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Synergistic integration of sonochemical and electrochemical disinfection with DSA anodes

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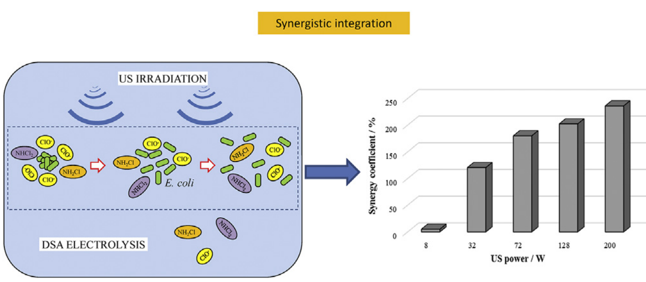
GRAPHICAL ABSTRACT



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ABSTRACT

This work focuses on the disinfection actual urban wastewater by the combination of ultrasound (US) irradiation and electrodisinfection with Dimensionally Stable Anodes (DSA). First, the inactivation of *Escherichia coli* (*E. coli*) during the sonochemical disinfection was studied at increasing ultrasound power. Results showed that it was not possible to achieve a complete disinfection, even at the highest US power (200 W) dosed by the experimental device used. Next, the electrodisinfection with DSA anodes at different current densities was studied, finding that it was necessary a minimum current density of 11.46 A m^{-2} to reach the complete disinfection. Finally, an integrated sonoelectrodisinfection process was studied. Results showed a synergistic effect when coupling US irradiation with DSA electrodisinfection, with a synergy coefficient higher than 200% of the disinfection rate attained for the highest US power applied. In this process, hypochlorite and chloramines were identified as the main reagents for the disinfection process (neither chlorate nor perchlorate were detected), and the presence of trihalomethanes was far below acceptable values. Confirming this synergistic effect with DSA anodes opens the door to novel efficient disinfection processes, limiting the occurrence of hazardous disinfection by-products.

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1. Introduction

In recent years, electrochemical disinfection has attracted the interest of several researchers, being the main target the development of economic and safe disinfection technologies which prevent the occurrence of hazardous disinfection by-products (Mezule et al., 2013; Haaken et al., 2014; Cotillas et al., 2015). One of the milestones of these studies has been the applicability of boron-doped diamond (BDD) anodes for the treatment of synthetic

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wastewater polluted with *Escherichia coli* (*E. coli*) (Gusmão et al., 2010; Li et al., 2010; Cui et al., 2013; Lacasa et al., 2013). These anodes are classified into the group of non-active electrodes and exhibit high performance in disinfection and in the removal of several type of pollutants (Marselli et al., 2003; Martínez-Huitle et al., 2012; Panizza, 2014; Garcia-Segura et al., 2015).

Unfortunately, the electrochemically-assisted disinfection with BDD anodes favours the generation of chlorates and perchlorates (Bergmann and Rollin, 2007; Bergmann, 2010; Vacca et al., 2011) which are known to be harmful for human health. This fact limits the maximum value of current density and electric charge applicable in disinfection processes (Llanos et al., 2014). Opposite to electrolysis with diamond coatings, production of chlorate and perchlorate is expected to be limited with the well-known Dimensionally Stable Anodes (DSA) based on RuO_2, widely used in chlor-alkali industry for the production of hypochlorite from concentrated brines (Kiros et al., 2006; Mohaddes, 2008).

Another novel technology that may exhibit good features for disinfection of urban wastewaters is the application of ultrasound irradiation (Antoniadis et al., 2007). Disinfection can be achieved by different mechanisms: 1) the production of free radicals in the bulk (Juretic et al., 2015); 2) shearing forces produced by ultrasonic cavitation (Drakopoulou et al., 2009); 3) breakage of bacteria agglomeration, that favours the reaction between oxidants species and microorganisms and therefore, the disinfection processes (Hughes and Nyborg, 1962).

In this context, in a previous research it was found that ultrasonic cavitation dramatically increases the efficiency of the inactivation of *E. coli* working with BDD anodes and low current densities (in order to prevent the formation of chlorate and perchlorate) (Llanos et al., 2015). This improved performance was explained in terms of a more efficient production of oxidants and to the promotion of the dispersion of agglomerated cells, favouring the attack to microorganisms.

