ACETYLSALICYLIC ACID TREATMENT IN WASTEWATER BY ELECTROCOAGULATION USING ALUMINUM ELECTRODES: ELECTRICAL VOLTAGE EFFECT

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ABSTRACT

Electrocoagulation is a technique that uses electric current to induce oxidation-reduction reactions, forming hydroxides that act as coagulating agents for the removal of emerging contaminants. The general objective of this study is to evaluate the efficiency of the electrocoagulation process applied to the treatment of emerging contaminant acetylsalicylic acid by electrocoagulation. Emerging pollutants are chemicals, both natural and synthetic, and can be organic and inorganic, with high toxic potential and negative impacts on both the environment and human health. A factorial design of 33 times in triplicate was developed, the important variables were reaction time and electrical voltage. The dependent variables or response were concentration and operating cost. At the end of the treatments, the results indicated that the highest percentage of acetylsalicylic acid elimination at 5 volts was 58.67% in the total times of 10, 20 and 30 minutes at the initial concentration of 0.3 mg/mL. In the 11-volt tests, the best removal efficiency was 60.00%, with 30 minutes of removal time. As expected, the greatest wear of the electrodes was obtained through the tests subjected to 11 volts, and in relation to the cost of operation, the test with the highest cost was R$ 3.15/m3. For tests subjected to 5 Volts, the highest cost was R$ 0.73/m3. The results of the study indicated significant elimination of aspirin. Therefore, this study can be a reference for other researchers or manufacturers to evaluate and estimate the process on a larger scale using this system for the treatment of synthetic wastewater.

Keywords: Acetylsalicylic acid (ASA). Electrocoagulation. Emerging Pollutants. Wastewater Treatment.

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INTRODUCTION

The increase in the number of factories and production capacity, coupled with the growing demand for medicines, has led to an increase in wastewater generation. As a result, the massive discharge of pharmaceutical wastewater has become a cause for concern due to the considerable pollution of water bodies and toxicity, both chronic and subchronic, affecting aquatic ecosystems and humans, due to accumulation in the environment and food chain (Chen et al., 2023; Maniakova et al., 2020).

Emerging pollutants are compounds that have emerged due to poor waste management, such as pharmaceuticals, plastics, personal care products, cosmetics, dyes, among others, which end up being deposited in bodies of water, such as rivers, lakes, oceans and groundwater. (Geissen et al., 2015; Camargo et al., 2020; Jari et al., 2022).

Research indicates that small concentrations of these contaminants have been detected in water throughout the country. The U.S. Environmental Protection Agency has been striving to reduce the impact of various emerging contaminants, such as perchlorate, pharmaceuticals, PCP, and EDC (Water Quality Association, 2019).

Currently, the global consumption of medicines has increased, with antidepressants, antiseptics, and treatments for anxiety and overweight being the best-sellers in pharmacies and laboratories (Epi-Phare, 2020). Analgesics and nonsteroidal anti-inflammatory drugs (NSAIDs), such as ibuprofen, diclofenac, ketorolac, acetylsalicylic acid and paracetamol, are among the most widely consumed medicines worldwide (WHO, 2007). These drugs generate residues in wastewater, which are classified as emerging contaminants (ECs).

Emerging pollutants are compounds of diverse origins and chemical characteristics, whose presence in the environment and possible impacts have been largely ignored, generating environmental problems and health risks. Most of these are unregulated pollutants, although they could be subject to future regulations depending on studies on their potential health effects and monitoring data related to their frequency of occurrence (Gil et al., 2012; Mugs, 2017).

This poses a significant risk, and requires its regulation, depending on its possible eco-toxicological and toxicological effects, and its levels in the aquatic environment. Therefore, there is an urgent need to strengthen scientific knowledge and adopt appropriate technological and policy approaches to monitor emerging pollutants in water resources and wastewater as well as assess their potential risks to human health and the environment (UNESCO, 2017).

The environmental impact caused by the toxic substances present in emerging pollutants has led the scientific community to develop different methods of water treatment.

These methods include photocatalytic degradation (BALESTRIN et al., 2017), membrane separation (ultrafiltration, nanofiltration) (CRUZ et al., 2020; CARVALHO et al., 2021; FARTO et al., 2021), adsorption (MENDES et al., 2023; SILVEIRA., 2022; KUHL et al., 2021) and electrocoagulation (FONSECA et al, 2024; BEHLING, 2021). Although several of these processes are very effective in the disposal of different types of medicines, their large-scale implementation is limited due to high costs and complexity, especially in Wastewater Treatment Plants (WWTPs) (GOMES et al., 2017). In addition, some of these methods generate secondary contamination, since the byproducts of the transformation can be as toxic or more toxic than the original drugs (; FELIS et al., 2020).

One of the most effective methods for wastewater treatment is electrocoagulation (EC). This process works by electrically dissolving iron or aluminum ions from electrodes of the same material, which act as coagulants in the formation of ions. Metal ions are generated at the anode, while hydrogen gas is released at the cathode. The flocculated particles rise to the surface of the water thanks to hydrogen bubbles formed at the cathode (KHANDEGAR .et al, 2018; TANG et al, 2015).

