Ibuprofen removal from synthetic effluents using Electrocoagulation-Peroxidation (ECP)

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Abstract Concerning water resources, several ordinances and legislation determine standards and conditions for the discharge of effluents into water bodies. However, several contaminants are not covered by these guidelines due to little knowledge of their long-term effects and because they are found in low concentrations. These contaminants are called emergent and this category includes drugs, such as anti-inflammatory drugs. The electrocoagulation process associated with advanced oxidation comes up as an alternative to conventional effluent treatment processes, and the objective of this study was to evaluate this process using scrap iron as sacrificial electrodes in the treatment of synthetic effluents containing ibuprofen. High-performance liquid chromatography was used to quantify the drug in synthetic effluents. The Central Rotational Composite Design $2^4$ was used in an experimental design, considering independent variables the concentration of contaminants, applied current, the concentration of the primary oxidizing agent $\text{H}_2\text{O}_2$, and the reaction time. The optimized conditions determined by statistical analysis were drug concentration of 5 mg L$^{-1}$, $\text{H}_2\text{O}_2$ concentration of 200 mg L$^{-1}$, current of 5 A, and 150 min. The removals obtained under these conditions were higher than 92% in the aqueous phase, showing that electrocoagulation peroxidation technique has the potential to treat contaminants such as drugs present in effluents and waters.

Keywords Emerging contaminants · Scrap iron electrode · Hydrogen peroxide · High-performance liquid chromatography · Electrochemical process · Medicines

Introduction

One of the main concerns of society today is the preservation of water resources, in view of the importance of water for the life of all species on the planet. Among the various contaminants studied that may affect this natural resource, the so-called emerging contaminants have stood out in the last decade. One of the main sources of environmental contamination is sanitary sewage, in addition to all the pathogens present in this is a continuous entry of emerging compounds. In addition to effluents specifically, the overflow of sewage in several regions of the world results in pollution of water bodies, such as rivers and streams, aggravating the process of degradation of water quality, and thus increasing environmental and
public health risks (Sojobi & Zayed, 2022). Sojobi (2016) in his study showed the presence of several contaminants not only in surface waters, but also in underground waters, being possible to observe that the contamination caused by anthropic actions has more severe effects, changing the characteristics of subsoil waters, being able to leave them unsuitable for consumption and in this way compromising food security.

Emerging contaminants are defined as a class of contaminants that has evolved as a result of the development of new products; most of these are not inserted in monitoring programs or environmental legislation. Emerging contaminants are chemical substances (synthetic or natural) that were recently detected in the environment and there is little information about the environmental and public health risks that they may cause (Naidu et al., 2016). Among the emerging contaminants, which are produced on a large industrial scale, drugs stand out, as they are directly linked to human health (Ebele et al., 2016). Among the drugs most used by self-medication in Brazil, ibuprofen stands out (Arrais et al., 2016). The continuous disposal of drugs in water bodies is a worldwide socio-environmental problem.

Chronic exposure to drugs can cause unexpected effects on the human organism and other living beings (Torres et al., 2012). That said, much research and discussion has been carried out regarding the presence of drugs in water, underground and surface bodies. These compounds, when discarded improperly in the environment, are almost unchanged (Aranami & Readman, 2007). This contamination is a major challenge for water distributors and public health. Some research has shown the consequences of emerging contaminants in the environment, such as hormonal damage in aquatic beings, impaired development of insects and invertebrates, and inhibition of algae photosynthesis; in humans, there is an increase in the incidence of breast, testicular, and prostate cancer; infertility; and endometriosis (Machado et al., 2016; Naidu et al., 2016).

The concentration of these drugs in the effluent treatment stations can reach the level of μg L⁻¹ and, in some treatment stations, aimed at the treatment of drug effluents, it can reach levels of milligrams per liter (Santos et al., 2009; Sim et al., 2011; Zhao et al., 2019). Among the various drugs that can be found in supply and wastewater, one of the most common and of unrestricted use is ibuprofen, so several treatment methods have been adopted in recent years, given the high incidence of this drug in low concentrations in water. Table 1 presents some studies that address the removal of Ibuprofen in recent years.

