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ELECTROCHEMICAL ADVANCED OXIDATION PROCESSES FOR REMOVAL OF TOXIC/PERSISTENT ORGANIC POLLUTANTS FROM WATER

# Anodic oxidation of benzoquinone using diamond anode

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**Abstract** The anodic degradation of 1,4-benzoquinone (BQ), one of the most toxic xenobiotic, was investigated by electrochemical oxidation at boron-doped diamond anode. The electrolyses have been performed in a single-compartment flow cell in galvanostatic conditions. The influence of applied current (0.5–2 A), BQ concentration (1–2 g $dm^{-3}$), temperature (20–45 °C) and flow rate (100–300 $dm^3\ h^{-1}$) has been studied. BQ decay kinetic, the evolution of its oxidation intermediates and the mineralization of the aqueous solutions were monitored during the electrolysis by high-performance liquid chromatograph (HPLC) and chemical oxygen demand (COD) measurements. The results obtained show that the use of diamond anode leads to total mineralization of BQ in any experimental conditions due to the production of oxidant hydroxyl radicals electrogenerated from water discharge. The decay kinetics of BQ removal follows a pseudo-first-order reaction, and the rate constant increases with rising current density. The COD removal rate was favoured by increasing of applied current, recirculating flow rate and it is almost unaffected by solution temperature.

**Keywords** Benzoquinone degradation · BDD anode · COD removal · Electrochemical oxidation · Water treatment

## Introduction

1,4-benzoquinone (BQ) is a multifunctional molecule with highly toxic characteristics causing erythema and localised tissue necrosis; it is irritating to the eyes and respiratory system, and it inhibits the function of biological wastewater plant. There are two main causes of the presence of BQ in the environment: (i) it is released in effluent during its use in the production of hydroquinone, dyes, insecticides, photographic processes, pharmaceuticals and leather industries; and (ii) it is an intermediate in the oxidation of benzene and other phenols.

Despite BQ is one of the most toxic xenobiotic and it is widely found in wastewaters, only few studies have been reported on its treatment method. These include electrochemical coagulation (Can and Bayramoglu 2010), photochemical oxidation (Shevchuk and Kirsho 1981), biodegradation (Kumar et al. 2011) and electrochemical oxidation (Pulgarin et al. 1994; Feng et al. 1995; Houk et al. 1998; Bock and MacDougall 1999; Yoon et al. 2007).

The anodic degradation of BQ was studied for the first time by Pulgarin et al. (1994) using $Ti/IrO_2$ and $Ti/SnO_2$ anodes showing that the nature of the anode is the most important parameter. With $Ti/IrO_2$, the ring rupture occurred resulting in the accumulation of carboxylic acids as final products, while at $Ti/SnO_2$ BQ and carboxylic acids are oxidised to $CO_2$.

Feng et al. (1995) and Houk et al. (1998) studied the incineration of BQ using a quaternary metal oxide anode in the absence of a soluble electrolyte, using a solid-polymer electrolyte. The anode showed a good reactivity for the incineration of BQ, but the electrolysis time was excessively long (i.e. 64 h).

Acidic solution of BQ has been treated using amorphous Ni–Nb–Pt–Sn anode, obtaining the removal of 23 % of BQ after 3 h of electrochemical oxidation (Pierna et al. 2001).

Yoon et al. (2007) studied the degradation of BQ in a flow-through cell with carbon fibre and found that the removal efficiency increased with applied current and time. In particular after 12 h of electrolysis at 175 mA, 99.23 % of BQ was removed but only 41.65 % were mineralised to $CO_2$.

In the last decades, many papers demonstrated that the complete mineralization of organic compounds can be

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obtained with high efficiency by direct electrolysis using synthetic boron-doped diamond (BDD) as anode material (Panizza and Cerisola 2009a, 2005; Martínez-Huitle and Ferro 2006). With this electrode, which present an inert surface with low adsorption properties and a wide potential window for water discharge, the organics and their intermediates are oxidised to $CO_2$ by $^{\bullet}OH$ radicals electrogenerated from water discharge:

$$2H_2O \rightarrow 2^{\bullet}OH + 2H^+ + 2e^- \quad (1)$$

It is reported that many organic compounds such as phenols (Elaoud et al. 2011; Panizza and Cerisola 2009b; Rodrigo et al. 2001; Iniesta et al. 2001; Canizares et al. 2005; Morao and Lopes 2004; Panizza et al. 2000), dyes (Panizza and Cerisola 2008; Canizares et al. 2006; Faouzi et al. 2006; Martinez-Huitle and Brillas 2009; Hammami et al. 2008; Panizza and Cerisola 2007), drugs (Guinea et al. 2009; Flox et al. 2006; Brillas et al. 2005; Ozcan et al. 2008; Ciriaco et al. 2009; Zhao et al. 2009) and real effluents (Martínez-Huitle et al. 2012; Panizza and Martinez-Huitle 2013; Panizza et al. 2006; Canizares et al. 2007a; Canizares et al. 2009, 2007b) have been successively removed using BDD, even with current efficiency approaching 100 %.

