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Ciprofloxacin Removal Using Electrocoagulation Process: Optimization of Operating Parameters Using Response Surface Methodology (RSM)

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Abstract

The electrocoagulation process is an effective technique for treating various types of wastewater. In this work, the treatment of Ciprofloxacin (CIP) wastewater was optimized using Response Surface Methodology (RSM) with central composite design (CCD). RSM-CCD optimized the key factors such as solution pH, current intensity, and electrolysis time to attain maximum removal efficiency of (CIP). The second-order regression model and the empirical data were reasonably fit by ANOVA, which demonstrated a high coefficient of determination value (R^2=0.9594). RSM-CCD estimation demonstrated that a pH of 4, a current intensity of 2 mA/cm^2, and an electrolysis time of 60 min were the best operating parameters that achieved the maximum removal efficiency.
Keywords: Electrocoagulation, Optimization, Aluminum electrodes, Central Composite Design (CCD).

1. Introduction

The existence of emerging pollutants like "Personal Care Products", "Pharmaceutically Active Compounds", artificial sweeteners, and other substances has recently attracted awareness, even if they present in a trace quantity that may adversely impact the ecosystem [1]. Antibiotics, hormones, analgesics, and antidepressants are an example of pharmaceuticals that have become a necessary element of a modern lifestyle.

These contaminants can easily penetrate the water bodies through different sources such as pharmaceutical firms, clinics, medical wastes, and specified usage of different drugs. Pharmaceuticals are characterized by high COD and nitrogenous compounds that necessitate more treatment. Without appropriate treatment, releasing such effluent into an environment can harm flora and fauna [2]. Continuous exposure to such contaminants has the potential to result in both acute and chronic contamination [3, 4], behavioral changes [5], reproductive harm [6], microbiological dangers [7, 8], chemical hazards [9], and agricultural consequences [10, 11], due to its ability to enter the food chain. Ciprofloxacin (CIP) is a synthetic antibiotic that belongs to the second generation of fluoroquinolones (FQs) and is extensively used to treat bacterial diseases and illnesses in animals and human beings.

The scarcity of proper treatment methods in traditional WWTP of medication factories and hospitals and, on the other side, Improper disposal of unused or expired CIP raised the CIP contaminants of surface water in the latest ten years [12]. However, the treatment of antibiotic pollutants is a very difficult task. It encounters many restrictions in finding a proper treatment procedure due to its stubborn behaviors and the cost of treatment associated with its power consumption. Standard wastewater treatment methods include physical, biological, and chemical processes based on different kinds of wastewater. Since antibiotics contain a variety of chemicals and functional groups, which hinder the action of microbes [13, 14], this feature causes them unsuitable for biological treatment. The chemical treatment requires additional chemicals, which increases the operation's cost and the danger of secondary contamination. Therefore, the main treatment approaches for pharmaceutical wastewater rely on physio-chemical treatment.

Electrocoagulation (EC) is the most eco-friend technique for wastewater treatment because of its easy setup, lesser footprint, and the ability to treat large amounts of water without chemicals [15]. EC is an environmentally friendly process since the ‘electron’ is the main reagent and does not need chemical additions. That, in turn, will lower sludge production and eventually eradicate some harmful substances utilized as coagulants in conventional techniques [16]. EC involves coagulation that integrates electrochemical, chemical, and physical mechanisms based on electrochemically disintegrating a consumable anode only when an electric current is applied [17], The main reactions in this procedure are metal disintegration at the anode and the generation of the hydroxyl anion at the cathode [18]. The process occurs in three subsequent steps: (i) coagulant production via the sacrificed electrode's electrolytic oxidation., (ii) destabilization of the impurities, and (iii) flocs formation from an assemblage of the destabilized phases [16].

The electrode used in the process should be nontoxic and sustainable, which is why aluminum iron, stainless steel, mild steel, etc., are used as they are safe, very beneficial, readily available and affordable [19]. Different operative parameters influence the process, such as the density of current, solution pH, operation time, the distance between the electrodes, electrode surface area, agitation speed, and electrode arrangement. EC has successfully eliminated various contaminants from wastewater such as laundry, tannery, refinery wastewater, bacteria, arsenic and fluoride, pesticides, heavy metals and oils, chemical oxygen demand, color and organic substances.

