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*CORRESPONDENCE Arturo Estrada-Vargas, 🛛 arturo.evargas@academicos.udg.mx

[†]These authors have contributed equally to this work and share first authorship

RECEIVED 01 October 2022 ACCEPTED 10 May 2023 PUBLISHED 23 May 2023

CITATION

Covarrubias-Del-Toro R, Huerta-Rocha M, Lezama L, García EXM and Estrada-Vargas A (2023), Hydrogen peroxide formation in carbon clothes for enhancement of an electro-oxidation tertiary treatment for tequila vinasse wastewater. *Front. Environ. Sci.* 11:1059259. doi: 10.3389/fenvs.2023.1059259

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Hydrogen peroxide formation in carbon clothes for enhancement of an electro-oxidation tertiary treatment for tequila vinasse wastewater

Rafael Covarrubias-Del-Toro[†], Marisol Huerta-Rocha[†], Luis Lezama, Edith Xio Mara García and Arturo Estrada-Vargas*

Departamento de Estudios del Agua y de la Energía, Universidad de Guadalajara, Guadalajara, Mexico

Carbon-based electrodes have been extensively used as cathodes for the electrogeneration of H₂O₂, which is used in the production of the hydroxyl radical •OH to mineralize organic compounds in several types of wastewater. Carbon cloth has been also used in combination with activated carbon for the combined adsorption and oxidation of persistent organic compounds present in tequila vinasse wastewater. Whereas most of the works regarding the H₂O₂ electro-generation involve relatively complex processes to enhance the catalytic activity or the electrochemically active area of these electrodes, calcination by itself represents a simple and low-cost option to enhance these cathodic and anodic functions, especially in the fabrication of large area electrodes that could be needed to treat the large amount of tequila vinasse wastewater that is produced daily. In this work, the effect of calcination at 300°C and 600°C of carbon clothes in the oxidation current and H₂O₂ production at different potentials in H₂SO₄ was studied. Oxidation current increased 700% upon calcination at 600°C, compared to no calcination. H₂O₂ was produced only in a narrow range of polarizations, whereas calcination at 600 °C increased the generation rate from 7.1 \pm 0.3 to 17.8 \pm 0.4 mg L⁻¹ h⁻¹.

KEYWORDS

Tequila vinasse wastewater, tertiary treatment, electro-oxidation, hydrogen peroxide generation, Fenton reagent

1 Introduction

Tequila is an alcoholic beverage with appellation from middle west region in Mexico (SAGARPA, 2017). In 2021, a total of 527 million liters of Tequila were produced (Consejo Regulador del Tequila, 2021). For each liter of tequila, between 10 and 12 L of vinasse wastewater are produced (López-López et al., 2010). Due to its corrosive nature and high content of total suspended and dissolved solids, discharge of wastewater from tequila vinasse into water bodies could represent a major environmental problem.

Although the contents of wastewater that is discharged into water bodies are regulated in Mexico since 1996, only a fraction of this wastewater receives the proper treatment before being discharged (López-López et al., 2010). Recently it has been estimated that $69\% \pm 14\%$ of the vinasse wastewater in Jalisco, the state of Mexico where most of tequila is produced, receive full treatment and is discharged into water bodies or used for irrigation, $25\% \pm 5\%$

receives incomplete treatment and is discharged into the soil or used for irrigation, and $4\% \pm 1\%$ is treated by an external company (Zurita et al., 2022). The main reason for the discharge without treatment could be the economic limitation of distilleries to achieve treatment technologies (Díaz-Vázquez et al., 2021).

