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Ciprofloxacin Removing from Aqueous Solutions Using Batch Reactor Electrocoagulation Process with Aluminum Electrodes

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ABSTRACT

Increasing the reliance on pharmaceuticals such as analgesics, antibiotics, antidepressants, and other medications harms the environment and human health. The electrocoagulation process is a modern and crucial technology for treating various pollutants. This paper uses electrocoagulation technology (EC) to remove the most widely used antibiotic, ciprofloxacin (CIP) from an aqueous solution. The proposed approach was experimentally implemented in a batch reactor equipped with (aluminium sheets) that act as electrodes (cathode and anode) arranged vertically in a monopolar parallel mode (MP-P). Different operating parameters were considered, in this work, including inter-electrode distance (IED), pH of the solution, current density (CD), electrolysis time (ET), initial concentration of CIP (Co), and concentration of supporting electrolyte NaCl. Several experiments were performed, and the results revealed that EC has successfully applied with a high removal efficiency of 98.48% under optimum operating conditions: a gap between electrodes = 1 cm, current density = 1.5 mA/cm^2, electrolysis time = 60 min, pH = 5, initial CIP concentration = 50 mg/l, and NaCl = 500 mg/l. The experimental results confirmed that the EC process provides a strategy for removing CIP from wastewater with a high removal efficacy and low energy consumption, additionally offering an increased opportunity for using Al-EC cells to treat antibiotic contaminants.

Keywords: Aluminum electrode, antibiotics, electrocoagulation, monopolar connection

INTRODUCTION

The presence of emerging contaminants such as "Personal Care Products" (PCPs), "Pharmaceutically active Compounds" (PhACs), and others, even if they are present in small amounts, adversely affect the ecosystem (Khan et al., 2020). Ciprofloxacin ($C_{17}H_{18}FN_3O_3$) is an example of an antibiotic frequently used to treat respiratory tract infections, gastrointestinal infections, and urinary tract infections caused by bacteria (Kraemer, Ramachandran, & Perron, 2019). Such antibiotics' occurrence, alteration, and fate may cause serious environmental risks. The traditional methods of wastewater treatment before discharging into rivers include advanced oxidation treatment (Boczkaj & Fernandes, 2017), biological treatment (Huang et al., 2017; Liew, Kassim, Muda, Loh, & Affam, 2015), physicochemical treatment (Bhuptawat, Folkard, & Chaudhari, 2007; Sher, Malik, & Liu, 2013), in addition to developed technologies such as membrane filtration (Dickhout et al., 2017; Teng et al., 2018) and adsorption (Amosa et al., 2016).

Advanced oxidation methods need intense oxidants that make wastewater treatment unsafe and expensive. On the other hand, biological treatment requires highly regulated conditions with longer detention periods, higher footprints, and the production of unwanted byproducts. Chemical treatment needs chemical addition, which raises the expense of the process, as well as, complicates the downstream process, and increases the danger of secondary pollution. Adsorption and membrane filtration can only treat wastewater successfully if they are combined with pre-treatment methods (Tahreen et al., 2020). Of all techniques, EC technique is the most economical for treating wastewater due to their uncomplicated setup, lower footprint, and capability to treat enormous quantities of water without considerable chemical treatment (Sahu et al., 2014).

Other advantages of the EC approach include inexpensive process and maintenance, no release of harmful substances, small amounts of TDS and secondary pollutants, and eliminating the tiniest size of colloidal particles (Ahmadzadeh et al., 2017).

EC technique includes a coagulation process when the sacrificial anode disintegrates, producing in situ coagulant agents (Khandegar & Saroha, 2013). It is a complex process that involves numerous chemical and physical processes that rely on sacrificial electrodes to provide the ions in a water stream. The process involves the following steps: (1) the creation of coagulants through electrolytic oxidation of the consumable electrode, (2) destabilizing of the pollutants, and (3) flocs formation from the aggregation of the destabilized phase (Eyvaz, Gürbulak, Kara, & Yüksel, 2014).