Ibuprofen is a weak organic acid that when ingested is metabolized and excreted in the form of metabolites and their conjugates. About 15% of the compound is excreted in the parental form and 26% and 43% in the form of hydroxyl-ibuprofen and carboxy-ibuprofen metabolites (Farré et al., 2008; Luz et al., 2022). In water bodies, ibuprofen is of potential risk, as it can cause oxidative stress affecting the reproduction and maintenance of aquatic species (Luz et al., 2022; Stancova et al., 2017; González-Pérez et al., 2016).

Most of the water used for human supply in Brazil originates from surface water treatment, requiring advanced treatment to eliminate various pollutants. But most treatment plants use conventional processes, inefficient for emerging contaminants (Machado et al., 2016). Another source of contamination is the inappropriate disposal of drugs, which is of greater

| Emerging compounds       | Removal methods                          | Degradation efficiency (%) | Reference                  |
|--------------------------|------------------------------------------|----------------------------|---------------------------|
| Ibuprofen                | Adsorption mesoporous carbon microspheres| About 90.0                 | Ulfa et al. (2020)         |
| Ibuprofen and acetaminophen | Electrocoagulation                      | 78.0                      | Negarestani et al. (2020)  |
| Ibuprofen                | Nano-heterojunction                      | 99.7                      | Sruthi et al. (2021)       |
| Ibuprofen and sulfamethoxazol | Biosorption with nanoparticles        | 86.33–85.80               | Priyan and Narayanasamy (2022) |
| Ibuprofen                | Ozonation e photocatalyst                | 93.0                      | Alhumede et al. (2022)     |
| Ibuprofen and triclosan  | UV direct photolysis and UV/H₂O₂         | 97.0, 39.0                | Luz et al. (2022)          |
relevance in view of the growing consumption of medicines by the population. Figure 1 shows possible pharmaceuticals paths in the environment.

In order to minimize environmental impacts, it is necessary to develop treatment technologies that combine low cost and high efficiency. In this context, the electrocoagulation (EC) process appears as an alternative to conventional effluent treatment processes and it can be associated with the presence of a primary oxidizing agent, which may enhance the process of removing contaminants, as it associates the electrochemical process with advanced oxidative processes, where the formation of hydroxyl radicals (·OH) that present a high oxidative potential occurs and it turns the EC process more effective.

Several electrochemical methodologies are applied in wastewater treatment, including electrocoagulation, which was highlighted due to its advantages (Deghles & Kurt, 2016). Electrocoagulation is an electrolytic process that consists of dissolving a sacrificial electrode after applying a current between two electrodes to treat liquid wastewater containing organic pollutants (Khemila et al., 2018).

According to Aquino Neto et al. (2011), the electrocoagulation process occurs in three stages. Initially, the cations that are generated in the oxidation of the sacrificial metal anode will react with the water molecules to form hydroxides and polyhydroxides. Iron and aluminum are the metallic materials most used as sacrificial electrodes, due to their low cost and high efficiency. At the same time, electrolysis of water occurs and the formation of oxygen and hydrogen microbubbles, which will later serve to load the flocculated material to the surface. Then, the hydroxides that have been formed are adsorbed into colloidal particles that will cause the formation of the floccules, which during their transport end up in contact with the impurities. Flotation occurs due to the formation of microbubbles of oxygen at the anode and hydrogen at the cathode, due to water electrolysis. The pollutants end up being dragged to the surface, clarifying the effluent.

The electrochemical reactions of the metal as anode and cathode can be summarized according to Eqs. 1 to 6 (Chen, 2004; Gabriel, 2017; Mollah et al., 2004):

For the iron anode:

\[
\text{Fe}_{(s)} \rightarrow \text{Fe}^{3+}_{(aq)} + 3\text{e}^{-} \quad (1)
\]

\[
\text{Fe}^{3+} + 3\text{OH}^- \rightarrow \text{Fe(OH)}_3 \quad (2)
\]

![Fig. 1 Possible pharmaceuticals paths in the environment](image)
4Fe^{2+} + O_2 + 2H_2O \rightarrow 4Fe^{3+} + 4OH^- \quad (3)

Cathode reactions and formation of oxygen and hydrogen are:

Fe^{3+} (aq) + 3e^- \rightarrow Fe(s) \quad (4)

2H_2O(l) \rightarrow O_2 + 4H^+ + 4e^- \quad (5)

2H_2O(l) + 2e^- \rightarrow H_2(g) + 2OH^- \quad (6)

Electrocoagulation has been widely used in the treatment of industrial effluents, such as effluent from the textile industry (Tones, 2015), refrigeration (Eryuruk et al., 2018), tannery (Pavão et al., 2018), dairy (Behling et al., 2018), oily effluent (Cometti et al., 2014), paper and cellulose (Carvalho et al., 2015), biodiesel (Cordeiro et al., 2015), and drugs (Olvera-Vargas et al., 2021) among other types. It is a particularly effective technique for a wide range of pollutants, such as heavy metals, organic compounds, microorganisms, and several others; due to this, it is receiving greater prominence today.

