems [32–35]. Then, the obtained results

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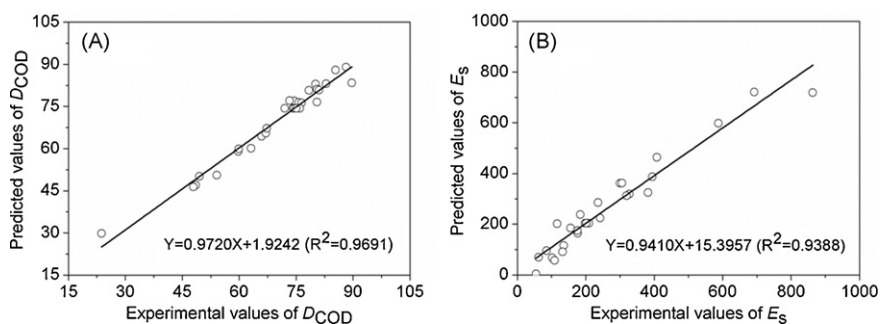


Fig. 1. Comparison between experimental and predicted values of D_{COD} (A) and E_s (B) in the smaller BDD anode system.

were used to guide the operation of a larger BDD anode system (anode area = 2904 cm²). The BDD anode system was successfully scaled up by 121 times without performance deterioration.

2. Materials and methods

2.1. BDD anode systems

Two BDD anode systems of similar configurations were constructed. For the smaller system, a Nb/BDD plate with a working area of 24 cm² (3 × 8 cm, bought from CONDIAS GmbH, Germany) was used as the anode, and a piece of stainless steel with the same size was as the cathode. The gap between the electrodes was set to be 1.55 cm, and thus the working volume of this system was 37.2 mL. As for the larger system, a Nb/BDD plate with a working area of 2904 cm² (33 × 88 cm) was used as the anode and the stainless steel of the same size was as the cathode. The gap between the electrodes was also set to be 1.55 cm, so the working volume of this system was 4501 mL.

Continuous mode electrochemical oxidation of phenol simulated wastewater was performed in these two BDD anode systems under galvanostatic condition at room temperature. Phenol simulated wastewater was chosen as the studied object, because phenol is a common and typical organic pollutant in many industrial

effluents [1,2,5]. During electrochemical oxidation process, phenol simulated wastewater was continuously pumped through the reactor at a certain flow rate (determined by retention time) which ranged in 0.42–4.65 mL min⁻¹ for the smaller BDD anode system and 51.15–562.62 mL min⁻¹ for the larger BDD anode system.

2.2. Response surface methodology

The response surface methodology (RSM) was used for the first time to describe and optimize the electrochemical oxidation process of BDD anode system. In RSM, the most popular class of second-order designs called central composite design (CCD) was used for the experimental design, since the CCD is well suitable for fitting a quadratic surface, which usually works well for the process optimization [32,35].

The retention time (X_1), current density (X_2), initial COD (X_3), and conductivity (X_4) were chosen as independent variables, because studies have demonstrated that they significantly influenced the electrochemical oxidation processes [7,36–38]. The COD degradation efficiency and specific energy consumption were as output variables or responses. Independent variables and their experimental ranges are given in Table 1. The initial COD was controlled by phenol concentration and the conductivity was kept by certain Na₂SO₄ quantity.