The direct discharge of vinasse wastewater into water bodies or soil represent a serious threat to these ecosystems, since they have high temperature (70°C or more), acidic pH (3.0-4.5) and a large amount of total suspended (2,000-8,000 mg L⁻¹) and dissolved (23,000-42,000 mg L⁻¹) solids, modifying the color, pH and conductivity of water and adding putrescible organic compounds (Prajapati and Chaudhari, 2015; Castillo-Monroy et al., 2020). Additionally to these physicochemical properties, Biochemical Oxygen Demand (BOD₅) and Chemical Oxygen Demand (COD) of tequila vinasse wastewater have received special attention, since water with a high BOD or COD load decreases the dissolved oxygen concentration, affecting the aquatic life. Between 1996 and 2021, Mexican regulation allowed a maximum of 150 mg L⁻¹ of BOD₅, whereas COD was not regulated (SEMARNAT, 1996). Distilleries with a conventional wastewater treatment consisting of a combination of biological and physicochemical steps such as coagulation, flotation, aerobic and/or anaerobic digestion, etc., were able to comply with this regulation. More than 100 volatile compounds have been identified in tequila vinasse, which are mainly removed by the anaerobic digestion process (Rodriguez Arreola et al., 2020). However, tequila vinasse wastewater has a small content of persistent organic pollutants, which remain after conventional treatment and maintain the COD values between 300 and 1,000 mg L⁻¹ (Castillo-Monroy et al., 2020). These compounds correspond mainly to phenolic species (Colin et al., 2016).

In 2022, a limit of 150 mg L⁻¹ COD value was published in Mexican regulation (SEMARNAT, 2022). This new COD limit encouraged distilleries to adopt tertiary treatment approaches capable of reaching regulatory discharge standards. Although physicochemical treatments such as adsorption have been implemented to remove the persistent pollutants and lower the COD, these methods imply the formation of solid waste. Processes that aim to oxidize at least partially these persistent pollutants have been recently explored, such as Advanced Oxidation Processes (AOPs) (Ferral-Pérez et al., 2016; Castillo-Monroy et al., 2020; Rodriguez Arreola et al., 2020).

Among AOPs, the Fenton reagent constitutes a popular approach (Fenton, 1894). Briefly, the Fenton mixture consists of H_2O_2 and Fe^{2+} in acidic medium that react to produce the hydroxyl radical (•OH), as stated in Eq. 1:

$$\mathbf{F}\mathbf{e}^{2+} + \mathbf{H}_2\mathbf{O}_2 \to \mathbf{F}\mathbf{e}^{3+} + \bullet\mathbf{O}\mathbf{H} + \mathbf{O}\mathbf{H}^- \tag{1}$$

The Fenton process presents cost, stability and safety issues regarding the acquisition and handling of H_2O_2 reagent. In this context, electrochemically assisted AOPs (E-AOPs) present a solution to these problems by a double approach: Firstly, they allow the direct electro-oxidation of the persistent pollutants on the anode surface. Secondly, the E-AOP effect can be enhanced by the production of the solution, eliminating some safety risks of H_2O_2 transportation and handling and reducing the reagents cost (Oturan and Brillas, 2007; Fang et al., 2017; Moreira et al., 2017). Enhanced electro-oxidation of

vinasse wastewater has been studied by Castillo-Monroy et al. (2020). However, the effect of anodic removal of pollutants and electro-Fenton process could not be studied separately since the complex composition of vinasse wastewater hinders the determination of •OH formation.

 H_2O_2 can be produced by electrochemical reduction of O_2 in the cathode as stated in Eq. 2:

$$\mathbf{O}_2 + 2\mathbf{H}^+ + 2\mathbf{e}^- \to \mathbf{H}_2\mathbf{O}_2 \tag{2}$$

The formation of H_2O_2 in carbonaceous materials such as vitreous carbon, graphite and carbon felt, sponge, and cloth has been extensively studied (Pérez et al., 2017). More recently, H_2O_2 production rates of 1,387, 30 and 1,035 mg l⁻¹ h⁻¹ have been achieved by using respectively modified carbon cloth and carbon black as electrodes (Pérez et al., 2017; Zárate-Guzmán et al., 2019; Zhang et al., 2019). These works regard the modification of carbon-based materials to improve the catalytic activity or the electroactive area of the electrode and therefore the H_2O_2 generation rate.

Calcination of carbonaceous materials has shown to introduce oxygen functional groups in carbon cloth and carbon black, enhancing desired functionality (Gu et al., 2018; Zhang et al., 2019). Although some of the mentioned works include calcination of carbon-based electrodes as a step of the modification process, only the work of Zhang et al. (2019) regards the calcination temperature effect. Taking into consideration the large amounts of vinasse produced in tequila industry, calcination could represent a simple and low-cost modification process that facilitates the scaling up of electrode size for E-AOP-based wastewater treatment processes. In this regard, this work aims to study the effect of calcination *per se* in the electro-generation rate of H_2O_2 by using carbon clothes as electrodes, and to analyze the possibility to extend these results to other carbonaceous materials.