Electrocoagulation has multiple potential advantages, such as its flexibility, ease of operation, absence of chemical additions, rapid start-up, easy control, and its ability to treat various contaminants, which has increased interest in its application (JAMRAH et al, 2005; TAHREEN et al, 2020; KADIER et al, 2022).

Since effluents with emerging contaminants, such as acetylsalicylic acid, are highly complex and contain elevated levels of chemical contaminants, the aim of this research was to evaluate the efficiency of the electrocoagulation process using aluminum electrodes to remove this emerging contaminant acetylsalicylic acid. Important variables such as reaction time, electric current intensity, concentration, applied voltage, and operating cost were studied.

METHODOLOGY

DESCRIPTION OF THE STUDY SITE

The research experiments were carried out at the Air Pollutants Laboratory, the Water Resources and Environmental Sanitation Laboratory and the Multiuser Laboratory of the Londrina Campus (LabMult-LD), located in the city of Londrina, PR, on the campus of the Federal Technological University of Paraná (UTFPR-LD), block S. In addition, it had the support and collaboration of the laboratories and supplies of the Chemical Engineering career of the State University of Maringá.

EXPERIMENTAL PROCEDURE

The experiments were carried out according to the statistical designs given in Table 1 and Table 2. Two different experimental designs were executed with independent variables of 5 and 11 volts of electrical voltage.

In the first statistical design, electrocoagulation experiments were performed using a 3 3 factorial design, evaluating the effects of independent variables at three levels (-1, 0, 1). The experiments were conducted in triplicate, with a total of 27 trials. The independent variables in this design were acetylsalicylic acid concentration (mg/L) and electrocoagulation time (min) at 5 volts of electrical voltage, as presented in Table 1.

In the second experiment, a 3 3 factorial design was applied. The tests were performed with the independent variables concentration of acetylsalicylic acid (mg/L) and electrocoagulation time (min) at 11 volts of electrical voltage. The arrangements of the experiments are detailed in Table 2.

Table 1 presents the values of the independent variables used in the experimental designs. The independent variables are acetylsalicylic acid concentration (mg/L) and electrocoagulation time (min) at 5 volts of electrical voltage.

Table 1. Values of independent variables (5 volts)

Statistical planning					
Practice	Concentration (mg/mL)	Time (min)	Amperage	Absorbance (ABS)	Acetylsalicylic acid (mg/mL)
1	0.1	10	0.03	2.398	0.08
2	0.1	20	0.03	2.132	0.07
3	0.1	30	0.02	2.035	0.067
4	0.2	10	0.05	3.211	0.109
5	0.2	20	0.02	3.336	0.114
6	0.2	30	0.02	3.336	0.114
7	0.3	10	0.06	3.336	0.114
8	0.3	20	0.04	3.336	0.114
9	0.3	30	0.03	3.336	0.114
10	0.1	10	0.03	2.308	0.077
11	0.1	20	0.02	2.17	0.072
12	0.1	30	0.02	2.282	0.076
13	0.2	10	0.04	3.211	0.109
14	0.2	20	0.03	3.336	0.114
15	0.2	30	0.02	3.336	0.114
16	0.3	10	0.08	3.336	0.114
17	0.3	20	0.04	3.512	0.12
18	0.3	30	0.03	3.336	0.114
19	0.1	10	0.03	2.398	0.08
20	0.1	20	0.03	2.132	0.07
21	0.1	30	0.02	2.035	0.067
22	0.2	10	0.05	3.211	0.109
23	0.2	20	0.02	3.336	0.113
24	0.2	30	0.02	3.335	0.112
25	0.3	10	0.06	3.334	0.114
26	0.3	20	0.04	3.336	0.115
27	0.3	30	0.03	3.336	0.114

Table 2 presents the values of the independent variables used in the experimental designs. The independent variables are acetylsalicylic acid concentration (mg/L) and electrocoagulation time (min) at 11 volts of electrical voltage.

Table 2. Values of independent variables (11 volts)

Statistical planning					
Practice	Concentration (mg/mL)	Time (min)	Amperage	Absorbance (ABS)	Acetylsalicylic acid (mg/mL)
1	0.1	10	0.07	2.385	0.085
2	0.1	20	0.06	2.26	0.081
3	0.1	30	0.06	2.222	0.079
4	0.2	10	0.1	3.301	0.118
5	0.2	20	0.07	3.426	0.123
6	0.2	30	0.06	3.301	0.118
7	0.3	10	0.11	3.426	0.123
8	0.3	20	0.13	3.426	0.123
9	0.3	30	0.08	3.426	0.123
10	0.1	10	0.07	2.385	0.085
11	0.1	20	0.07	2.204	0.079
12	0.1	30	0.06	2.28	0.082
13	0.2	10	0.09	3.426	0.123
14	0.2	20	0.07	3.301	0.118
15	0.2	30	0.06	3.301	0.118
16	0.3	10	0.08	3.426	0.124
17	0.3	20	0.17	3.426	0.124
18	0.3	30	0.15	3.301	0.118
19	0.1	10	0.07	2.385	0.084
20	0.1	20	0.06	2.26	0.08
21	0.1	30	0.06	2.222	0.078
22	0.2	10	0.1	3.301	0.117
23	0.2	20	0.07	3.426	0.122
24	0.2	30	0.06	3.301	0.117
25	0.3	10	0.11	3.426	0.122
26	0.3	20	0.13	3.426	0.122
27	0.3	30	0.08	3.426	0.122

Preparation of Synthetic Water

The water used in the study was prepared with acetylsalicylic acid produced by the pharmaceutical company Adria, following the AOAC method (Association of Official Analytical Chemists, 1995).