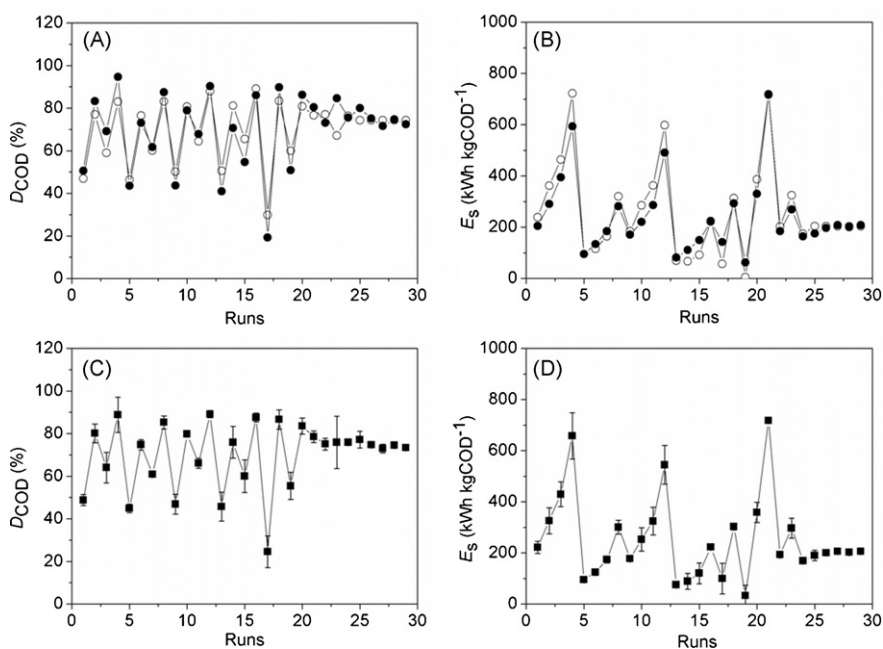


Fig. 2. Comparison between experimental (●) and predicted (○) values of D_{COD} (A) and E_s (B) in the larger BDD anode system. (C) and (D) For D_{COD} and E_s with standard deviation between experimental and predicted values.

Table 1
Experimental range and levels of the independent factors for electrochemical oxidation of phenol simulated wastewater.

Independent factors	Factor X_i	Range and levels				
		–2	–1	0	1	2
Retention time (min)	X_1	8	28	48	68	88
Current density (mA cm^{-2})	X_2	6	13	20	27	34
Initial COD (mg L^{-1})	X_3	100	300	500	700	900
Conductivity (mS cm^{-1})	X_4	3	6	9	12	15

For statistical calculations, the variables X_i were coded as x_i according to the following relationship:

$$x_i = \frac{X_i - X_0}{\delta X} \quad (1)$$

where X_0 is value of the X_i at the center point, and δX presents the step change.

Experimental data were analyzed using the response surface regression procedures by means of the software package Minitab 15 and fitted to the following second-order polynomial model:

$$Y = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_4x_4 + b_{11}x_1^2 + b_{22}x_2^2 + b_{33}x_3^2 + b_{44}x_4^2 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{14}x_1x_4 + b_{23}x_2x_3 + b_{24}x_2x_4 + b_{34}x_3x_4 \quad (2)$$

where Y is response variable, b_0 is constant, b_1 , b_2 , b_3 , and b_4 are regression coefficients for linear effects, b_{11} , b_{22} , b_{33} , and b_{44} are quadratic coefficients, and b_{12} , b_{13} , b_{14} , b_{23} , b_{24} , and b_{34} are interaction coefficients.

2.3. Analytical methods

The influent and effluent COD of phenol simulated wastewater was measured by a titrimetric method using dichromate as the oxidant in acidic solution at 150 °C for 2 h (Hachi, USA). The specific energy consumption (E_s) was calculated using the following

equation:

$$E_s = \frac{1000U It}{(\text{COD}_{\text{in}} - \text{COD}_{\text{out}})V} \quad (3)$$

where E_s is the specific energy consumption (in kWh kgCOD^{-1}), U is the voltage (in V), I is the current (in A), t is the retention time (in h), COD_{in} and COD_{out} are the influent and effluent COD of wastewater (in mg L^{-1}), and V is the volume of reactor (in L).

3. Results and discussion

3.1. Performance of the smaller BDD anode system

Electrochemical oxidation of phenol simulated wastewater was performed in the smaller BDD (24 cm^2) anode system according to the full factorial central composite design (CCD) (Table 2). The COD degradation efficiency (D_{COD}) and specific energy consumption (E_s) for every run were also shown in Table 2. Under different conditions, the D_{COD} was in the range of 23.71–89.68% and the E_s was in 54.43–863.39 kWh kgCOD^{-1} .

RSM was used to analyze the obtained data. The experimental data were fitted to the second-order polynomial model (Eq. (2)) using the response surface regression procedures. The following empirical RSM models were obtained for D_{COD} (Eq. (4)) and E_s (Eq.

