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Applying electro-Fenton process as an alternative to a slaughterhouse effluent treatment

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article info abstract

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Electrochemical oxidation of the effluent of a slaughterhouse facility by electrolysis under galvanostatic control was studied using Ti-RuO₂/carbon felt (CF), Ti-PdPtOx/CF and BDD/CF cells by electro-Fenton at constant current density. Electrode material and the applied current density are significant parameters on oxidative degradation of pollutants in wastewater. It was found that pollutants of the effluent were oxidized to simple substrates and $CO₂$ with different results, obtaining good removal efficiencies of chemical oxygen demand of 83.5%, 77.9% and 74.3%, for the Ti-RuO₂/CF, Ti-PdPtOx/CF and BDD/CF systems, respectively. Trends in degradation depend on the production of hydroxyl radicals ('OH) on the anodic materials and coagulation as confirmed by this study. Efficiency of electrochemical oxidation process was tested by carrying out chemical oxygen demand (COD) determinations. Additional operating parameters such as pH, initial ferrous concentration and electrolysis time were also evaluated.

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1. Introduction

Currently, the increasing demand for water by the world population, as reflected by the existence of more than 40 countries which are experiencing a water deficit [\[1\]](#page--1-0), leads to the requirement to optimize efficient use, while seeking technological options that fulfill regulatory compliance and facilitate recycling. As the world demand for water increases, the participation of society and industry in identifying solutions is critical. Within the country of Mexico, there are 1141 meat processing facilities with the average consumption for processing being 1000 L/cow and 450 L/pig processed [\[2\]](#page--1-0). The wastewater from this process, rich in organic matter and salts, is usually treated by biological methods, as does the slaughterhouse implemented in this study which is located in the Mexican state of Guanajuato, in the city of León. This facility is investigating depuration options for the generated effluent which currently is treated by a combination of anaerobic digester and batch activated sludge.

An interesting alternative to biological, chemical and photochemical methods for the treatment of organic compounds in aqueous media is the electrochemical oxidation (EOx) [3–[11\]](#page--1-0) by using metallic oxides

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<http://dx.doi.org/10.1016/j.jelechem.2015.07.002> 1572-6657/© 2015 Elsevier B.V. All rights reserved. or boron doped diamonds (BDD), as anodes. Different studies on these materials have shown increased performance levels to the conversion and/or combustion of a wide variety of organic compounds. In the conversion process the organic compounds are only transformed to biocompatible subproducts which can be sufficiently eliminated in a successive biological treatment. The oxidation mechanism proceeds through an oxygen introductory step into the oxide lattice, resulting in the conversion of the organics and a change of the oxidation number in the metals (such as $IrO₂$ and $RuO₂$). On the other hand, the electrochemical combustion yields $CO₂$ and $H₂O$ by direct surface oxidation of the undesired organic compounds and, therefore, the residual solution does not require additional purification. In the previous case, the accumulation of free hydroxyl radicals (• OH) on the electrode surface is the determinant factor for the electrochemical combustion. Anodic materials with these characteristics are $SnO₂$ and $PbO₂$ with or without doping species as well as BDD [\[4,12](#page--1-0)–16].

The electro-Fenton process is another alternative to electrochemical technique, with advantages such as low sludge generation, high removal efficiency of pollutants and on site generation of hydrogen peroxide $(H₂O₂)$ [17–[20\].](#page--1-0) Hydrogen peroxide is continuously generated by the reduction of dissolved oxygen in the cathode in acid medium according to Eq. [\(1\)](#page-1-0) and through Fenton's reaction (Eq. [\(2\)\)](#page-1-0), catalyzed by the ferrous ion (Fe²⁺), produces the free 'OH in the bulk [\[21\],](#page--1-0) which undergoes degradation reactions such as given in Eqs. [\(3\) and \(4\)](#page-1-0) [\[22\].](#page--1-0) The free 'OH radical is the second oxidant, only after fluorine

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 $(E^{\circ}$ ($\text{OH}/\text{H}_2\text{O}) = 2.8 \text{ V}$ vs SHE) [\[23\].](#page--1-0) It is a non-selective specie, which may oxidize organic pollutants to water $(H₂O)$ and carbon dioxide $(CO₂)$ or more simple compounds, Eq. (4) .

$$
O_{2(g)} + 2H^{+} + 2e^{-} \rightarrow H_{2}O_{2}
$$
\n⁽¹⁾

$$
Fe^{2+} + H_2O_2 + H^+ \rightarrow Fe^{3+} + H_2O + \text{`OH}
$$
 (2)

$$
^{\bullet}OH + Fe^{2+} \rightarrow Fe^{3+} + OH^{-} \tag{3}
$$

$$
^{\bullet}OH + organic matter \rightarrow final products. \tag{4}
$$

An advantage of this process is the cathodic reduction of ferric ions $(Fe³⁺)$ as presented in Eq. (5). There are other important waste reactions affecting the efficiency of the process, because they compete with the basic ones, Eqs. (1) and (5). These reactions involve the evolution of oxygen at the anode by Eq. (6) and the evolution of hydrogen at the cathode by Eq. (7) [\[22,24\]](#page--1-0). The efficiency of the process also depends on the cathode, recommending the use of those of carbonaceous nature due to their greater capacity for generation of H_2O_2 [\[25,26\]](#page--1-0).