The aim of this work is to study the degradation of a solution containing BQ using a flow cell with a BDD anode. The effect of the main operating parameters, such as current density, flow rate, organic concentration and temperature on chemical oxygen demand (COD) decay and BQ removal has been investigated.

## Experimental

The synthetic solution was prepared dissolving different amounts of BQ ($C_6H_4O_2$) in distilled wastewater in 0.5 M $HClO_4$. $HClO_4$ was chosen as supporting electrolyte, because it does not generate some oxidising species liable to react with organics, as occurs using $Cl^-$ medium (i.e. generation of $Cl_2$) or $SO_4^{2-}$ medium (i.e. production of $S_2O_8^{2-}$).

The boron-doped diamond (BDD) thin-film electrode was supplied by Adamant Technologies (Switzerland). It was synthesised by hot filament chemical vapour deposition technique (HF CVD) on single crystal p-type Si wafers. The filament temperature ranged from 2,440 to 2,560 °C, while the substrate temperature was 830 °C.

The obtained diamond film had 1 μm thickness. In order to stabilise the electrode surface and obtain reproducible results, the diamond electrode was pre-treated by anodic polarisation in 1 M $HClO_4$ at 10 mA $cm^{-2}$ for 30 min. Following this treatment the surface became hydrophilic.

Bulk oxidations were performed in a one-compartment electrolytic flow cell under galvanostatic conditions using an AMEL 2055 potentiostat/galvanostat. BDD was used as the anode, and stainless steel as the cathode. Both electrodes were square, each with 50-$cm^2$ geometrical area and 1-cm inter-electrode gap. The solution was stored in a 0.4-$dm^3$ thermoregulated glass tank and circulated through an electrochemical reactor by a centrifugal pump with different flow rates in the range of 100–300 $dm^3$ $h^{-1}$.

Current efficiency (CE) for anodic oxidation was calculated from COD values, using the following relationship (Panizza et al. 2008):

$$CE(\%) = \frac{(COD_t - COD_{t+\Delta t})}{8 \cdot I \cdot \Delta t} F \cdot V \cdot 100 \quad (2)$$

where $COD_t$ and $COD_{t+\Delta t}$ are chemical oxygen demands at times $t$ and $t+\Delta t$ (in $g_{O2}dm^{-3}$) respectively, $I$ is the current (A), $F$ is the Faraday constant (96,487 $Cmol^{-1}$), $V$ is the electrolyte volume ($dm^3$), and 8 is the oxygen equivalent mass ($geq.^{-1}$).

The specific energy consumption ($E_C$, in kWh $m^{-3}$) was obtained as follows:

$$E_C = \frac{U_{cell} I t}{V 3600} \quad (3)$$

where $U_{cell}$ is the average cell voltage (V), $I$ is the current (A), $t$ is the electrolysis time (s) and $V$ is the volume of the treated solution ($dm^3$).

Evolution of BQ concentrations during electrolyses was monitored by high-performance liquid chromatography (HPLC) using spectra monitor 3100 chromatograph equipped with UV detector at 254 nm and fitted with an Alltech C18 5u column (length 250 mm, i.d.4.6 mm) at 25 °C. The analyses were carried out isocratically with an acetonitrile/water 25:75 (v/v) mixture as mobile phase at a flow rate of 1 $cm^3$ $min^{-1}$.

## Results and discussion

The effect of several operational parameters including applied current, initial organic concentration, flow rate and temperature on COD and BQ removal was studied during the electrochemical treatment using BDD electrodes.

Figure 1 shows the trend of the COD and current efficiency (inset) with time during the anodic oxidation of 2 g $L^{-1}$ of BQ at different current densities. For all the applied currents, the COD decreases up to the detection limit (about 15 mg $L^{-1}$), indicating complete mineralization (>98 % COD removal) from overall reaction (Eq. 4) by means of its reaction with

$^{\bullet}$OH electrogenerated on BDD surface from water discharge (Eq. 1):

$$C_6H_4O_2 + 24^{\bullet}OH \rightarrow 6CO_2 + 14H_2O \quad (4)$$

Increasing the current from 0.5 to 1 A resulted in a faster COD removal rate (i.e. dCOD/dt), meaning that at low current, the process is under charge transfer control because a greater charge passing into the cell favours the oxidation of the pollutant and its intermediates.

