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RESEARCH NOTE

Ibuprofen Removal from a Pharmaceutical Wastewater using Electro-Fenton Process: An Efficient Technique

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ABSTRACT

The aim of this research is to evaluate the effective parameters such as pH, current density (mA/cm^2), H_2O_2/Fe^{2+} molar ratio, volume ratio of H_2O_2 to pharmaceutical wastewater (PhW) (ml/l) and reaction time (min) on the electro-Fenton process for the ibuprofen (as a pharmaceutical waste in water) removal. Since a synthetic wastewater with the same concentration of ibuprofen in a real pharmaceutical wastewater (400 ppm) was chosen in this research, the sample was tested in terms of the chemical oxygen demand (COD). The parameters were statistically optimized under response surface mythology (RSM). The software was also applied to minimize the number of runs. The optimum conditions for 98.29% COD removal experimentally were at pH of 2.43, current density of 23.08 mA/cm^2, H_2O_2/Fe^{2+} molar ratio of 2.69, volume ratio of H_2O_2/PhW of 1.84 ml/l and reaction time of 28.08 min.

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1. INTRODUCTION

One of the drugs that are widely used for muscle pain and inflammatory disorders is ibuprofen. Ibuprofen (2-[4-(2-methylpropyl) phenyl] propanoic acid) structural formula is shown in Figure 1 [1].

Pharmaceutical factories have a wastewater containing large amounts of medicines. These materials can be dangerous for human and animal health [2]. So, treatment process on this type of wastewater would be necessary. There are several methods such as biological, physical, chemical and their combination for the pharmaceutical wastewater treatment. Furthermore, there are other treatment techniques such as treatment by O_3 and O_3/H_2O_2 [3], activate sludge [4], up-flow aerobic stage reactor [5], submerged membrane bioreactors [6], RO/NF membrane [7], membrane and activated carbon [8]. Advanced oxidation processes are also suggested for the organic pollutants treatment in aqueous media [9]. In this area, the most common method was the electro-Fenton process [10] due to some dramatic benefits such as short reaction time and large amount of pollutant removal [11, 12]. Hydroxyl radical obtained from Fe^{2+} ions and hydrogen peroxide reaction decomposes the organic compounds [13, 14]. The cyclohexadienyl radicals readily react with dissolved O_2 (peroxy radicals formation). The peroxy radicals may eliminate HO_2•. They may also undergo ring-opening reaction. Therefore, the linear dicarboxylic acid molecules can be formed. The ring-opening reaction is an important step of mineralization. In fact, the radical scavenging reaction opens a new reaction channel for degradation. Furthermore, the intermediates cannot be transformed back to the starting molecules [15]. The product of this process is shown in Figure 2 [1].

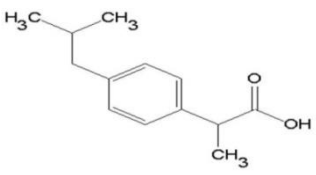

Figure 1. Ibuprofen structural formula [1]

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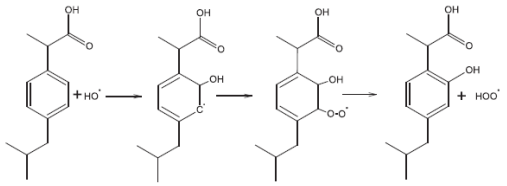


Figure 2. Ibuprofen degradation by hydroxyl radical (during electro-Fenton process) [1]

In this paper, electro-Fenton process was applied to decompose ibuprofen as a sample of pharmaceutical waste in the water. Effect of five parameters on this process were considered together and optimized.

2. MATERIALS AND METHODS

2. 1. Wastewater Source and Characteristics According to a report obtained from Parand Daru Co. (Saveh, Iran), ibuprofen concentration in wastewater was around 400 ppm. However, ibuprofen solubility in water is around 21 ppm at 25 °C [15], but, according to a mass balance reported by the company there is 400 ppm ibuprofen in the company wastewater which a part of it is in the solid phase as sediment [16]. Therefore, an aqueous solution with the same concentration was prepared (ibuprofen 400 mg tablet was purchased from Parand Daru Co. and dissolved in distilled water) and homogenized by a magnetic stirrer (400 rpm) during the electro-Fenton process. Since coagulation, sedimentation and mineralization assist this process [9], immiscible ibuprofen will be added to the sludge phase during the process. Moreover, for preventing any error regarding various pharmaceuticals conflicting on each other, a synthetic wastewater containing ibuprofen (as a pharmaceutical sample) was only prepared. The product was saved in a plastic vessel and stored in a cold room at 4°C. The characteristics of this wastewater are reported in Table 1.