Response surface methodology aims to optimize the response of interest which is influenced by numerous variables. It is a useful statistical method for the optimization of chemical reactions and/or industrial processes and it is widely used for experimental design, in this technique the response surface is optimized that is affected by process parameters [20].

This work employed the EC technique to achieve a higher CIP removal efficiency using response surface methodology RSM in an eco-friendly manner. RSM-CCD was employed to analyze and optimize the impact of the factors as independent variables involving solution pH, current density, and electrolysis time on the dependent variable (removal efficiency).

2. Experimental

2.1 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

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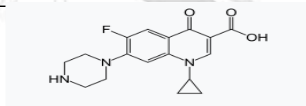
wavelength of 272 nm used as a model pollutant in this work. A set of stock solutions containing 750 mg of CIP per 1L is prepared by dissolving the CIP powder in the distilled water. The structure and characteristics of CIP are listed in Table 1. The pH of the solutions is changed by using hydrochloric 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.

Table 1: The characteristics of CIP.

Chemical name	Ciprofloxacin
Chemical structure	
solubility	30 mg/ml at 20^0 C
Molecular Formula	$C_{17}H_{18}FN_3O_3$
Molecular Mass(gr/mol)	331.34
Water solubility	30 mg/ ml at 20^0 C
PKa	5.9 , 8.89
Therapeutic Class	Fluoroquinolones
wavelength	272 nm

2.2 Instruments

In this work the following instruments were employed:
1. pH meter: pH meter with a range of (0.0 - 14.0), type HANNA (Romania), was used in this work to continuously measure the acidity and alkalinity of wastewater to be treated .
2. Magnetic stirrer: A Magnetic stirrer type (DAIHANLABTECH CO., LTD) providing a range of stirring speeds (100 – 400) rpm was employed to provide proper mixing to create a more homogenous mixture.
3. Electronic balance: An electronic balance type Sartorius (M-power) (Germany), with an accuracy of four digits was used to weigh all chemical materials used in the study.
4. UV Spectrophotometer: A double beam 6800 UV-Visible spectrophotometer type JENWAY, as was used to determine the absorbance of CIP solutions at the predetermined maximum wavelength of 272 nm.
5. Direct power supply (DC): A direct current power supply type (PS-305D), was used in this work to feed the cell with the desired potential (0–5 A) and voltage (0–30 V). Voltage of (5 volt) was used in all experiments.

2.3 Experimental set up

The EC unit (Figure 1) is a glass rectangular reactor consists of six aluminum electrodes placed vertically in a monopolar parallel mode. The electrode lengths are (100 × 100 × 3) mm and placed vertically in the center of the reactor.
The electrodes are connected to a DC power supply style (PS-305D) to deliver the proper current density (0-5A) and voltage of 5 volt, then the electrodes are thoroughly scrubbed with sandpaper prior to each run to control the electrode passivation and then they are washed with an acidic solution and clarified water to eliminate any precipitates on the surfaces after each run. A magnetic stirrer type (DAIHANLABTECH CO., LTD) was utilized to provide a homogenous mixing at a steady rate of 100 rpm.

Figure 1: The experimental system used in the study

2.4 Experimental approach

The pH value of the samplings was changed to the proper value utilizing HCl (1M) and NaOH (1M), and the solution's conductivity was then measured. Sodium chloride (NaCl) is added to the solution to enhance the conductivity and improve the ion transfer in the solution. The current density is changed to the desired value via the power supply. At the end of each EC run, the sample is left to settle for 60 min and then drawn from the middle of the supernatant for the analysis.

2.5 Experimental design

Response surface methodology (RSM) is one of the typical methods utilized to evaluate and examine the optimal conditions. RSM model focuses on the relation between the independent factors, such as solution pH, electrolysis time and density

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of current, and the response, such as CIP removal efficiency. Design of Expert version. 13 is used to analyze the experimental results using the CCD method.
A 20-experiment set based on three independent factors, such as pH (A), electrolysis time (B) and current density (C), with two levels, were codified to be optimized. Table (2) lists the identified factors and their levels.

