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Development of electrooxidation cells with recovered carbon, for its possible application in the removal of 17-α-ethinyl estradiol from water

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ABSTRACT

In this work, various electrochemical cells were developed from carbons recovered from mechanical seal carbons, electric motor brush carbons (MBC) and AA battery carbons (BC). After the different types of carbons were conditioned, three electrooxidation devices were assembled, where the carbons were used as anode, and the cathode was stainless steel 304 SS. In each of the devices, three different separations between the anode and the cathode were considered: 1/8″, 3/16″ and 1/4″. The electrical characterization of each of the cells was carried out using a sodium bisulfite solution ($NaHSO_3$), with and without system agitation. The results indicated that agitation was not a factor that significantly affected the voltage conduction. The voltametric behavior revealed that the highest current density was presented by the MBC (39.2 mA/cm^2) for a separation of 1/8″, followed by the BC (17.45 mA/cm^2) for the same separation. To select the best arrangement, the premature wear of the anode was also considered. In this instance the BC did not present carbon detachment, so it was selected to carry out the degradation of 17-α-ethinyl estradiol. The degradation efficiency was 74% in 2 h. The identification of by-products by gas chromatography coupled to mass spectrometry revealed the majority presence of carboxylic acids.

Keywords: Urban waste; Recovered coal; Electro-oxidation

1. Introduction

The generation of urban solid waste (USW) worldwide in 2012 was calculated at around 1,300 million tons/d, and it is estimated that it could grow to 2,200 million by the year 2025 [1]. In 2010, it was determined that about 44% of the USW produced globally originated in countries with the most developed economies of the Organization for Economic

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Cooperation and Development, followed by the Pacific Region and East Asia (21%). The countries of Latin America and the Caribbean contributed 12% of the total [2].

In Mexico, the most recent figure published in 2015 indicates that the generation of USW reached 53.1 million tons, which represented an increase of 61.2% compared to 2003 (10.24 million tons more generated in that period) [3]. Regarding the composition of the waste in general, organic waste (food waste, gardens and similar organic materials) predominates, contributing 52.4%, followed by paper and cardboard waste (13.8%), plastics (10.9%), glass (5.9%), aluminum (1.7%), ferrous metals (1.1%), other metals (0.6%), and other types of garbage (12.1%).

One of the alternatives to mitigate environmental impacts at a global level is the reuse and recycling of materials, which to a certain extent reduces the pressure on ecosystems and other sources of natural resources [2].

There is a problem in Mexico due to the generation of batteries and cells caused by the increase in portable technology. These batteries and cells are, in general, thrown together with the rest of the household waste in open-air dumps, sanitary landfills, vacant lots, or bodies of water, causing severe environmental impacts.

In a diagnosis carried out by the National Institute of Ecology and Climate Change (INECC, by its acronym in Spanish) on Comprehensive Waste Management (CWM), it was estimated that between 2006 and 2012, Mexico generated an annual average of about 34,122 tons of batteries, comprised of approximately 1,534 million pieces. This means that, considering the national population of 2010, each inhabitant generates, on average, the equivalent of 307 g of batteries per year. Of the total number of batteries generated in the period, 70.1% corresponded to carbon-zinc batteries, 19% to alkaline batteries, 6.2% to lithium batteries and the remaining 4.7% to mercury oxide, zinc-air, and silver carbon oxide batteries [4].

Another important source of USW contamination is the generation of waste from engines and mechanical seals. Currently there are no studies in Mexico on the generation of industrial waste from mechanical seals or motor brushes (both carbons). These come mainly from centrifugal pumps, submersible pumps, electric tools, and household appliances with direct current motors, among others.

Recently, much more concern has been concentrated on the presence of organic pollutants with estrogenic activity due to reproductive effects on human beings and also with aquatic organisms [5]. The compound 17-α-ethinyl estradiol (EE2) (Fig. 1), excreted in urine by humans, is mainly from female oral contraceptives [6,7] and possesses potentially more estrogenic effects in comparison with counterparts in natural steroid estrogens, even at a low concentration [8–10].

Several advanced oxidation processes have been proven to effectively decompose EDCs (endocrine disrupting compounds) [11–13]. Electrochemical oxidation (EO), which can generate $^{\bullet}OH$ active radicals without secondary pollution, can be an alternative choice for EDC removal [14,15].

EO of organic pollutants occurs either indirectly or directly and is highly dependent upon the nature of the electrode material, water matrix, system parameters and electrolyte. Indirect oxidation occurs via an electroactive mediator, which transfers electrons from the electrode to the organic pollutant [16]. During EO, oxidants (hydroxyl radicals, sulfate radicals, carbonate radicals, free chlorine) are generated in situ, which facilitates indirect oxidation [17–18]. Direct oxidation takes place through electron transfer on the surface of the electrode without the involvement of any other substance or through an adsorbed mediator oxidant on the surface of the electrode [16].