The dose of acetylsalicylic acid was determined by the spectrophotometric method, in which a UV spectrophotometer - model DR 5000 was used

Preparation of HCl Solution 0.1 mol/l

Hydrochloric acid (HCl) was considered the "target" of the sample and was used to calibrate the spectrophotometer. HCl solutions were prepared at 0.1 mol/L, taking into account that its molar mass is 36.46 g/mol. Thus, the mass (g) needed to prepare 5 liters of the solution was calculated.

Developing Standard Solutions

Crude acetylsalicylic acid (ASA) standards (0.1 mg/mL, 0.2 mg/mL, and 0.3 mg/mL) were prepared in 2000 mL volumetric flasks. The respective masses of the reagent were weighed and dissolved in a solution of 0.1 mol/L HCl. The volume of the flasks was then filled with the acidic solution. For the preparation of these solutions, the masses (g) of acetylsalicylic acid (ASA) to be weighed were calculated on the basis of a solution of 100 mL at 0.3 mg/mL. The molar mass of acetylsalicylic acid (ASA) was considered to be 180.158 g/mol, as determined by Merck (2001).

Performing the Calibration Curve and Sample Analysis

Table 3 presents the data necessary for the construction of the calibration curve of acetylsalicylic acid (ASA) solutions, taking into account the dilute concentration of each sample (mg/mL) and its respective absorbance. Absorbance was measured at a wavelength of 230 nm using a model DR 5000 spectrophotometer . Thus, it was possible to construct the calibration curve A x C (mg/mL) and calculate the specific absorbance of acetylsalicylic acid (AAS).

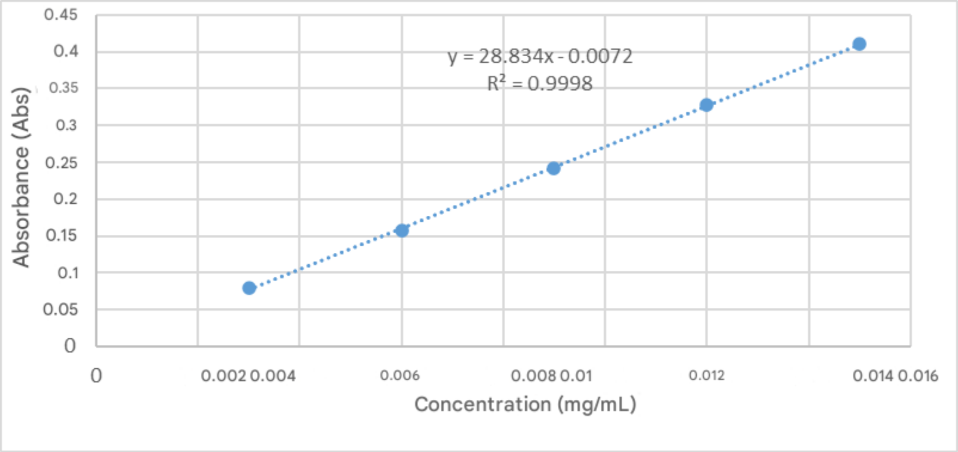
Table 3. Support for calibration curve performance and sample analysis

	Acetylsalicylic Acid (AAS) (mL) Standards	Volumen HCl 0,1 mol/ mL(mL)	Deionized H2O	Concentration Standard (mg/mL)	Absorbance (Abs)
White	-	25	Completed by 100 mL	0	-
1	1	25		0,004	0,080
2	2	25		0,007	0,158
3	3	25		0,010	0,243
4	4	25		0,013	0,329
5	5	25		0,016	0,412

From the data in Table 3, the calibration curve A x C (mg/mL) could be made. Data on the standard concentration of each sample in relation to its absorbance were used. From the curve and equation of the line ($y=28.834x-0.0072$) obtained, it was possible to determine the value of the concentration of acetylsalicylic acid (ASA) in the x-axis by substituting the absorbance value of the sample in the y-axis.

In Figure 1, it represents the calibration curve, to know the concentration through absorbance.