Table 2: Experiment domain of CCD

Independent variables	Unit	Coded variables	Levels		
			Level -1	0	Level +1
Initial pH	___	A	4	9	11
Electrolysis time	min	B	10	35	60
Current density	mA/cm^2	C	0.5	1.25	2

The quadratic polynomial equation, which is a second-order model, was used to fit the response variable, as shown below [21] :

$$Y = bo + b_1A + b_2B + b_3C + b_{12}AB + b_{13}AC + b_{23}BC + b_1^2A^2 + b_2^2B^2 + b_3^2C^2 \quad (5)$$

Where: Y denotes the responses (removal efficiency); bo indicates the mean value of the practical responses; *bi* describes the main impact of factor i on the response A: initial pH, B: initial electrolysis time, C: current density. The suitability of the model was verified and summarized the experimental results obtained in Table (2).

3. Results

3.1 Effect of process parameters

3.1.1 Effect of initial pH and current density

Figure 2 demonstrates that CIP removal up to 87.14% required an acidic medium with a pH of 4 and the smallest current density of just 0.5 mA/cm^2. Since a medium to acidic pH can more effectively remove CIP by electrocoagulation [22]. In contrast, an increase in pH to 9 and a density of current of 2 mA/cm^2, the removal efficiency becomes around 74.25% only, indicating the significance of pH. However, raising the density of current from 0.5 mA/cm^2 to 2 mA/cm^2 with the same pH of 9 improves the removal of CIP by about 63.8 to 74.25%. The same results with the significant role of pH were obtained by [22]. Different monomeric compounds are produced within a wide range of pH; therefore, aluminum ions (Al^{+3}) produced due to anode disintegration are quickly subjected to spontaneous hydrolysis reactions. At lower pH, the anode dissolution creates Al^{+3} and $Al(OH)_2$ that are directly transformed into $Al(OH)_3$ at a proper pH value. These $Al(OH)_3$ characterized by large surface area, which aids in absorbing metal oxides and organic pollutants effectively [23]. Furthermore, the predominant aluminum hydroxide in neutral and slightly acidic environments is $Al(OH)_3$, this type of

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aluminum hydroxide is characterized by high adsorption capacity. Thus, when the initial pH is maintained 4, all aluminum cations generated at the anode form the insoluble coagulants, resulting in a more effective treatment [24]. On the other hand, in alkaline media, the prevailing aluminum hydroxide is $(OH)^{-4}$, which has limited adsorption capacity as compared with Al(OH)3 [25]. Therefore, the removal efficiency lowered as pH increased to 9.

