



Electrochemical promotion of strong oxidants to degrade Acid Red 211: Effect of supporting electrolytes



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ABSTRACT

Solutions of the synthetic dye Acid Red 211 (AR211), commonly used in many industries, were comparatively degraded using different supporting electrolytes under current-controlled electrolysis conditions. The efficiency of the electro-oxidation (EOx) approach with boron doped diamond electrodes (EOx/BDD) was examined by applying 100, 200, 300 and 400 mA with HClO₄, H₂SO₄ or Na₂SO₄ as a supporting electrolyte. Under similar experimental conditions, a diamond electro-Fenton (EF/BDD) system was used for treating synthetic dye solutions and was compared with the removal efficiencies obtained by EOx/BDD. The results clearly showed that when AR211 was oxidized by employing the EF/BDD process with generation of free ·OH from the electrogeneration production of hydrogen peroxide (H₂O₂) and addition of Fe²⁺, the oxidizing power increased to 97% under the best conditions, with higher current efficiencies and lower energy consumption than those obtained by EOx/BDD. In this study, we also attempt to discuss critical evidence regarding the viability of the use of different supporting electrolytes because the results obtained in this research clearly demonstrated that the effect of the electrogenerated strong oxidant species depends on parallel mechanisms followed on the BDD surface, as well as the particular production of strong oxidants, such as the case of BDD(·OH) or BDD(SO₄^{·-}).

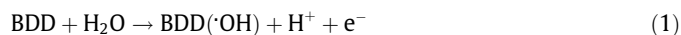
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1. Introduction

Worldwide, different industries produce a large quantity of wastewater that is highly colored and contains high levels of organic matter [1]. The use of large quantities of dyestuffs during the dyeing stages of leather manufacturing processes is a cause of such pollution that causes major aesthetic and environmental problems [2,3].

Technologies such as biological, physical and chemical processes have been carried out for organic matter and color reduction [4]. However, most disposed synthetic dyes are of a non-biodegradable nature, and direct biological treatment of the colored effluents is not effective [2,4]. Therefore, it is necessary to propose effective techniques to treat these effluents, both in terms of limited water resource management and the need for

environment preservation. Among these techniques, so-called advanced oxidation processes (AOPs) are powerful treatment technologies which generate highly reactive free hydroxyl radicals (·OH) able to mineralize several toxic and non-biologically degradable compounds [5,6]. Nevertheless, in recent years, electrochemical advanced oxidation processes (EAOPs) have been considered promising technologies which offer favorable approaches for the prevention of pollution problems from industrial effluents [2,3,7–12]. The most important EAOP is electrochemical oxidation (EOx), where organics are oxidized via ·OH produced from water discharge at the surface [9–11,13]. The anodes (with a high O₂-overpotential) principally used for this electrochemical approach are PbO₂, SnO₂ and, more recently, boron doped diamond electrodes (BDD) [9–11], according reaction (1). BDDs are considered the best anodes for EOx due to their wide electrochemical window, low adsorption ability, large chemical stability and higher O₂-overpotential than other electrodes [13].



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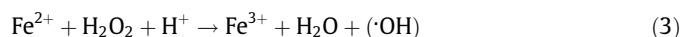
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Thanks to these properties, organics can be incinerated by the large amounts of weakly physisorbed $\cdot\text{OH}$ generated from water oxidation at high current. On the other hand, indirect electro-oxidation methods based on Fenton's reaction chemistry such as electro-Fenton (EF) and solar photoelectro-Fenton (SPEF) are being developed for the remediation of wastewaters with organic pollutants [12–16]. In the case of EF, H_2O_2 is electrochemically produced by the two-electron reduction of injected O_2 at the appropriate cathode from reaction (2) [12,13]:



However, efficient production of H_2O_2 is achieved when carbonaceous cathodes are used, such as carbon nanotubes–polytetrafluoroethylene (PTFE) [17,18] carbon-felt [19–21] carbon sponge [21], graphite-felt [22] carbon-PTFE gas O_2 or air diffusion electrodes [23–27] and BDD electrodes [28,29]. Moreover, the oxidation power of H_2O_2 is improved by adding catalytic quantities of Fe^{2+} to produce Fe^{3+} and, after that, the production of free $\cdot\text{OH}$ is promoted in the bulk from the Fenton reaction (3), under an optimum range of pH from 2.8 to 3.0 [12,28]. The advantages of this EAOP compared with the chemical Fenton reagent is the in situ H_2O_2 production and the regeneration of Fe^{2+} catalysts at the cathode from Fe^{3+} reduction by reaction (4) [12,24–28]:



When an undivided cell with a BDD anode and BDD cathode is used (BDD/BDD), organics are mineralized into water, carbon dioxide and inorganic ions by both BDD($\cdot\text{OH}$) formed from reaction (1) and $\cdot\text{OH}$ produced in the bulk from the Fenton reaction (3).