2. 2. Experimental Set-up A glass beaker with 400 cm^3 capacity (as reactor) equipped with a magnetic stirrer was used. The parallel electrodes were made of iron (with a gap of 3 cm. Each electrode had an area of 2×0.5 cm^2 with effective surface area of 1 cm^2). In each run, 250 cm^3 of PhW was taken and poured in the reactor; pH of each run was set by H_2SO_4 (0.1 N) and NaOH (0.1 N) and controlled by a pH-meter (METTLER-TOLEDO 320). The electrods were connected to a DC power supply (fabricated by Kala Gostaran-e-Farada supplier, 30 V and 3 A). For each experiment, a spcifed amount of iron salt ($FeSO_4.7H_2O$, Merck grade) was added to the reactor. The stirrer speed was fixed at 400 rpm (without any vortex observation) and at ambient temperature (25 °C). H_2O_2 (purchased from Merck with purity of 30%) as a reaction accelerator was added to the reactor and power current was switched on. At the end of each run, power source was turned off and the sampel was unmoved around 15 min (for settling solid particles). Then, the sample was filtered by filter paper and placed in a UV-Vis spectrophotometer (HACH, US) with wavelength of 300 nm which carefully measured and validated [17] for ibuprofen. Moreover, initial sampel of PhW was taken and diluted in five different volumes. Absorption mounts for these concentrations were obtained (at 300 nm) for the calibration chart. After each run, the electrods were washed with distilled water to remove any gross solids.

TABLE 1. Characteristics of the used wastewater

Parameter	Unit	Value
Chemical oxygen demand (COD)	mg/l	685
Total dissolved solid (TDS)	mg/l	288
pH	-	4.5

2. 3. Experiments Design The response surface methodology (RSM) was used to minimize the number of experiments and optimize the operating conditions [18]. The Design-Expert software (version: 7.0.0) was used for this purpose [19]. Box-Behnken design (BBD) which is under RSM was applied in this research [20]. Forty six experiments were designed and the goal was to maximize the ibuprofen removal. The independent variables were pH (X_1), current density (X_2), H_2O_2/Fe^{2+} molar ratio (X_3), volume ratio of H_2O_2/PhW (X_4) and reaction time (X_5). COD removal was chosen as a dependent variable. All of the variables were coded and they were illustrated as low level number of -1, medium level number of 0 and high level number of 1 in Table 2.

This work was performed by the following equation:

$$X_i = (x_i - x_0)/\Delta x \quad (1)$$

Removal percentage of PhW (ibuprofen or COD) was calculated by the following equation:

$$\text{Removal (\%)} = (C_i - C_0)/C_i \quad (2)$$

TABLE 2. Independent variables and their levels obtained from the BBD

Symbol	Factor	Coded levels of variables		
		-1	0	1
X_1	pH	2	2.5	5
X_2	Current density (mA/cm^2)	20	50	80
X_3	Molar ratio H_2O_2/Fe^{2+}	0.5	2.75	5
X_4	H_2O_2/PhW (ml/l)	0.3	1.4	2.5
X_5	Reaction time (min)	10	55	100

where, C_i and C_0 are initial and final ibuprofen concentration, respectively. Table 3 shows the matrix design obtained from the software and responses obtained from the experiments (Actual) and software (Predicted).