EC process is highly influenced by many operational parameters like current density, pH of the solution, electrolysis time, applied voltage, the gap between the electrodes, etc. EC system utilizes a power source (DC/AC) between metal electrodes immersed in polluted water (Khandegar & Saroha, 2013). The electrical current leads disintegration of metal plates that are usually made of iron or aluminum into wastewater(Barışçı & Turkay, 2016). The metal ions appear at the anode, whereas the hydrogen gas is released from the cathode (Parsa, Panah, & Chianeh, 2016). At a proper pH, the metal ions produce a variety of coagulated kinds and metal hydroxides that destabilized and collect the suspended molecules or adsorb and precipitate dissolved contaminants. The flocculated particles could also be floated out of the water by hydrogen gas (Daneshvar, Khataee, Ghadim, & Rasoulifard, 2007).When a DC electric field is applied, the following reactions are expected in the vicinity of the aluminium electrodes (Eyvaz et al., 2014):

$$\text{Anode:} \quad Al \rightarrow Al^{+3} + 3e^{-} \tag{1}$$

$$2H_2O \rightarrow O_2\,(g) + 4H^{+} + 4e^{-} \tag{2}$$

$$\text{Cathode:} \; 3H_2O + 3e^{-} \rightarrow 3OH^{-} + 3/2H_2\,(g) \tag{3}$$

$$Al^{+3}(g) + 3H_2O \rightarrow Al\,(OH)^{3} + 3H^{+}\,(g) \tag{4}$$

This paper aims to determine the optimum treatment conditions through the study of influencing factors on the removal of ciprofloxacin (CIP) and estimate the energy consumption under monopolar electrical relation with static aluminium electrodes.

MATERIALS AND METHOD

Chemicals

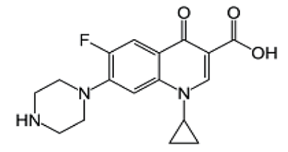
Ciprofloxacin with the molecular formula ($C_{17}H_{18}FN_3O_3$), purity ≥ 98%, solubility in water 30 mg/ml at 20 ^{0}C, molecular weight 331.34 g/mol, and a wavelength of 272 nm was used as a model pollutant in this work. A set of stock solutions containing 750 mg of CIP per 1L is prepared by dissolving the CIP powder in the distilled water. The structure and characteristics of CIP are listed in Table 1. The pH of the solutions is changed by using hydrochloric acid (1M HCl) and sodium hydroxide (1M NaOH). The electrical conductivity of solutions improved by adding a specific amount of NaCl into each 1 L of the sample.

Experimental Setup and procedure

In the proposed approach EC system (Figure1) consists of a DC power supply (PS-305D), an electrochemical reactor made of glass (10

Figure 1. Real experimental system used in the study

Table 1. The characteristics of CIP

Property	Value
Chemical structure	
Solubility	30 mg/ml at 20 °C
Molecular formula	$C_{17}H_{18}FN_3O_3$
Molecular mass (gr/mol)	331.34
Water solubility	30 mg/ml at 20 °C
PKa	5.9, 8.89

mm wall thickness), a magnetic stirrer (Daihanlabtech CO., LTD), and pairs of sacrificial electrodes made of aluminum. The Electrochemical reactor is designed with the dimensions of 20 cm length × 20 cm height × 15 cm width. The reactor has equipped with three pairs of sacrificial electrodes made of aluminum plates. Each plate with a size of 10×10×0.3 cm, immersed vertically in a batch reactor and arranged in a monopolar parallel mode (MP-P) with an effective surface area of 167.8 cm^2, and act as an anode and cathode. The electrodes are connected to a direct power supply DC 0–5 A, and 0–30 V. At the beginning and the end of each run, the electrodes are washed with the water, dipped in 1 M HCl solution for 10 min, rinsed again with the tap water, and then dried. A stock solution of 750 mg/l is prepared by dissolving 0.75 gm of CIP powder in 1000 ml of distilled water. The concentrations of synthetic wastewater 25, 50, 100, and 150 mg/l are prepared by dilution of a suitable amount of stock solution with dribbled water. In all experiments the solution is continuously stirred at 100 rpm via a magnetic stirrer. After each run, the samples are pipetted from the middle of the supernatant portion after a detention period of 60 min and then filtrated using a 0.42 μm pore-sized filter paper (Whatman), and finally analyzed laboratory using a UV-Vis Spectrophotometer (PD-303UV) at a maximum wavelength (λmax =272 nm) of CIP. In all runs, a constant temperature (25 ± 2 C°) was maintained. The efficiency of antibiotic removal is calculated based on Eq. 5 (Mousazadeh et al., 2021):

$$R(\%) = \left[\frac{C_o - C_e}{C_o}\right] \times 100(\%) \qquad (5)$$

where: R – is moval efficiency, C_O – the initial contaminant concentration (mg/l),
C_e – represents the final contaminant concentration (mg/l).

