



Cold incineration of sucralose in aqueous solution by electro-Fenton process



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ABSTRACT

The effective removal of artificial sweeteners sucralose (SUC) from water was carried out for the first time by an electrochemical advanced oxidation method. Electro-Fenton process was used to generate hydroxyl radicals, a highly powerful oxidant species that are able to oxidize organic pollutants until their mineralization at room temperature. The effect of catalyst (Fe^{2+}) concentration and applied current on the mineralization of SUC during electro-Fenton treatment was evaluated. Results showed that the electro-Fenton process is capable to mineralize SUC solution effectively. A higher mineralization rate was obtained with BDD than Pt anode. Mineralization current efficiency results pointed out better mineralization efficacy (MCE) at relatively short electrolysis time and low applied current values. The formation and evolution of the concentration of carboxylic acids and released inorganic ions, as a consequence of mineralization process, were followed along the treatment. Finally, the toxicity of SUC and its oxidation intermediates products was assessed along the mineralization process using Microtox method to show the detoxification capacity of the process.

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1. Introduction

Artificial sweeteners, representing 62% of the commercial sweetener market, have been used in a wide variety of low calorie food and drink products, confectionery, as well as pharmaceuticals [1,2]. Sucralose (SUC), which is a chlorinated disaccharide derived from sucrose, is one of the most popular artificial sweeteners and has shown increasing trend of consumption [1]. SUC is about 600 times sweeter than the parent molecule, sucrose [3,4]. Nowadays, SUC is approved in more than 80 countries and is used widely in food products and pharmaceuticals [3]. It is not metabolized by the human body due to the orientation of the glycosidic linkage, and consequently, up to 92% of the consumed SUC is excreted unchanged in both urine and feces [1,3]. However, this molecule can develop potential adverse effects on health, since it provokes symptoms, such as, increase in blindness, mineralization of pelvic area and epithelial hyperplasia [5]. Nowadays, SUC has been detected in wastewaters and rivers in many countries, such as American, France, Italy and China [5,6]. Recently, SUC is considered

as an emerging contaminant by the U.S. Environmental Protection Agency (US-EPA) due to its occurrence in environmental waters and persistence (half-life up to several years) [5,7,8]. Unfortunately, it is hard to degrade by conventional wastewater treatments processes [3,9,10]. Therefore, it is crucial to develop powerful water treat technologies for the removal of SUC and other sweeteners from polluted effluents.

During the last decades, advanced oxidation processes (AOPs) have attracted great attention for the treatment of organic pollutants in aqueous solutions [11]. AOPs involve the in-situ generation of hydroxyl radicals ($\cdot\text{OH}$) which are a highly powerful oxidizing agents ($E^0 = 2.80 \text{ V/SHE}$) through chemical, photochemical or electrochemical techniques to bring about chemical degradation of organic pollutants [12,13]. Different AOPs have been employed to remove SUC from water, including ozonation [14,15], H_2O_2 photolysis [16], photo-Fenton and TiO_2 photocatalysis [5] processes. UV irradiation at around 254 nm has been shown to be inefficient because of its very low molar extinction coefficient ($<20 \text{ M}^{-1} \text{ cm}^{-1}$) in the UV region and almost no absorbance above 230 nm [3,15,16]. Ozonation was found to be not enough efficient in SUC removal due to the absence of any evident sites for direct oxidation [13]; however its oxidative degradation was carried out by $\cdot\text{OH}$ generated in the oxidative system such as $\text{hv}/\text{H}_2\text{O}_2/\text{O}_3$ [3,15]. On

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the other hand, the processes involving generation of $\cdot\text{OH}$ like homogeneous photo-Fenton and TiO_2 based heterogeneous photocatalysis were able to remove SUC efficiently from water [5]. More recently, Xu et al. studied the mineralization of SUC by UV/peroxydisulfate (PDS) system reaching its almost complete mineralization when using 30-fold excess of PDS [17]. These studies showed that $\cdot\text{OH}$ based AOPs are able to eliminate SUC from water in contrast of conventional treatment processes.

Among the AOPs, electro-Fenton process has attracted great attention and has been applied successfully to the destruction of toxic and biorefractory organic pollutants [18]. This process is based on the electrochemical generation of homogeneous $\cdot\text{OH}$ from on-site production of Fenton's reagent (H_2O_2 and Fe^{2+}). H_2O_2 is generated at the cathode of an electrochemical reactor from 2-electron reduction of dissolved O_2 (Eq. (1)) [19]. The cathodes which favor the generation of H_2O_2 are carbon felt [20–22], carbon sponge [23], activated carbon fiber [24] and O_2 diffusion cathode [25,26].



The oxidizing power of H_2O_2 is enhanced in the presence of Fe^{2+} through classical Fenton's reaction (Eq. (2)) which leads to the production of $\cdot\text{OH}$ [27–30].



Moreover, the Fe^{2+} consumed in Fenton's reaction can be regenerated via cathodic reduction of Fe^{3+} (Eq. (3)) in electro-Fenton process [18,31].