TABLE 3. Experimental matrix design

Run	pH	Current density (mA/cm^2)	Molar ratio (H_2O_2/Fe^{2+})	H_2O_2/PhW (ml/l)	Reaction time (min)	COD removal (%)	
						Actual	Predicted
1	3.5	20	2.75	1.4	100	92.431	94.576
2	3.5	50	2.75	2.5	10	98.642	103.342
3	3.5	80	5	1.4	55	91.234	89.263
4	3.5	80	0.5	1.4	55	91.238	89.585
5	2	50	2.75	2.5	55	88.268	77.484
6	5	50	0.5	1.4	55	91.207	87.297
7	5	20	2.75	1.4	55	91.121	86.227
8	5	50	2.75	1.4	100	91.241	89.410
9	3.5	20	5	1.4	55	91.211	90.021
10	3.5	50	2.75	0.3	10	15.988	20.879
11	2	50	2.75	1.4	10	91.237	94.129
12	3.5	50	2.75	1.4	55	91.172	91.169
13	3.5	50	2.75	1.4	55	91.15.8	91.169
14	3.5	20	2.75	0.3	55	41.150	40.500
15	3.5	50	0.5	1.4	100	91.242	92.712
16	3.5	50	2.75	0.3	100	58.762	56.555
17	2	80	2.75	1.4	55	91.246	95.775
18	3.5	50	5	1.4	10	87.675	86.796
19	3.5	50	5	2.5	55	91.244	88.565
20	3.5	80	2.75	1.4	10	90.951	88.224
21	3.5	50	2.75	1.4	55	91.154	91.169
22	3.5	80	2.75	0.3	55	42.427	40.376
23	3.5	50	0.5	1.4	10	84.608	83.872
24	3.5	50	2.75	1.4	55	91.18.1	91.169
25	2	20	2.75	1.4	55	93.472	99.28
26	5	50	5	1.4	55	78.462	80.598
27	2	50	2.75	0.3	55	79.656	64.671
28	3.5	80	2.75	2.5	55	91.246	92.116
29	2	50	0.5	1.4	55	91.238	91.223
30	3.5	20	2.75	1.4	10	91.207	88.743
31	3.5	50	5	1.4	100	91.192	89.047
32	3.5	50	2.75	1.4	55	91.172	91.169
33	3.5	50	5	0.3	55	33.721	37.973
34	3.5	20	0.5	1.4	55	91.223	90.350
35	3.5	50	2.75	1.4	55	91.172	91.169
36	5	50	2.75	2.5	55	98.152	106.739
37	5	50	2.75	1.4	10	83.455	81.432
38	3.5	50	0.5	0.3	55	30.147	36.513
39	3.5	80	2.75	1.4	100	91.689	93.572
40	3.5	50	0.5	2.5	55	91.242	90.677
41	5	50	2.75	0.3	55	10.414	14.799
42	5	80	2.75	1.4	55	90.507	88.057
43	3.5	20	2.75	2.5	55	91.243	88.057
44	2	50	5	1.4	55	91.240	97.280
45	3.5	50	2.75	2.5	100	81.247	78.848
46	2	50	2.75	1.4	100	94.247	97.332

3. RESULTS AND DISCUSSION

3. 1. Consideration of Regression Model and Analysis of Variance (ANOVA) The following equation was used to predict this work response [21]:

$$Y = \beta_0 + \sum_{i=1}^{k} \beta xi + \sum_{i=1}^{k} \beta ii x_i^2 + \sum_{i=1} \sum_{i \neq j=1} \beta ij x_i x_{ij} + \varepsilon \quad (3)$$

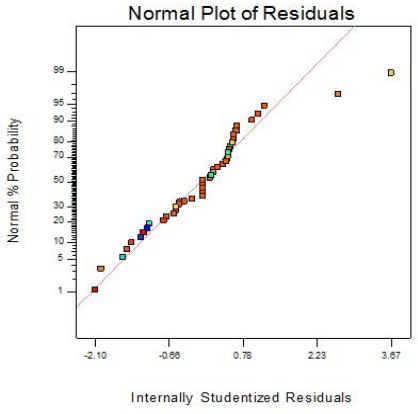
where Y and β_0 are COD removal percentage and constant coefficient, respectively. β_i, β_{ii} and β_{ij} are the liner regression coefficients. x_i and x_j are the coded variables for the independent variables and ε is the random error.

The following equation was investigated for COD removal percentage (R%) by software.

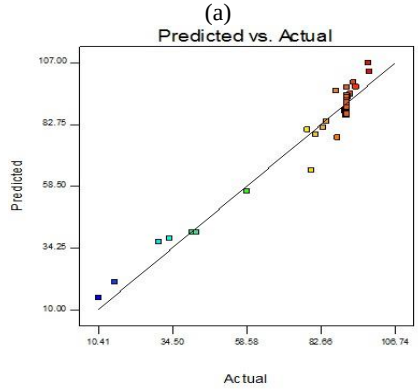
$$R\% = 91.17 - 5.15A - 0.38B - 0.16C + 26.19D + 2.80E + 1.30AB - 3.19AC + 19.78AD + 1.19AE + 2.025E - 003BC - 0.32BD - 0.12BE - 0.89CD - 1.67CE - 15.04DE + 0.21A^2 + 0.92B^2 - 2.28C^2 - 25.46D^2 - 0.81E^2 \quad (4)$$

where, A, B, C, D and E are pH, current density (mA/cm^2), H_2O_2/Fe^{2+} molar ratio, volume ratio of H_2O_2/PhW (ml/l) and reaction time (min), respectively.

