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Characterization of a new cartridge type electrocoagulation reactor (CTECR) using a three-dimensional steel wool anode

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Abstract

In this work a new electrocoagulation reactor with cylindrical geometry using a three-dimensional steel wool anode has been developed. The architecture of the Electrocoagulation reactor is closely related to a filter cartridge, modified in order to contain the electrodes. The complete system includes the Cartridge Type ElectroCoagulation Reactor (CTECR) and its housing. The residence time distribution (RTD) was used as tool to study the flow behavior of the electrolyte within the reactor. The new reactor has been successfully used in the removal of a textile dye (Remazol Red RB 133) working in continuous mode of operation, where the color elimination rate reaches 99 %. Moreover, its design allows both an easy replacement of the cartridge when the steel wool anode is consumed, and the collection and storage of the hydrogen generated on the cathode.

Highlights

A new Cartridge type reactor for electrocoagulation is tested.
Electrocoagulation is performed using 3D electrodes made of steel wool anodes.
Removal of textile dye Remazol Red RB 133 is used as test reaction.
Color removal efficiency reaches 99%.

Keywords

Electrocoagulation; Cartridge type reactor; RTD; Dye removal; Wastewater treatment

1.- INTRODUCTION

For the last decades, the social concern about the environmental impact caused by industry is growing and new laws demanding more strict environmental protection are being approved. For this reason the search of “greener” and more efficient methods for wastewater treatment is increasing.

Among the different techniques for wastewater treatment, electrochemical methods have achieved a relevant place [1-3]. Electrocoagulation (EC) is an electrochemical technique closely related to chemical coagulation, that involves the supply of coagulant ions (Al^{3+} , Fe^{3+}) by the application of an electrical current to a sacrificial anode (made of aluminum or iron) placed into an electrochemical reactor [4-6]. The metallic ions produced by the corrosion of Al or Fe behave in a similar way to the aluminum or ferric ions employed in chemical coagulation. However, the characteristics of the particle aggregates (flocs) generated during the electrocoagulation process differ dramatically from those generated by chemical coagulation. Thus, flocs generated in an electrocoagulation process tend to contain less bound water and are both more shear resistant and more readily filterable. On the other side, the amount of chemicals needed is much lower and the salinity of the wastewater does not increase.

Although EC has been implemented for the second part of the 20th century with limited success and popularity, this technology has been increasingly used for treatment of industrial wastewaters from different origins in the last decade [4, 7-9]. Thus, EC has been applied to treat wastewaters containing oil emulsions [10, 11], food waste [12-15], dyes [16-19], and other type of pollutants [20, 21]. Also, the electrical energy needed for the corrosion of Al or Fe can be obtained from classical or renewable electricity sources [22-24].

Electrocoagulation reactors have been built in a number of cell geometries ranging from open tanks to filter press cells. Different electrode shapes have also been used (plates, cylindrical, mesh, etc.) [2, 4, 5]. Each system has its own set of advantages and disadvantages. The selection of a particular reactor configuration should consider factors including from the mode of operation (continuous vs batch) to operational parameters like floc buoyancy, bubble formation, passivation of the electrodes, etc.

The aim of this work is to study the behavior of a novel EC reactor design using a three-dimensional anode. The cylindrical geometry of the EC reactor is closely related to filter cartridges, where steel wool is used as sacrificial anode in continuous mode of operation to form the called Cartridge Type Electro Coagulation Reactor (CTECCR). Since EC is widely used in the treatment of wastewater from the textile industry, a textile dye has been employed as pollutant model for testing the efficiency of the new reactor design. The chosen dye is the Remazol Red RB 133, which has been previously employed in different works as a model pollutant. These studies demonstrated the feasibility of color removal from a Remazol Red RB 133 containing solution when treated by electrocoagulation using Al anodes [22, 25] and Fe anodes[26].

Firstly, in order to characterize the reactor, a Residence Time Distribution (RTD) study was carried out at several flow rates. Next, the removal efficiency of Remazol Red RB was studied at different ratio of current intensity to flow rate. Finally, other possible advantages of using this EC reactor were studied, including: i) monitoring of the sacrificial anode up to the total consumption and subsequent replacement, and ii) collection and storage of the hydrogen generated on the cathode.