The energy consumption was calculated as follows (Ghosh, Medhi, & Purkait, 2011):

$$E = \frac{I\,V\,t}{Vol} \qquad (6)$$

where: E – the energy consumption in (kWh/m^3),
I – the current (A), V – the voltage (V),
t – denotes the electrolysis time (h),
Vol – solution volume (m^3).

RESULTS AND DISSCUSION

Effect of pH

The pH is one of the important factors that significantly affect the EC process performance, especially the speciation of coagulating agents (Gu et al., 2023). In the proposed approach, a series of experiments were conducted using synthetic wastewater with various initial pH values 4, 5, 7, and 9 to assess the impact of this parameter on the EC process and specify the optimal initial value that attains the best CIP removal rate. The

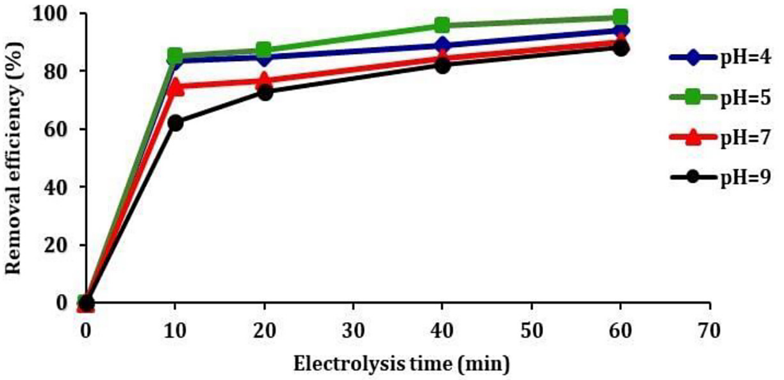


Figure 2. The effect of initial pH on removal efficiency of CIP
(IED = 1 cm, CD= 1.5 mA/cm^2, Co = 50 mg/l, and NaCl = 500 mg/l)

other operating parameters were kept constant. Figure 2 depicts the effect of pH on the removal efficiency of CIP. Figure 2 shows that the preferable pH value for CIP removal falls in the range 5–7, and the maximum removal efficiency occurs in the acidic condition corresponding to a pH value of 5. As can be seen, the removal efficiency increases with pH increasing from 4 to 5 and then decreases as pH increases from 5 to 9. That is because some types of wastewater give the highest EC efficiency in acidic media (Barrera-Díaz, Frontana-Uribe, & Bilyeu, 2014). Conversely, some EC processes are more efficient in relatively more alkaline pH environments (Nawarkar & Salkar, 2019). Based on this fact, EC is an adaptable treatment process that can effectively operate in a wide range of pH (acidic, base, and neutral). This change in the CIP removal rate can also be attributed to the alteration of the amphoteric characteristics of aluminium hydroxide since the pH value governs the nature of the flocs created, which are responsible for pollutant's adsorption.

Furthermore, the prevalent aluminum hydroxide in neutral and little acidic environments is Al(OH)3, characterized by high adsorption ability. Therefore, maintaining the initial pH in the range of 4–8 makes all aluminum cations generated at the anode form indissoluble coagulants, resulting in a more effective treatment (Shafaei, Rezayee, Arami, & Nikazar, 2010). On the other hand, in alkaline media, the prevailing aluminium hydroxide is OH^{-4}, which has limited adsorption capacity as compared with $Al(OH)_3$ (Hashim et al., 2019). Therefore, the removal efficiency lowered as pH increased to 9.

The figure also demonstrates that the CIP removal efficiency increases with the electrolysis time increases. A fast increase in removal efficiency occurred in the first ten minutes; after ten minutes, the removal efficiency increased gradually with time. Based on Faraday's law, increasing the ET leads to an increase in both the concentration of metal ions and hydroxide flocs. After 60 min ET, the removal efficiency of CIP was found to be 98.48% at pH 5. The same results are proved by (Mohammed, M-Ridha, Abed, & Elgharbawy, 2021).

EFFECT OF IED

IED is one of the most important factors for properly functioning electrocoagulation cell. Distance between electrodes represents the gap formed between the anode and cathode. The impact of this parameter on removal efficiency was examined using different IED values 0.5, 1, 1.5, and 2 cm. The other operating conditions were kept constant. Figure 3 depicts the impact of IED on the removal efficiency.