One of the ways to potentiate the effect of electrocoagulation is the use of oxidizing agents such as hydrogen peroxide, since in addition to the removal by the formation of the coagulant, it can accentuate the mineralization of the contaminants and its decomposition generates ·OH radicals, which present high oxidative potential and thus has the ability to degrade these compounds reaching mineralization.

Due to their remarkable mineralization efficiency and because they are considered ecologically correct, advanced electrochemical oxidation processes stand out among the most promising technologies for treating refractory organic pollutants, including pharmaceutical compounds (Olvera-Vargas et al., 2021).

The presence of hydrogen peroxide promotes the production of the hydroxyl radical (OH) and, therefore, the oxidation potential can be improved (Bashir et al., 2018; Sun et al., 2017). Hydroxyl radicals are powerful secondary oxidizing agents; they are not selective, reacting quickly with organic compounds by means of hydroxylation with the addition of a hydroxyl group to an unsaturated bond or dehydrogenation, through the loss of a hydrogen atom, following a mechanism with radical until the conclusion of its mineralization, through the transformation of initial pollutants into carbon dioxide, water, and inorganic ions (Asghar et al., 2015; Bashir et al., 2018; Boye et al., 2003).

Thus, the best conditions are sought to improve the efficiency of the peroxidation electrocoagulation process in the removal of contaminants of pharmaceutical origin, without the need to use large amounts of energy and thus optimizing the current density, the concentration of H_2O_2, and the reaction time in the process. About this perspective, the objective of this work was to evaluate the application of EC associated with advanced oxidation and electrocoagulation peroxidation (ECP) to remove ibuprofen from synthetic effluents.

Materials

Synthetic effluent containing ibuprofen

Synthetic effluents were prepared containing ibuprofen diluted in deionized water in varying concentrations according to the experimental planning matrix. Reagents with a pharmaceutical standard acquired in a handling pharmacy were used to prepare synthetic effluents. In preparing the solutions, it was necessary to add 5 mL of methanol in order to improve the dilution of the contaminant. In this study, analytical standards were used for high-performance liquid chromatography (HPLC) analysis (Sigma Brand) to build the standard curve for quantification.

Experimental module

As electrodes for the electrocoagulation system, iron scrap was used supplied by the Cercena Foundry Industry, located in the city of Erechim, RS, Brazil. The electrodes used had the dimensions of 12 cm × 9 cm × 3 mm. In the ECP process, an electrochemical reactor on a laboratory scale made of glass was used, with dimensions 15 cm × 30 cm and capacity for a volume of 3 L (2 L of synthetic effluent was used for each treatment (Fig. 2)). The density of the electric current applied to the treatments was based on the literature, varying from 1 to 5 A; the distance between the electrodes was fixed at 3 cm. For a better conductivity in the synthetic effluent, 2 g L^{-1} of NaCl was added to each treatment and this value was obtained in preliminary tests as the most
suitable for the system under study and then fixed for all tests. All tests were performed at room temperature (20–25 °C), under agitation of 100 rpm, without the use of a light barrier, so that the conditions used were closer to a full-scale condition. The pH of the effluent remained with the natural value of the solution, close to neutral (6.5–7.5). It is worth mentioning that preliminary tests were performed without the addition of $H_2O_2$, where there was no significant removal of the drugs.

