

# An experimental investigation of chemical oxygen demand removal from the wastewater containing oxalic acid using three-phase three-dimensional electrode reactor

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Accepted 18 August 2001

## Abstract

The removal of chemical oxygen demand (COD) from wastewater containing oxalic acid was experimentally investigated using a three-phase three-dimensional electrode reactor. The experimental results show that the removal efficiency depends on the applied cell voltage, airflow, electrolysis time and the number of repeated runs. The former four factors all had a positive effect while the last factor generally had a negative effect on the COD removal in the experimental range. However, for electrolyses under the conditions of 30.0 V cell voltage, 7.0 l min<sup>-1</sup> airflow and 60.0 min, the negative effect was negligible. After 50 runs, the electrochemical reactor could maintain a COD removal efficiency of more than 90%. © 2002 Elsevier Science Ltd. All rights reserved.

**Keywords:** Electrochemistry; Three-dimensional electrode; Wastewater; Oxalic acid

## 1. Introduction

The last several decades have witnessed a growing awareness of the fragile state of the earth's drinking water resources (Legrini et al., 1993). The growing pollution of our hydrosphere has stimulated the need for developing new technologies for water and wastewater treatment (Mukherjee and Ray, 1999). Electrochemistry, with its unique ability to oxidize and reduce compounds at a well-controlled electrode po-

tential, offers many interesting possibilities in environmental engineering (Simonsson, 1997). Recently, electrochemical methods for the treatment of wastewater have attracted a great deal of attention (Coin et al., 1996; Rajeshwar et al., 1994; Simonsson, 1997). In particular, three-dimensional electrode systems, such as packed and fluidized bed electrodes, are gaining increased technical importance. This is because they have a more extensive interfacial electrode surface (Bockris and Kim, 1997; Brown et al., 1994; Tissot and Fragniere, 1994; Tennakoon et al., 1996) and allow the application of considerably higher current density than two-dimensional electrodes (Kreysa et al., 1975).

These electrochemical technologies have been successfully used to remove metal ions from waste streams,

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however, their application to eliminating organic pollutants from wastewater is still rarely used industrially to date (Boscoletto et al., 1994). The main problem is that the electrochemical oxidation of organic pollutants is very slow, in practice, as a consequence of kinetics, although the electrochemical oxidation of all organic pollutants is theoretically possible before  $O_2$  evolution (Zor et al., 1998). In order to increase the electrochemical efficiency of organic pollutant removal, we are developing a new electrochemical reactor; a three-phase three-dimensional electrode cell. The cell is based on a combination of a packed-bed electrode and a gas-diffusion electrode, thus differing from the reported three-dimensional electrode reactor which has only solid and liquid phases (Bockris and Kim, 1997; Brown et al., 1994; Tissot and Fragniere, 1994; Tenakoon et al., 1996).

Oxalate is one of the important intermediates in the complete mineralization of many organic pollutants (Alvarez-Gallbergos and Pletcher, 1999; Huston and Pignatello, 1999; Kotronarou et al., 1992; Liakou et al., 1997; Pignatello and Sun, 1995; Sharma et al., 2000; Stern et al., 1997; Vinodgopal et al., 1998; Weavers et al., 2000), and is also resistant to oxidation (Alvarez-Gallbergos and Pletcher, 1999). However, it also exists in some actual industrial wastewater. For example, oxalate is an important toxic pollutant in Bayer liquor during alumina processing (Bangun and Adesina, 1998; Williams and Perrotta, 1998). In addition, the electrochemical oxidation of oxalate on carbon electrodes has been reported to occur via an irreversible  $2e^-$  process to yield  $CO_2$  as the sole product and no intermediate species complicate the oxidation process (Byrne and Eggins, 1998). As a result, it is frequently used as a model organic pollutant in the study of the treatment of organic wastewater (Alvarez-Gallbergos and Pletcher, 1999; Bangun and Adesina, 1998; Byrne and Eggins, 1998; Byrne et al., 1999; Chollier et al., 1999; Krysa et al., 2000; Kulas et al., 1998; Marsen et al., 1993). The aim of the present work is to investigate the chemical oxygen demand (COD) removal from synthetic wastewater containing oxalic acid using the three-phase three-dimensional electrode reactor. Special attention is paid to observing changes in COD removal efficiency of the electrochemical reactor in the process of continuous use in view of practical application.