Note that, the removal efficiency increased from 87.49% to 98.48% when the distance between electrodes changed from 0.5 to 1 cm. This result can be explained as follows, the narrow path between electrodes results in a high electrostatic attraction causing the breaking of the metal hydroxides formed by collision with one another (Boinpally et al., 2023). In addition, the time becomes inadequate to produce the flocs (interaction of metal ions with hydroxyl group OH^-. Figure 3 also shows that the removal efficiency increased as electrolysis time increased. Following this, it was found that after an electrolysis time of 60 min, the removal efficiency was found to be 98.48% with a 1cm distance between electrodes.

From the other side, the Figure shows that an increase in the gap length beyond the optimal distance (1 cm) reduces the removal

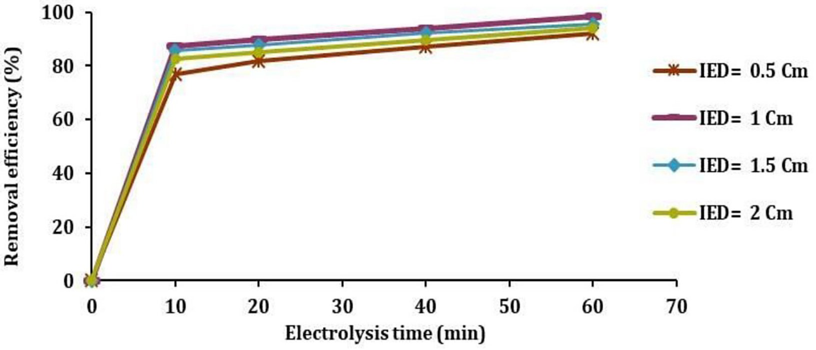


Figure 3. The effect of inter-electrode (IED) distance on removal efficiency (pH = 5, CD = 1.5 mA/cm^2, Co = 50 mg/l, and NaCl = 500 mg/l)

efficiency of CIP since the electrical attraction drops with the increasing of ions travelling path and, consequently, metal ions take a long time to interact with the hydroxyl groups (OH^-) to create the flocs. The obtained results agreed with the other studies (Khandegar & Saroha, 2013).

Effect of current density and electrolysis time

The current density is the most crucial parameter for controlling the reaction rate. This parameter affects the EC process's performance and the electrodes' lifetime. This work tested different current densities of 0.5, 1, 1.5, and 2 mA/cm^2. The other parameters were kept constant. Figure 4 illustrates the variation of CIP removal efficiency with the studied current densities.

As shown in the figure, CIP removal efficiency increased noticeably as the current density increased from 0.5 to 1.5 mA/cm^2. The increase in the removal rate is attributed to the increase in the generation rate of metal ions AL^{+3}. Moreover, as the current density increase, the number of generated Al+ cations increases, too, as well as the formation rate of $Al(OH)_3$ (Aoudj et al., 2010). From the results, it is clear that the increase in removal efficiency occurs within the limited range of current density, reaching the max or optimal rate. It can also be seen that beyond the optimal value of the current density, the removal rate starts to decrease from its best value of 98.48% to 96%. The reason may be that the hydrogen gas rate emitted from the cathode increases as current density increases, in addition to the increasing amount of aluminium hydroxides in the medium and flocking producing rate. As the number of bubbles of hydrogen increases, they stick to the coagulant crystals more, resulting in the floatation of these crystals on the reactor surface more quickly (floatation effect). This impact minimizes the remaining period of flocking matter in the reactor and reduces the potentiality of admixing with pollutants, decreasing the efficiency. The figure also shows that the current density is directly proportional to the electrolysis time since the removal efficiency increased from 78.43 to 98.48% as electrolysis time increased from 10 to 60 min.

Nevertheless, some researchers justified that applying higher currents might increase by-products and additional environmental risks. (Bilińska et al., 2020; Xu et al., 2020). Using too high curent may lead to electrode passivation and accelerate polarization (Fu et al., 2021), increasing power consumption. Therefore, the balance between removal efficiency, electrode consumption, and energy consumption is necessary. As a result, the CD of 1.5 mA/cm^2 was utilized in all experiments. A similar scenario happened in the previous studies. (Holt et al., 2002).