2.- EXPERIMENTAL

2.1.- EC experiments using a conventional tank reactor.

EC experiments using iron plates as sacrificial anodes were performed in batch mode of operation. The laboratory EC cell consisted of a methacrylate parallelepiped tank, where the electrodes stack was submerged. The electrode stack assembly consisted of three parallel-plate iron electrodes: two anodes alternating with one cathode. The outer sides of both anodes were electrically isolated, so that only the inner sides –facing the cathode-, were electrochemically active. The dimensions of the electrodes were 130 mm x 90 mm x 2 mm. The total anodic area was 234 cm², the interelectrode gap was 1cm and the volume of solution was 650 cm³. Experiments were performed at room temperature. A magnetic stirrer was used to stir the solution. A continuous current supply (Blausonic Power Supply 0-30V 2.5A DC) was employed.

2.2.- EC experiments using a new CTECR reactor with a three-dimensional steel wool anode.

The EC reactor developed in this work is based on a system of cartridge and its housing, CTECR. **Figure 1** shows a section drawing of both elements (cartridge EC reactor and housing) and a detailed view of the components of the CTECR. The dimensions of the elements of the cartridge EC reactor are shown in **Table 1**.

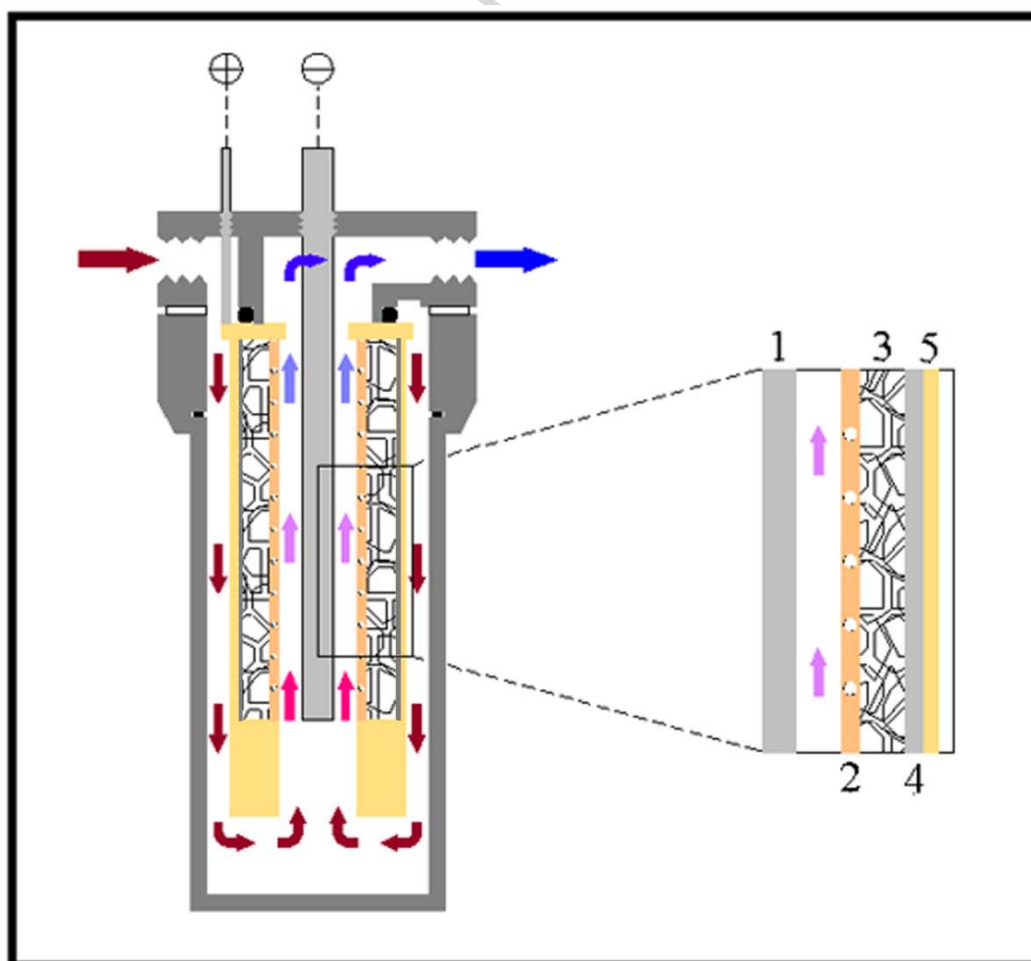


Figure 1. Schematic cross-sectional view of the EC reactor cartridge and housing.
 1) Aluminum cathode. 2) Polyethylene mesh. 3) Steel wool anode. 4) Stainless steel mesh. 5) External plastic cartridge.

Table 1. Dimensions of the CTECR reactor elements.

DESCRIPTION	DIMENSIONS
Cartridge EC reactor volume	650 mL
Outer casing	
Diameter	6.5 cm
Height	25 cm
Steel wool anode	
Height	20 cm
Thickness	1.25 cm
Plastic mesh	
Diameter	4 cm
Height	20 cm
Cathode	
Diameter	1 cm
Height	20 cm

As shown in figure 1, the cartridge is the electrocoagulation reactor. It has a cylindrical geometry and starting from the outside has the following cylindrical elements:

1.- Plastic outer casing open at the lower part, so that the solution flow therein is forced upward.

2.- Current collector made of a stainless steel mesh from Alson's Filters. The electrical connection is made using an isolated wire drawn at the top of the housing (see figure 1).

