



# Is it worth the use of bipolar electrodes in electrolytic wastewater treatment processes?



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## HIGHLIGHTS

- Application of bipolar electrodes can help to optimize electrolytic treatments.
- Current efficiency and energy efficiency depend on the concentration of compound.
- Bipolar electrodes show improvement when the mediated oxidation becomes relevant.
- Electrolysis of wastewater carried out in the lab scale should be easily scaled-up.

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## ABSTRACT

In this work, it is studied the advisability of the application of bipolar electrodes (consisting of boron doped diamond coatings) for the electrolytic treatment of wastewater. Results are discussed in terms of the success in two different reactions: the depletion of an organic pesticide (2,4-dichlorophenoxyacetic acid) and the oxidation of chlorides to perchlorates. This later reaction should be prevented in an actual environmental remediation process because of the hazardousness of perchlorate, but it has been investigated in this work because it gives valuable information about formation of oxidants during electrolytic processes as chlorates and perchlorates behaves as inert species in the oxidation of 2,4-D. Results pointed out the importance of the optimization of the number of bipolar electrodes to be included in each cell for a given application. Bipolar configuration can help to improve the results obtained in the removal of organic compounds by direct processes and it also influences on the formation oxidizing species that can improve the results of this electrolytic process. However, it increases significantly the cell voltage and hence the power consumption. In this work, it is demonstrate that each bipolar electrode saves around 22% of the electric charge provided by the single cell to deplete 2,4-D and to generate oxidants, although this positive effect is masked by the increase in the cell voltage. Energy required to oxidize organic increases with the number of bipolar electrodes installed in the cell while energy efficiency in the production of oxidants shows a maximum for the cell configuration with 1 bipolar electrode.

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## 1. Introduction

During the last decades, electrochemical oxidation of wastewaters has been a hot topic in environmental electrochemistry thanks to the development of novel anode materials. Among them, boron doped diamond (BDD) has become the most promising electrode for this application. These electrodes have shown very high efficiencies in the use of electricity and high chemical and electrochemical stability [1,2]. The hydroxyl radicals, and many other oxidants produced at high concentrations on the surface of diamond, are responsible for the harsh oxidation conditions attained

and help to explain the higher efficiencies obtained in the degradation of organics in comparison with other well-known Advanced Oxidation Processes [3,4]. Their main drawback seems to be their bad mechanical properties but lack of scale-up studies makes difficult to determine how they could perform in an industrial process. Most papers related to the electrochemical oxidation of wastewater polluted with organics with BDD are carried out at a lab or bench scale, and they are mainly focused on the reaction of oxidation, particularly on:

- the study of the chemical species involved in the reaction of removal of a pollutant, trying to elucidate reaction mechanisms and occurrence of hazardous intermediates or final products in the reaction medium, and

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- the characterization of the effect on efficiency of the treatment of single operating parameters (such as temperature and current density) or waste composition parameters (pH, electrolyte composition) in very simple electrochemical cells, typically consisting of single mixed tanks or one-compartment flow pass cell.

For this reason, they typically show the changes during the progress of the treatment of the concentration of pollutants, intermediates and final products and, sometimes, they propose a very preliminary calculation of efficiency. Little attention is paid to the use of commercial cells and even less to the effect of the cell design on the results of a treatment. As a consequence, most of these studies should be classified as lab-scale regardless of the size of the cell used. It is important to take in mind that scale-up should not be understood as a single increase in the volume or flow rate of wastewater treated, but as the study of other aspects of the processes such as flow-conditions, electrode arrangements, auxiliary services, construction materials, etc.

Hence, it is clear that many efforts have to be done during the next years in the scale-up of wastewater electrolytic treatment processes, in order to determine if the technology could be applied at the full scale. Next step should be to improve system performance by a proper cell design, mainly using commercial cells. This stage is carried out at the bench (and sometimes pilot plant) scale and up to now very few works have been focused on this topic [5–7].

At this point, the use of bipolar configuration is a matter of the major significance in the scale up of electrochemical processes and hence on those processes based on the electrochemical removal of pollutants. The objective of the present investigation is the study of the bipolar cell configuration for the treatment of wastewaters using two reactions that are going to develop simultaneously in an electrolytic cell with diamond electrodes: the depletion of an organic pesticide and the oxidation of chlorides to perchlorates. The aim is not to look for the optimization of the environmental remediation process (obviously in such an application production of perchlorate should be prevented) but simply to increase knowledge about scale up of this type of processes taking advantage of the simultaneous occurrence of both reactions during the electrolysis of a synthetic wastewater.