## 2. Materials and methods

### 2.1. Materials

A commercial charcoal- and vegetable-based granulated activated carbon (GAC) from KEBO LAB Company, Sweden, was used in this study. It has an average

particle size of approximately 3 mm, a specific surface of  $925 \text{ m}^2 \text{ g}^{-1}$  according to the BET method and a specific weight of  $0.4375 \text{ kg l}^{-1}$ . Oxalic acid dihydrate (99.5–100.5%) was obtained from Riedel-De Haën AG, Germany. All solutions were prepared with deionized water.

### 2.2. COD and surface area measurement

The COD was determined using a LP1W spectrophotometer, Dr Lange standard test tube and a Dr Lange procedure (Dr Bruno Lange GmbH, Düsseldorf, Germany). It reflects the amount of organic pollutants in the sample. The surface area of GAC was determined with a Micromeritics ASAP 2400 instrument.

### 2.3. Experimental set-up

The experimental apparatus is a batch rectangular undivided three-phase three-dimensional electrode reactor as shown in Fig. 1. The reactor support was made from plastic. The anode and cathode (feeder electrodes), situated 11.0 cm apart from each other, were made from stainless steel plates. The 77.0 g GAC was packed between the two feeder electrodes as a bed electrode with a bed height of approximately 4 cm. The volume of the three-dimensional electrodes is  $11.0 \times 4.0 \times 4.0 \text{ cm}^3$ . Compressed air was sparged into the bed electrodes by a micropore plate from the bottom of the reactor. The electric power was supplied with regulated DC power supply, PAC 30-6, Japan.

### 2.4. Experimental method

A simulated organic wastewater containing oxalic acid was prepared to a COD concentration of 529.2 ppm. The pH value was not adjusted and no electrolyte was added.

A total of 100.0 ml of the simulated wastewater was

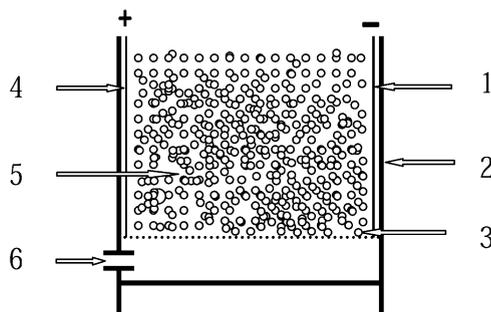


Fig. 1. Schematic diagram of the three-phase three-dimensional electrode reactor (1, cathode; 2, support; 3, micropore plate; 4, anode; 5, particle electrodes; 6, compressed air).

fed into the three-dimensional electrode reactor prior to each run. The reactor was timed starting when the d. c. power and compressed air supply were switched on. The resulting solution was filtered to remove any traces of activated carbon and then was analyzed for COD. In order to probe the behavior of the GAC bed electrode for COD removal in the process of continuous use, every GAC bed electrode was repeatedly used 50 times under the same experimental conditions (in the following discussion,  $N$  is used to represent the number of repeated runs). The experimental results were assessed mainly based on COD removal efficiency (%), i.e. the ratio between COD removal and initial COD.

### 3. Results and discussion

#### 3.1. Effect of cell voltage

In aqueous medium, GAC has considerable electrochemical activity (Kastening et al., 1997a,b; Putzien, 1984), in addition to its strong adsorption to a lot of pollutants. In the present study, the GAC was employed as a bed electrode (working electrode) material. Hence, it was expected that the electrochemical reactor used in the present work could remove oxalic acid by both electrochemical oxidation and adsorption.