In the present study an electrooxidation system was developed from waste batteries, which was used for the removal of 17-α-ethinyl estradiol from water.

2. Materials and methods

Initially, the carbon pieces selected were obtained from mechanical seal carbons present in pumps (MSC), electric motor brush carbons (MBC) and AA battery carbons (BC) (Fig. 2). These pieces were used as anodes.

2.1. Preparation of recycled carbon anodes

The carbon materials used as anodes underwent a cleaning process according to the methodology recommended

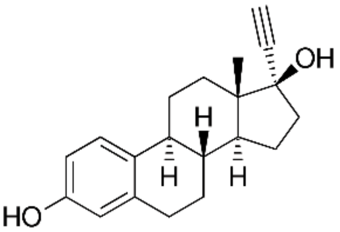


Fig. 1. Molecular structure of 17-α-ethinylestradiol.

Fig. 2. Recovered carbons that were used as anodes: (a) mechanical seal carbons, (b) motor brush carbons and (c) AA battery carbons.

in patent No. WO 2012/155285 [19]. Initially, the materials were sanded with different types of sandpaper, from coarse sandpaper grains (No. 200 and 600) to fine sandpaper grains for polishing (No. 1500 and 2000), until a mirror finish was achieved. Subsequently, all the carbons were immersed in ethyl ether, then washed with distilled water and sonicated for 15 min. Finally, the coals were boiled in a beaker with a 0.3% H_2SO_4 solution for 30 min, until no carbon residues were observed (Fig. 3).

2.2. Electromechanical cell arrangement to fix anode and cathode

Considering that the brush carbons are the ones with the largest area (Fig. 3b), a basic arrangement was made with four carbons. Taking into account the areas of the walls that were in reaction against the anodes, an approximate area of 7,339 mm^2 was calculated, determining that 12 seal carbon rings and 12 AA battery carbon cylinders were required. With this we obtained similar areas between the three materials. Subsequently, the design of the electromechanical arrangements of the cells was carried out. To guarantee the separation between anode and cathode three different distances were established: 1/8″ (3.17 mm), 3/16″ (4.77 mm) and 1/4″ (6.35 mm). Stainless steel 304 was used as cathode in the form of studs and meshes. Fig. 4 shows the general scheme of each device according to the shapes of the recycled carbon materials.

2.3. Manufacture of electrooxidation device

Once the electromechanical arrangements were developed, the device containing each of these established arrangements (electrooxidation cell) was prepared. This cell was constructed with 3.5 mm transparent acrylic for the cylinder and supports, and 1/2″ (12.7 mm) thick acrylic for flanges. A terminal strip was installed to facilitate internal and external connection of direct current (DC) power source, voltmeter, ammeter, power supply, and submersible pump operation (Fig. 5).

2.4. Electrical characterization of the electrodes

In this stage tests were carried out at different voltages for each of the developed arrangements. According to the design of the cells, the three materials, MBC, MSC and BC, were tested as anode and the cathode was stainless steel 304

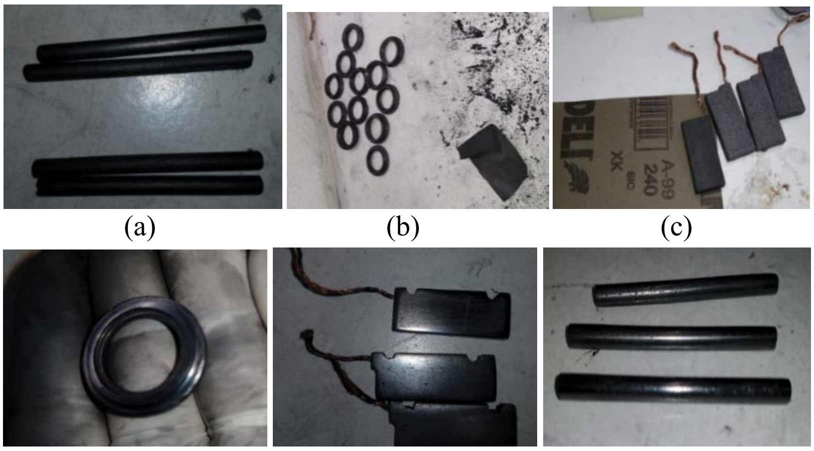


Fig. 3. Sanded with different grits, (a) 200, (b) 600 and (c) 1,500 sanded with 2,000 grit mirror polished finish.