The increase to 2 A does not produce significant improvement in the oxidation rate with respect to 1 A, and this behaviour indicates that at high current, the oxidation of BQ became under mass-transport control and an increase of the applied current mainly favours the secondary reaction of hydroxyl radicals decomposition to oxygen:

$$2^{\bullet}OH \rightarrow O_2 + 2H^+ + 2e^- \quad (5)$$

This was confirmed by the evolution of the current efficiency (Fig. 1, inset). In fact, at 0.5 A, at the beginning of the electrolysis, the CE remained about 100 %; this is a characteristic of electrolysis performed at a current below the limiting one, and under these conditions the oxidation of BQ is controlled by the rate at which electrons are delivered at the anode. On the contrary, at 2 A, CE is always below 100 % and it decreases with time, meaning that the oxidation was carried out at current density higher than the limiting one and the process was under mass-transport control.

The decay of BQ concentration during the electrolyses at different current densities has been followed by reversed-phase HPLC, and the results are reported in Fig. 2. It can be observed that BQ removal is slightly influenced by applied current, and in any condition, the BQ decays could be very satisfactory described by pseudo-first-order kinetics (Fig. 2, inset). The pseudo-first-order reaction can be attributed to the fact that in galvanostatic conditions, a stationary concentration of $^{\bullet}$OH radicals is reached at the electrode surface. From this analysis, it was found that the pseudo-first-order rate constant increases from $5.65\times10^{-4}$ s$^{-1}$ to $6.69\times10^{-4}$ s$^{-1}$ and $8.28\times10^{-4}$ s$^{-1}$ with increasing current from 0.5 to 1 and 2 A, respectively. From the data of Fig. 2, it is also possible to calculate the specific charge (Ah dm$^{-3}$) necessary for the complete removal of BQ from the solution. The specific energy was 2.8, 4.5 and 9.4 Ah dm$^{-3}$ for the electrolyses at 0.5, 1 and 2 A respectively. These values were lower than that obtained by Pulgarin et al. (1994) for the oxidation of BQ using $IrO_2$ or $SnO_2$ anodes (i.e. 100 and about 14 Ah dm$^{-3}$, respectively). By comparing the results reported in Figs. 1 and 2, it is worth noting that BQ is removed more rapidly than the COD of the solution, meaning that some oxidation intermediates are formed. Hydroquinone and aliphatic carboxylic acids such as maleic, fumaric, acetic and oxalic acids have been identified as the main intermediates. These findings fit very well with the results reported by Pulgarin et al. (1994) using a $SnO_2$ anode for the anodic oxidation of BQ. Figure 3 shows the influence of the current density on the evolution of

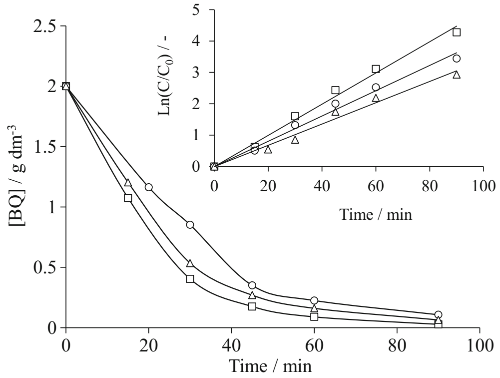

**Fig. 2** Effect of applied current on the evolution of BQ concentration during the electrolysis of 1 g dm$^{-3}$ of BQ aqueous solution at applied current of (*circle*) 0.5 A, (*triangle*) 1 A and (*box*) 2 A. Conditions: *T*= 20 °C; flow rate=300 dm$^3$ h$^{-1}$. The *inset* presents the corresponding kinetic analysis assuming a pseudo-first-order reaction

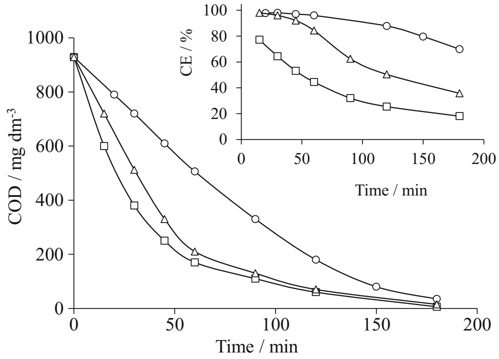

**Fig. 1** Effect of applied current on the evolution of COD and CE (*inset*) during the electrolysis of 1 g dm$^{-3}$ of BQ aqueous solution at applied current of (*circle*) 0.5 A, (*triangle*) 1 A and (*box*) 2 A. Conditions: *T*= 20 °C; flow rate=300 dm$^3$ h$^{-1}$