According to Figures 3a and 3b, the quadratic model is suitable for this work, because the actual data are very close to the straight line.



(a)



(b)

Figure 3. Normal probability vs. internally studentized residuals values (a) and predicted vs. actual values (b) for COD removal

Table 4 shows ANOVA data obtained from the software. According to this table, R^2, R^2 adjusted (Adj) and R^2 predicted (Pred) were close to one. The adequate precision (AP) was more than four and F-value was a large amount. The coefficient of variance percentage (CV%) was less than ten. All of these parameters confirmed that the quadratic model properly works in this research [21-24].

3. 2. Three Dimensional Plots for the Regression Model Figure 4a shows interaction of current density and pH on COD removal. As shown in this figure, maximum removal can occur at minimum pH and current density. Figure 4b shows interaction between pH and H_2O_2/Fe^{2+} molar ratio. According to this figure, molar ratio effect is more than that of the pH. Furthermore, Figure 4c shows a weak effect of pH against volume ratio of H_2O_2/PhW on the COD removal. Interaction between reaction time and pH is shown in Figure 4d, as well. As shown in this figure, COD removal increased with increasing the reaction time and pH reduction. As illustrated in Figure 4e, a middle range of pH and H_2O_2/Fe^{2+} molar ratio can improve COD removal. This output can be found from Figures 4a and 4b. In fact, the reaction rate decreased when pH was much more than that of the optimum point (that was around 2.43 in this research) [23, 25]. Hydroxyl radicals cannot be easily produced in the alkaline conditions [26]. Therefore, the removal efficiency will be decreased. Furthermore, the current density has a significant role in production of hydroxyl radicals and metal ions [27]. Ferrous irons regeneration (from ferric ions) on cathode is a contributing factor [28]. An increase in the current density above its optimum value (23.08 mA/cm^2) can activate the competitive reactions and inhibit the main electro-Fenton reactions [23, 29, 30].

TABLE 4. Quadratic model ANOVA results responses

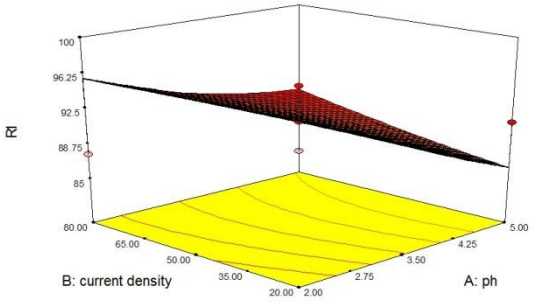
Variable	COD removal
Standard deviation	5.780
Mean	81.630
Coefficient of variance (CV%)	7.070
Press	335.64
R-Squared	0.961
Adj R- Squared	0.930
Pred R-Squared	0.845
Adequate precision	23.561
F-value	31.100
P-value	< 0.0001

As shown in Figure 4f, COD removal increased with the volume ratio of H_2O_2/PhW (ml/l) increment when the current density was at mid-range. Figure 4g shows interaction of current density and reaction time on the COD removal. The response increased with increase in both parameters. Figure 4h shows interaction between H_2O_2/Fe^{2+} molar ratio and H_2O_2/PhW (ml/l) on COD removal. This figure indicates that a middle range of these independent variables will increase the removal. Figure 4i shows that reaction time (against H_2O_2/Fe^{2+} molar ratio) has a positive effect on the removal, while reaction time reduction [against volume ratio of H_2O_2/PhW (ml/l)] has a good effect on the removal where the volume ratio increases (as shown in Figure 4j).

The optimum values of H_2O_2/Fe^{2+} molar ratio and volume ratio of H_2O_2/PhW were at 2.69 and 1.84 ml/l, respectively. Further increase in these two parameters can active the side reactions between H_2O_2 and OH [31]. Moreover, reaction time normally increases COD removal, although it decreases after the optimized reaction time (28.08 min). Its reason is due to H_2O_2 decomposition to O_2 and H_2O [14].