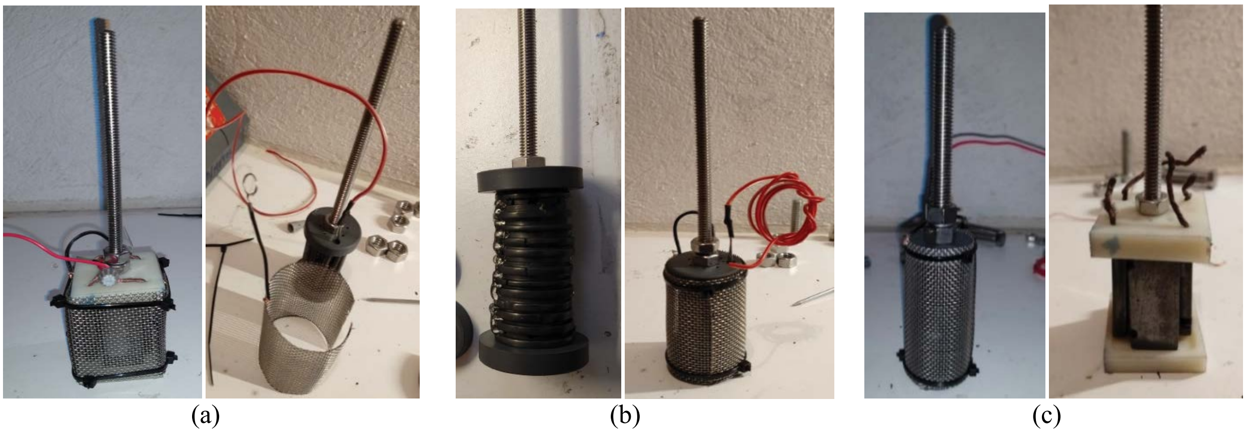


Fig. 4. Final assembly of electromechanical anode and cathode arrangements. (a) Mechanical seal carbon, (b) motor brush carbon and (c) AA battery carbon.

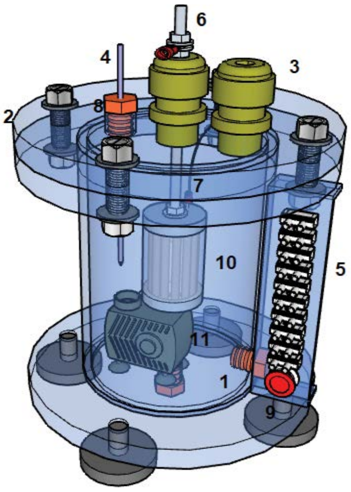


Fig. 5. Electrooxidation device, main compounds: (1) main body of the device, acrylic cylinder; (2) main body flanges of the device, acrylic; (3) 1/2″ gland connectors for sealing cables and electrodes; (4) stainless steel rod. As a voltage measurement electrode in the fluid; (5) connection board; (6) anode electrical connection with stainless steel screw; (7) anode connection (carbons); (8) brass connector; (9) switch for submersible pump operation; (10) electromechanical arrangement of anode and cathode; (11) submersible pump.

(SS 305). For each material, the different previously established anode-cathode separations were tested: 1/8″, 3/16″ and 1/4″. The electrical characterization was performed with deionized water, which had an electrical conductivity of 0.02 µS/m at 25°C. The test began after making the appropriate connections for each of the separations. The applied voltages ranged between 0 and 8 V with measurements made every 0.5 V. For each arrangement the current and the voltage produced by the source of DC were measured, as well as the current of the circuit directly within the fluid. This was done by means of a stainless-steel electrode connected in series with the circuit to an ammeter. The voltage of the anode and cathode arrangement was measured by an ammeter connected in parallel.

2.5. Evaluation of the carbon-stainless steel cell

In this stage, for each anode–cathode cell arrangement, the evaluation was carried out using a sodium bisulfite solution ($NaHSO_3$), following the methodology established by Aguirre-Quintana [19]:

- The three selected carbon materials (anodes), MBC, MSC and BC, were tested.
- The three separations between anode and cathode, 1/8″, 3/16″ and 1/4″ were established.
- The $NaHSO_3$ solution used to evaluate the oxidation-reduction process had a concentration of 140 mg/L in a 12% ethanol-water mixture and was adjusted to pH = 4 with H_2SO_4.
- The electrical connections were made, as mentioned in the previous paragraph.
- Each experiment lasted 20 min, using a voltage of 5 mV/s, with voltage increments of 300 mV every minute for 20 min. The voltage and current at the output of the DC source was recorded.
- With the data obtained for voltage and current in the source and in the circuit, graphs were made to obtain the behavior of each arrangement and to calculate the power demand (W) and current density (j).
- Finally, the cell with the best performance was selected.

2.6. Evaluation of degradation kinetics

For the degradation tests, synthetic solutions of 17-α-ethinyl estradiol were prepared at a concentration of 50 mg/L. These were added to the developed device (750 mL) and the pH was adjusted to 6. A cell with battery carbons as anode and stainless-steel mesh as cathode was used with a separation of 3/16″. The applied voltage was 4,000 mV with constant stirring throughout the degradation process. To evaluate the kinetics, a 20 mL sample was taken every 20 min, until reaching 120 min.