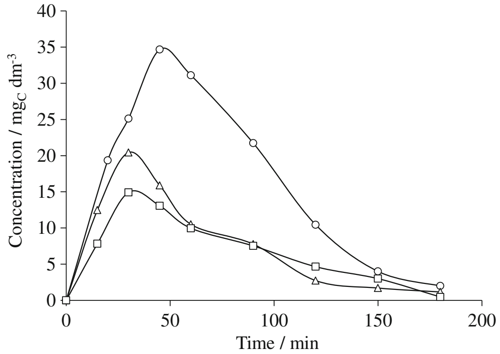

**Fig. 3** Time evolution of the reaction intermediates, expressed as mg$_C$ dm$^{-3}$, during the oxidation of g dm$^{-3}$ of BQ at different current densities. Conditions: *T*=20 °C; flow rate=300 dm$^3$ h$^{-1}$; applied current (*circle*) 0.5 A; (*triangle*) 1 A and (*box*) 2 A

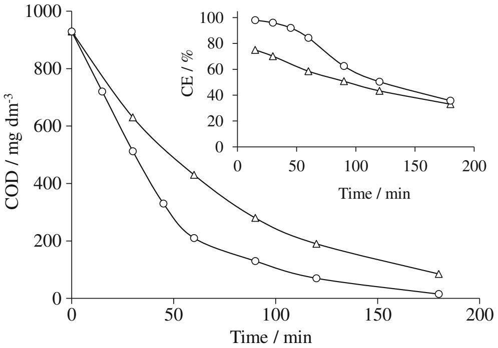

**Fig. 4** Effect of recirculation flow rate on the evolution of COD and CE (*inset*) during the electrolysis of 1 g dm$^{-3}$ of BQ aqueous solution. Conditions: $T$=20 °C; applied current, 1 A; flow rate, (*triangle*) 100 dm$^3$ h$^{-1}$. (*circle*) 300 dm$^3$ h$^{-1}$

oxidation intermediates, expressed as mg of carbon dm$^{-3}$. At higher current density, lower amounts of intermediates are accumulated in the medium, since BQ oxidation is accelerated by reaction with more hydroxyl radicals. However, after 180 min, all the intermediates are mineralised to $CO_2$ and water.

The influence of the recycle flow rate on COD removal during BQ oxidation at 1 A is shown in Fig. 4. The overall COD depletion is lower as the recycle flow rate is diminished; thus, meaning that at low recycle flow rate, the oxidation becomes a diffusion-controlled process. In fact, the increase in the recycle flow rate accelerates the transport of the organics towards the electrode surface, where they react with the electrogenerated hydroxyl radicals, and, consequently, they disappear from the solution more quickly and with higher current efficiency (Fig. 4, inset).

Figure 5 shows the effect of the initial BQ concentration on COD and CE evolution during electrolysis at 25 °C by applying a current of 1 A and with a recycle flow rate of 300 dm$^3$ h$^{-1}$. Overall COD removal is achieved in all cases and the time for total mineralization increased with BQ concentration as expected from the presence of a greater amount of organic in the solution.

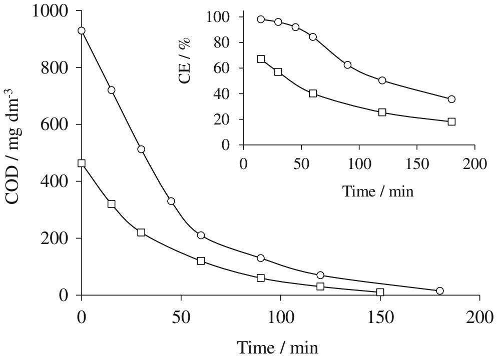

**Fig. 5** Effect of initial BQ concentration on the evolution of COD and CE (*inset*) during the electrolysis at $T$=20 °C; applied current, 1 A; flow rate=300 dm$^3$ h$^{-1}$. BQ concentration, (*box*) 1 g dm$^{-3}$ and (*circle*) 2 g dm$^{-3}$