In the EC process, the removal efficiency of the pollutant is highly dependent on the ion concentration of the solution. As seen in the plot, as operating time increases, the degradation rate of CIP increases, too, due to the additional ion concentration in the solutions (Varank et al., 2014). Increasing the electrolysis time not only leads to an increase in the generation rate of aluminium complexes but also raises the hydrogen bubbles production through the electrodissolution of the anode and reduction in the cathode (Alam et al., 2021). According to the results illustrated in Figure 4, the electrolysis time of 60 min represents the optimal time as it gave the maximum removal rate of CIP

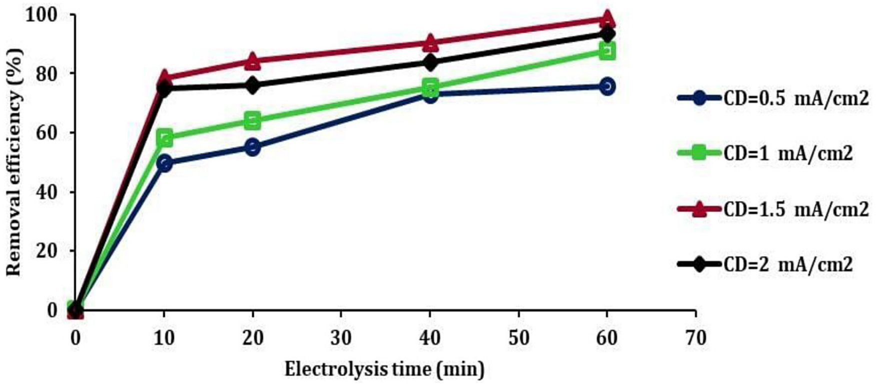


Figure 4. The effect of current density on the efficiency (IED = 1 cm, pH = 5, Co = 50 mg/l, and NaCl = 500 mg/l)

(98.48%). The optimization of electrolysis time is essential to avoid the loss of both energy and resources. Therefore, in this study, considering one hour of reaction time is somewhat enough to remove pollutants. The same results were obtained by (Almukdad et al., 2021).

Effect of intial CIP concentaration (Co)

The EC process highly depends on the solution's initial pollutant concentration. In this work, several experiments were performed with initial concentrations of 25, 50, 100, and 150 mg/l to evaluate the impact of this parameter on the removal efficiency. The remaining operating conditions were kept constant. Figure 5 presents the impact of Co on the removal of CIP.

It can be noticed that the removal efficiency of CIP decreased with the increasing of initial CIP concentration since the same current density is utilized; consequently, the quantity of dissolved aluminium ions at the anode remains the same for all CIP concentrations. Based on this, the metal hydroxide complexes (coagulants) produced at the same CD were insufficient to adsorb all antibiotic molecules in the solution, decreasing the removal efficiency. The decrease in removal efficiency may also be attributable to the lack of active sites in the metal hydroxide flocs, which are responsible for trapping an increasing number of CIP molecules (Oulebsir et al., 2020). The active surface area for these coagulants becomes saturated with the molecules of CIP with no more active surface left to capture more molecules at high initial concentrations (Hashim et al., 2017). After 60 minutes and with an initial concentration of 50 mg/l, the highest removal efficiency was found to be 98.48%.

Effect of NaCl addition

The supporting electrolyte is important in EC operations because it contributes to the intensification of solution conductivity, minimizing the drop in ohmic characteristics and electricity consumption. (Hakizimana et al., 2017). In this work, the electrical energy consumption (EEC) and removal efficiency were investigated using solutions with different values of NaCl concentration ranging from 100 to 600 mg/l. The other operating parameters were kept constant. The effect of NaCl on removal efficiency is shown in Figure 6.

Note that after 60 minutes of electrolysis, the removal rate increased from 61.25% to 98.48% as NaCl concentration increased from 100 to 500 mg/l. This result can be explained as follow: using NaCl to increase the conductivity improves the removal efficiency since the presence of NaCl provides antipassive Cl- ions that can damage the passive oxide layer generated on the surface of the anode, increasing the metal's anodic dissolution rate. In addition, these antipassive ions could remarkably reduce the adverse impact of other anions existing in the solution, such as HCO^{-3} and $SO4^{-2}$. The presence of carbonate anion would lead to the precipitation of Ca^{+2} ion. These ions can form an insolation layer on the cathode surface, increasing the electrochemical cell's ohmic resistance.(El-Ashtoukhy et al., 2016).