3.- The anode is a three-dimensional electrode made of extra fine steel wool. Steel wool, also known as wire wool or wire sponge, consists of bundles of fine steel filaments. It is commonly used as an abrasive in finishing and repair work for polishing wood or metal objects, cleaning household cookware, cleaning windows and sanding surfaces. The chemical composition of the steel wool is 99 % iron, 0.12-0.15 % carbon and 0.8-1 % manganese, and the wire thickness is approximately 0.030 mm. The piece of wool employed is 20 x 20 cm, rolled to form a cylinder and located between the outer casing and the plastic mesh.

4.- Polyethylene mesh charged with the task of pressing the anode against the current collector in order to minimize IR drops and avoid the contact between anode and cathode. The wire diameter is 0.1cm and mesh size is 0.5 cm.

The cathode is an aluminum rod (diameter 1 cm) placed at the axis of the cartridge. However, it is anchored to the housing such that when the exhausted cartridge is replaced, the cathode is not extracted from the system. The dimensions of the cathode are: diameter 1cm and height 20 cm (the rest of the aluminum cylinder is electrically insulated using a thin silicone sheath)

EC experiments with Remazol Red RB 133 solutions were carried out in a continuous mode of operation without effluent recirculation. **Figure 2** shows a scheme from the experimental set employed. The treated solution was collected at the electrocoagulation cell exit. It is important to note that in every assay, a volume equivalent to three times the reactor volume ($3 \times 650 \text{ cm}^3$) of treated solution were neither collected nor analyzed in order to allow the EC system to reach steady state. After that, samples were decanted and the supernatant liquid was analyzed. The experiments

were carried out at room temperature and effluent temperature was measured at the electrocoagulation cell exit.

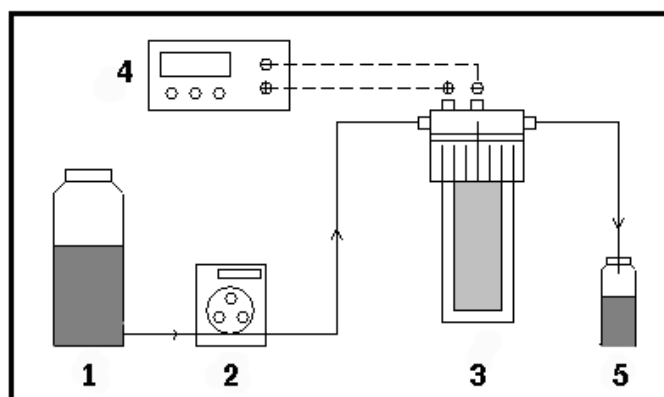


Figure 2. Experimental system diagram. (1) Solution tank. (2) Peristaltic pump. (3) Electrocoagulation reactor. (4) Power supply. (5) Treated solution.