Curve (d) in Fig. 2 shows the dependence of COD removal efficiency on  $N$  with a constant airflow of  $7.0 \text{ l min}^{-1}$  but no applied cell voltage. In other words, it is a kind of adsorption curve for adsorption of oxalic acid on a repeatedly used GAC bed under air bubbling. It exhibits a typical trend relative to the number of runs for GAC bed. When  $N$  is 1, most of the oxalic acid is adsorbed and the COD removal efficiency reaches 85.2%. As  $N$  increases, the COD removal efficiency rapidly decreases. But after  $N > 40$ , the declining of the curve gets very slow, suggesting that the GAC bed is close to reaching its maximum adsorption capacity. The other curves in Fig. 2 show the results of COD removal by electrolysis with various cell voltages (10 V, 20 V and 30 V) and a constant airflow of  $7.0 \text{ l min}^{-1}$ . It can be seen from these curves that the COD removal efficiencies all decrease with increase in  $N$ , but the decrease is intensively dependent on the applied cell voltage. The higher cell voltage, the less the decrease. When a cell voltage of 30.0 V was applied, the decrease in COD removal efficiency of curve (a) was only 9.7%, which is much less than that of curve (d), in the process of 50 electrolysis runs. These experiments indicate that the COD removal by adsorption on the GAC bed decreased rapidly in the process of repeated use and finally approached zero due to saturation of the GAC.

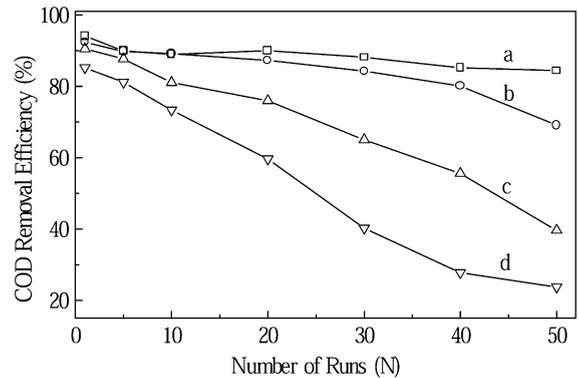


Fig. 2. Dependence of COD removal efficiency on the number of runs [operating conditions: airflow =  $7.0 \text{ l min}^{-1}$ , the treatment time of each run = 30.0 min and applied cell voltage = 30.0 V (a), 20.0 V (b), 10.0 V (c) and 0.0 V (d)].

In contrast, the three-phase three-dimensional electrode cell using GAC as a bed electrode can be expected to keep a high COD removal efficiency for wastewater bearing oxalic acid during the continuous use of the bed electrode as long as a suitable cell voltage is employed.

Fig. 3 gives the COD removal efficiencies for the 50th electrolysis ( $N = 50$ ) under various applied cell voltages. The COD removal efficiency increased significantly with an increase in the applied cell voltages. For a cell voltage of 0.0 V, i.e. under the condition of open circuit, the COD removal efficiency is 23.7% while it is 84.4% for a 30.0 V cell voltage. The latter is 60.7% higher than the former. These enhancement effects are attributed to the increase in the driving force of the electrode reaction, in addition to the increase in current, with increasing voltage. This is because potential is the major driving force for the respective phenomena of interest in electrochemical reactors (Card et al., 1990).

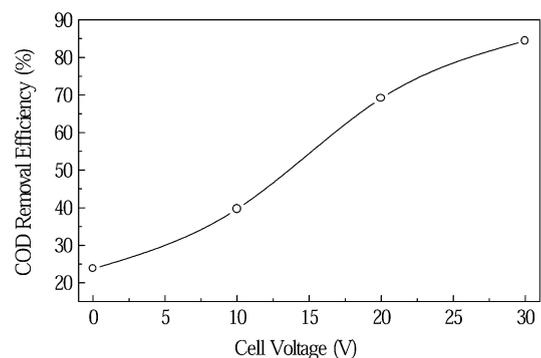


Fig. 3. Effect of cell voltage on COD removal (operating conditions: 50th runs, airflow =  $7.0 \text{ l min}^{-1}$  and treatment time = 30.0 min).

### 3.2. Effect of airflow

During electrolyses, the compressed air was uniformly sparged into the cell by a micropore plate. The sparged air serves two purposes. One is to agitate in order to speed mass transfer. Another is to supply the essential oxygen for electrochemical reactions. Some authors have reported that the oxygen can be changed into a stronger oxidizing agent,  $H_2O_2$ , on activated carbon electrodes by the two-electron reduction of oxygen (Alvarez-Gallbergos and Pletcher, 1999; Foller and Bombard, 1995; Tatapudi and Fenton, 1993; Yeager, 1984). The three-phase three-dimensional electrodes can simultaneously make use of anodic oxidation and cathodic electrogenerated  $H_2O_2$  to degrade organic pollutants. As a result, it is expected that the sparged compressed air will play an important role in the degradation of oxalic acid.