2 Materials and methods

2.1 Materials

Calcination of carbon clothes (Grupo ROOE) was developed at 300 or 600°C in a simple furnace. H_2SO_4 , FeSO₄•7H₂O, FeCl₂•4H₂O (Sigma-Aldrich), dry air 99% (Grupo Infra, S.A. de C.V.) were used as received. Conventionally treated tequila vinasse wastewater was collected from the effluent at the exit of the treatment plant of a tequila distillery from Tequila, Jalisco, Mexico, and stored at 2°C.

2.2 Fenton reaction

Conventionally treated tequila vinasse wastewater (170 mg L^{-1} COD) was mixed with H_2O_2 in excess and enough FeSO₄•7H₂O or FeCl₂•4H₂O to produce •OH that stoichiometrically would remove the mentioned COD.

2.3 Cyclic voltammetry

A three-electrode-cell was built by using a carbon cloth with an exposed area of 3.63 cm^2 as working electrode, a Pt mesh of $5 \text{ cm} \times 5 \text{ cm}$ as counter electrode and a Ag[AgCl wire as reference electrode.



This wire is considered a pseudo-reference electrode since it is directly immersed in the solution (Inzelt, 2013). A PalmSens4 potentiostat was used to control the cell potential. An air stone (Pawfly) connected to a dry air tank was used to bubble dry air in the cell when necessary.

2.4 H₂O₂ generation

Six carbon clothes with a total area of 15 cm² were immersed and used as electrodes in 110 mL of 0.100 M H₂SO₄. The potential/ current was controlled with a 42AH0477 Newark power supply. Dry air was bubbled from the bottom of the cell. Figure 1 shows a schematic diagram of the electrochemical cell. Polarization between 0.6 and 3.6 V was applied for about 12 min in order to generate H_2O_2 in the cathode.

2.5 H₂O₂ determination

Samples of 3 mL from the solution in the H_2O_2 generation cell were taken and mixed with 10 μ L TiOSO₄ solution in order to form the Ti•H₂O₂ complex (Ribeiro et al., 2009). The mixture absorbance at 405 nm was measured by means of a V-770 UV-Vis spectrometer (JASCO) in order to determine the complex concentration with a previously developed calibration curve by using QtiPlot[®] software.

2.6 COD determination

Tequila vinasse wastewater was sampled and put in digestion vials (Hach) for 2 h in a digestor (Hanna Instruments). The COD of digested samples was measured with a DR900 colorimeter (Hach) (Oxygen Demand, Chemical USEPA 1 Reactor Digestion Method 2 Method 8000 Test preparation Instrument-specific information, 2021).





Cyclic voltammograms of carbon clothes calcinated at different temperatures and immersed in 0.020 M $\rm H_2SO_4.$

3 Results

3.1 Fenton reagent

Fenton reagent has shown previously its potential to oxidize persistent organic compounds in vinasse wastewater (Castillo-Monroy et al., 2020). However, in the industry it is known that the persistent compounds concentration for vinasse from different sources or even from the same source and different batch process could vary. In this regard, the oxidation experiment had to be repeated for the specific vinasse wastewater sample collected for this work. Tequila vinasse wastewater with $COD_0 = 170 \pm 7 \text{ mg L}^{-1}$ was mixed with H_2O_2 with or without $FeSO_4$ or $FeCl_2$ salts to compare the COD removal with or without the expected generation of \bullet OH. The final CODs in mg l⁻¹ of different mixtures were: H_2O_2 : greater than 1,500; $H_2O_2 + FeSO_4$: 29 ± 11 ; $H_2O_2 + FeCl_3$: 293 ± 3 .



3.2 H₂O₂ generation

Figure 2 shows a cyclic voltammograms at 50 mV s⁻¹ of a carbon cloth used as working electrode with exposed area of 3.63 cm^2 . An increment of about 0.6 mA cm⁻² is observed in the reduction peak at -0.6 V vs. Ag|AgCl as dry air is bubbled, indicating that an oxygen-consuming reduction reaction occurred. A control experiment by bubbling N₂ instead of dry air was performed to ensure that the increase in the current was due to the presence of oxygen in the electrolyte, and not to the electrolyte agitation by bubbling (not shown).