Table 2
A 2^4 full factorial CCD with five replicates of the centre point for electrochemical oxidation of phenol simulated wastewater in the smaller BDD anode system.

Runs	x_1	x_2	x_3	x_4	D_{COD} (%)	E_s (kWh kgCOD^{-1})
1	–1	–1	–1	–1	48.53	183.93
2	1	–1	–1	–1	74.55	299.94
3	–1	1	–1	–1	59.77	407.66
4	1	1	–1	–1	80.11	692.35
5	–1	–1	1	–1	47.98	84.89
6	1	–1	1	–1	75.69	135.11
7	–1	1	1	–1	63.12	176.64
8	1	1	1	–1	82.89	327.80
9	–1	–1	–1	1	49.53	155.36
10	1	–1	–1	1	78.40	236.01
11	–1	1	–1	1	65.90	305.96
12	1	1	–1	1	85.37	587.42
13	–1	–1	1	1	54.10	61.53
14	1	–1	1	1	80.49	101.75
15	–1	1	1	1	67.02	132.15
16	1	1	1	1	88.17	241.93
17	–2	0	0	0	23.71	108.82
18	2	0	0	0	89.68	318.95
19	0	–2	0	0	59.88	54.43
20	0	2	0	0	81.04	394.63
21	0	0	–2	0	80.52	863.39
22	0	0	2	0	73.32	115.82
23	0	0	0	–2	67.25	381.62
24	0	0	0	2	76.41	175.63
25	0	0	0	0	74.17	206.23
26	0	0	0	0	74.58	205.69
27	0	0	0	0	71.97	209.21
28	0	0	0	0	75.96	197.91
29	0	0	0	0	75.07	201.13

(5):

$$Y(D_{\text{COD}}) = 74.350 + 13.402x_1 + 5.225x_2 + 0.121x_3 + 2.277x_4 - 4.435x_1^2 - 0.993x_2^2 + 0.622x_3^2 - 0.651x_4^2 - 1.766x_1x_2 + 0.020x_1x_3 + 0.127x_1x_4 + 0.175x_2x_3 + 0.300x_2x_4 + 0.241x_3x_4 \quad (R^2 = 0.9703) \quad (4)$$

$$Y(E_s) = 204.034 + 63.935x_1 + 95.575x_2 - 129.249x_3 - 37.425x_4 - 4.789x_1^2 - 2.128x_2^2 + 64.141x_3^2 + 11.396x_4^2 + 33.749x_1x_2 - 25.714x_1x_3 - 5.623x_1x_4 - 38.932x_2x_3 - 11.736x_2x_4 + 7.003x_3x_4 \quad (R^2 = 0.9410) \quad (5)$$

The obtained models had high values of coefficient of determination (R^2). Additionally, good agreements between the experimental and predicted values of D_{COD} and E_s were observed (Fig. 1). Experimental values were presented in X axes, and Y axes was for corresponding predicted values. The data were fitted to equation $Y = aX + b$. For both D_{COD} and E_s , the values of "a" and the coefficients of determination (R^2) were very close to 1, which indicated the satisfactory predictions. These results demonstrated that RSM was very suitable to describe the electrochemical oxidation process of BDD anode system.

3.2. Performance of the larger BDD anode system

The obtained D_{COD} and E_s models by RSM demonstrated that both COD degradation efficiency and specific energy consumption would be at the same level once the retention time, current density, initial COD, and conductivity were same, whatever the BDD anode system was enlarged by any times. Therefore, a larger BDD anode (2904 cm²) system was constructed with similar configuration, i.e., the BDD anode system was scaled up by 121 times. The performance of this system in electrochemical oxidation of phenol simulated wastewater was investigated under the same conditions as those for the smaller BDD anode system (listed in Table 2).

Fig. 2 shows the experimental and predicted values of D_{COD} and E_s in the larger BDD anode system. Good agreement with relative small standard deviation (0.2–12%) was observed between experimental and predicted values by RSM, which implied that BDD anode system could be successfully scaled up over 100 times through controlling the same retention time, current density, initial COD, and conductivity conditions.