Fig. 4 presents the COD removal efficiencies for the electrolyses with a constant cell voltage of 30.0 V and various airflows. As shown in the figure, the COD removal efficiencies all decrease with increase in  $N$  in all the runs, but to different degrees. The greater airflow corresponds to less reduction in COD removal efficiency. For example, COD removal efficiency progressively decreased from 94.5 to 84.4% for an airflow of  $7.0 \text{ l min}^{-1}$ , while COD removal efficiency dropped from 92.7 to 68.9% in the absence of airflow. The net reduction of the former is only 10.1% while that of the latter is 23.8%.

With respect to the 50th electrolysis, Fig. 5 clearly shows that the COD removal efficiency increased rapidly below  $5.0 \text{ l min}^{-1}$  but changed slowly over  $5.0 \text{ l min}^{-1}$ , gradually approaching the limiting value. The maximum enhanced COD removal efficiency reached

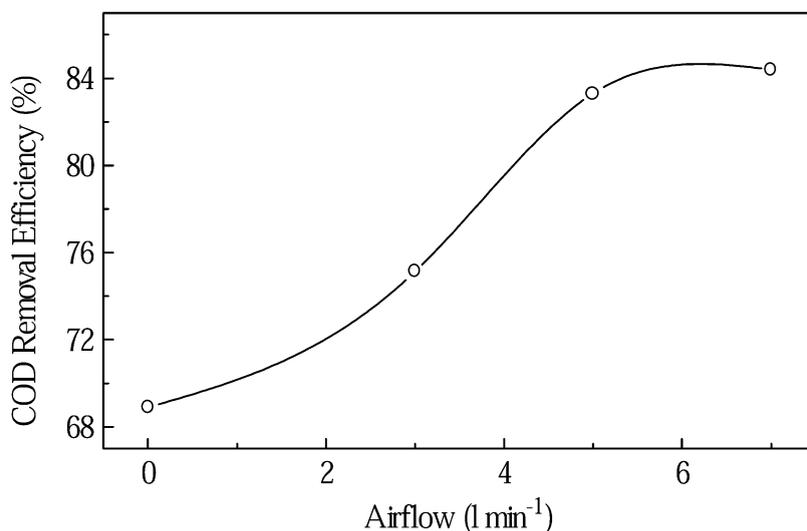


Fig. 5. Effect of airflow on COD removal (operating conditions: 50th runs, applied cell voltage = 30.0 V and treatment time = 30.0 min).

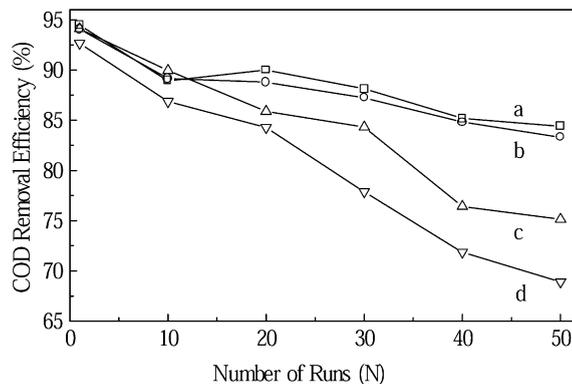


Fig. 4. Dependence of COD removal efficiency on the number of runs [operating conditions: applied cell voltage = 30.0 V, the treatment time of each run = 30.0 min and airflow =  $7.0 \text{ l min}^{-1}$  (a),  $5.0 \text{ l min}^{-1}$  (b),  $3.0 \text{ l min}^{-1}$  (c) and  $0.0 \text{ l min}^{-1}$  (d)].

15.5%. The enhancement effect of the sparged air indicates experimentally that the three-phase three-dimensional electrode is somewhat superior to the common three-dimensional electrodes in COD removal from organic wastewater.