2.7. Extraction and analysis of EE2 degradation products

All the fractions obtained in each of the samples were passed through solid phase extraction cartridges before the GC-MS analysis. Sep-Pak C18 cartridges were preconditioned with 5 mL of ethyl acetate and 5 mL of methanol, followed by 3 mL × 5 mL of ultrapure water (UPW). For extraction, the filtered samples were passed through cartridges at a flow rate of 3 mL/min. Then the cartridges were washed with 3 mL × 5 mL of 10% methanol in UPW (v/v) and dried under a vacuum for 2 h. The target compounds were eluted from cartridges using 3 mL × 5 mL of ethyl acetate. The eluate was evaporated until it was nearly dry under a gentle stream of nitrogen and resuspended in *n*-hexane. Because of the large polarity of the target compounds, it was necessary to reduce their polarity and improve the stability of the substances and sensitivity of the chromatographic analysis by derivatization before the GC-MS analysis. The derivatization reagent [N-methyl-N-(trimethylsilyl)trifluoroacetamide] (TMS) was added to the samples at 60°C and maintained for 30 min. EE2 and degradation products were derivatized and cooled to room temperature for the GC-MS analysis. The GC-MS analyzed EE2 and degradation products in the full scanning mode (selected ion monitoring, SIM), and the scanning range was approximately m/z 50–600 for the ion scanning mode. This was carried out according to the methodology established by Ronderos-Lara et al. [20]. For the quantification of residual EE2, a calibration curve with five points was made in a concentration range between 0.05 and 100 mg/L. The limits of detection (LOD) and quantification (LOQ) were calculated using signal-to-noise criteria (S/N); LOD = 3 (S/N) and LOQ = 10 (S/N) [21]. The LOD limit was 0.025 mg/L while the LOQ was 0.075 mg/L.

3. Results and discussion

3.1. Electrical characterization of the circuit for the different established arrangements

The characterization was carried out for the three proposed separations between anode and cathode (1/8″, 3/16″ and 1/4″) for each of the recovered carbons (Fig. 6).

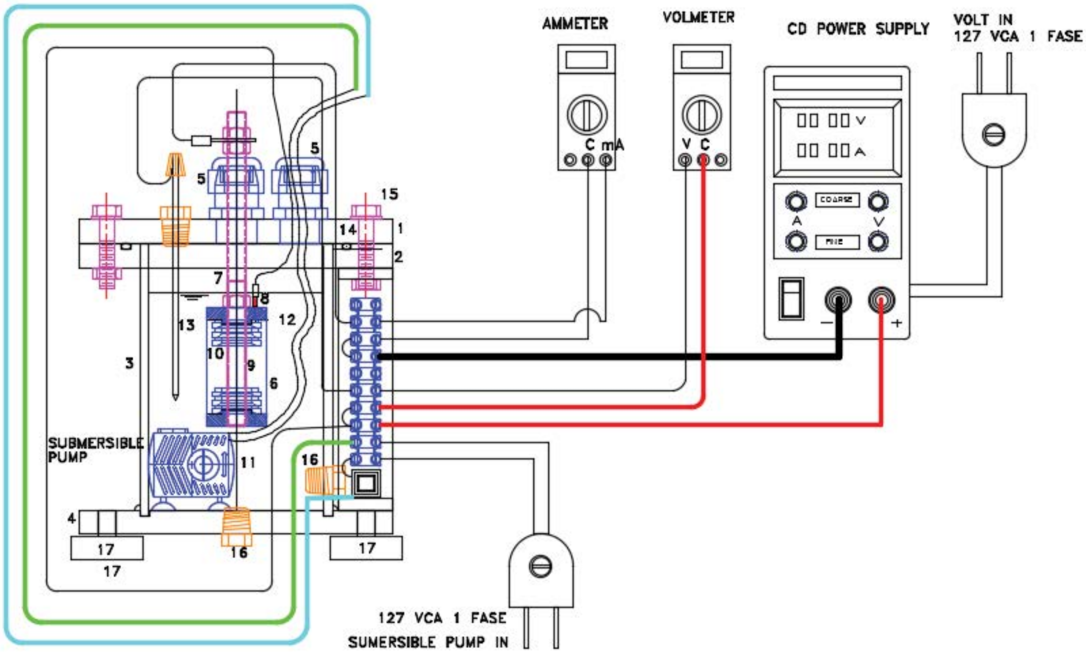


Fig. 6. Connection diagram and device components: (1) cover flange; (2) top flange; (3) reactor body; (4) base flange; (5) electromechanical arrangement of oxidation; anode and cathode; (6) screw for cathode connection; (7) anode connector; (8) stainless steel cathode; (9) recycled carbon anode; (10) recirculation submersible pump; (11) submersible recirculation wire; (12) circuit voltage stainless steel electrode for measurement in aqueous solution; (13) sealing "O" ring; (14) stainless steel screw; (15) bronze stopper; (16) PVC bases.

The results indicate small changes in the voltage. These differences are attributed to the resistance of the materials, which increases when all the elements are connected in a circuit, including the measuring instruments. The test was performed with and without fluid agitation, which indicated that agitation was not a factor that significantly affected voltage conduction (Fig. 7).