3.3. Optimization of the larger BDD anode system

The D_{COD} and E_s in the larger BDD anode system were significantly different under special conditions. The influences of primary factors (retention time t , current density j , initial COD C_0 , and conductivity S) were clearly depicted by the contour plots of D_{COD} (Fig. 3) and E_s (Fig. 4).

As for D_{COD} , the interaction between retention time and current density was obvious (Fig. 3A). To achieve the same degradation target, longer retention time required lower current density, and vice versa. Furthermore, it can be observed that the influence of retention time was more apparent than those of current density (Fig. 3A), initial COD (Fig. 3B), and conductivity (Fig. 3C). The role of current density was more important than those of initial COD (Fig. 3D) and conductivity (Fig. 3E). The initial COD was more significant than conductivity (Fig. 3F). The role order of factors was $t > j > C_0 > S$. These figures also showed that higher degradation efficiency was obtained at longer retention time, larger current density,

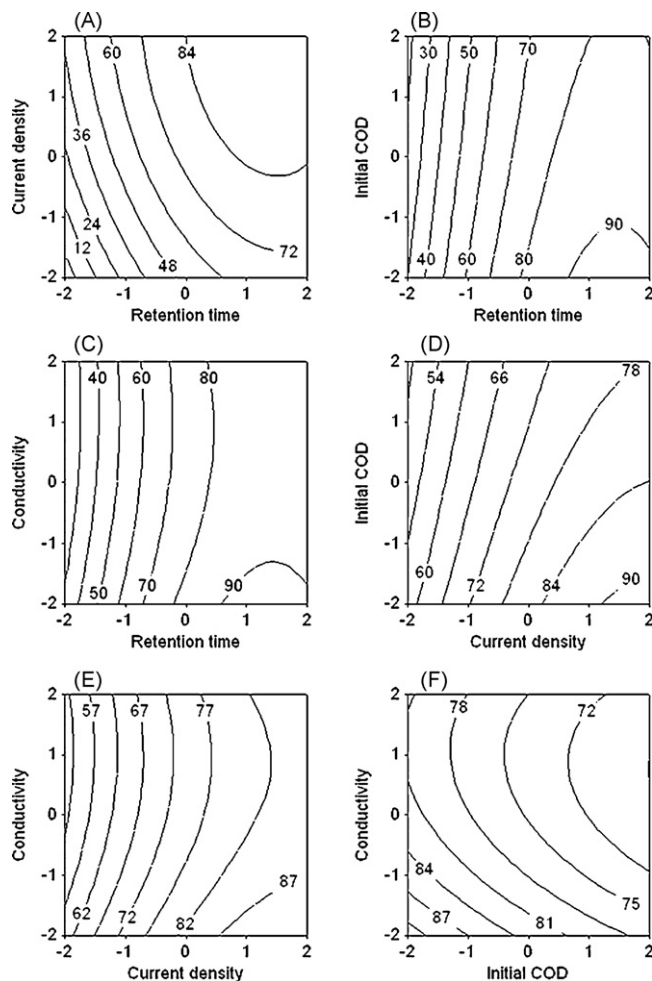


Fig. 3. Contour plots of D_{COD} in the larger BDD anode system vs. two shown factors: (A) retention time and current density, (B) retention time and initial COD, (C) retention time and conductivity, (D) current density and initial COD, (E) current density and conductivity, and (F) current density and conductivity.

and lower initial COD. The conductivity mostly did not affect the degradation efficiency.

E_s was affected by retention time, current density, initial COD, and conductivity (Fig. 4). It can be observed that the effect of initial COD was more obvious than those of retention time (Fig. 4B), current density (Fig. 4D), and conductivity (Fig. 4F). The current density was more important than retention time (Fig. 4A) and conductivity (Fig. 4E). The role of retention time was more significant than that of conductivity (Fig. 4C). The important order of factors was distinguished as $C_0 > j > t > S$. Lower specific energy consumption was shown at shorter retention time, smaller current density, higher initial COD, and larger conductivity, which was almost opposite to those for higher degradation efficiency. Therefore, the integrated influences on degradation efficiency and energy consumption should be considered to determine operating conditions for an electrochemical oxidation process, and RSM was very effective to describe and optimize this complex system.