**Methods**

**Experimental procedure**

For the experimental design, the Central Rotational Composite Design (CCRD) was used (Rodrigues & Iemma, 2009). Four independent variables were worked on: electric current, treatment time, ibuprofen concentration, and hydrogen peroxide concentration. A complete factorial design $2^4$ was carried out, including 8 axial points and 4 repetitions in the central points, totaling 28 tests. The efficiency of EC was based on the percentage of drug removal. Table 2 shows the coded values of the independent variables. It is noteworthy that the values of the independent variables were defined from preliminary tests with the effluent. Table 3 shows the matrix of the experimental design. The influence of the independent parameters was determined from the efficiency of removing the response or dependent parameter. From the empirical models obtained with the CCRD, the condition of optimization of the treatment process is obtained, in which the maximization of efficiency in the removal of response variable is sought.

**Analytical determination of ibuprofen**

The determination of the concentration of the drug ibuprofen during the tests was done by means of high HPLC. For this purpose, a chromatograph of the Shimadzu brand model LCMS-2020 was used, equipped with a C18 column, 5 μm in diameter, 250 mm in length, and 4.6 mm in internal diameter, and a SPD-M20A photodiode network detector. The analysis occurred by isocratic elution, with the mobile phase consisting of 80% methanol HPLC (≥99.9%) and 20% ultrapure water.

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**Table 2** Levels studied for the independent variables of the experimental design matrix

| Independent variables | Levels |
|-----------------------|--------|
|                       | $-2$   | $-1$ | 0 | 1 | 2 |
| Ibuprofen concentration (mg L$^{-1}$) | 5 | 10 | 15 | 20 | 25 |
| $H_2O_2$ concentration (mg L$^{-1}$)    | 150 | 200 | 250 | 300 | 350 |
| Current (A)            | 1 | 2 | 3 | 4 | 5 |
| Time (min)             | 30 | 60 | 90 | 120 | 150 |
(mili-Q®), acidified with 0.1% formic acid, flow 0.8 mL min⁻¹, injection volume of 20 μL, analysis time of 20 min, and oven temperature of 30 °C, with the same temperature for the column. The quantification of the compound was performed through analytical curves with solutions of the analytical standards for each compound.

To create the analytical curve, standard solutions were prepared with eight concentrations in the range of 0.05 to 20 ppm. Before all injections in the chromatograph, the raw and treated effluent went through a filtration process with a 0.45 μm PTFE filter.

**Sludge analysis**

The analysis of the contaminants in the sludge was performed after the peroxidation electrocoagulation with the optimized variables. At the end of the process, excess liquid was removed, and sludge was collected in Falcon tubes, totaling 23 samples. These samples were centrifuged for 10 min at 1500 rpm. The solid was deposited at the bottom of the tubes, facilitating the removal of the supernatant for disposal. The sludge was resuspended using 10 mL of methanol in each tube followed by stirring. The tubes were centrifuged once again under the same

| Table 3 Experimental design matrix | Test | Coded variables | Independent variables |
|------------------------------------|------|----------------|-----------------------|
|                                    |      | IC (mg L⁻¹) | H₂O₂ (mg L⁻¹) | C (A) | T (min) |
| 1                                 | −1   | −1   | −1   | −1 | 10  | 200 | 2  | 60 |
| 2                                 | −1   | −1   | −1   | 1  | 10  | 200 | 2  | 120 |
| 3                                 | −1   | −1   | 1    | −1 | 10  | 200 | 4  | 60 |
| 4                                 | −1   | −1   | 1    | 1  | 10  | 200 | 4  | 120 |
| 5                                 | −1   | 1    | −1   | −1 | 10  | 300 | 2  | 60 |
| 6                                 | −1   | 1    | −1   | 1  | 10  | 300 | 2  | 120 |
| 7                                 | −1   | 1    | 1    | −1 | 10  | 300 | 4  | 60 |
| 8                                 | −1   | 1    | 1    | 1  | 10  | 300 | 4  | 120 |
| 9                                 | 1    | −1   | −1   | −1 | 20  | 200 | 2  | 60 |
| 10                                | 1    | −1   | −1   | 1  | 20  | 200 | 2  | 120 |
| 11                                | 1    | −1   | 1    | −1 | 20  | 200 | 4  | 60 |
| 12                                | 1    | −1   | 1    | 1  | 20  | 200 | 4  | 120 |
| 13                                | 1    | 1    | −1   | −1 | 20  | 300 | 2  | 60 |
| 14                                | 1    | 1    | −1   | 1  | 20  | 300 | 2  | 120 |
| 15                                | 1    | 1    | 1    | −1 | 20  | 300 | 4  | 60 |
| 16                                | 1    | 1    | 1    | 1  | 20  | 300 | 4  | 120 |
| 17                                | −2   | 0    | 0    | 0  | 5   | 250 | 3  | 90 |
| 18                                | 2    | 0    | 0    | 0  | 25  | 250 | 3  | 90 |
| 19                                | 0    | −2   | 0    | 0  | 15  | 150 | 3  | 90 |
| 20                                | 0    | 2    | 0    | 0  | 15  | 350 | 3  | 90 |
| 21                                | 0    | 0    | −2   | 0  | 15  | 250 | 1  | 90 |
| 22                                | 0    | 0    | 2    | 0  | 15  | 250 | 5  | 90 |
| 23                                | 0    | 0    | 0    | −2 | 15  | 250 | 3  | 30 |
| 24                                | 0    | 0    | 0    | 2  | 15  | 250 | 3  | 150 |
| 25                                | 0    | 0    | 0    | 0  | 15  | 250 | 3  | 90 |
| 26                                | 0    | 0    | 0    | 0  | 15  | 250 | 3  | 90 |
| 27                                | 0    | 0    | 0    | 0  | 15  | 250 | 3  | 90 |
| 28                                | 0    | 0    | 0    | 0  | 15  | 250 | 3  | 90 |