A Laboratory Power Supply Elektro Automatic MODEL EA-PS 2016-100 DC current supply was employed. The solution was circulated by a Heidolph PumpDrive 5206 peristaltic pumps. The pH of the treated samples was measured by a CRISON microPH 2000 pH-meter, whereas conductivity was measured by a CRISON 525 Conductivity meter.

2.3.- Residence Time Distribution (RTD) curves

2.3.1.- Experimental assembly and procedure.

The RTD experiments were carried out in the CTECR and housing using the experimental system shown in figure 2 with the following modifications. First, a conductivity meter was used to determine the NaCl tracer concentration at the reactor exit. Also, centrifugal pumps (Rule 500GPH and Sanso PMD311) and a flowmeter were used.

All RTD experiments were carried out under room conditions with distilled water using NaCl as tracer. Distilled water from the effluent reservoir was fed into the reactor at different inlet flow rates and 5 ml of NaCl brine were injected in a pulse input into the entrance of the EC cartridge cell. To do this, a silicone tube was placed in the housing so that the tracer was injected just at the bottom inlet of the EC cartridge cell. The corresponding conductivity of the raw water was measured using a conductivity meter with Galvanic Isolated Output 4-20 mA, connected to a data acquisition module. The experiment ended when the value of the conductivity decreased to the normal distilled water level.

2.3.2.- RTD characteristics.

The Residence Time Distribution (RTD) of a chemical reactor is a probability distribution function that describes the amount of time a fluid element could spend inside the reactor. RTD curves are used to characterize the mixing and flow within reactors [27-29].

RTD curves were determined experimentally using the classical pulse tracer response technique. The conductivity of the solution at the exit of the reactor is registered thus obtaining the concentration of tracer and the curve $C(t)$. The obtained curve of $C(t)$ can be transformed into a dimensionless residence time distribution curve by the following relation:

$$E(t) = \frac{C(t)}{\int_0^{\infty} C(t) \cdot dt} \quad (1)$$

$E(t)$ is the function that describes in a quantitative manner how much time different fluid elements have spent in the reactor. Other characteristic parameters of flow dynamics in the reactor can be calculated from the following equations:

$$t_m = \int_0^{\infty} t \cdot E(t) \cdot dt \quad (2)$$

$$\tau = \frac{V_r}{Q} \quad (3)$$

where t_m =mean residence time (s); τ =hydraulic residence time (s); V_r =reactor volume (l); and Q =volumetric flow rate (Ls^{-1}).

The RTD profiles can be used to calculate some quantitative relationships between the hydraulic residence time (τ), peak time (t_p), and the mean residence time (t_m). These quantitative parameters can be calculated from the following relations:

$$\text{Plug flow index (PFI)} = t_p / \tau \quad (4)$$

$$\text{Dead zone index (DZI)} = t_m / \tau \quad (5)$$

2.4.- Chemicals and analysis.

The working solution for the color removal test was prepared by dissolving Remazol Red RB 133 dye (DyStar S.A.) in ultra-pure water. Conductivity and pH were adjusted adding the necessary amount of HCl 35 % Merck and NaCl Panreac 99 %. In every sample the concentration of NaCl was 2 g L^{-1} .

Remazol RB concentrations were determined using a working curve of absorbance versus concentration at long-wave absorption maximum λ_{\max} (518 nm for Remazol Red RB 133). The decolorization efficiency, E , is calculated as:

$$E = \frac{C_i - C_f}{C_i} \times 100 \quad (6)$$

where C_i is the initial dye concentration (mg/L) and C_f the final dye concentration (mg/L).

The concentration of Remazol Red RB 133 in the solution was determined spectrophotometrically at 518 nm wavelength, using a HACH DR 2000 spectrophotometer. Treated samples were filtered before analysis in order to remove flocs formed during the treatment. There was no color decrease when the wool was submerged within the samples, so we have no evidence of dye adsorption on the wool.

3.- RESULTS AND DISCUSSION

3.1.- CTECR reactor characterization.