Figure 3 shows cyclic voltammograms at 50 mV s⁻¹ of carbon clothes previously calcinated at different temperatures, immersed in 0.020 M H_2SO_4 with exposed area of 3.63 cm² and a 5 cm × 5 cm Pt mesh as counter electrode. Solid lines and marks represent respectively no bubbling and bubbling air during the experiment. Although greater oxidation and reduction currents were observed as the calcination temperature increased, the increase in reduction current due to air bubbling at -0.6 V vs. Ag|AgCl was not proportional to the current for each calcination temperature.

The H_2O_2 production as a function of the polarization for carbon clothes calcinated at different temperatures and used as electrodes for the electrochemical cell in section 3.4 is shown in Figure 4. For all calcination temperatures, H_2O_2 is formed only above certain potential that ranges from 2.4 to 2.8 V. Maximum generation rates of 7.1 ± 0.3, 14.3 ± 0.2 and 17.8 ± 0.4 mg l h⁻¹ were achieved by using clothes without calcinating and calcinated at 300°C and 600°C respectively. At polarizations above the maximum H_2O_2 generation potential for each calcination temperature, the generation decreased.

4 Discussion

4.1 Fenton reagent

The Fenton reaction was attempted to be performed with excess of H_2O_2 and Fe^{2+} salts. The $[H_2O_2]/[Fe^{2+}]$ ratio was high, although optimal concentrations to maximize •OH generation were not used

(Bouafia-Chergui et al., 2010). Given the final COD values, apparently only $FeSO_4$ removed COD with an efficiency of 83%. It should be noted that since Fe^{3+}/Fe^{2+} concentrations were below the solubility limit of all Fe salts, thereby no acid was added to lower the pH and prevent precipitation. This achieved COD removal is lower than the 90% at high pH reported previously by Castillo-Monroy et al. (2020). However, the purpose of the experiment was to ensure that Fenton reagent could remove the COD of the vinasse wastewater collected for this work.

 H_2O_2 added to vinasse wastewater possibly did not react with the persistent organic compounds, and therefore reduced Cr^{6+} in the vial during digestion, leading to an apparently higher COD than the original vinasse wastewater sample (Kawanishi et al., 1986). H_2O_2 mixed with FeCl₂ presented an apparently higher COD than vinasse wastewater possibly due to the excess of H_2O_2 , which could promote the regeneration of Fe²⁺ according to Eqs 3, 4, therefore consuming the Cr⁶⁺ of digestion vials to oxidate Fe²⁺ to Fe³⁺ (Bouafia-Chergui et al., 2010).

$$\mathbf{F}\mathbf{e}^{3+} + \mathbf{H}_2\mathbf{O}_2 \to \mathbf{F}\mathbf{e}^{2+} + \mathbf{H}^+ + \bullet\mathbf{O}\mathbf{H}_2 \tag{3}$$

$$\mathbf{F}\mathbf{e}^{3+} + \bullet \mathbf{O}\mathbf{H}_2 \to \mathbf{F}\mathbf{e}^{2+} + \mathbf{H}^+ + \mathbf{O}_2 \tag{4}$$

$4.2 H_2O_2$ generation

The increase in reduction current due to air bubbling at -0.6 V vs. Ag|AgCl in Figure 2 could correspond to oxygen-consuming reactions, although clearly only a fraction corresponds to H₂O₂ formation as set in Eq. 2. From Figure 2, we can infer that applying potentials below -0.6 V vs. Ag|AgCl could diminish the H₂O₂ production due to the presence of side reactions such as (5) and (6), or even worse, H₂O₂ reduction reactions, such as (7), as reported previously (Brillas and Casado, 2002; Luo et al., 2015).

$$\mathbf{O}_2 + 4\mathbf{H}^+ + 4\mathbf{e}^- \to 2\mathbf{H}_2\mathbf{O} \tag{5}$$

$$2\mathbf{H}^{+} + 2\mathbf{e}^{-} \to \mathbf{H}_{2}\mathbf{O} \tag{6}$$

$$\mathbf{H}_{2}\mathbf{O}_{2} + 2\mathbf{H}^{+} + 2\mathbf{e}^{-} \rightarrow 2\mathbf{H}_{2}\mathbf{O}$$
 (7)