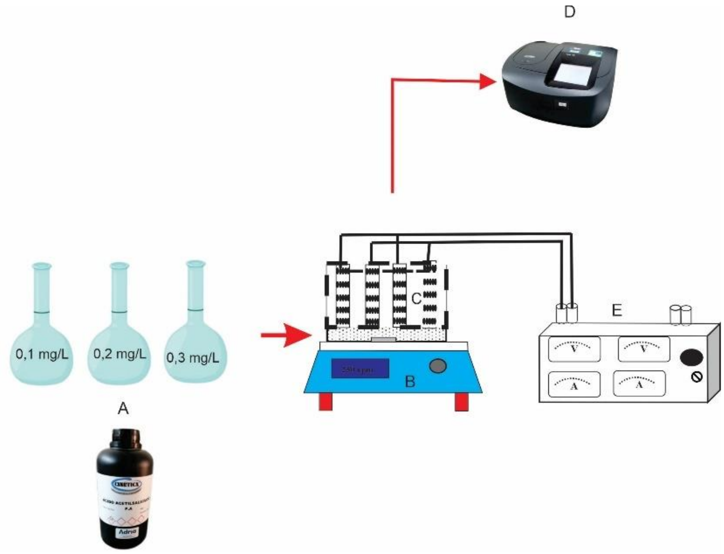
Figure 1. Calibration curve



ELECTROCOAGULATION PROCESS SCHEME ADAPTED FOR ACETYLSALICYLIC ACID

Figure 2 presents a process diagram: A) Preparation of synthetic water in different concentrations (AAS) B) Magnetic stirrer C) Reactor with aluminum electrodes D) UV spectrophotometer - model DR 5000 E) Power source (5 and 11 volts)

Figure 2: Electrocoagulation process diagram



The electrocoagulation process diagram consisted of 1 polypropylene cylindrical reactor with a volume of 200 mL. Four aluminium electrolytic plates were inserted into the reactor, connected in parallel and spaced 2 cm apart. The plates, which refer to the electrodes, are 7.5 cm high, 4 cm wide and 3 mm thick and were fixed with PVC (polyvinyl chloride) screws. In addition, plates of the same polarity were interconnected by a metal screw.

OPERATIONAL COSTS

Electrode Wear Calculation

According to Meneses et al. (2012), electrode wear in the electrochemical formation process of the coagulant agent is related to the consumption of electrons, the generation of which is directly related to Faraday's Laws. Therefore, equation 2 can be used to calculate the amount of electrode consumed.

$$m_{el} = \frac{i \times t \times M}{F \times z} \qquad (2)$$

Where:

mel = maximum amount of electrode consumed (g);

i = applied current (A) - the average of the currents of each test was used;

t = application time of the current(s) - which was constant at 30 minutes;

M = molar mass of the predominant element in the electrode (g/mol) - aluminium (26.98 g/mol)

z = number of electrons involved in the oxidation reaction of the anode element - aluminium (3)

F = Faraday's constant (96,500 C/mol).

Electrode Wear Time

To obtain the actual weight of the electrodes, without the presence of residues from the electrolysis process, they will be cleaned with phosphoric acid. After cleaning, the electrodes will be weighed on a precision balance. According to Wiendl (1998), the estimated electrode wear time can be calculated using equation 3:

$$t = \frac{M_0}{\alpha \times i} \qquad (3)$$

Where:

t = electrode wear time (h);

Mo = initial molar mass of aluminium (g) - 57.35 g

α = electrochemical equivalent of electrode material (g/A.h) - aluminum (0.335 g/A.h)

i = applied current (A) - the average of the currents of each test was used.

Electricity Consumption

According to Kobya et al. (2006), the electricity consumption in an electrocoagulation system can be expressed by the following Equation 4:

$$C_{energy} = \frac{U \times i \times t}{V} \qquad (4)$$

Where:

Cenergy = energy consumption (W.h/m³);

U = electrical voltage applied to the system (V);

i = electric current applied to the system (A) - the average of the currents of each test has been used;

t = current application time (h) - 0.5 hours;

V = volume of treated effluent (m³) - 0.00025 m³

System Operating Cost Calculation

According to Donini et al. (1994) and Kobya et al. (2006), electrode material and electricity costs account for most of the cost of operating an electrocoagulation system, and can be calculated using equation 5:

$$C_{operacion} = a.C_{energy} + b.C_{electrode} \qquad (5)$$

Where:

C operation = Operating cost (R$/m³ of effluent);

a = energy cost (R$/kWh) - R$ 0.63/kWh;

C energy = energy consumption (kWh/m³ of effluent);

b = Mass cost of the plate (R$/kg of electrode) - aluminum (R$ 5.40/kg);

C electrode = electrode consumption (kg/m³ of effluent)

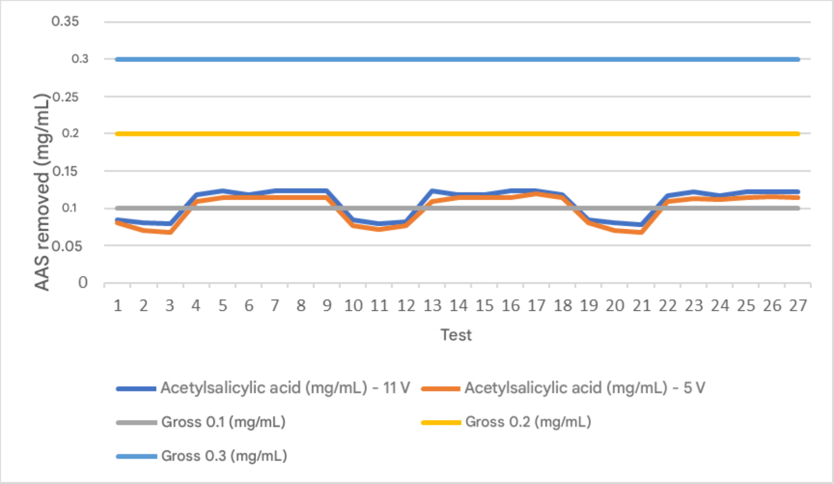
RESULTS AND DEBATE

RESULT OF THE PARAMETERS EVALUATED

After treating the emerging contaminant, the parameters were evaluated using graphs based on the laboratory data. This allowed to analyze the elimination efficiency of the emerging contaminant acetylsalicylic acid, as well as to evaluate the results with the cost of operation, using electrocoagulation.