IC: ibuprofen concentration, H₂O₂: concentration, C: current, T: time
conditions. The supernatant methanol was removed, filtered with a 0.45 PTFE filter, and the final volume was combined and the sample was then injected into the chromatograph. Equation 7 can be used to check the concentration of total contaminants that has been removed.

\[
[C_{TR}] = [C_I] - ([C_{FL}] + [C_L])
\]  

(7)

Meaning:

\([C_{TR}]\) total contaminant concentration removed

\([C_I]\) initial contaminant concentration

\([C_{FL}]\) contaminant concentration in the liquid phase after ECP treatment

\([C_L]\) contaminant concentration in the sludge formed in the treatment

Analysis of residual hydrogen peroxide

For the analysis of residual hydrogen peroxide in the samples, the MQuant® colorimetric kit (Merck) was used, which detects the peroxide in the concentration range of 0.5–25 mg L\(^{-1}\) and 1–100 mg L\(^{-1}\).

Determination of total iron

To determine the total dissolved iron in the treated effluent, an analysis was performed by mass spectrometry with inductively coupled plasma, only in the sample under the optimized conditions.

Statistical analysis

For the statistical analysis of the results of the CCRD matrix, the Statistica\(^{\circledR}\) 7 program was used to perform analysis of variance (ANOVA) and obtain the graphical representation of the model, using a response surface graph and contour profile, which assists in determining the optimal operating region for EC.

Since the model is valid for the response variables, the global desirability function, available in the Statistica\(^{\circledR}\) 7 program, was applied; its function is based in the simultaneous optimization of all the variables studied in the electrocoagulation/peroxidation process.

Results and discussion

Optimization of the ECP process

With the objective of verifying the efficiency of the electrocoagulation treatment with the use of scrap iron electrodes applied in the synthetic effluents containing ibuprofen, the removal percentage was calculated for each proposed test. The results obtained can be seen in Table 4.

According to the results of Table 4, simultaneously analyzing all the tests, there were high removals of