Optimal conditions to simultaneously achieve high D_{COD} and low E_s were obtained from RSM models and calculated by Minitab 15 software. At the optimized conditions ($t = 80$ min, $j = 8.5$ mA cm⁻², $C_0 = 633$ mg L⁻¹, $S = 3$ mS cm⁻¹), the D_{COD} and E_s in the larger BDD anode system were 77% and 63 kWh kg COD⁻¹, respectively. That is, the COD of phenol simulated wastewater was reduced from 633 mg L⁻¹ to 145 mg L⁻¹ (<150 mg L⁻¹, National Dis-

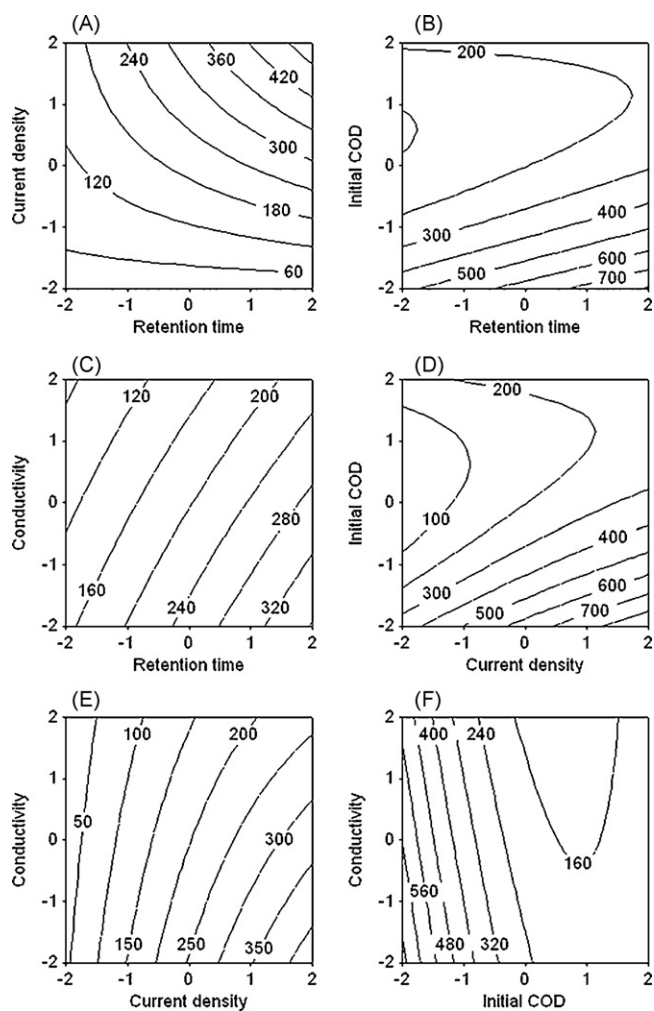


Fig. 4. Contour plots of E_s in the larger BDD anode system vs. two shown factors: (A) retention time and current density, (B) retention time and initial COD, (C) retention time and conductivity, (D) current density and initial COD, (E) current density and conductivity, and (F) current density and conductivity.

charge Standard of China) during 80 min, while the specific energy consumption was only 63 kWh kgCOD⁻¹ (i.e., 31 kWh kgCOD⁻¹), which demonstrated that the larger BDD anode system was of great potential in electrochemical oxidation of bio-refractory wastewater.

4. Conclusions

The scale-up of BDD anode system is the next significant step to allow electrochemical oxidation as a practical technology for wastewater treatment. Using the response surface methodology (RSM), the effects of scale-up were investigated on BDD anode systems with similar configurations. As a result, the smaller BDD anode (24 cm²) system was successfully scaled up by 121 times through controlling the primary influence factors, such as retention time, current density, initial COD, and conductivity. Expected COD degradation efficiency and energy consumption were obtained in the scaled up BDD anode (2904 cm²) system. At the optimized conditions, the COD of phenol simulated wastewater in the scaled up BDD anode system was effectively reduced from 633 mgL⁻¹ to 145 mgL⁻¹ (<150 mgL⁻¹, National Discharge Standard of China) during 80 min, while the specific energy consumption was only 31 kWh kgCOD⁻¹.

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