In order to evaluate the effect of the flow rate on the hydraulic behavior of the CTECR reactor, RTD studies were carried out at several flow rates (5-130 L h⁻¹).

Unfortunately, for the lower flow rates studied (5.6 and 8.7 L h⁻¹) RTD data and curves were not reproducible due to the fact that the injection of the tracer caused great turbulences inside the reactor. In these cases, when flow rate is low, the turbulences generated are stronger than the liquid flow and the injection changes the hydrodynamic pattern significantly. For higher flow rates the tracer injection flow does not modify the hydrodynamics of the reactor.

Figure 3 shows the representation of $E(t)$ vs t for different flow rates. The figure shows that for all the flow rates studied a single peak of similar shape was obtained. Whatever was the flow rate, the value of $E(t)$ increases sharply to a peak maximum and, after this, $E(t)$ goes back to the baseline only after a long time. The increase in flow rate results in higher values of $E(t)$ at the peak maximum, and a shortening of the posterior tail. The appearance of a single peak also indicates that there are no recirculation or parallel paths.

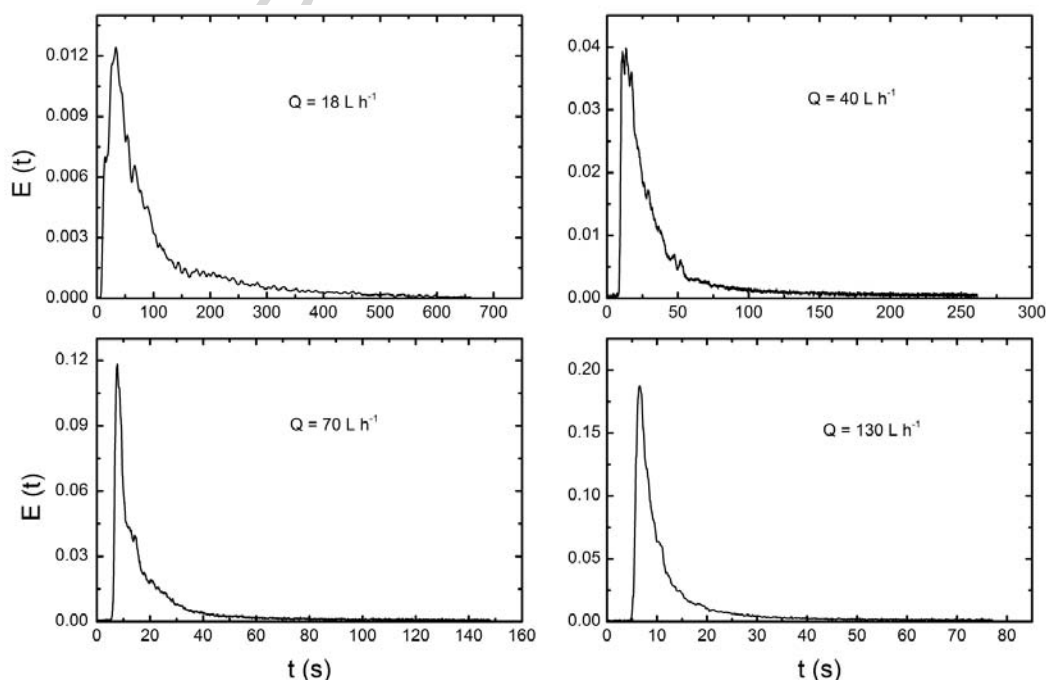


Figure 3. RTD $E(t)$ vs t curves for different flow rates: a) 18, b) 40, c) 70, and d) 130 Lh⁻¹

The sharp increase of $E(t)$ is characteristic of a plug flow. By increasing the solution flow rate, both an increase in the value of $E(t)$ at the peak maximum, and a decrease in the minimum residence time

(when first signals of traced appear) are observed. It seems that plug flow is thus enhanced by higher flow rates. These results are supported by table 2, where an increase of PFI is observed on increasing the flow rate.

