

Evaluation of electro-fenton process for removal of amoxicillin from simulated wastewater

Ghazi Faisal Naser*, Ihsan H. Dakhil and Ali A. Hasan

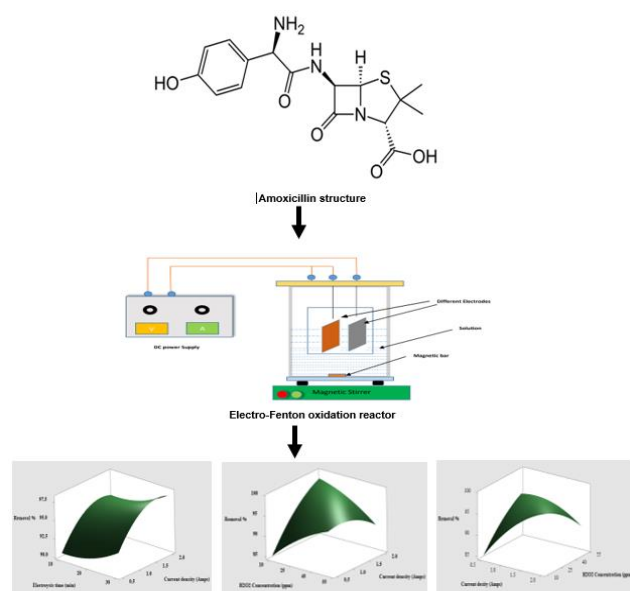
Chem. Eng. Dep., Faculty of Engineering, Al Muthanna University–Iraq

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*to whom all correspondence should be addressed: e-mail: ghazi_faisal@mu.edu.iq

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Graphical abstract



Abstract

The purpose of this work is to examine the efficiency of the Electro-Fenton process (Fenton oxidation) for the elimination of amoxicillin from simulated wastewater through response surface methodology (RSM). The effect three operating parameters that were selected for this evaluation were electrolysis time, hydrogen peroxide concentration, and the current density. The Electro-Fenton method was assessed by a set of experiments that were designed using the Box-Behnken model. The results conclude optimum parameters conditions, at 30 min electrolysis time, 30 ppm H₂O₂, and current density 2 amps by fixing the other two parameters; 20 ppm concentration of amoxicillin and pH of 3. The maximum removal efficiency at these conditions was 98.11%. The mathematical model concluded has a high correlation coefficient ($R^2 = 98.3$). Depending on the results, the Electro-Fenton process has proven an excellent method for the elimination of antibiotics from wastewater.

Keywords: Antibiotics; wastewater treatment; electro-fenton process; rsm; optimization; isotherms

1. Introduction

Nowadays, therapeutic compounds remain usually rummage-sale toward luxury numerous illnesses in persons, animals, in addition, other organisms. These compounds of medical have remained documented by way of developing contaminants in the water aimed at the previous three decades, are extensively rummage-sale in medicine, medicine of veterinary, and feed of animal using development organizers to 15% of all medicines rummage-sale universal are devoted to antibiotic (Hasani *et al.* 2020, Al-zobai *et al.* 2020). Amongst the different medical crops, antibiotics, once current in wastewaters has involved additional care because of eco-toxicological belongings and the potential toward allowing the resilient pathogenic microorganisms cohort, via a hereditary assortment of species, foremost toward a main public health subject (Carvalho and Moraes 2021). The manufacture of mass, misappropriation, and antibiotics remainder produced a negative result on the environment. In addition, numerous antibiotics are defecated through faunae and human finished feces and urine by way of the parental compounds might remain complete or incomplete metabolites and alteration products. The release of antibiotics leads toward a possible danger to the environment even at low concentrations and it has remained noticed in the water environment for example rivers, ponds, and wastewater (Anjali and Shanthakumar 2019; Dakhil 2013). Educations have long-established the incidence of pharmaceutical crops in mud, earth, residues, surface water, and terrestrial animals and floras. the accumulation of antibiotics has developed the main reason for anxiety toward scientists and ecologists (Dutta. and Mala 2020; Taher *et al.* 2009; Dakhil and Ali 2021). The aquatic environment squalor caused through these contaminants necessity remain prevented and regulator remains chiefly challenging owing to the extensive dispersal of their release sources ranging from national or manufacturing waste to landfills (Jafer and Hassan 2019). Actual regularly, because of the demands of their project, they are comparatively non-recyclable complexes. Therefore, the conservative action of lively sludge, extensively rummage-sale in treatment of wastewater, is