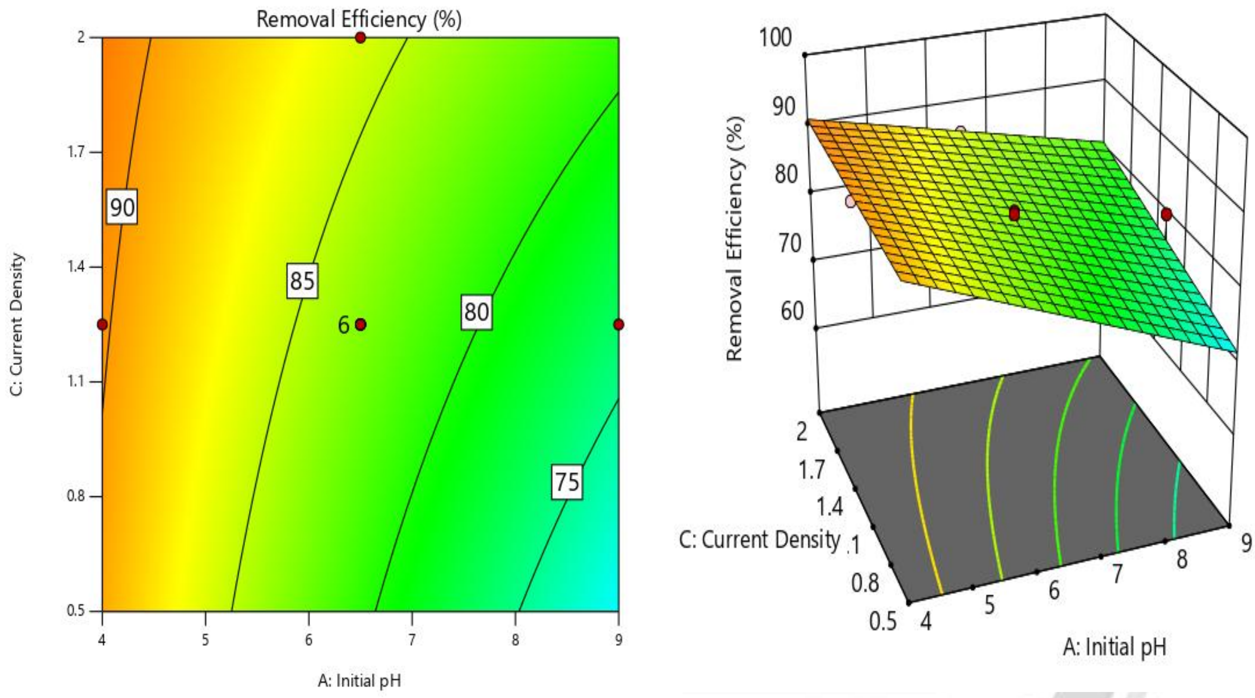

Figure 2: The effect of initial pH and applied current density on CIP removal.

3.1.2 Effect of electrolysis time and pH value

The lost electrode mass naturally increases over time. Thus, Al^{+3} ions released from aluminum electrodes depend largely on the electrolysis time, hence affecting the EC process [26]. CIP removal is increased by increasing the electrolysis time because longer electrolysis times result in more hydroxyl radicals and their flocs, which aid in coagulation and flocculation and then promote CIP removal [27]. The plots depicted in Figure.٣ indicate that maintaining the acidic medium with a pH of 4 and electrolysis time of just 10 min resulted in a removal efficiency of 8٧.14%. On the other hand, increasing the operation time by about 10 to 3٥ min while maintaining the same pH value of 4 gave a sensible increase in removal rate of about 89.14%. However, the impact of electrolysis time increasing from 10 to 3٥ min was relatively influential.

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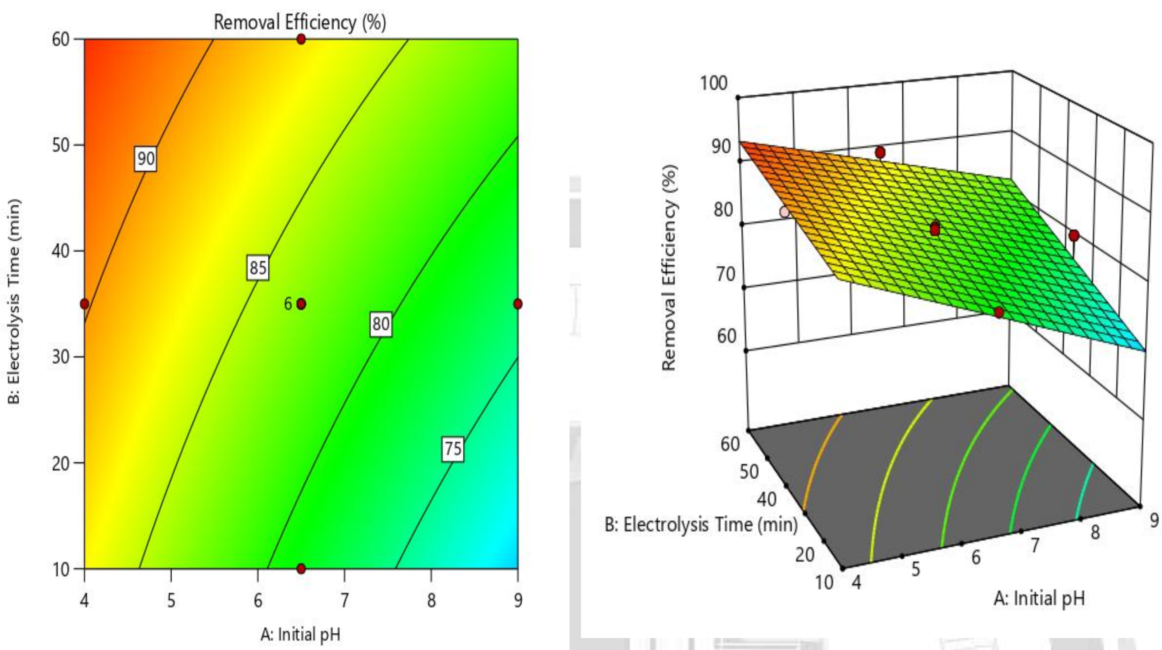