According to the literature, the other techniques such as ultrasonic, solar photo-catalysis, photo-Fenton could remove ibuprofen from a pharmaceutical wastewater at 98, 98 and 40%, respectively [32-34].

3. 3. Optimization and Validation The optimization process was statistically carried out with COD removal enhancement (as a goal). The operating conditions were at current density of 23.08 mA/cm^2, Fe^{2+}/H_2O_2 molar ratio of 2.69, volume ratio of H_2O_2/PhW of 1.84 (ml/l) and reaction time of 28.08 min for 99.740% of COD removal. For validation, an experiment was carried out at the same operating conditions. According to this experiment, COD removal was around 98.290%. These data are presented in Table 5.



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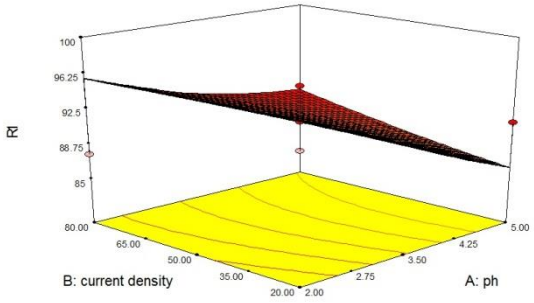

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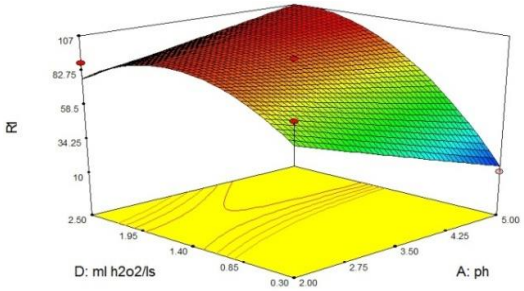

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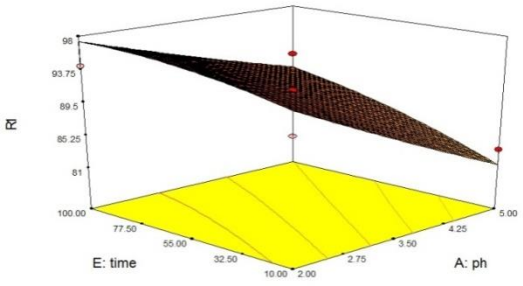
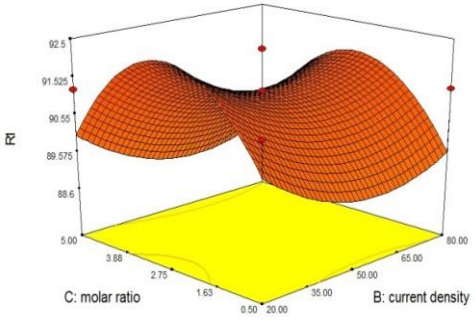
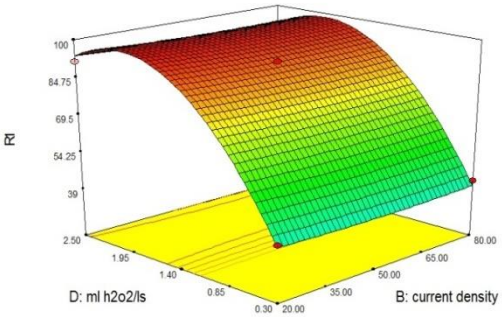


(d)



(e)



(f)