inadequately aimed at the elimination of these compounds (Cuerda-Correa *et al.* 2020; Dakhil 2020; Ali and Dakhil 2012). There are numerous approaches obtainable aimed at removing antibiotic compounds, including adsorption, membrane, and bio methods. Though, pollutants are not detached through such approaches; rather, they are impartially transferred from one phase to another (Jafer *et al.* 2019). Advanced oxidation processes (AOPs) for example photocatalytic and photo Fenton oxidation are valuable. In such approaches, free radicals are rummage-sale aimed at the oxidation of resistant substances, and it converts them into the inoffensive crops water and carbon dioxide (Olama *et al.* 2018; Hassan and Al-zobai 2019; Sultan *et al.* 2020). AOPs lead to the squalor and oxidation of amoxicillin. Fenton process remains unique of the extremely practical action technique because of high competence, ease of skill, and unimportant poisonousness of reactants (Naeem *et al.* 2018; Hassan *et al.* 2018; Naser *et al.* 2021). In the Fenton process, H₂O₂ and ferrous sulfate are careful by way of reductants and oxidants, correspondingly. eq (1) mostly happened in this method (Dehghani *et al.* 2016):



The Fenton oxidation scheme, an environment friendship and capability method remained rummage-sale toward making the oxidized pollutant in wastewater finished adding the ferrous sulfate and hydrogen peroxide (Alakoul *et al.* 2021). Electro-Fenton oxidation must rose ended the preceding period by way of the unique attractive process aimed at the action of a used variety of pollutants that do not essential the outline of material owing to its cohort after redox responses (Hammouda *et al.* 2019). This study utilizes electrochemical oxidation (electro-fenton process) for removing the organic material (Amoxicillin) from simulated wastewater and the best value was found from different variables such as H₂O₂, current, and electrolysis time. The amoxicillin chemical structure is presented in Figure 1.

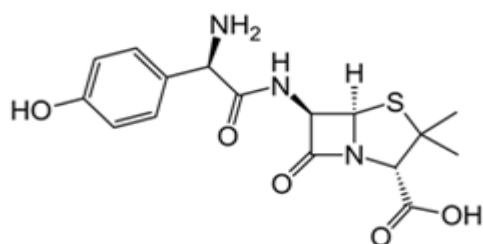


Figure 1. Amoxicillin structure

2. Materials and methods

2.1. Chemical and analytical analysis

H₂O₂ (45% wt/wt Germany), NaCl (purity 99 %), hydrochloric acid (purity 98 %), and NaOH (Thomas baker), were used in this study. At the end of each experiment, the change of the amoxicillin concentration in simulated wastewater by the electro-Fenton oxidation treatment was measured using a UV spectrophotometer at 254 nm.

The amoxicillin removal efficiency can be calculated through the following equation (2):

$$R \% = \frac{A_o - A_t}{A_o} \times 100 \quad (2)$$

where R acts amoxicillin removal efficiency; A_o and A_t acts measured concentrations of amoxicillin before and after the treatment in the unit of (ppm), respectively.

2.2. Electrodes

The electrodes of the electro-oxidation reactor were graphite and iron as anode and cathode correspondingly. Dimensions of graphite and iron electrodes were (9cm * 5cm * 0.14cm) and (14cm * 8cm * 0.3cm), respectively. The active area was kept 30 cm² and the inside space of the electrode was supported by way of 6 cm the design of the electro-Fenton oxidation reactor is shown in Figure 2.