From Figure 3, the cathodic current at -0.6 V vs. Ag|AgCl increased with the calcination temperature, from 1 to 7 mA cm⁻² at no calcination and calcination at 600°C respectively. This result agreed with the one from Zhang et al. (2019), where reduction current increased from 8.1 to 117.6 mA with calcination. Additionally, the inset showed that both reduction and oxidation current increased with calcination. This result suggests that calcination has the potential to enhance the electro-oxidation of persistent organic compounds in carbon-based anodes. A constant reduction current increase of about 0.6 mA cm⁻² as dry air is bubbled, compared to the experiment without bubbling, regardless of the calcination temperature, could be observed. This suggests that the enhancement of H₂O₂ electro-generation in the cathode could be significatively lower than the electro-oxidation enhancement.

Figure 4 showed the behaviour of H_2O_2 generation rate with applied polarization for electrodes without calcination and calcinated at 300°C and 600°C in the electrochemical cell described in section 3.4. The minimum polarization value for H_2O_2 formation (2.4–2.8 V) differed substantially from

the -0.6 V potential needed for observing oxygen-consuming reaction in cyclic voltammetry. This can be explained since H₂O₂ generation experimental setup corresponded to a two-electrode cell, without reference electrode and using the carbon clothes as cathode and anode, instead of using a Pt mesh with greater area as counterelectrode. Therefore, higher overpotentials were needed for reduction reactions to occur in anode-cathode cells used in industry than three-electrode cells used typically in the laboratory. H₂O₂ production reached a maximum and then diminished at higher polarizations, suggesting that it is mainly produced only at a small range of potentials, as concluded previously by Zárate-Guzmán et al. (2019). A possible explanation is the fact that H₂O₂ reduction presented in Eq. 7 occurs slowly in carbon-based materials, however, high overpotentials could increase the reaction kinetics and therefore reduce H₂O₂ in the cathode as it is formed according to Eq. 2 (Luo et al., 2015). Additional experiments at potential differences greater than 3.6 V for 12 min were performed after electro-generating H₂O₂ in carbon clothes at 3.0 V for 12 min. The H₂O₂ concentration decreased totally after the experiments at higher potential differences.

Furthermore, Figure 4 showed that the H₂O₂ formation rate increased with calcination temperature. This result has been previously explained by considering that calcination introduces carbon oxidized surface groups such as C-O-C, C-OH, C=O and O=C-OH, enhancing the oxygen reduction reaction in the electrode surface and increasing the specific surface area (Gu et al., 2018; Zhang et al., 2019). The maximum H₂O₂ production rate was achieved by applying a polarization of 2.7 V using carbon clothes calcinated a 600°C. This rate of 17.8 \pm 0.4 mg l⁻¹ h⁻¹ is still below to the 31 mg l-1 h-1 rate achieved in carbon felt by Zárate-Guzmán et al. (2019), and far below the 1,051 and 1,387 mg l^{-1} h^{-1} rates obtained respectively in black carbon by Zhang et al. (2019) and in carbon cloth modified with carbon black by Pérez et al. (2017). This result can be explained by the small specific area of carbon cloth compared to those of carbon felt and carbon black. However, these results showed that calcination by itself without further electrode modification increased the H2O2 generation rate, opening the possibility to enhance the oxidation of persistent compounds in tequila vinasse wastewater by a simple method, which is easier to scale up in electrode size than other surface modification techniques. Aditionally, these results open the possibility to study the enhancement of H2O2 production by further calcination in other modification processes such as the one proposed by Zárate-Guzmán et al. (2019), or to study the effect of calcination at higher temperatures than 300°C in the modification process proposed by

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Data availability statement

The raw data supporting the conclusion of this article will be made available by the authors, without undue reservation.

Author contributions

EM and AE-V contributed to conception and design of the study. RC-D-T and MH-R performed the experiments. LL, EM, and AE-V wrote sections of the manuscript. All authors listed have made a substantial, direct, and intellectual contribution to the work and approved it for publication.

Funding

The authors acknowledge Secretaría de Educación Pública (SEP, Mexico) for funding this research work through grant FCA-23212-2020, and Universidad de Guadalajara (Mexico) for funding the open access publication fee. Scholarships for RC-D-T. and LL from Consejo Nacional de Ciencia y Tecnología (CONACYT, Mexico) are also fully acknowledged.

Conflict of interest

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