### 3.3. Effect of treatment time

The experimental results for various electrolysis times are shown in Fig. 6. It can be seen from the figure that the COD removal efficiencies all decrease with increasing  $N$ , but for various treatment times the difference in the decrease is rather evident. The curve (a), corresponding to the 10-min electrolyses, declines

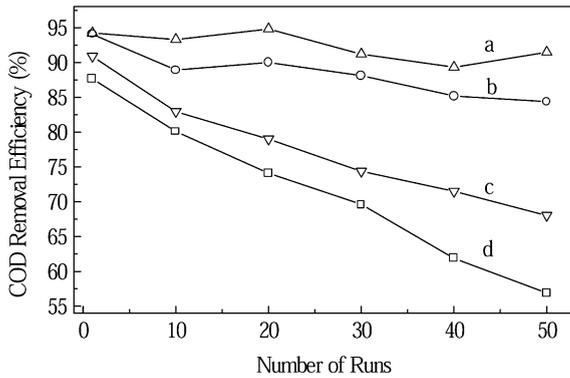


Fig. 6. Dependence of COD removal efficiency on the number of runs [operating conditions: applied cell voltage = 30.0 V, airflow = 7.0 l min<sup>-1</sup>, the treatment time of each run = 60.0 min (a), 30.0 min (b), 20.0 min (c) and 10.0 min (d)].

rapidly. During 50 runs, its COD removal efficiency decreased from 87.7 to 56.9%, i.e. the net reduction reached 30.8%. For 60-min electrolyses, its COD removal efficiency changed from 94.2 to 91.5% and the net reduction was only 2.7%. This variation can be considered negligible.

Pletcher (1982) and Tennakoon et al. (1996) assessed the performance of some three-dimensional electrodes for the generation of carbon dioxide using space-time yield, i.e. the amount of CO<sub>2</sub> formed/(time × cell volume). In the present case, the behavior of three-dimensional electrodes for COD removal was assessed using a similar quantity, space-time efficiency (STE):

$$STE = \frac{\text{Amount of COD removal}}{\text{Time} \times \text{Cell volume}} \quad (1)$$

where *STE* is the rate of COD removal per unit cell volume. Fig. 7 presents simultaneously the dependence of COD removal efficiency and *STE* for the 50th electrolysis on the electrolysis time. As can be seen, the COD removal efficiency and *STE* display a contrary change trend. The former increases with time while the latter declines with time. Based on these variations, it is suggested that an electrolysis duration of approximately 30 min is more suitable to optimize COD removal efficiency and SET during the repeated use of GAC bed electrode.

### 3.4. Effect of current

Constant-current electrolyses at various currents were carried out to investigate the effect of current on the COD removal efficiency of the three-phase three-dimensional electrode in the present study. It is apparent in Fig. 8 that the high applied current resulted in high efficiency of COD removal although the removal

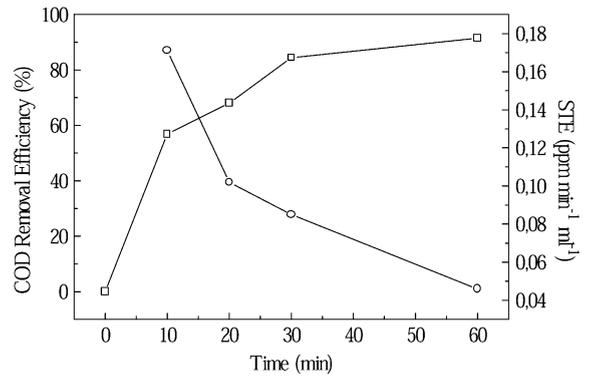


Fig. 7. Effect of treatment time on COD removal (operating conditions: 50th runs, applied cell voltage 30.0 V and airflow = 7.0 l min<sup>-1</sup>).

efficiencies always decreased with increasing *N* in our experimental range.

With respect to the 50th electrolysis, an increase in the applied current from 0.05 to 0.20 A led to an increase in COD removal efficiency from 37.3 to 58.1%, as shown in Fig. 9. However, the enhancement of the COD removal efficiency was much less than that of the applied current. This result implies that Faradic efficiencies of the COD removal may not increase with increase in applied current, although COD removal efficiency increased. To complement the understanding of this phenomenon, the apparent Faradic efficiencies of the COD removal were calculated using the following formula (Polcaro et al., 1999; Szpyrkowicz et al., 2000):

$$\eta_F = \frac{\Delta COD \times V_w \times F}{8 \times I \times \Delta t} \quad (2)$$