| Test | IC (mg L\(^{-1}\)) | \(\text{H}_2\text{O}_2\) (mg L\(^{-1}\)) | C (A) | T (min) | % Ibuprofen removal |
|------|-----------------|----------------------|------|--------|---------------------|
| 1    | 10              | 200                  | 2    | 60     | 57,537              |
| 2    | 10              | 200                  | 2    | 120    | 88,339              |
| 3    | 10              | 200                  | 4    | 60     | 95,125              |
| 4    | 10              | 200                  | 4    | 120    | 89,551              |
| 5    | 10              | 300                  | 2    | 60     | 92,766              |
| 6    | 10              | 300                  | 2    | 120    | 96,146              |
| 7    | 10              | 300                  | 4    | 60     | 94,728              |
| 8    | 10              | 300                  | 4    | 120    | 90,991              |
| 9    | 20              | 200                  | 2    | 60     | 80,359              |
| 10   | 20              | 200                  | 2    | 120    | 71,794              |
| 11   | 20              | 200                  | 4    | 60     | 94,109              |
| 12   | 20              | 200                  | 4    | 120    | 84,505              |
| 13   | 20              | 300                  | 2    | 60     | 93,105              |
| 14   | 20              | 300                  | 2    | 120    | 95,681              |
| 15   | 20              | 300                  | 4    | 60     | 87,513              |
| 16   | 20              | 300                  | 4    | 120    | 95,589              |
| 17   | 5               | 250                  | 3    | 90     | 88,969              |
| 18   | 25              | 250                  | 3    | 90     | 94,831              |
| 19   | 15              | 150                  | 3    | 90     | 72,657              |
| 20   | 15              | 350                  | 3    | 90     | 97,702              |
| 21   | 15              | 250                  | 1    | 90     | 86,353              |
| 22   | 15              | 250                  | 5    | 90     | 99,882              |
| 23   | 15              | 250                  | 3    | 30     | 96,902              |
| 24   | 15              | 250                  | 3    | 150    | 96,641              |
| 25   | 15              | 250                  | 3    | 90     | 90,033              |
| 26   | 15              | 250                  | 3    | 90     | 96,004              |
| 27   | 15              | 250                  | 3    | 90     | 88,011              |
| 28   | 15              | 250                  | 3    | 90     | 94,538              |

\(IC\) ibuprofen concentration, \(\text{H}_2\text{O}_2\) concentration, \(C\) current, \(T\) time
ibuprofen, reaching more than 99% removal. Khadir et al. (2020) applied electrocoagulation in water contaminated with ibuprofen and, under the optimized conditions of 110 min, pH 5, 2 A of current, and 3 cm of distance between the electrodes, the maximum removal was 63%. It can be seen that there is a lot of variation in the removals; this may have happened because the electrodes are made of scrap iron and may differ from each other. It is worth mentioning that only in the tests with the lowest current and the shortest time (tests 21 and 23), the effluent, after treatment, presented residual hydrogen peroxide, with concentrations of 100 mg L\(^{-1}\).

The final pH of the treated effluent remained in the range close to neutral (6–7.5). According to Zaied et al. (2020), pH is a significant parameter in the performance of EC in the treatment of drug effluent, which removal efficiency reduces when the pH of the solution becomes alkaline or acid. It was concluded that the removal efficiency with pH in the range of 5.74 to 7.48 is greater in comparison with the pH value of 7.48 to 8.26. In the study by Khadir et al. (2020), where electrocoagulation was applied to remove ibuprofen, they analyzed the removal efficiency with pH values of 2, 3, 5, 7, and 9. The removal efficiency of 28.75% and 11.5% was reached at pH 2 and 9, respectively. At pHs 3, 5, and 7, the removal efficiency was 46.55, 63%, and 35.75%, respectively, with pH 5 having the highest removal.

To validate the adjustment proposed by the results obtained, ANOVA was performed based on the model provided for the experimental design for the removal of ibuprofen. The first stage of validation took place using the Pareto graph, shown in Fig. 3, where it is possible to identify the parameters and interactions that significantly influence the dependent variables, with 95% confidence, represented by the red line. Linear variables are represented by (L) and quadratic

![Pareto Chart for Ibuprofen](image)

Fig. 3 Pareto Chart for Ibuprofen

\[ \text{Standardized Effect Estimate (Absolute Value)} \]

\[ p = 0.05 \]
variables by (Q), the positive signs next to the bars indicate an increase in the removal of the parameters and the negative signs reduce the parameters.

According to the simultaneous analysis of the Pareto graphs, the H$_2$O$_2$ concentration and the current in linear terms proved to be efficient in the 95% confidence interval for removing Ibuprofen. The interaction between these two parameters in linear terms is also efficient in removing ibuprofen. ANOVA results are shown in Table 5.

For the ibuprofen removal variable, the $p$-value was statistically satisfactory at 95% confidence. It is noteworthy that the value of $F_{\text{calculated}}$ is greater than $F_{\text{tabulated}}$, which also proves that it is significant. In addition, the regression is greater than the residuals for all variables, proving that the proposed statistical model is valid. Several studies in different areas of knowledge such as Awolusi et al. (2019), Zhang et al. (2019), Sojobi et al. (2021), and Sojobi and Liew (2022) have demonstrated the versatility of the application of the response surface and contour curves to help predict the optimal conditions of a study. In the present work, this methodology was also applied in order to obtain the optimal region within the levels evaluated for the removal of ibuprofen.