As can be seen in the plot, the removal rate decreased to 95% as NaCl became 600 mg/l since the excessive addition of NaCl leads to excessive Cl^{-}, which can accelerate the oxidation capacity of the anode (Serna-Galvis et al., 2017). In addition, excessive Cl^{-} may increase the corrosion pitting rate resulting in excessive consumption of Al electrodes (Silva et al., 2022). Therefore, the

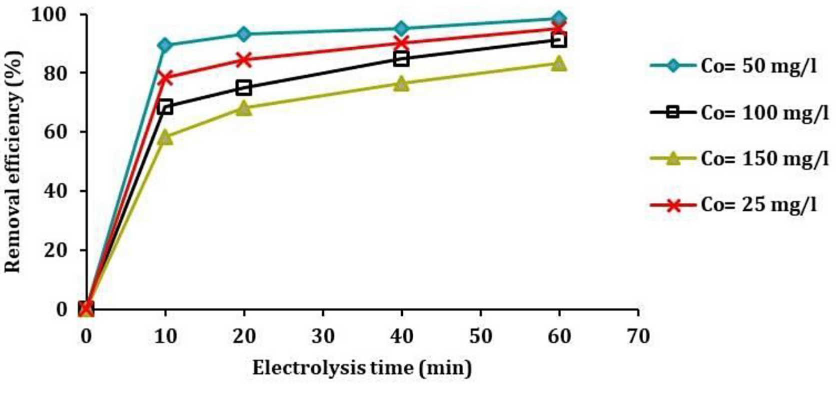


Figure 5. The effect of initial concentration of CIP on removal efficiency (IED = 1 cm, pH = 5, CD= 1.5 mA/cm^2, and NaCl=500 mg/l)

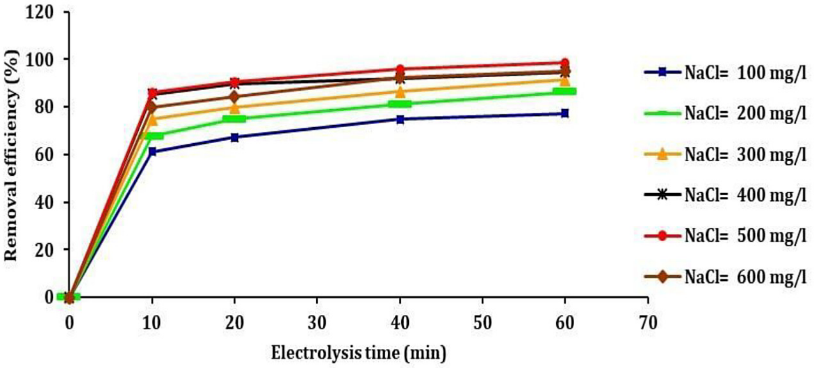


Figure 6. The effect of NaCl on removal efficiency (IED = 1 cm, pH = 5, CD= 1.5 mA/cm^2, and Co = 50 mg/l)

amount of supporting electrolyte to the solution should be controlled, and thus it is limited to 500 mg/l. The same analysis was obtained by (Mohammed et al., 2021).

On the other hand, adding NaCl impacts electrical energy consumption (EEC) at constant current density. The reduction in the EEC can be attributed to the decrease of solution resistance (IR-drop) in the EC cell, which is also related to a rise in the conductivity of the solution at fixed CD (Daneshvar et al., 2010). After 60 min, it is noted that the energy consumption decreased from 0.91335 kWh/m^3 to 0.3322 kWh/m^3 as the concentration the NaCl increased from 100 to 500 mg/l.

CONCLUSIONS

This paper investigates the suitability of employing electrocoagulation technique to treat synthetic wastewater with different concentrations of Ciprofloxacin antibiotic. The proposed approach was experimentally implemented in a batch reactor equipped with three pairs of aluminium electrodes installed vertically in a monopolar parallel mode (MP-P) connected externally to a power supply DC. Different operating parameters are considered in this work, including inter-electrode distance, pH of the solution, current density, electrolysis time, initial concentration of CIP, and concentration of supporting electrolyte NaCl.

The study proved that an increase in the value of electric current density, electrolytic concentration (NaCl), and electrolysis time increases the removal percentage of CIP. While the removal percentage decreased when the initial CIP concentration and pH value increased. It is also observed that the solution conductivity significantly affected the CIP removal efficiency and the electrical energy consumption.

Through several experiments, the results revealed that EC has successfully applied with the high removal efficiency of 98.48% under optimum operating conditions: IED = 1 cm, pH = 5, CD = 1.5 mA/cm^2, ET = 60 min, Co = 50 mg/l, and NaCl = 500 mg/l. The results showed that as the conductivity increased, energy consumption decreased. It was found that energy consumption decreased when NaCl concentration increased.

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