Figure 3: The impact of initial pH and electrolysis time on CIP removal

3.1.3 Effect of current density and electrolysis time

The density of current is one of the most effective parameters in the EC process. Raising the current density induces the increased release of Al ions from the anode and hydrogen bubbles from the cathode, causing an increased number of upward flocs, and improving CIP removal [28]. When the current density becomes 2 mA/cm^2, and electrolysis time of 10 min, the removal efficiency reaches up to 87.14%. However, the best removal rate of up to 94.6% was achieved at an electrolysis time of 60 min and a current density of 2 mA/cm^2. Figure ٤ shows that with an initial pH of 4 and a density of current of 0.5 mA/cm^2, the removal efficiency of CIP becomes 87.1٤% while raising the current density to 2 mA/cm^2 and maintaining the same pH value improved the removal efficiency by 9٤.6%. The current density continuously affects the number of aluminum ions released from the anode resulting in higher CIP removal. [29], improved that increasing the current density increased the CIP reduction.

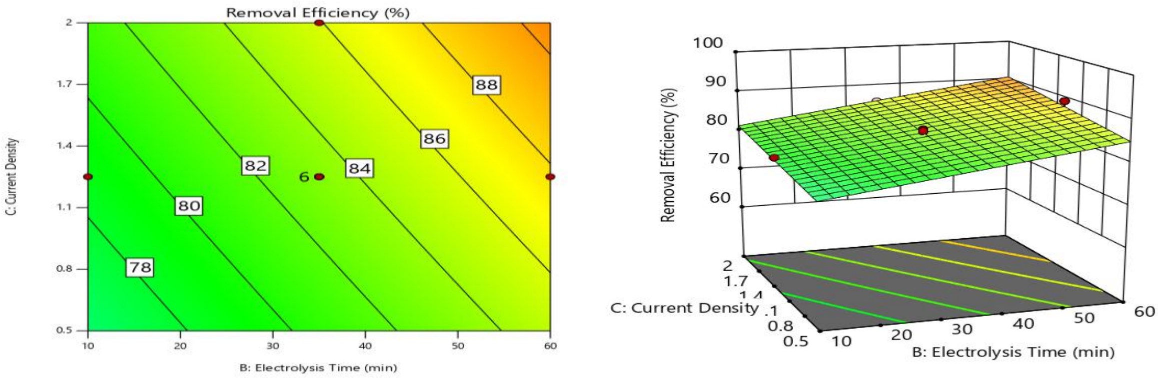

Figure 4: The impact of operation time and applied current density on CIP removal.

Generally, when an electric current is used, the subsequent reactions are anticipated nearby aluminum electrodes [16]:

Anode: $Al \rightarrow Al^{+3} + 3e^-$ (1)

$2H_2O \rightarrow O_2\ (g) + 4H^+ + 4e^-$ (2)

Cathode: $3H_2O + 3e^- \rightarrow 3OH^- + 3/2\ H_2\ (g)$ (3)

$Al^{+3}(g) + 3H_2O \rightarrow Al\ (OH)_3 + 3H^+\ (g)$ (4)

3.2 Statistical design and analysis

Three experimental parameters were selected namely the solution pH initial concentration, current density and operating time. The experimental conditions are summarized along with the results obtained from 20 experiments according to (RSM) in Table (2). The satisfactoriness of the models was also checked by the correlation factor (R^2). The correlation coefficient (R^2) values for the quadratic equations were 0.9594 for CIP. Figure 5 compares expected and experimental response values from the model. The figure shows that the data points are close to the slope line of CIP removal, and the expected values approximated the actual values.