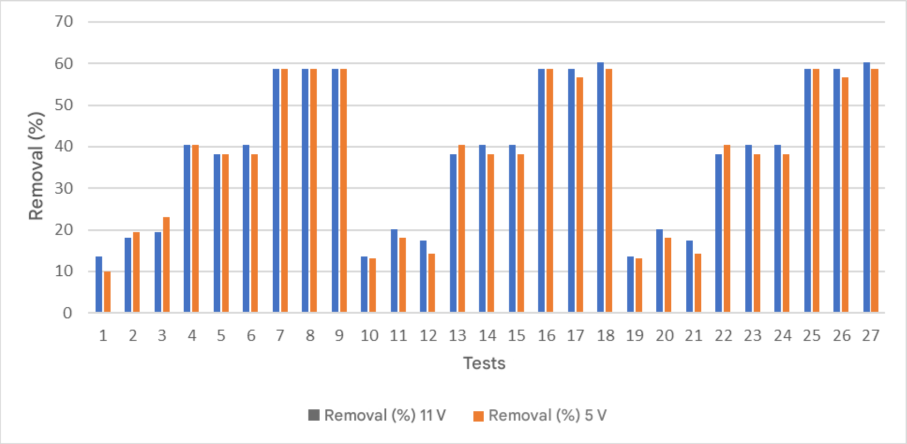
In Fig. 3 . The concentration of acetylsalicylic acid removed in the electrocoagulation process is compared under two different voltage values: 5 volts and 11 volts. The term "ASA (mg/mL)" refers to the concentration of acetylsalicylic acid removed, and horizontal lines indicate the concentrations of contaminated raw water samples. The removal of the substance with both voltages was similar, indicating that there were no significant differences between them.

Figure 3. Concentration of Acetylsalicylic Acid Removed



In Figure 4.- It can be seen that some trials have achieved even greater efficacy in the removal of acetylsalicylic acid, this was the case of trials 7, 8, 9, 16, 17, 18, 25, 26 and 27, which presented the highest concentration of the drug in the crude sample (0.3 mg/mL). The best result was obtained in tests 18 and 27, which were subjected to a voltage of 11 volts and had an electrocoagulation time of 30 minutes, resulting in a removal of acetylsalicylic acid of 60.0%.

Figure 4. Salicylic acid removal efficiency



RESPONSE SURFACE GRAPHS

Figure 5 shows the graph of the response surface of tests subjected to a voltage of 5 volts.

As expected, the surface graph of the electrocoagulation process at 5 volts was similar to the process with a voltage of 11 volts. However, the coefficient of determination presented a slightly better result, with R2 = 0.99134

Figure 5. Surface graph of CE process behavior at 5 volts

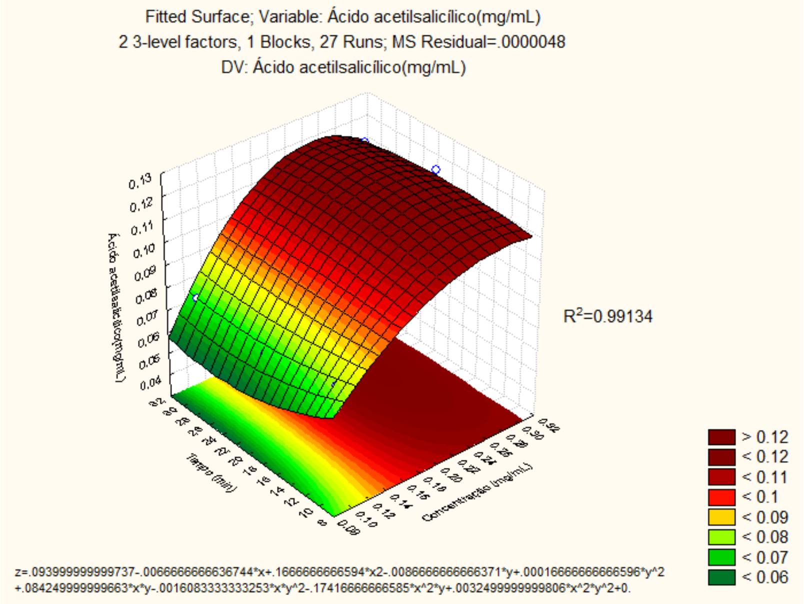
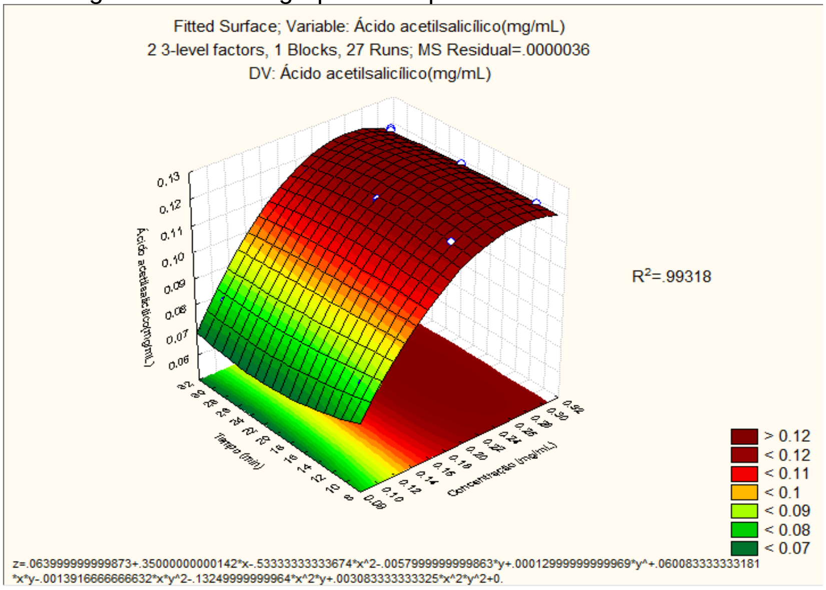