Subsequently, the response surface and contour profile graphs are shown (Fig. 4). The graphs express the behavior of the percentage of removal of Ibuprofen in function of the independent variables concentration of hydrogen peroxide and applied current, the two variables that significantly influence in the removal of Ibuprofen.

From Fig. 4, it can be seen that when using hydrogen peroxide concentrations around 200 mg L$^{-1}$, associated with an average applied current (4.5–5 A), there is an increase in efficiency of removing ibuprofen. Also, it can be seen that higher values of hydrogen peroxide are associated with greater removal of

| Table 5 Analysis of variance of the model foreseen for the removal of ibuprofen from effluent treated by EC with iron electrode at a significance level of 95% ($p < 0.05$) |
|---------------------------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Parameter                       | Quadratic model | Sum of squares  | Degrees of freedom | Means  | $F_{\text{calculated}}$/$F_{\text{tabulated}}$ | $p$-value |
|---------------------------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| % Ibuprofen removal             | Regression      | 2347.165        | 14              | 167.655         | 3.692/2.554     | 0.01204         |
| Residuals                       | 590.28          | 13              | 45.406          |                 |                 |                 |
| Total                           | 2937.445        | 27              | 213.061         |                 |                 |                 |

Fig. 4 a Response surface and b contour profile of Ibuprofen removal considering hydrogen peroxide concentration and applied current
Ibuprofen as well. According to the study by Khadir et al. (2020), the greatest removal of ibuprofen by electrocoagulation was 54.75% using a current of 2 A, for 110 min. This trend can be attributed to the phenomenon that with increasing current density, the rate of dissolution of the anode increases, leading to greater removal. Through Fig. 4, it is possible to verify the regions where the H$_2$O$_2$ concentration and current ratio favor the removal of ibuprofen under the conditions evaluated and in higher intensity currents associated with concentrations above 200 mg/L result in greater removal of Ibuprofen.

Global desirability

As observed in the statistical analysis, the response variable for removing ibuprofen was significant at the 95% confidence level. Thus, it was decided to use the function Desirability, from the software Statistica® 7, which allows the optimization of multiple response variables, to determine the optimized operational values of the independent variables aiming at greater removal of the dependent variables. Figure 5 shows the optimal work values. The overall desirability value varies from 0 to 1, the closer it is to 1 the closer
the response obtained to that intended. The overall function obtained was equal to 1, thus indicating an excellent response.

The red line in the graphs indicate the optimized values of the variables drug concentration, hydrogen peroxide concentration, current, and treatment time at 5 mg L$^{-1}$, 200 mg L$^{-1}$, 5 A, and 150 min, respectively, aiming at greater removal possible of ibuprofen. From these conditions, 100% drug removal is expected to occur. Mission et al. (2011) obtained the greatest removal of ibuprofen using a current of 21.4 Velasquez et al. (2019) removed 87.61% of ibuprofen using a current of 8 A and 20 min. Negarestani et al. (2020) obtained the greatest removal of ibuprofen with the optimized values of 110 min and 2 A and drug concentration of 40 ppm. The treatment is enhanced with the addition of H$_2$O$_2$, allowing greater removal of the contaminant in a shorter time (Pereira, 2018).

Validation of the proposed model

Through the electrocoagulation test using the optimized condition obtained through the Global Desirability test, it is possible to validate the results obtained in Fig. 5. A removal close to 100% was expected; however, the actual removal obtained using the optimized conditions was 92.91%, still resulting in a concentration of 0.4 mg L$^{-1}$ for ibuprofen. In their study, Yoosefian et al. (2017) applied electrocoagulation with iron electrodes to remove the antibiotic ciprofloxacin. With the variables' initial concentration, pH, current, time, and distance between electrodes optimized, the expected removal was 99% and the experimental one was 100%. Irdemez et al. (2006) applied electrocoagulation to remove phosphate from effluents with aluminum electrodes. With the parameters optimized, the estimated and experimental phosphate removal from wastewater was 76% and 99%, respectively. With another configuration, the actual removal was 93% and 99% predicted. The difference between the actual and the estimated value may be related to the fact that the electrodes used in this work are made of scrap iron material.