Table 3: Experimental Condition based on CCD matrix and the results of CIP removal.

Run	Initial pH	Electrolysis time min	Current Density mA/cm^2	Removal Efficiency %
1	6.5	35	2	85.7395
2	6.5	35	1.25	83.9395
3	6.5	35	1.25	83.9395
4	6.5	35	1.25	83.656
5	4	60	2	94.637
6	4	35	1.25	89.1421
7	6.5	35	1.25	84.1421
8	6.5	10	1.25	79.3255
9	4	10	0.5	87.1421
10	4	10	2	87.1421
11	9	10	0.5	63.823
12	6.5	35	0.5	79.145
13	6.5	35	1.25	81.145
14	9	35	1.25	79.8964
15	4	60	0.5	91.637
16	6.5	35	1.25	83.4521
17	6.5	60	1.25	89.1421
18	9	60	2	85.0329
19	9	10	2	74.251
20	9	60	0.5	76.632

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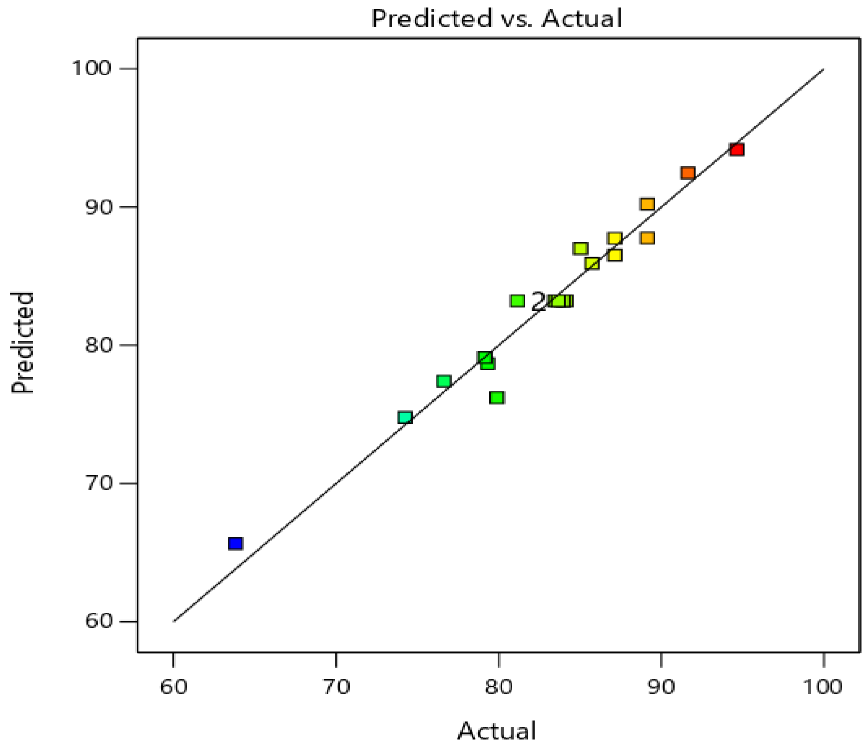

Figure 5: The predicted vs. actual for CIP removal

3.3 Analysis of variance (ANOVA)

(ANOVA) was employed to evaluate the acceptance of the model. Based on F and P values, significant terms for the model were found. The calculated R square indicated a good match for the models of CIP removal efficiency.

Table.3 displays (ANOVA) results that show the influence of independent factors on targeted responses. The quadratic model provides good fits to the data, and the R^2 for CIP removal is reasonably high (0.9594) in addition to high F-values and low p-values (<0.05). There was no statistically significant improper fit for the generated models for any of the responses, as can be seen in Table (3).