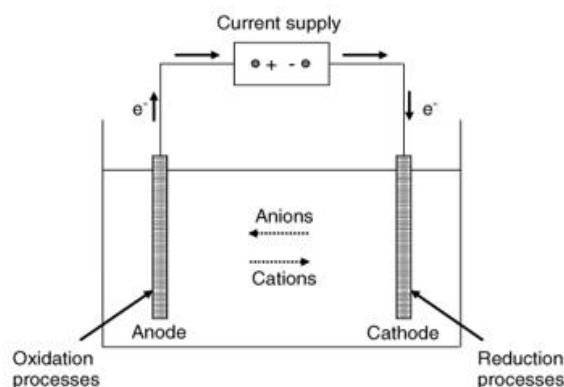


Figure 2. Electro- Fenton oxidation reactor

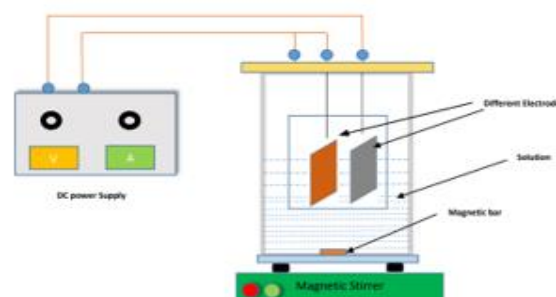


Figure 3. Electro-Fenton oxidation reactor

2.3. Electro-Fenton oxidation treatment

It is obvious from the Electro-Fenton oxidation procedure that 400 ml of simulated wastewater was loaded to the batch reactor (800 ml in volume) with constant concentration at 20 ppm amoxicillin. The simulated wastewater was prepared by dissolving amoxicillin (C₁₆H₁₉N₃O₅S.3H₂O) in distilled water and pH was adjusted to 3 through 1N HCL and NaOH. DC power source (RXN-305D) was used for electrodes and conserved by way of 29.6 V, and 0.25 gm of NaCl was used for every run. The electro batch reactor was used to spread the metal ions and then add various amounts of oxide agent at 200 rpm to complete the experiments as shown in Figure 3. After each run, the sample was withdrawn and put in a centrifuge at 2000 rpm for five minutes.

Table 1. Operating parameters limits

Operating parameter	Unit	Minimum value	Maximum value
Time of electrolysis	min	10	30
H ₂ O ₂ concentration	ppm	10	50
Current density	amps	0.5	2

Table 2. Results of experimental of Box-Behnken model to remove Amoxicillin

Run	X ₁ Electrolysis time (min)	X ₂ H ₂ O ₂ concentration (ppm)	X ₃ Current density (Amps)	Amoxicillin removal (%)	Final pH	E (kW/m ³)
1	15	20	0.88	91.11	3.51	12.81
2	25	20	0.88	92.52	4	21.36
3	15	40	0.88	93.66	4.2	12.81
4	25	40	0.88	95.12	3.26	21.36
5	15	20	1.63	96.55	3.27	23.81
6	25	20	1.63	96.58	3.57	39.67
7	15	40	1.63	93.82	3.42	23.80
8	25	30	1.63	95.88	3.25	39.67
9	10	30	1.25	95.11	3.76	12.20
10	30	10	1.25	95.58	4.1	36.62
11	20	50	1.25	95.22	3.27	24.42
12	20	30	1.25	92.93	3.51	24.41
13	20	30	0.5	91.66	3.2	9.77
14	20	30	2	98.11	3.41	39.06
15	20	30	1.25	96.05	3.48	24.41
16	20	30	1.25	96.45	3.51	24.41
17	20	30	1.25	95.89	3.44	24.35
18	20	30	1.25	95.99	3.35	24.41
19	20	30	1.25	96.00	3.25	24.37
20	20	30	1.25	95.95	3.28	24.35

Energy consumption remains a real significant feature in such type of process methods, so, it is essentially using the following equation (3) (Feng *et al.* 2021)

$$E = (U.I.t) / (1000 V) \quad (3)$$

where U represents the voltage (volt), I: current density (amps.), t: electrolysis time (h), and V: reactor volume (m³).