3.2. Voltametric behavior of the established arrangements

The results indicate that in the MSC array the behavior for the three arrays was very similar with power consumption after 20 min of reaction around 6 W (Fig. 8).

For the BC arrangement, the highest power consumption was observed for the 1/8″ gap (17.5 W), followed by the 3/16″ gap (8 W) and finally 1/4″ (6 W, approximately) (Fig. 9).

In the MBC arrangement, the highest power consumption was presented for the 1/8″ separation at approximately 25 W, while for the 1/4″ and 3/16″ separations it was 20 and 16 W, respectively (Fig. 10).

3.3. Selection of the best arrangement

According to the voltametric behavior, the average power (*W*) and *j* (mA/cm^2) of each experiment were obtained (Table 1).

The results indicate that in the arrays with MSC, the *W* and *j* do not have a considerable variation for the three separations between electrodes, which suggests that this type of carbon does not have good electrical properties. In the case of the BC array, the 1/8″ gap had higher power consumption and current density, while the 1/4″ gap array had the lowest power consumption. Finally, the lowest power was found in the 3/16″ separation for the MBC arrangement.

This allowed us to observe that the cell with the highest efficiency regarding *W* and *j* was the 3/16″ separation cell for MBC; however, the detachment of carbon would cause premature wear. For this reason the BC cell with the same separation of 3/16″ was selected ($j = 8.77$ mA/cm^2), which did not present wear, and the power consumption was similar.

The role played by the current density (*j*) in the degradation of organic compounds is fundamental since it favors an increase of $^•$OH radicals [22]. For example, Ricardo et al. reported values of *j* of 5 mA/cm^2 for the degradation of estrone with efficiencies between 80% and 90% for electrolysis times of 47 and 60 min, respectively. Meanwhile, for values of *j* of 10 mA/cm^2, they found degradation efficiencies close to 98% after 30 min of electrolysis, using a boron doped diamond anode [23]. Likewise, Feng et al. [24] reported degradation efficiencies of 95% of EE2 using current densities of 10 mA/cm^2 in a glass reactor made up of a Ti/SnO$_2$ electrode (anode) and a stainless-steel cathode, these voltametric behaviors were similar to what was observed in our design.

3.4. Application of the device developed for the degradation of 17-α-ethinyl estradiol

Once the best arrangement (BC 3/16″) was selected, the degradation efficiency of a synthetic solution of 17-α-ethinyl estradiol at a concentration of 50 mg/L, prepared with tap water at pH 6, was determined. The degradation was carried out at a current density of 10 mA/cm^2, and every 20 min an aliquot of 20 mL of the remaining solution in the reactor

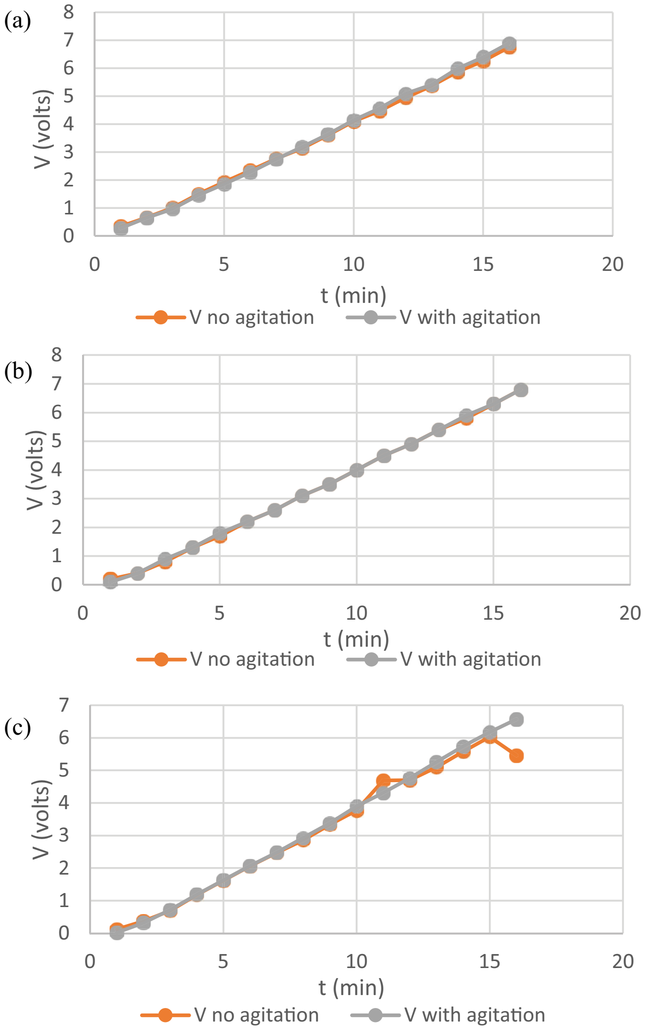


Fig. 7. Example of the incidence of the agitation of the fluid on the behavior of the voltage. Input voltage vs mechanical seal carbon anode circuit voltage for the different spacings (a) 1/8″, (b) 3/16″ and (c) 1/4″.