Therefore, a catalytic amount (0.1–0.2 mM) of Fe^{2+} is needed in electro-Fenton contrarily to classical Fenton process that requires high amount of iron(II) salt. Thanks to the electrocatalytic regeneration of Fe^{2+} (catalyst), electro-Fenton process overcomes the drawbacks of classical Fenton process avoiding the use of large quantities of Fe^{2+} and formation of large ferric hydroxide sludge [22,32,33].

Meanwhile, hydroxyl radicals can also be generated from water oxidation at a high O_2 -overtoltage anode (M) such as PbO_2 , Pt and BDD (Eq. (4)) [34–40].



Therefore, in the case of using a high O_2 -overtoltage anode in electro-Fenton process, organic pollutants can be destructed simultaneously and quickly both by $\cdot\text{OH}$ generated from Fenton's reaction (Eq. (2)) in the bulk solution and $\text{M}(\cdot\text{OH})$ formed from oxidation of water at the surface of anode (Eq. (4)) [41].

In this study, we investigated the effective removal of artificial sweetener SUC from water, taking advantages of the electro-Fenton technology [12,18]. To the best of our knowledge, this study is the first investigation on complete mineralization (cold incineration) of SUC in water at room temperature by an electrochemical AOP. For this purpose, we examined carefully the effect of the Fe^{2+} (catalyst) concentration and applied current on the mineralization degree of SUC solutions. Then, the effect of applied current on the mineralization was calculated under best operating conditions. As a consequence of mineralization procedure, the formation and evolution of short-chain aliphatic carboxylic acids and the release of inorganic ions were monitored by ion-exclusion chromatography and ion chromatography (IC). The evolution of solution toxicity of SUC solution and its intermediates was assessed throughout the treatment by Microtox method.

2. Materials and methods

2.1. Chemicals

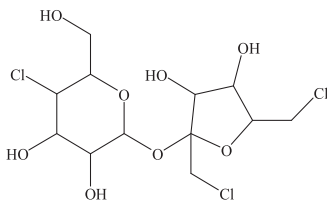
Sucralose (SUC, $\text{C}_{12}\text{H}_{19}\text{Cl}_3\text{O}_8$) was purchased from Sigma-Aldrich. The chemical structure and main characteristics of SUC were presented in Table 1. Analytical grade anhydrous sodium sulfate and heptahydrated ferrous sulfate were obtained from Sigma-Aldrich and Acros Organics, respectively. Analytical grade carboxylic acids and other chemicals used for chromatographic analysis were provided by Acros, Merck, Sigma, Riedel-de Haën and Fluka. Ultrapure water used for the preparation of the working solutions and HPLC eluting solutions was obtained from a Millipore Milli-Q (simplicity 185) system with resistivity $>18 \text{ M}\Omega \text{ cm}$ at room temperature.

2.2. Electrochemical apparatus and procedures

Bulk experiments were conducted in an open, undivided and cylindrical glass cell of 250 mL capacity containing 220 mL SUC solution at room temperature (20–22 °C). Either a cylindrical Pt mesh (4.5 cm height, i.d. = 3.1 cm, from Plateaxis, France) or a 25 cm² thin-film BDD electrode (COMDIAS GmbH, Germany) was used as anode, and a 105 cm² piece of carbon felt (17.5 × 6 cm, Carbon-Lorraine, France) was used as cathode. In all electrolyses, the anode was centered in the cell, surrounded by the carbon felt cathode covering the totality of the inner wall of the electrochemical reactor. H_2O_2 was generated from the reduction of O_2 dissolved in the solution (Eq. (1)). The continuous saturation of oxygen at atmospheric pressure was assured by bubbling compressed air through a frit at about 0.5 L min⁻¹, starting 5 min before the beginning of electrolysis. All the electrolytic trials were conducted under constant current conditions by a Hameg HM8040-3 triple power supply (Germany).

The degradation experiments were performed using 0.2 mM SUC solution with constant stirring by using a magnetic stirrer (Stuart, Britain). This concentration is significantly higher than its concentration in natural water streams or the industrial effluents. It is chosen in order to perform easily the mineralization (TOC removal measurements), and also to show the capability of the process to achieve the treatment even in case of high concentration, such as the elimination of stocks out of date. 50 mM Na_2SO_4 was added into the SUC solution as supporting electrolyte. A catalytic quantity of ferrous ion was added into the solution before the beginning of electrolysis. The initial pH (pH_0) of SUC solutions was measured with a CyberScan pH 1500 pH-meter (Eutech Instrument, USA) and set at 3.0 (± 0.1), adjusting by the addition of 1 M sulfuric acid.

Table 1
Chemical structure and main characteristics of SUC.

Name	Sucralose
Chemical structure	
Molecular formula	$\text{C}_{12}\text{H}_{19}\text{Cl}_3\text{O}_8$
CAS number	56038-13-2
Molecular weight	397.63
Water solubility (g/L)	283 (20 °C)

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