2.4. Experimental design

In this research, optimization of new variables for amoxicillin wastewater treatment through electro-Fenton oxidation is obtainable through the response surface method (RSM). The Minitab-19 was utilized for experimental design, data processing, and graphical charting (Dakhil *et al.* 2021). The parameters of electro-Fenton oxidation were; X1 acts time of electrolysis, which is ranged from 10 min to 30 min, X2 acts hydrogen peroxide concentration, which is ranged from 10 ppm to 50 ppm and X3 represents current density also ranged between 0.5 and 2 amps as shown in Table 1.

3. Results and discussions

3.1. Regression models

Relationships between responses and variables were obtained in this work using a 2nd-order model and a least-squares technique (Hassan A.A. *et al.* 2020; Rashid A. H. *et al.* 2020; Dakhil I.H. *et al.* 2021):

$$\begin{aligned} \text{Amoxicillin Removal \%} = & 73.72 - 0.233 X_1 + 0.513 X_2 \\ & + 20.33 X_3 + 0.0075 X_1^2 - 0.00275 X_2^2 \\ & - 3.41 X_3^2 + 0.00199 X_1 * X_2 - 0.003 X_1 * X_3 - 0.289 X_2 * X_3 \end{aligned} \quad (4)$$

Where X1 is electrolysis time, X2 is hydrogen peroxide concentration and X3 is the current density. The correlation coefficient of this mathematical model (R² = 98.3). Table 2 demonstrates the values of operating variables and responses, amoxicillin removal, the final value of pH, and energy consumption (E).

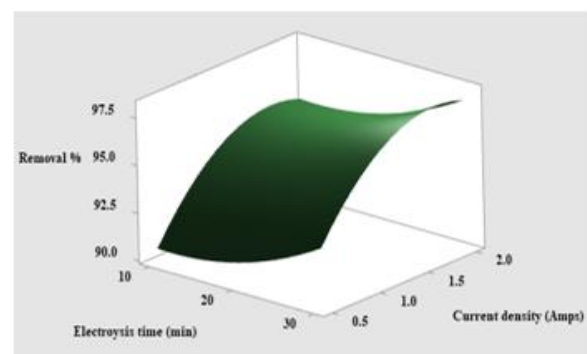


Figure 4. The effect of electrolysis time on amoxicillin removal efficiency

3.2. Electrolysis time effect

Experiments were conducted with operating variables that were selected with fixing two parameters; 20 mg/L of amoxicillin concentration and 3 pH to evaluate the effect of electrolysis time in electro-Fenton treatment. Figure 4

shows the surface plot of electrolysis time with current density concerning the removal efficiency of amoxicillin. The high elimination was attained 95.8%, as shown in this figure. The removal percentage of organic components from simulated wastewater increases as the electrolysis time increases. This result is in agreement with the finding of (Alturki *et al.* 2021). Also, longer oxidation periods make gradual detoxification possible without creating carbon dioxide and inorganic materials (Yong *et al.* 2017).