was taken. Each fraction was processed as described in the methodology and analyzed by gas chromatography coupled to mass spectrometry. The degradation results for three experiments reveal that, at 120 min, the efficiency reached approximately 74.0% (Fig. 11). These results were lower than those reported by Murugananthan et al. [25] in the degradation efficiencies of E2, with an efficiency of 90%, using boron-doped diamond electrodes (BDD) and by Feng et al. [24] (95%), applying Ti-based SnO_2 (Ti/SnO_2) electrode as the anode. Comparing these results with those obtained using our device, we can suggest that the scale up of electrochemical cells to an industrial level is promising at a low cost, considering that they can be prepared with battery waste.

As is known, the anodic oxidation of organic compounds is highly dependent on the anode material. Electrochemical oxidation of organic contaminants can result from adsorbed active oxygen ($^{\bullet}OH$) or chemisorbed active oxygen (on the surface of the electrode). In general, $^{\bullet}OH$ is more effective than active oxygen (O) for the oxidation of organic

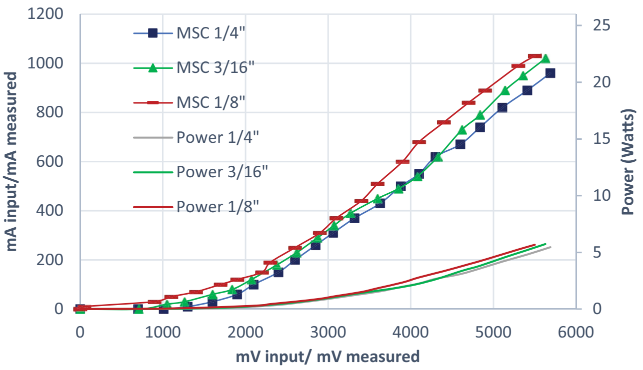


Fig. 8. Voltametric behavior of the three separations 1/8, 3/16 and 1/4 with the mechanical seal carbon anode.

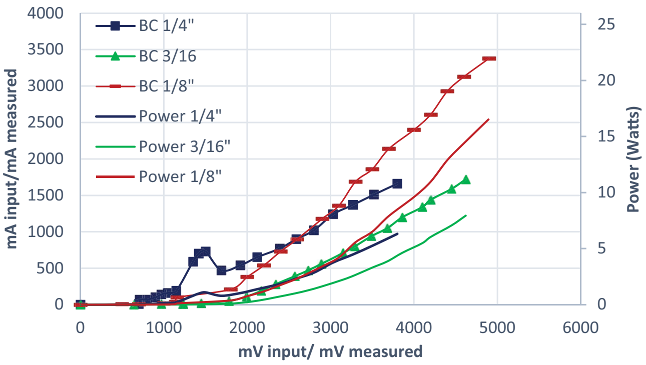


Fig. 9. Voltametric behavior of the three separations 1/8, 3/16, 1/4 with the battery carbon anode.

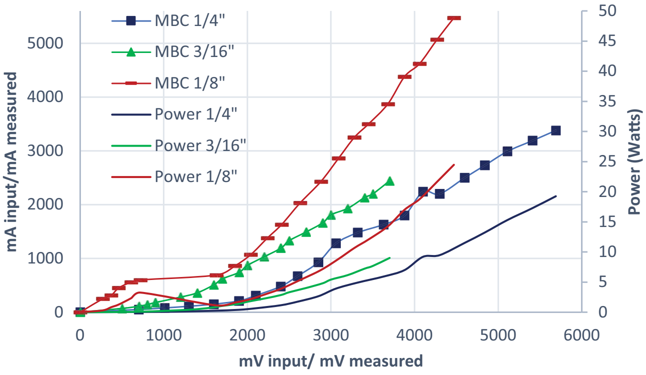


Fig. 10. Voltametric behavior of the three separations 1/8, 3/16, 1/4 with the motor brush carbon anode.

Table 1
Voltametric results of the total of the 9 electrode arrangements with the three separations

Separation (inch)	Mechanical seal carbon		Battery carbon		Motor brush carbon	
	W	*j*	*W*	*j*	*W*	*j*
1/4	1.69	5.22	1.69	8.78	6.2	19.35
3/16	1.79	5.66	2.43	8.77	2.96	14.37
1/8	1.75	5.72	4.93	17.45	7.99	39.2