where  $\Delta COD$  is the COD change before and after the

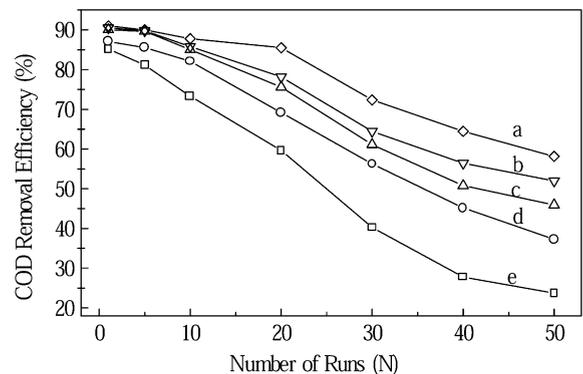


Fig. 8. Dependence of COD removal efficiency on the number of runs [operating conditions: airflow = 7.0 l min<sup>-1</sup>, the treatment time of each run: = 30.0 min and applied current = 0.2 A (a), 0.15 A (b), 0.10 A (c) and 0.0 A (d)].

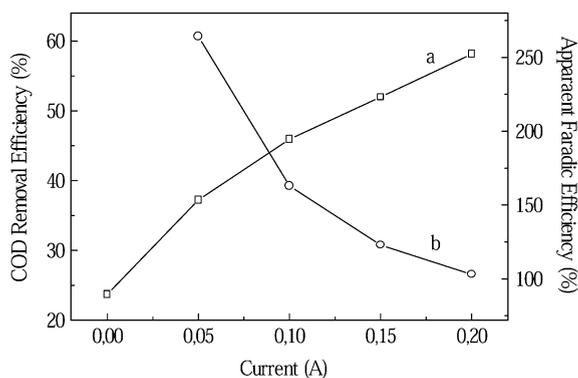


Fig. 9. Effect of applied current on COD removal (operating conditions: 50th runs, airflow = 7.0 l min<sup>-1</sup> and treatment time = 30.0 min).

treatment (g l<sup>-1</sup>),  $V_w$  is the volume of the treated wastewater (l),  $F$  is Faraday's constant (96487 C equiv.<sup>-1</sup>), 8 is the equivalent weight of oxygen,  $I$  is the applied current (A) and  $\Delta t$  is treatment time (s).

The calculation results for the 50th electrolyses are presented in curve (b) of Fig. 9. It can be seen from the curve that the apparent Faradic efficiency decreased from 264.2 to 160.1% as the applied current increased from 0.05 to 0.20 A, which is consistent with the above analysis. This reduction in apparent Faradic efficiency suggests that some side reactions concomitant to electrooxidation of oxalic acid, such as oxygen and hydrogen evolution and heat generation, etc., wasted much more current under the condition of high current than small current. Consequently, in practical application, it is necessary to limit the current of operation, as proposed by Tennakoon et al. (1996), to gain a high current efficiency.

In addition, it is also observed in Fig. 9 that the apparent Faradic efficiencies all exceed 100% in our experimental range. Such high apparent Faradic efficiencies can be attributed to the combination of pollution electrooxidation, including the direct oxidation on the anode and the indirect oxidation of cathodic electro-generated H<sub>2</sub>O<sub>2</sub>, and the adsorption of the GAC bed electrode. The efficiency of the current used for the oxidation of oxalic acid is lower than these apparent current efficiencies.

#### 4. Conclusions

A new electrochemical reactor, a three-phase three-dimensional electrode cell, was used to remove COD from simulated wastewater containing oxalic acid. It was confirmed that the removal efficiency depended on the applied cell voltage, airflow, electrolysis time and the number of repeated runs. An encouraging result

observed is that for electrolysis with 30.0 V cell voltage, airflow of 7.0 l min<sup>-1</sup> and time of 60.0 min, the electrochemical reactor could maintain a COD removal efficiency of more than 90% after 50 repeated runs. It is expected that the electrochemical technology based on the three-phase three-dimensional electrode can be applied to treat organic wastewater.

#### Acknowledgements

This project was supported by the National Natural Science Foundation of China (29977030), the Natural Science Foundation of Guangdong Province (990274) and the Science Technology Project of Guangdong Environmental Protect Agency (199914).

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