In order to verify whether the ECP process enabled the effective removal of contaminants, an analysis was carried out on the sludge formed after treatment with the optimized values. The result obtained was residual Ibuprofen of 0.628 mg L$^{-1}$. Thus, it was possible to verify through Eq. 7 that there was a removal of 79.4% of the contaminant.

Some studies report the possibility of removing 95% of organic contaminants just by applying the electrochemical process (Urtiaga et al., 2014); however, it must be emphasized that the characteristics of the system, as well as the concentration of the contaminant and its structural peculiarities that will define the efficiency of the process. It should be noted that the presence of ibuprofen in the sludge shows that the physical–chemical processes can assist in the treatment of this type of contaminant and, if associated with other methods, their efficiency can be enhanced. There are several current technologies aimed at removing emerging contaminants, studies such as Nasir et al. (2022) present the use of adsorption for the removal of these compounds; however, it is known that tertiary processes such as adsorption result in a new challenge in the disposal of contaminated adsorbent material.

The results obtained in this study showed the potential for using the electrocoagulation/peroxidation process, as compared to similar technologies (Table 1); the removals were within the expected range of about 90–92%.

Total iron residual

The result obtained for the total iron residual was 132.4 mg L$^{-1}$. The high concentration can be attributed mainly to the high current that was employed (Giordanni, 2017). Ryan et al. (2020) used electrocoagulation with iron electrodes to remove disinfection by-products in the water. The residual iron after the sludge filtration was between 4.6 and 5.8 mg Fe L$^{-1}$. Pereira and Brito (2018) obtained, at the end of the treatment process for the removal of laboratory effluents, the concentration of residual total soluble iron was 6.40 mg L$^{-1}$. In Fig. 6, it is possible to see the difference between the synthetic effluent after the treatment with a color change due to the presence of the sludge containing the iron and after it is filtered with a 0.45-µm PTFE filter.

The Brazilian Resolution of the National Environment Council No. 430, of May 13, 2011 (CONAMA), presents conditions and standards for the discharge of effluents. It provides that the effluents from any polluting source can only be released directly into the receiving body if they have a dissolved iron value.
below 15 mg L$^{-1}$. Although iron is not toxic, its presence in high concentrations can affect aquatic life, public supply, and human health. Therefore, secondary treatment is necessary to enable the effluent to be released into the receiving water body.

**Conclusions**

Electrocoagulation using scrap iron electrodes proved to be an efficient treatment for the removal of ibuprofen from effluents, with an average removal of more than 90% and with no residual hydrogen peroxide in most of the tests. Analysis of variance showed that the model is valid with 95% confidence in removing ibuprofen. The analysis of the global desirability function and the response surface made it possible to determine the optimized values of the independent variables, and the actual removal for ibuprofen was 92% in the liquid phase, showing that the treatment is efficient. Still, the importance of using industrial waste in the manufacture of the electrodes used in this study is emphasized, adding value to scrap iron. The total iron concentration after the electrocoagulation/peroxidation process and after sludge separation was 4.6–5.8 mg L$^{-1}$. The removal of emerging contaminants is of great importance at the industrial level because it can facilitate obtaining higher quality water, since some industries in their specificities need greater control of the conditions in the water used in their processes, as well as enabling the practice of reuse of wastewater within the industry itself.

It is suggested a post-treatment step to remove dissolved iron that is in disagreement with the limit stipulated in environmental legislation and to determine the intermediate compounds formed from this process, in order to enable the verification of the mineralization capacity of this system.

**Abbreviations**

- A: Ampere; AEOPs: Advanced electrochemical oxidation processes; AOPs: Process with advanced oxidative processes; C: Current; CCRD: Central Rotational Composite Design; EC: Electrocoagulation; ECP: Electrocoagulation-peroxidation; IC: Ibuprofen concentration; ICP-MS: Mass spectrometry with inductively coupled plasma; H$_2$O$_2$: Peroxide hydrogen concentration; HPLC: High-performance liquid chromatography; T: Time

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**Data availability** All data generated or analyzed during this study are included in this published article [and its supplementary information files].

**Declarations**

- **Ethics approval and consent to participate** Not applicable.

- **Consent for publication** Not applicable.

- **Competing interests** The authors declare no competing interests.
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