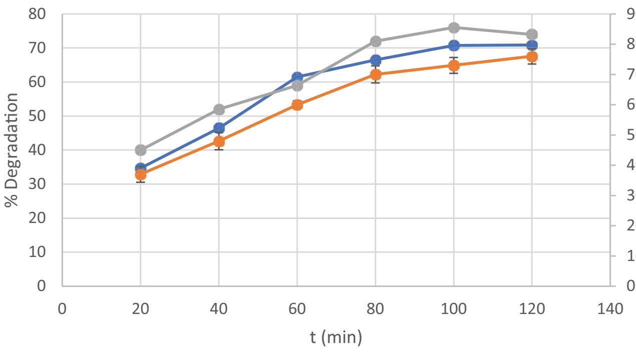


Fig. 11. Degradation percentage of 17-α-ethinyl estradiol in the electro-oxidation experiment with battery carbon cells separation of 3/16″.

contaminants. For this reason, a higher anodic potential can generate a higher electron capture activity, which favors both the direct oxidation of organic contaminants on the anode surface [26] and •OH production from water electrolysis for OH-mediated reactions [27]. More free or adsorbed •OH produced could cause an electrophilic attack on compounds that have an aromatic ring in their structure and its derivatives [28,29].

The possible mechanism of electrochemical degradation of EE2 is described below. The first step is the electrolysis of water, forming •OH hydroxyl radicals and OH^- anions, which are transferred to the anode as a function of positive charge to produce •OH radicals:

$$2H_2O - 2e^- \rightarrow 2\,{}^{\bullet}OH + 2H^+ \tag{1}$$

$$OH^- - e^- \rightarrow {}^{\bullet}OH \tag{2}$$

The electrogenerated •OH radicals can be involved in the formation of two other intermediate groups; H_2O_2 and $HO_2^{\bullet}$. Hydroxyl radicals may react with each other, forming hydrogen peroxide near the anode [Eq. (3)], which is then oxidized to oxygen [Eq. (4)]. Meanwhile H_2O_2 will react with •OH radicals in solution to yield the $HO_2^{\bullet}$ group which can combine •OH radicals to produce oxygen [Eqs. (5) and (6)].

$$2\,{}^{\bullet}OH \rightarrow H_2O_2 \tag{3}$$

$$H_2O_2 \rightarrow O_2 + 2H^+ + 2e^- \tag{4}$$

$$H_2O_2 \rightarrow {}^{\bullet}OH \rightarrow H_2O_2^{\bullet} + H_2O \tag{5}$$

$${}^{\bullet}OH + HO_2^{\bullet} \rightarrow H_2O + O_2 \tag{6}$$

3.5. Degradation kinetic

The degradation process of EE2 obeyed first-order reaction kinetics in the electrochemical degradation process (Fig. 12), which was represented by Eq. (7).

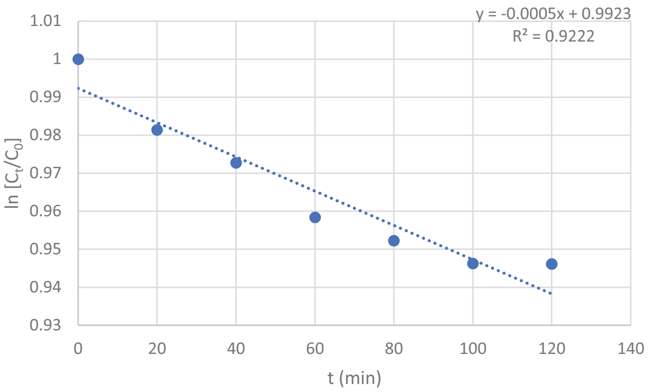


Fig. 12. Kinetic representation for the first-order reaction.

$$-\ln\left(\frac{C_t}{C_0}\right) = kt \tag{7}$$

where C_t/C_0 plotted as a function of the electrolysis time provided an exponential decay curve conforming to first-order kinetics, and the rate constants were evaluated from the slope of the plots of $\ln(C_t/C_0)$ vs. the reaction time (t). This suggests that the generation of •OH is constant, which means that EE2 degradation corresponds to a bimolecular reaction between EE2 and •OH, and that the oxidation of EE2 was probably a diffusion controlled reaction [30].

3.6. Identification of EE2 and by-products obtained from degradation

The mass spectrum of the EE2-TMS derivative, as well as the suggested fragmentation mechanism, are shown in Fig. 13. The ion m/z = 232[M–208]$^+$ (base peak) corresponds to the ion formed from the double cleavage in cyclohexane B, at the C11–C12 and C8–C14 bonds. The ion m/z = 425[M–15]$^+$ (qualification peak) corresponds to the loss of a methyl group from the TMS group, which is linked to the aromatic ring. The ion m/z = 196[M–208]$^+$ corresponds to the ion formed due to the double cleavage in cyclohexane B at the C12–C13 and C8–C14 bonds. The ion m/z = 440 corresponds to the molecular ion of the derivative EE2-TMS.

Fig. 14 shows the final chromatogram (the boxes indicate the new peaks) with their respective retention time and the most probable compound according to the NIST database. The highest results, in terms of coincidence percentages between the chromatograms obtained from each new peak and those of the databases, are shown in Table 2, as well as their respective retention times and mass-charge ratio (m/z).