Figure 6 shows the graph of the response surface of tests subjected to a voltage of 11 volts. As expected, the surface graph of the electrocoagulation process at 5 volts was similar to the process with a voltage of 11 volts. However, the coefficient of determination showed a slightly better result, with R^2 = 0.99318. That means that there was a greater removal of the emerging contaminant acetylsalicylic acid at 11 volts.

Figure 6. Surface graph of EC process behavior at 11 volts

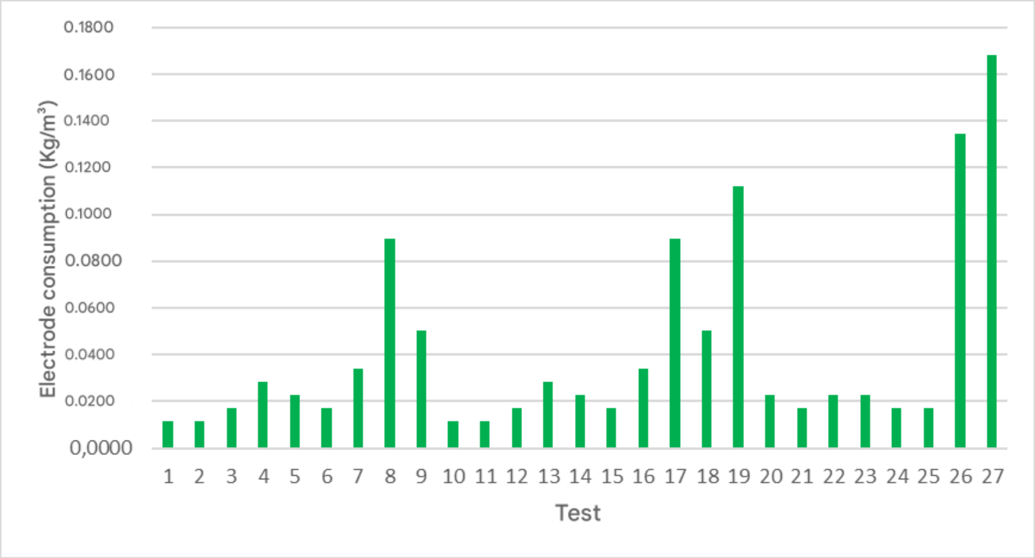


ELECTRODE CONSUMPTION

Figure 7 shows the data obtained for the tests subjected to the elimination of acetylsalicylic acid at a voltage of 11 volts. The highest electrode consumption was reached in test 27, reaching 0.165 kg/m3. According to the experimental plan, this test has an electrocoagulation time of 30 minutes.

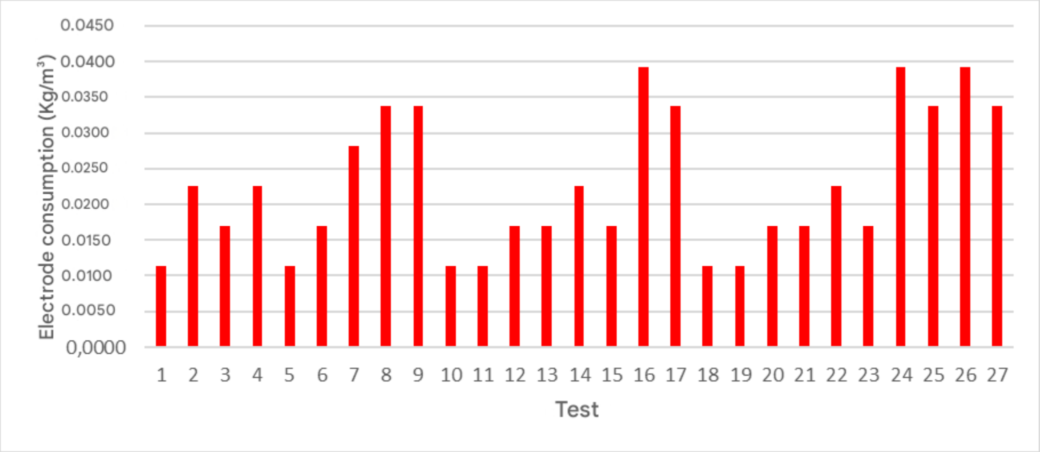
Electrode consumption (kg/m3) was calculated using Equation 2.