The shape of the curves indicates that the behavior of the reactor assimilates to the combination of a plug flow, a mixed tank and a dead volume. The three types of pattern correspond to the different regions inside the reactor. The plug flow corresponds to the center of the cartridge, between the cathode and the polyethylene mesh, where the liquid flows without any obstacle. The region corresponding to the mixed tank is the area near the mesh, where the matter exchange between the anode and the solution takes place. The dead volume may correspond to the region inside the wool mesh, where the liquid is blocked up. When the flow rate increases, the blocked volume decreases due to the higher stirring in the interface between the different zones [27].

For high flow rates (higher than 70 L h^{-1}), the plug flow behavior is stronger and it is interesting when the mode of operation of the CTECR – batch or continuous – includes a recirculation of the solution. On the other hand, for systems with no recirculation is more interesting to perform the treatments at low flow rates, because the residence time increases, the matter exchange is better and the coagulant species have time enough to trap the dye molecules.

Table 2. Calculated parameters using RTD experimental data.

Q (L h^{-1})	t_m (s)	τ (s)	t_p (s)	PFI	DZI
18	115	130	26	0.2	0.77
40	49	57	13	0.23	0.74
70	24	31	8	0.26	0.67
130	14	19	6	0.32	0.57

3.2.- Elimination of Remazol Red RB 133

Once the reactor was characterized, the treatment of a simulated wastewater containing a dye was tested.

3.2.1. Batch reactor

Previous studies demonstrated the feasibility of color removal from a Remazol Red RB 133 containing solution when treated by electrocoagulation using Al anodes [22, 25]. The optimal conditions found for the elimination of color in a 650 cm^3 mixed tank where: j : 10 mA cm^{-2} and pH^0 : 6, for a $[\text{RB}]^0$: 250 ppm. In order to test the behavior of the treatment when Iron anodes are employed, some experiments were carried out under different values of time, Q and j.

The treatment was performed at different treatment times, in the range of 5-30 min. When a current density of 10 mA cm^{-2} is applied, a treatment time of 5 min is enough to achieve a color removal of 99.8 %. **Figure 4** shows the results of the experiments under different values of j, where values higher than 2.5 mA cm^{-2} allow achieving elimination rates higher than 99.5 %. The voltage values measured at the reactor were in the range 1.5 - 6 V. These results confirm that the elimination of color by electrocoagulation using Fe anodes is feasible in the range of j values studied, similar to those obtained using Al anodes in a batch reactor.

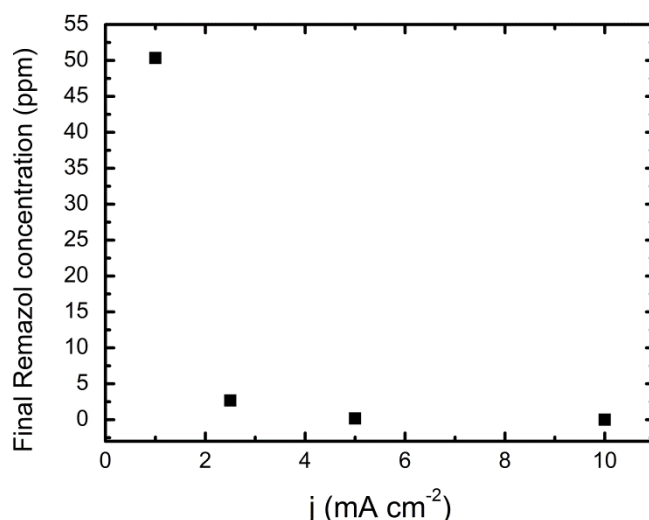


Figure 4. Final concentration of Remazol vs j. t: 10 min. [RB]^o: 100 ppm. pH^o: 4.4. k^o: 2.7 mS cm⁻¹.

3.2.2. Cartridge type Electrocoagulation reactor (CTECCR)

The removal of the dye using the cartridge type reactor was tested and optimized in order to demonstrate its proper behavior. Among the most important parameters to control when EC is employed for wastewater treatment are the current density and flow rate. For this study, samples of a solution of Remazol Red RB 100 ppm were treated by applying the same range of current densities employed in section 3.2.1., and varying flow rate from 4.9 to 18.6 L h⁻¹. The current densities applied to the three dimensional electrodes are calculated for the geometric area of the mesh. This flow rate range allows working under hydraulic residence times similar to those studied at the batch experiments, as seen in **table 3**.