The results shown in Table 2 reveal that the main by-products identified in the degradation of EE2 correspond to carboxylic acids, which is consistent with those reported in other studies [31,32].

A possible explanation of the by-products obtained from the degradation of EE2 could be inferred as follows: EE2 was oxidized by the hydroxyl radical to hydroxylation products by hydrogen substitution or an additional reaction, and then the ring was opened; the products were then oxidized to macromolecular organic carboxylic acids and small organic carboxylic acids [24].

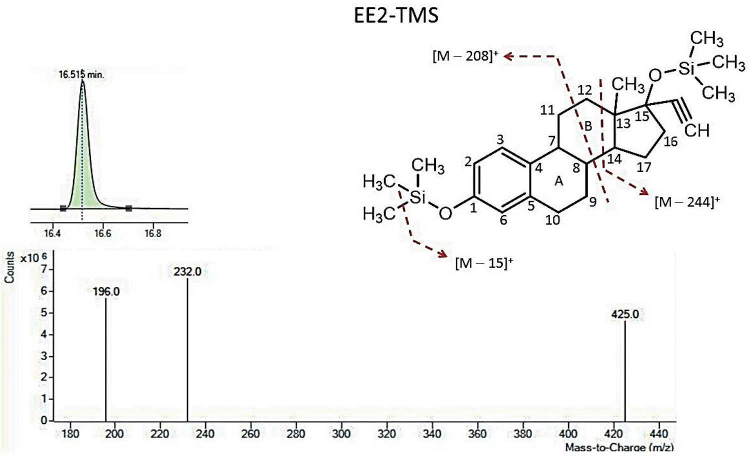


Fig. 13. Identification of the 17-α-ethinyl estradiol-[N-methyl-N-(trimethylsilyl)trifluoroacetamide] derivative and its fragmentation mechanism.

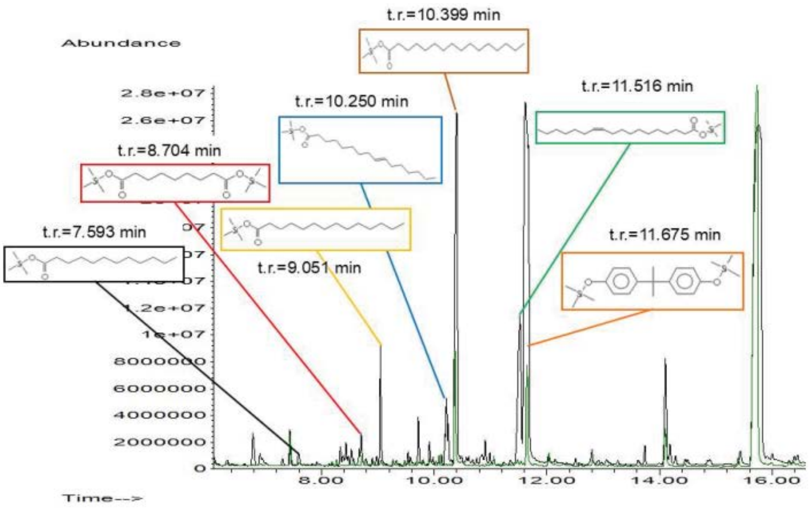
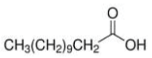
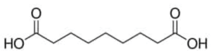
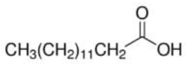
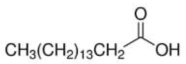
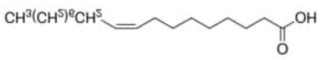
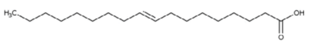


Fig. 14. Final chromatogram with by-products obtained after electrochemical degradation.

Table 2
Probable species formed in the electrooxidation reaction of 17-α-ethinyl estradiol

tr	Compound	*m*/*z*	Structure
7.95	Dodecanoic acid	257	
8.704	Azelaic acid	317	
9.051	Tetradecanoic acid	285	
10.399	Hexadecanoic acid	313	
11.516	Oleic acid	339	
11.675	Elaidic acid	117	

4. Conclusions

Through the voltametric characterization of the three types of electrodes built, it was possible to observe that the electrode that gave the best performance from the point of view of current density and power was the one that was built with electric motor brushes (MBC). However, this presented wear, and for this reason the battery waste (BC) was selected with a separation between anode and cathode of 3/16".

The degradation efficiencies obtained in the present study of around 74% in just 2 h are highly satisfactory if we compare them with other electrochemical processes that, although they have better degradation efficiencies, use electrodes doped with another compound. This doping will increase cost in the implementation of large-scale wastewater treatment systems.

The degradation kinetics fit a first-order model, suggesting that hydroxyl radical generation remained constant during degradation. This means that EE2 degradation corresponds to a bimolecular reaction between EE2 and •OH, and the oxidation of EE2 was probably a diffusion-controlled reaction.

The by-products identified correspond mostly to carboxylic acids, which is consistent with the degradation route expected when the hydroxyl radical breaks an aromatic ring.

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