Thus, the aim of the present work is to study the potential synergistic effect of coupling ultrasound irradiation and electrolysis with DSA anodes to the disinfection of actual effluents from municipal wastewater treatment plants (WWTP). This study would widen the application of the combined sono-electrodisinfection process to higher values of current densities and applied electric charges, limiting the formation of hazardous disinfection by-products as chlorate. In a first approach, the efficiency of single sonodisinfection and electrodisinfection processes was studied, evaluating the influence of the applied ultrasound power and current density on the process performance. Next, US irradiation was coupled with the electrochemical disinfection with DSA anode in order to evaluate the potential synergies of both techniques.

2. Material and methods

2.1. Analytical techniques

Faecal coliforms were estimated using the most probable number (MPN) technique (APHA-AWWA-WPCF, 1998). Microorganism counts were carried out by the multiple-tube-fermentation technique (24 h of incubation at 44 °C) using 5 tubes at each dilution (1:10, 1:100, and 1:1000).

Nitrogen and chloride inorganic anions (NO_3^-, NO_2^-, Cl^-, ClO^-, ClO_2^-, ClO_3^-, ClO_4^-) were measured by ion chromatography using a Shimadzu LC-20A equipped with Shodex IC I-524A column; mobile phase, 2.5 mM phthalic acid at pH 4.0; flow rate, 1.0 mL min^{-1}). The peak of hypochlorite interferes with that of chloride; therefore, the determination of hypochlorite was carried out by titration with As_2O_3 in 2 M NaOH. The same ion chromatography equipment (Shodex IC YK-421 column; mobile phase, 5.0 mM tartaric, 1.0 mM dipicolinic acid and 24.3 mM boric acid; flow rate, 1.0 mL min^{-1}) was used to measure the nitrogen inorganic cation (NH_4^+).

Inorganic chloramines were measured following the DPD standard method described in the literature (APHA-AWWA-WPCF, 1998).

Occurrence of trihalomethanes ($CHCl_3$, $CHBrCl_2$, $CHBr_2Cl$, $CHBr_3$) was followed by gas chromatography with an electron capture detector (ECD). The column used was a SPB 10 column (30 m × 0.25 mm; macroporous particles with 0.25 μm diameter). The flow rate was 50 mL min^{-1} and the initial temperature was 50 °C during 5 min until reach a final value of 150 °C (6 °C min^{-1}). Injection volume was set to 1 μL during 5 min and hydrogen was used as carrier gas.

2.2. Experimental setup

The sonodisinfection, electrodisinfection and sono-electrodisinfection processes were carried out in a single-compartment electrochemical cell. Dimensionally Stable Anode (DSA) based on RuO_2 (Tianode, India) was used as anode and stainless steel (SS) (AISI 304) (Mervilab, Spain) as cathode. Both electrodes (anode and cathode) were circular with a geometric area of 78.5 cm^2 and the electrode gap was 10 mm.

Wastewater was stored in a glass tank (2.5 L). The ultrasound generator (UP200S, Hielscher Ultrasonics GmbH, Germany) is equipped with a horn (40 mm diameter, 100 mm length, 12 Wcm^{-2}, 24 kHz) that is located inside the glass tank. The maximum power of ultrasound irradiated is 200 W and the output can be continuous or pulsed with duty cycle ranging from 10 to 100%.

The system works in total recirculation mode, with a peristaltic pump (JP Selecta Percom N-M328) continuously recycling the target wastewater. The power supply is a Delta Electronika ES030-10. Temperature of the system is kept constant by means of a thermostatised bath (JP Selecta, Digiterm 100) and a heat exchanger. A schematic of the experimental set-up has been described elsewhere (Llanos et al., 2015).

2.3. Experimental procedure

Bench-scale electrolyses and sonoelectrolyses of 2000 mL of wastewater were carried out under galvanostatic conditions. The current density applied ranged from 6.37 to 25.46 A m^{-2}. The cell voltage did not vary during electrolysis, indicating that RuO_2 layers did not undergo appreciable deterioration or passivation phenomena. Prior to use in galvanostatic electrolysis assays, the electrode was polarized for 10 min in a 5000 mg L^{-1} Na_2SO_4 solution at 150 A m^{-2} to remove any kind of impurity from its surface.

Sonochemical disinfection of 2000 mL was carried out at ultrasound power ranged from 0 to 200 W.

2.4. Target effluents

The target wastewater used in this study is the effluent of the secondary clarifiers of the municipal WWTP of Ciudad Real (Spain). The WWTP treats the wastewater produced in an average-sized town (75,000 p.e.) located in the centre of Spain. The influent is domestic wastewater without a significant industrial contribution. However, the effluent obtained after the treatment presents high concentration of microorganisms and prevents the direct reuse of treated wastewater. The average characteristics of the samples used in this work are shown in Table 1.

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