Table 3. a. Current density values employed. b. Flow rates employed in the study of dye removal and their corresponding residence time.

j (mA cm ⁻²)	Q (L h ⁻¹)	τ (min)
1	4.9	7.9
2.5	6.9	5.6
3.75	9.6	4.0
5	13.3	2.9
7.5	18.6	2.1
10		

The experiments were performed in order to work under the conditions of a continuous mode treatment. Samples were taken and analyzed after a volume equal to three times the reactor volume was treated. The final Remazol Red RB concentration was measured and the values under the selected conditions are shown in **figure 5**. From these curves it can be confirmed that for current densities higher than 3.75 mA cm⁻², elimination rates are higher than 99% and do not depend on the flow rate, for the flow rate range studied. The lowest current densities have strong dependence on flow rate and only when j: 2.5 mA cm⁻² and Q: 5 L h⁻¹ good results are obtained. For the current densities higher than 3.75 mA cm⁻², every flow rate applied allowed to obtain removal rates higher than 99 %. It needs to be taken into account that for a real application, the most interesting is to achieve the highest treatment capacity with the lowest energy consumption. **Table 4** shows the

energy consumption values for these experiments. Taking into account conditions where efficiency is higher than 95 %, the most suitable conditions obtained for the treatment of a 100 ppm Remazol Red RB solution were j : 3.75 mA cm⁻² and Q : 13.3 L h⁻¹, with an energy consumption of 0.68 kW h m⁻³ for a 98 % efficiency.

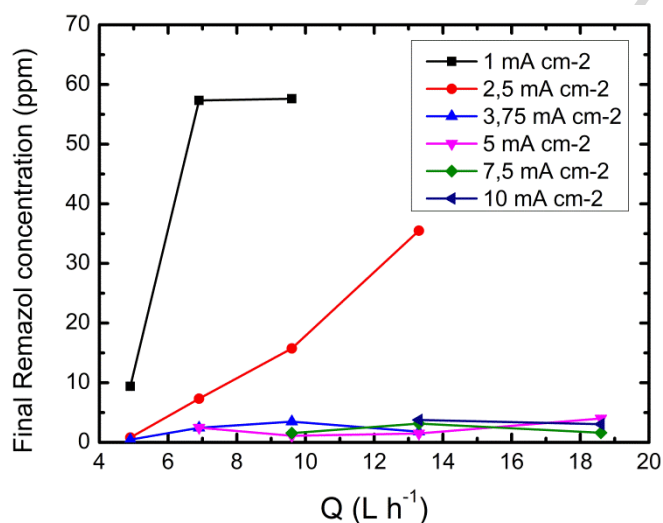


Figure 5. Final Remazol Red RB 133 concentration after the treatment at different values of flow rate and current density.

Table 4. Experimental data for EC treatment and calculated energy consumption.

j (mA cm ⁻²)	Voltage (V)	Q (Lh ⁻¹)	Final RB concentration (ppm)	Efficiency (%)	Energy consumption (kW h m ⁻³)
1.25	2.6	4.9	9.4	90.6	0.27
1.25	2.6	6.9	57.3	42.7	0.19
1.25	2.6	9.6	57.6	42.4	0.14
2.5	4.4	4.9	0.8	99.2	0.90
2.5	4.4	6.9	7.3	92.7	0.64
2.5	4.4	9.6	15.8	84.2	0.46
2.5	4.4	13.3	35.5	64.5	0.33
3.75	6	4.9	0.5	99.5	1.86
3.75	6	6.9	2.5	97.5	1.31
3.75	6	9.6	3.5	96.5	0.94
3.75	6	13.3	1.8	98.2	0.68
5	7.7	6.9	2.5	97.5	2.23
5	7.7	9.6	1.1	98.9	1.60
5	7.7	13.3	1.5	98.5	1.16
5	7.7	18.6	4.0	96.0	0.83
7.5	10.5	9.6	1.6	98.4	3.28
7.5	10.5	13.3	3.2	96.8	2.37
7.5	10.5	18.6	1.6	98.4	1.69
10	13.7	13.3	3.8	96.2	4.12
10	13.7	18.6	3.1	96.9	2.95