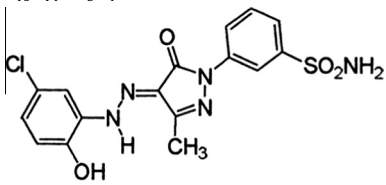
In light of this information, the present work reports a preliminary study on the degradation of the synthetic dye AR211 by using different supporting electrolytes (H_2SO_4 , HClO_4 and Na_2SO_4). One of the factors that influences the production of free radicals is the electrolytic medium in which they are generated; several studies have shown that a change of pH in the solution affects both the rate of degradation of organic compounds and the rate of free radical generation; however, $\cdot\text{OH}$ production has not been specifically studied under the influence of the supporting electrolyte. The effect of the media and the pH on the process efficiency was evaluated by total organic carbon (TOC) measurements during current-controlled electrolysis of synthetic dye AR211 solutions.

2. Experimental

2.1. Chemicals

Synthetic dye AR211 (Table 1) was provided by local industry. The chemicals used as H_2SO_4 , Na_2SO_4 , and ferrous iron ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ – catalyst) were supplied by Baker, while HClO_4 and titanium (IV) isopropoxide-97% were from Aldrich. All other reagents were

Table 1
Physico-chemical properties of the synthetic dye AR211.

Chemical formulation	$\text{C}_{16}\text{H}_{14}\text{ClN}_5\text{O}_4\text{S}$
Chemical structure	
Molecular weight (g/mol)	407.83
Registration number (CAS)	12239-05-3

of analytical grade, and these reagents were used without further purification. Solutions were prepared with high-purity water from a Millipore-Elix system, with resistivity $\geq 18 \text{ M}\Omega \text{ cm}$ at 25°C .

2.2. Electrochemical reactor and procedures

Electrochemical experiments were performed at room temperature in a 0.5 L undivided filter flow press reactor equipped with two electrodes. The anode and cathode electrodes have a geometrical area of 25 cm^2 of niobium plate with a diamond-coated film thickness of 2–7 μm . BDD electrodes were provided by Metakem GmbHTM, Germany. For the EOx process, BDD anodes and stainless steel cathodes were used. The inter-electrode gap was 5 mm. The synthetic solutions were placed in the reservoir and recirculated through the system using a diaphragm pump, as seen in Fig. 1. The flow rate is kept constant at 200 mL min^{-1} . The degradation of 100 mg L^{-1} aqueous synthetic dye AR211 solutions was carried out under current-controlled electrolysis conditions at 100, 200, 300 and 400 mA using a BK Precision–1688B power supply. Assays were monitored by TOC using a Shimadzu TOC-L. Prior to the electrolysis, compressed air was bubbled for 30 min through the solution. A catalytic amount of ferrous iron of 0.5 mM was added to solutions before starting the electrolysis. Supporting electrolyte concentrations were 0.1 mol L^{-1} for HClO_4 and H_2SO_4 , in the case of Na_2SO_4 0.05 mol L^{-1} were utilized. The perchloric acid medium was chosen in accordance with the literature revised [30], since no ferric or ferrous complexes have been determined in the presence of ClO_4^- ion.

Evaluation of H_2O_2 accumulation in the system was carried out by testing H_2O_2 generation in the different electrolyte solutions. The H_2O_2 concentration was determined using the $\text{Ti}(\text{SO}_4)_2$ titration method and spectrophotometric analysis at $\lambda = 407 \text{ nm}$.

3. Results and discussion

3.1. EOx/BDD experiments

EOx experiments applying different currents (100, 200, 300 and 400 mA) were carried out to understand the effect of the supporting electrolyte (H_2SO_4 , HClO_4 and Na_2SO_4) during the electrochemical degradation of AR211. As can be observed in Fig. 2, TOC decay was followed, as a function of time, at different current

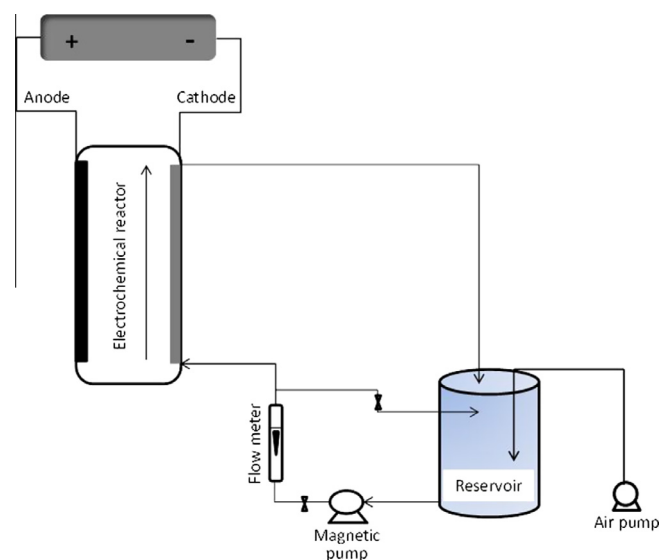


Fig. 1. Flow circuit for the electrochemical reactor.

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