Figure 7.- Electrode consumption in tests subjected to 11 volts



In Figure 8, as can be seen, tests 16, 24 and 26 showed the highest electrode wear for those subjected to 5 volts, reaching 0.038 kg/m3 in a 10-minute electrocoagulation process. As expected, the average of the tests subjected to 11 volts achieved a higher average electrode wear due to the application of a higher voltage to the system.

Figure 8.- Electrode consumption in tests subjected to 5 volts



ENERGY CONSUMPTION

In Figure 9, which shows the electrical consumption as a function of the electrode wear at 11 volts, the electricity consumption graph indicates that the test with the highest electrical demand was also test 27, with a value of 5.8 kWh/m3. This correlation makes a lot of sense, since the increased electrode wear was a consequence of a greater amount of energy in the system

Figure 9.- Test power consumption subjected to 11 volts

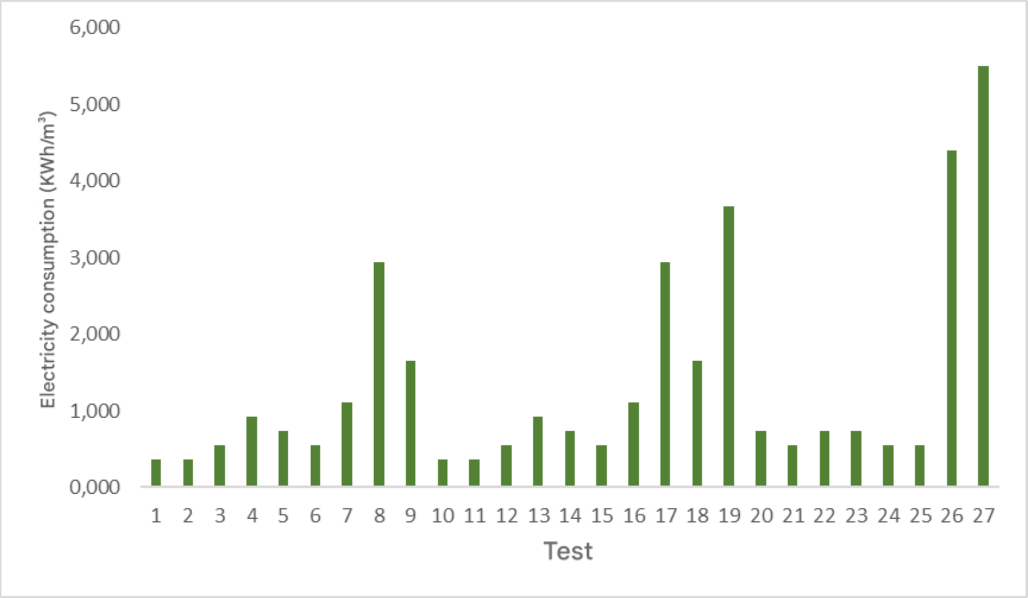
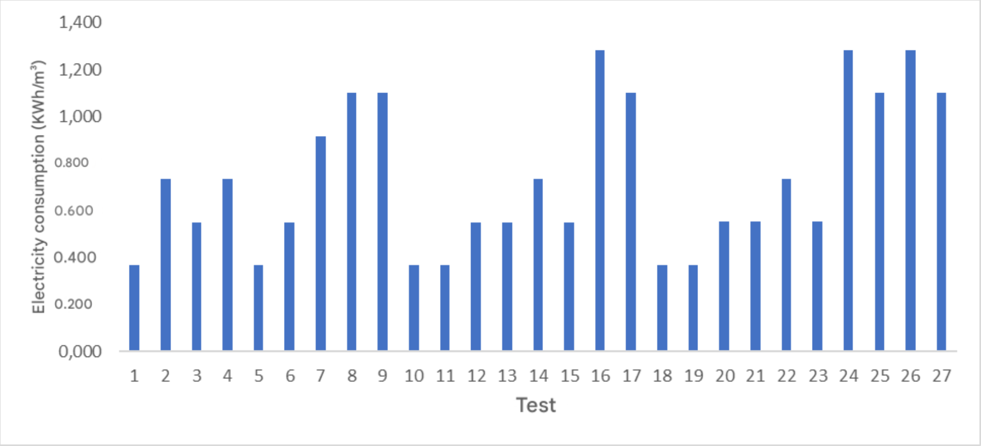


Figure 10 shows the electrical consumption as a function of electrode wear at 5 volts, tests 16, 24 and 26 were the ones with the highest energy consumption, reaching a value of 1.32 kWh/^{m3} and maintaining the correlation with the graph in Figure 9 on electrode wear.