$$
\text{Fe}^{3+} + \text{e}^- \rightarrow \text{Fe}^{2+} \tag{5}
$$

$$
H_2O \rightarrow \frac{1}{2}O_2 + 2H^+ + 2e^-
$$
 (6)

$$
2H^+ + 2e^- \rightarrow H_2. \tag{7}
$$

Therefore, the aim of this study is to evaluate the electrocatalytic activity of Ti-RuO₂, Ti-PdPtOx and BDD as anodic materials of the electro-Fenton process for the depuration treatment of a real slaughterhouse effluent at constant applied current density value in order to understand the role of the electrode material and their capacity to produce • OH radicals. In all cases the cathode was carbon felt (CF). There are studies of the treatment of similar wastewater, using processes based on free radicals • OH, such as the one reported by de Sena et al. [\[27\],](#page--1-0) in which the result for a combined treatment with the photo Fenton process was a total removal for chemical oxygen demand of 97.6%.

2. Materials and Methods

2.1. Sampling and Characterization of Slaughterhouse Effluent

We took a water sample from the effluent of the wastewater treatment system from a slaughterhouse facility with average flow of 350 m^3 /day and whose principal components were: an equalization tank, sieves, anaerobic digester and sludge batch reactor. The sample was preserved at 4 °C and was characterized by chemical oxygen demand (COD) 405.6 mg/L, total suspended solids (TSS) 150 mg/L, initial chlorine of 0 mg Cl₂/L, chloride of 364 mg Cl[−]/L, pH of 8.5 and conductivity of 3.8 mS/cm. COD was established as the variable to assess the depuration treatment and was measured using the closed reflux method [\[28\].](#page--1-0)

2.2. Chemicals

The ferrous sulfate for Fenton reaction (FeSO₄ \cdot 7H₂O) was provided by KEM, the titanium oxysulfate $(O_5STi \cdot xH_2SO_4)$ for UVdetermination of H_2O_2 was provided by Fluka Analytical and pure water (conductivity $\lt 1$ μS/cm) from a Millipore equipment model Elix 5 was used for solutions. Other chemicals such as sulfuric acid, o-phenanthroline and ferrous ammonium sulfate were of analytical grade purchased from Karal and KEM. A solution of H_2SO_4 at a concentration of 3 mol/L was used for the pH adjustment.

2.3. Electrode Materials

The dimensionally stable anodes (DSAs) of Ti-RuO₂ and Ti-PdPtOx used in this study were prepared using ASTM grade 2 Ti substrates for all samples. The first films were carried out at room temperature by either spraying or dipping the substrates in a 0.1 M alcohol $RuCl₃·xH₂O$ solution to form a coating. In all cases, each coating was dried at 100 °C during 10 min prior to deposition of the succeeding one. After the designated number of coatings was applied, a final longer annealing of 1 h at 450 or 500 °C, to form the ruthenium oxide phase by chloride ion displacement, was made. The electrode Ti-PdPtOx was prepared using the same method. The obtained materials were characterized by X-ray energy dispersive spectroscopy using SEM–EDS mode. A scanning electron microscope BROKER with an EDS system was used [\[29\].](#page--1-0) BDD films were provided by METAKEM™ (Germany) on a conductive niobium substrate (2 mm, standard) with boron doping level: 2000–4500 ppm, resistivity: 10–100 m Ω /cm and maximum current density: 500 mA/cm².

2.4. Instruments

A HACH Sension156 portable multiparameter was used for measuring of conductivity, pH and temperature and a DC Power supply BK (0–18 V and 5A) for electrochemical trials. The colorimetric analysis was conducted with a Thermo Scientific Evolution 300 UV–vis spectrophotometer and the chemical oxygen demand with a Hanna Instruments HI839800 COD system.

2.5. Electrochemical Measurements

Electrolytic tests were carried out using an electrochemical system as presented in Fig. 1, with a 1 L batch reactor, with an agitation of 350 rpm at a constant temperature of 23 °C. The system included a CF cathode (6 cm \times 10 cm \times 1 cm) with the form of a bag, which was tested with three different anodes: DSA of Ti-RuO₂ (15 cm \times 5 cm \times 1 mm), DSA of Ti-PdPtOx (15 cm \times 5 cm \times 1 mm) and a BDD electrode (15 cm \times 5 cm \times 1 mm). The electrodes had an area in contact with the solution of 60 cm^2 . The aeration of the system was carried out by using a fine bubbling from the bottom of the reactor and the inside of the CF cathode.

Fig. 1. Experimental set up for electrochemical tests.

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