## 2. Materials and methods

### 2.1. Experimental setup

All electrolyses were conducted in a DiaCell® type 401 supplied by Adamant Technologies (Switzerland) (Fig. 1). The DiaCell® module can be assembled with one, two, three and four compartments, each of them being fed with wastewater by internal parallel inlets. The boundary electrodes are monopolar Si/BDD and in case of using multiple compartments, the separating electrodes are floating bipolar Si/BDD electrodes. All electrodes are circular (100 mm diameter) and the inter electrode gap is 1 mm. Regarding the BDD electrodes, resistivity of the Si wafers was 100 mΩ cm and the thickness of the BDD coatings was 2–3 μm. Boron concentration in the coatings was 500 ppm and the ratio sp<sup>3</sup>/sp<sup>2</sup> around 150. Prior to electrolysis assays the BDD electrodes were cleaned during 10 min in a 1 M Na<sub>2</sub>SO<sub>4</sub> solution at 15 mA cm<sup>-2</sup>.

### 2.2. Analysis procedures and methods

The electrolyses were carried out galvanostatically (external current applied was 10 A) using 1.2 dm<sup>3</sup> of a solution containing

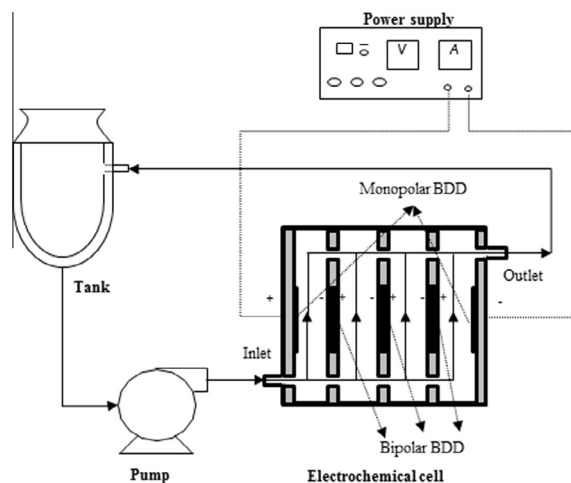


Fig. 1. Arrangement of the experimental set up. Detail of the electrochemical cell section.

100 mg dm<sup>-3</sup> 2,4-dichlorophenoxyacetic acid (2,4-D) and 3 g dm<sup>-3</sup> NaCl at natural pH (3.5). The electrolyte was stored in a glass tank and circulated through the electrolytic cell by means of a centrifugal pump. The electrolyte flow rate through the cell was 26.4 dm<sup>3</sup> h<sup>-1</sup>. A heat exchanger coupled with a controlled thermostatic bath was used to maintain the temperature at 25 °C. All electrolyses were performed in the same operating conditions in order to evaluate the efficiency of the different electrode arrangements.

All the samples extracted from electrolyzed solutions were filtered with 0.45 μm nylon filters from Whatman before their analysis. The decay of 2,4-D and the evolution of organic intermediates were followed by reversed-phase chromatography, total organic carbon (TOC) and chemical oxygen demand (COD). The chromatography system was an Agilent 1100 series coupled a UV detector. The analytical column Phenomenex Gemini 5 μm C18 was used. The analytics conditions were a mobile phase consisting of 60% acetonitrile and 40% water to 2% acetic acid with a flowrate of 0.4 cm<sup>3</sup> min<sup>-1</sup>, UV detection wavelength of 280 nm, temperature of the oven at 25 °C, and volume injection of 20 μL. The total organic carbon (TOC) concentration was monitored using a Multi N/C 3100 Analytik Jena analyzer. Measurements of pH were carried out with an InoLab WTW pH-meter. The chemical oxygen demand (COD) was monitored using a HACH DR200 analyzer.

Chlorine species (Cl<sup>-</sup>, ClO<sub>3</sub><sup>-</sup> and ClO<sub>4</sub><sup>-</sup>) were determined by ion chromatography using a Metrohm 7330 IC Separation Center coupled with a 752 pump unit and a 732 IC detector. The column used was a Metrosep A Supp 4. The mobile phase consisted of 1.8 mM Na<sub>2</sub>CO<sub>3</sub> and 1.7 mM NaHCO<sub>3</sub> with a flow rate of 1.0 ml min<sup>-1</sup>.

The current efficiency (CE) was calculated with Eq. (1) where COD<sub>t</sub> and COD<sub>t+Δt</sub> are the chemical oxygen demand (in g O<sub>2</sub> dm<sup>-3</sup>) at times t and t + Δt (in seconds), respectively, I is the current intensity (A), F is the Faraday constant (96,487 C mol<sup>-1</sup>), V is the volume of the electrolyte (dm<sup>3</sup>) and 8 is a dimensional factor for unit consistence ( $\frac{32 \text{ g O}_2 \cdot \text{mol}^{-1}}{4 \text{ mol e}^- \cdot \text{mol}^{-1} \text{ O}_2}$ ).

$$CE = \frac{[(COD)_t - (COD)_{t+\Delta t}]FV}{8I\Delta t} \quad (1)$$

The specific energy consumption (w, kWh m<sup>-3</sup>) was calculated by Eq. (2), where U is the cell potential (V):

$$w = \frac{Ult}{V} \quad (2)$$

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