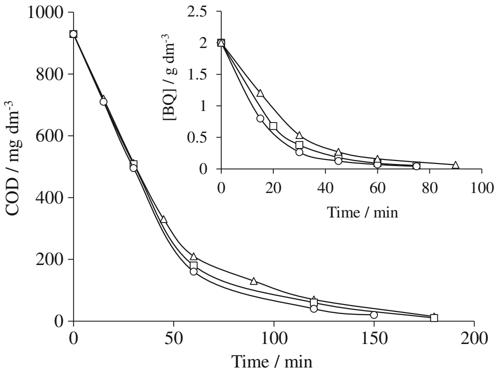

**Fig. 6** Effect of temperature on the evolution of COD and BQ concentration (*inset*) during the electrolysis of 1 g dm$^{-3}$ of BQ. Conditions: applied current, 1 A; flow rate=300 dm$^3$ h$^{-1}$. Temperature, (*triangle*) 20 °C, (*box*) 35 °C and (*circle*) 45 °C

However, it is interesting to observe that at high initial concentration, at the beginning of the electrolysis, the COD decreased linearly with specific charge and CE remained about 100% (Fig. 5, inset), indicating that the electrolysis was under charge transfer control. On the contrary, at low concentration, COD decreases exponentially and CE is below 100 %, meaning that the oxidation was carried out at current density higher than the limiting one and the process was under mass-transport control.

The variation of the COD and BQ concentration during the electrolysis at 20, 35 and 45 °C when applying a current density of 1 A and with a recycle flow rate of 300 dm$^3$ h$^{-1}$ is presented in Fig. 6. In a medium that cannot generate oxidising species, such as $HClO_4$, higher temperatures do not yield a significant increase of the oxidation rate of COD and BQ removal during the electrochemical process. This result is in agreement with other previous studies about the oxidation of naphthol (Panizza et al. 2001) and acid yellow (Rodriguez et al. 2009) on BDD. The small difference between the results obtained at different temperatures is only due

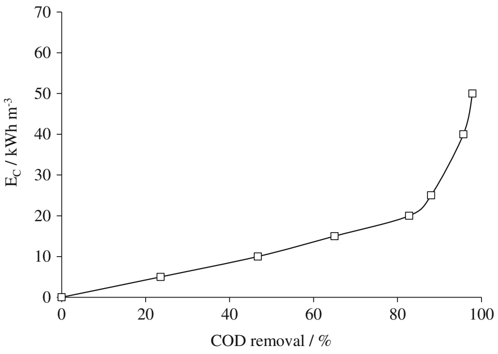

**Fig. 7** Evolution of specific energy consumption ($E_C$) with COD removal during the electrolysis of 1 g dm$^{-3}$ of BQ aqueous solution. Conditions: $T$=45 °C; applied current, 1 A; flow rate, 300 dm$^3$ h$^{-1}$

to an increase of the diffusion rate with rising temperature due to the decrease of the medium viscosity.

From this kinetic analysis, it was found that the pseudo-first-order rate constant for BQ oxidation increases from $6.69\times10^{-4}$ $s^{-1}$ to $8.51\times10^{-4}$ $s^{-1}$ and $9.42\times10^{-4}$ $s^{-1}$ with increasing temperature from 20 to 35 and 45 °C, respectively.

The apparent activation energy for the electrochemical oxidation of BQ at the BDD electrode was found to be 10 kJ $mol^{-1}$, which is close for a diffusion-controlled homogeneous reaction (typically less than 40 kJ $mol^{-1}$ (Elaoud et al. 2011)).

For large application, it is also very important to estimate the treatment costs, and thus Fig. 7 reports the variation of specific energy consumption, calculated from Eq. (3), as function of COD removal, in the best operating conditions found previously (i.e. flow rate 300 $dm^3$ $h^{-1}$; $T$=45 °C; $I$=1 A). For low COD removal, the specific energy consumption increases almost linearly, while for COD removal higher than 80 % $E_C$ increased exponentially. This behaviour can be probably explained by the formation of more refractory products, such as carboxylic acids which are hardly oxidisable intermediates, and the decrease of organic content in the solution. Energy costs for total mineralization was about 49.5 kWh $m^{-3}$.

## Conclusions

Electrochemical oxidation using a BDD anode has been successfully applied to treat a solution containing BQ. The experimental results showed that:

- Within the range studied, the almost total BQ and COD removal was obtained, regardless of the experimental conditions, due to the formation of hydroxyl radicals from the water discharge.
- The oxidation of BQ is well described by a pseudo-first-order kinetic, because in galvanostatic electrolysis there is a stationary concentration of $^{\bullet}$OH radicals on the BDD surface.
- At high BQ concentration, low current and high flow rate, the oxidation is controlled by charge transfer, while at low concentration, low flow rate and high current, it is under mass-transport control.
- At low current, high current efficiency (i.e. 100 %) was obtained but high amount of intermediates were formed, while at high current, the efficiency was lower but less amount of intermediates were formed.
- The COD removal rate increases with applied current and flow rate, while it is almost unaffected by solution temperature.

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