- Long-term experiments

This new reactor was conceived for working as a continuous mode reactor and to perform long experiments until the exhaustion of the anode. The reactor was also designed for an easy replacement of the exhausted anode. In order to test the anode lifetime, a long-term experiment was carried out. The conditions selected for this experiment were j : 3.75 mA cm^{-2} and Q : 5 L h^{-1} , because it is the lowest flow rate that guarantees a high elimination rate with a minimum reactive and sample consumption. The experiment lasted for 11 hours and the voltage was registered. The voltage increased continuously during the experiment, due to the loss of anode mass. The shortening of the anode means that the current is applied to a shorter surface, appearing higher overpotentials. When the voltage measured exceeded 25 % of the initial value, it was considered as the point when the anode was exhausted, and this is the moment for replacing anodes. At the end of the experiment, the reactor was open and it was verified that the anode was totally exhausted (**figure 6**). This situation is to be avoided, because current density and distribution are not the expected, energy consumption increases and the efficiency of the treatment falls down. The theoretical calculation of the time needed for the total dissolution of the three dimensional anode employed is 11.5 h, very similar to the actual time measured in the test.



Figure 6. Picture of the exhausted anode at the end of the long-term experiment.

- **Gas recovery study**

As it was previously explained, H_2 (g) is formed because of water reduction at the cathode, whereas O_2 (g) may be formed at the anode through the undesired reaction of water oxidation, which results in a decrease of the Fe dissolution efficiency and the H_2 (g) stream contamination. Commonly, the EC reactors are open structures where the gasses formed are able to escape to the atmosphere and are not easily recovered. In this case, the reactor built is closed and this allows driving the gasses to a separated tank, from where they can be recovered.

The gas produced during an EC experiment was collected, measured and compared to the theoretical volume of H_2 (g) produced if all the current was employed in the hydrogen generation. The experiment was carried out at j : 3.75 mA cm^{-2} and Q : 5 L h^{-1} . The theoretical gas production is 14.9 mL min^{-1} , whereas the gas production measured was 13.6 mL min^{-1} . This value represents an error of 8 % and the measured values were always below the theoretical, what means that the efficiency of the H_2 (g) production is lower than 100% and that probably the production of O_2 (g) is very short. These results confirm that the CTECR reactor allows the possibility of recovering the hydrogen generated, which can be employed as a fuel or a reagent. In the case that O_2 (g) is formed, it is possible to separate and purify the hydrogen stream. The recovery of hydrogen allows reducing the global cost of the treatment, making it more feasible for an industrial application.

4.- CONCLUSIONS

In this work a new system consisting of a Cartridge Type Electrocoagulation Reactor (CTECCR) and its housing has been developed. The CTECCR has a cylindrical geometry that is closely related to the geometry of a filter cartridge. The developed CTECCR uses a three-dimensional steel wool as sacrificial anode and works in continuous mode of operation.

Residence Time Distribution (RTDs) curves at different flow rates were used as tool to study the flow behavior of the electrolyte within the reactor. RTD curves showed that the behavior of the reactor assimilated to the combination of a plug flow, a mixed tank and a dead volume. The plug flow and dead volume regions correspond to the open region at the center of the cartridge and to the region deep inside the steel wool mesh respectively. The mixed tank behavior can be related to the "interface" between plug flow and dead volume regions.

The new reactor has been successfully used in the removal of a textile dye (Remazol Red RB 133) with removal efficiencies very similar to those obtained when an iron plate was used as anode in a conventional batch reactor. Other advantages of the developed CTECCR reactor are: i) its design allows an easy replacement of the cartridge when the steel wool anode is consumed, ii) long term experiments have demonstrated that the exhaustion of the steel wool anode takes place when approximately 95% of the active mass has been consumed, and iii) it has been demonstrated that the hydrogen generated on the cathode can be collected and stored.

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Highlights

A new Cartridge type reactor for electrocoagulation is tested.

Electrocoagulation is performed using 3D electrodes made of steel wool anodes.

Removal of textile dye Remazol Red RB 133 is used as test reaction.

Color removal efficiency reaches 99%.

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