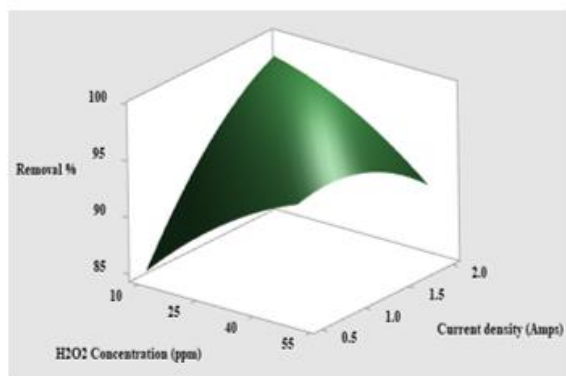


Figure 5. The effect of hydrogen peroxide on amoxicillin removal efficiency

3.3. Hydrogen peroxide concentration effect

Figure 5 shows the surface plotting of hydrogen peroxide and current density concerning removal efficiency. It is noticed that the elimination of amoxicillin augmented with the increase of the concentration of H_2O_2 in the ranges 10-28 mg/L and reached the complete elimination of 95.4% at 28 mg/L of hydrogen peroxide, 30 min of electrolysis time at room temperature. On the other hand, the extreme adding of H_2O_2 overhead 28 ppm had led to a negative influence on the elimination of organic obtainable in simulated wastewater. The elimination competence had reduced to 94.8% at 50 ppm of hydrogen peroxide for the reason that the continuous adding of hydrogen peroxide will react with rather than the organic contaminants, i.e. they are creating which remain not reactive like, which cause minimalize the presentation of the processing procedure. Furthermore, if hydrogen peroxide concentration was little, the creation of $\cdot OH$ will remain fewer, formerly the treatment will be incompetent. The quantity of free radicals formed in the Electro-Fenton process stays conventionally related with hydrogen peroxide, the concentration of H_2O_2 is an important factor that effectively belongs to the kinetic parameters. The same results were stated by Youssef *et al.* (2016) and Atiyah *et al.* (2020).

3.4. Current density effect

Figure 6 demonstrates the current density and hydrogen peroxide concentration concerning removal efficiency. The results show that the amoxicillin recovery increased with increasing the current density until the optimum values were attained about 98%, 92.55%, and 91.66% at the varied current of 2 amp, 0.88, and 0.5 amps, correspondingly. When the current rises, the functional current proportionately increases. Consequently, the

generation rate of H_2O_2 remains augmented, so the rate of free radical generation increases following the better elimination of amoxicillin at 2 amps compared with 0.88 and 0.5 amps. Additional current increases to 2 amps increased the elimination of organics to more than 98 percent due to undesired side reactions: (i) Four electrons of O_2 transfer to H_2O molecules instead of hydrogen peroxide (ii) H_2 released at the cathode, (iii) hydrogen peroxide was oxidized at anode (Divyapriya *et al.* 2018). Similar observations were found complete via Ahmed, Hassan (Ahmed *et al.* 2021).