Table 4: ANOVA results for CIP removal

Source	Sum of Squares	df	Mean square	F-Value	P-Value	
Model	**826.05**	**6**	**137.68**	**21.25**	**< 0.0001**	**significant**
A-Initial Ph	490.91	1	490.91	182.73	< 0.0001	
B-Electrolysis Time	206.09	1	206.09	76.71	< 0.0001	
C-Current Density	80.79	1	80.79	30.07	0.0001	
AB	16.82	1	16.82	6.26	0.0265	
AC	31.32	1	31.32	11.66	0.0046	
BC	0.1183	1	0.1183	0.044	0.837	
A^2	54.8	1	54.8	19.93	0.0012	
B^2	82.42	1	82.42	29.98	0.0003	
C^2	19.39	1	19.39	7.05	0.0241	

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Residual	**34.92**	**13**	**2.69**			
Lack of Fit	28.64	8	3.58	2.85	0.132	**significant**
Pure Error	6.28	5	1.26			
Cor Total	860.98	**19**				
R^2	**0.9594**					
Adjusted R^2	**0.9407**					

The model's F-value of 21.25 and lower possibility value (P value = 0.0001 < ٠.٠٥) show that it is significant for CIP removal, as shown in the table.
The following second-order polynomial equation represents the last regression model considering coded factors.

$$Y = 83.66 - 7.01A + 4.54\,B + 2.84\,C + 1.45\,AB + 1.9813\,\,AC + 0.1216\,\,BC + 0.4493\,A^2 + 0.1638\,B^2 - 0.4493\,C^2$$

4. Conclusions

This work used the EC procedure to remove CIP from synthetic wastewater. The system is characterized by a quiet and simple procedure and design. Many operating factors were examined: pH, current density and electrolysis time to determine their impact on CIP removal. (CCD) created a quadratic model and experimental runs. The model aims to maximize CIP removal efficiency. An acidic medium with a proper electrolysis time was an optimized and convenient condition for the EC procedure. The highest removal efficiency of 94.6% was accomplished with a current density of 2mA/ cm^2 for an electrolysis time of 60 min at a solution pH of 4. However, it was found that increasing the current density and electrolysis time resulted in higher removal efficiency. In this work, the results indicated that RSM was an appropriate manner to improve the operational conditions. ANOVA proved a high coefficient of determination value (R^2=0.9594), achieving a reasonable fit of the empirical data to the second-order regression model. The best conditions for reaching the 94.6% CIP removal were 2 mA/cm^2 of current density, pH value of 4 and electrolysis time of 60 min.

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مجلة جامعة بابل للعلوم الهندسية مجلة جامعة بابل للعلوم الهندسية مجلة جامعة بابل للعلوم الهندسية

إزالة الملوث (سيبروفلوكساسين) باستخدام عملية التخثير الكهربائي: تحسين عوامل التشغيل باستخدام منهجية استجابة السطح(RSM)

رغـــــد ماجـــــد عبيـــــس اســـــراء سعـــدي عبـــد الاميـــر سماكـــه

قسم الهندسة البيئية/ كلية الهندسة/ جامعة بابل

الخلاصـــة

تعتبر عملية التخثير الكهربي تقنية فعالة لمعالجة أنواع مختلفة من مياه الصرف الصحي. في هذا العمل، تم تحسين معالجة مياه الصرف الصحي التي تحتوي على السيبروفلوكساسين (CIP) باستخدام منهجية استجابة السطح (RSM) مع التصميم المركب المركزي (CCD). يقوم RSM-CCD بتحسين العوامل الرئيسية مثل الرقم الهيدروجيني للمحلول , كثافة التيار , ووقت التحليل الكهربائي لتحقيق أقصى كفاءة إزالة لـــ (CIP). اظهرت نتائج ANOVA ان البيانات التجريبية مناسبة بشكل معقول لنموذج الانحدار من الدرجة الثانية من خلال معامل التحديد عالي القيمة (R^2 = 0.9594). أظهر تقدير RSM-CCD أن الرقم الهيدروجيني ٤، وكثافة التيار ٢ ملي أمبير/سم^{٢}، ووقت التحليل الكهربائي ٦٠ دقيقة كانت أفضل عوامل للتشغيل و التي حققت أقصى كفاءة للإزالة .

الكلمات الدالة: التخثير الكهربائي, ايجاد افضل القيم لعوامل التشغيل, اقطاب الالمنيوم, التصميم المركب المركزي (CCD) .