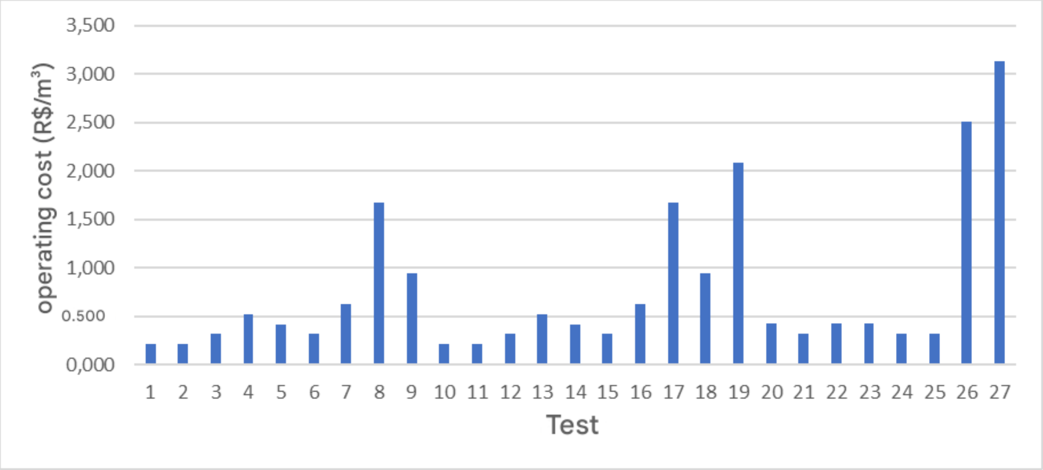
Figure 10.- Test power consumption subjected to 5 volts



COST OF OPERATION

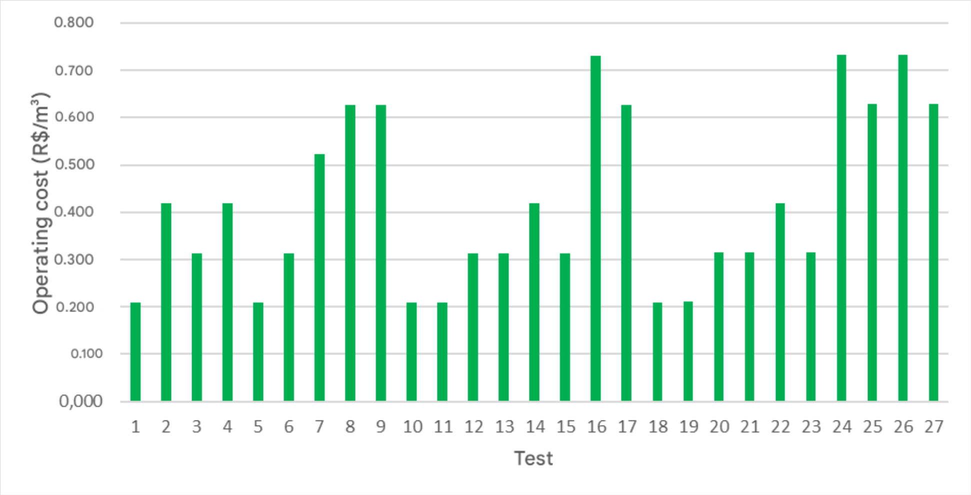
In Figure 11, as can be seen, for the 11 volt voltage, the operating cost was higher for test 27, reaching R$ 3.15/m3

Figure 11. Process operating costs subject to 11 volts



In Figure 12, as can be seen, for the 5 volt voltage, tests 16, 24 and 26 were higher than the others, reaching R$ 0.75/^{m3}. Importantly, test 16 only underwent a 10-minute process, however, it had the highest operating cost of those subjected to 5 volts.

Figure 12. Process operating costs subject to 5 volts



CONCLUSION

After the application of the electrocoagulation method with four aluminum electrodes, it can be concluded that the initial concentration of acetylsalicylic acid in the sample and the process time are important variables that affect the elimination of the emerging contaminant. Thus, the highest overall elimination percentage occurred in trial 27, which reached a removal rate of 60%, which was subjected to an electrocoagulation time of 30 minutes with an initial concentration of 0.3 mg/mL of acetylsalicylic acid. Overall, there were no significant differences between the elimination at the two voltages (5 and 11 volts).

In terms of operating cost, experiments subjected to a voltage of 5 Volts presented the highest cost-benefit of the study, since they had a lower cost to achieve a balanced removal efficiency than those subjected to 11 Volts. Test 27 (11 Volts), in relation to the cost of operation, the test with the highest cost was R$ 3.15/m^3. For tests subjected to 5 Volts, the highest cost was R$ 0.73/m3. It is also important to note that the greatest electrode wear occurred in test 27 (30 minutes at 11 volts), reaching 0.170 kg/m3. In addition to electricity costs, the greater the electrode wear, the higher the aluminum consumption during the process, which is another factor to consider when controlling electrocoagulation costs.

It is recommended for future studies, to add filtration to each test after electrocoagulation, which could increase the efficiency of acetylsalicylic acid removal.

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