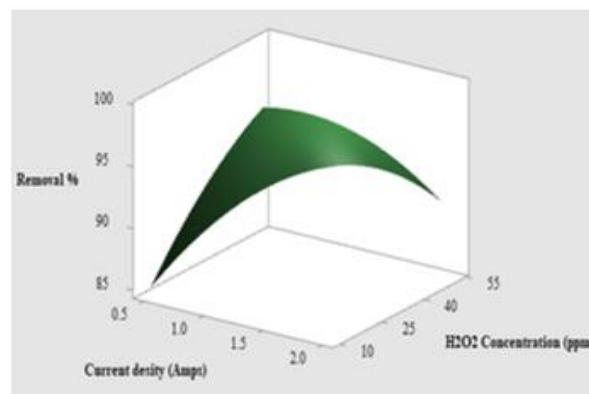


Figure 6. The current density effect on Amoxicillin removal

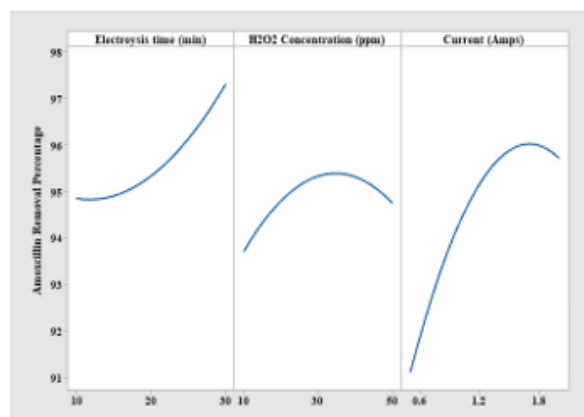


Figure 7. The optimum levels of the operating conditions

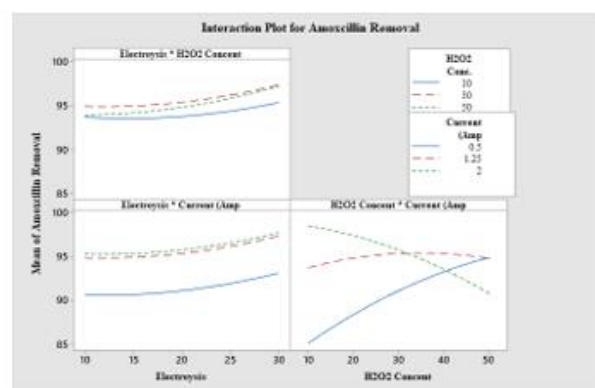


Figure 8. Interacting between the operating variables

3.5. Optimization and interacting of operating variables

The finest values of operational parameters for electro-Fenton oxidation were electrolysis time, hydrogen peroxide, and current density. The results of the D-

optimization analysis are shown in Figure 7 was found that the optimum value for removing organic material is over 98 percent. Figure 8 was represented the interaction among three operating parameters. From this figure, it is noticed that there was no interaction between electrolysis time versus H_2O_2 concentration and electrolysis time versus current density. On the other hand, there was an interaction between the concentration of H_2O_2 and current density at 30 and 40 ppm.

3.6. Energy consumption Estimation

The consumption of energy represents an important path of evaluating wastewater treatment by electro-Fenton oxidation technology, especially related to organic contaminant removal (Li *et al.* 2017). Figure 9 depicts the energy use results in the electrolysis period and current for amoxicillin removal through the electro-Fenton oxidation technique in wastewater. The values are related to the current and periodic time via electro-oxidation action, which is empirical and directed at all types of water (Da Silva A.J.C. *et al.* 2013).

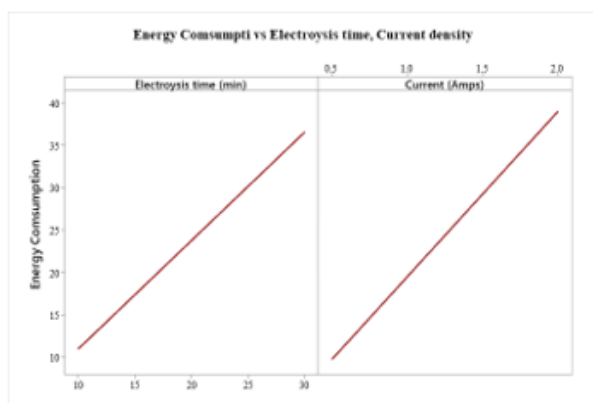


Figure 9. The result of the employed variables

The energy values and electrodes demand was nearly $38.67 \text{ kW}\cdot\text{m}^{-3}$ according to the best values of electrolysis (time and current) mentioned previously. Figure 8 exhibits the energy demand by way of example, the increase in energy consumption with current is more evident in comparison to the increase in electrolysis time, implying that the current is more important in the evaluation of energy consumption, as is well-known in the literature (Ganiyu S. O. *et al.* 2018).

4. Conclusion

The ability of the electro-Fenton oxidation process for the removal of organic pharmaceutical material from simulated wastewater has proven an efficient method by using graphite and iron electrodes. The mathematical relationship between operating parameters and responses was obtained in this work using a 2nd-order model and least-squares technique relations which creates high regression constants for all the considerate responses. The maximum removal efficiency and energy consumption at the optimum operating variables were 98.11% and $38.67 \text{ kW}\cdot\text{m}^{-3}$ respectively. In general, the Electro-Fenton process show clearly a clean and well-organized technique for removing amoxicillin from wastewater.

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