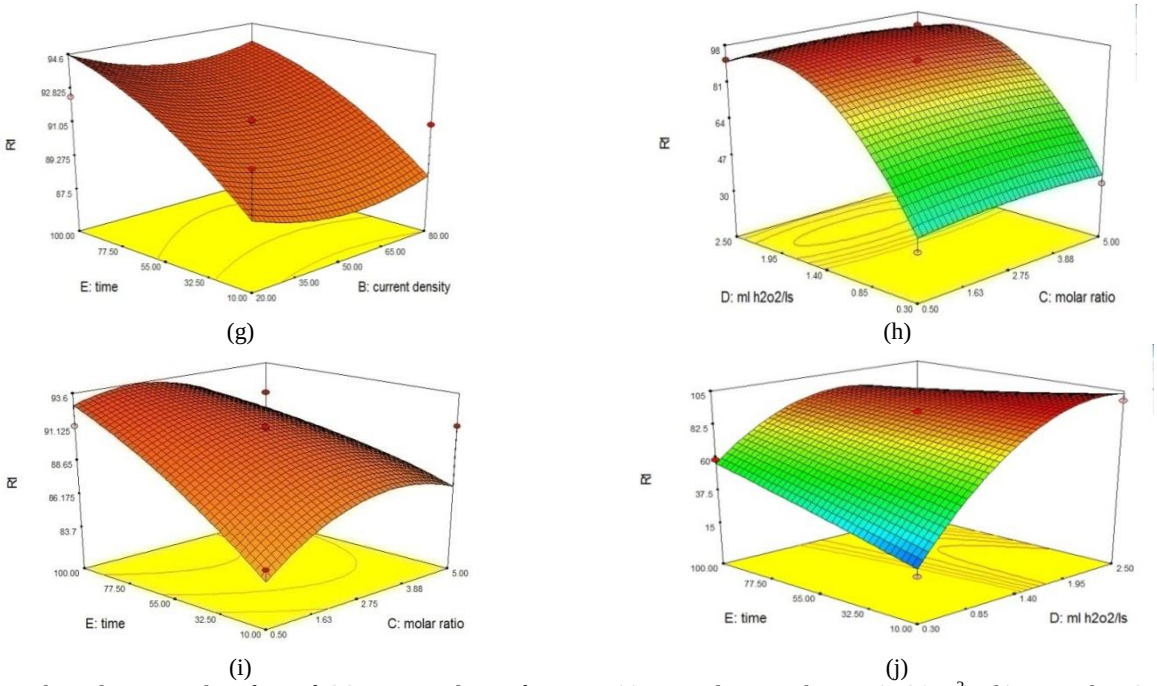


Figure 4. Three-dimensional surface of COD removal as a function: (a) pH andcurrent density (mA/cm^2), (b) pH and H_2O_2/ Fe^{2+} molar ratio, (c) pH and H_2O_2/PhW (ml/l), (d) pH and reaction time (min), (e) current density (mA/cm^2) and H_2O_2/Fe^{2+} molar ratio, (f) current density (mA/cm^2) and H_2O_2/PhW (ml/l), (g) current density (mA/cm^2) and reaction time (min), (h) H_2O_2/ Fe^{2+} molar ratio and H_2O_2/PhW (ml/l), (i) H_2O_2/Fe^{2+} molar ratio and reaction time (min), (j) H_2O_2/PhW (ml/l) and reaction time (min)

TABLE 5. Optimum conditions found by design expert and experimental verification for COD removal

pH	Current density (mA/cm^2)	Molar ratio (H_2O_2/Fe^{2+})	H_2O_2/PhW (ml/l)	Reaction time (min)	Removal% observed	Removal% predicted	**Error**
2.43	23.08	2.69	1.84	28.08	98.290	99.740	1.450

4. CONCLUSION

In this research, a synthetic pharmaceutical wastewater in terms of ibuprofen simulated with a real pharmaceutical wastewater was treated through electro-Fenton technique. Five independent variables were simultaneously considered on this process. Box-Behnken design under RSM was applied for number of runs minimization and optimization. A good correlation for removal of a pharmaceutical waste in water based on the independent variables was developed (with R^2=0.961). Ibuprofen concentration was decreased from 400 ppm (in the initial wastewater) to 6.840 ppm (in the final wastewater) through the optimum operating conditions.

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Ibuprofen Removal from a Pharmaceutical Wastewater using Electro-Fenton Process: An Efficient Technique

RESEARCH NOTE

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چکیده

هدف از انجام این پژوهش بررسی پنج پارامتر در فرایند الکتروفنتون در حذف بروفن از یک پساب دارویی است. این پارامترها pH، چگالی جریان، نسبت مولی H_2O_2/Fe^{2+} و نسبت حجمی H_2O_2/PhW (ml/l) و زمان واکنش هستند. در این تحقیق یک پساب سنتزی دست‌ساخت با همان غلظت ایبوبروفن در پساب واقعی (۴۰۰ ppm) انتخاب شد. نمونه‌ها بر اساس نیاز به اکسیژن شیمیایی (COD) آزمایش شدند. پارامترها تحت روش سطح پاسخ (RSM) بهینه-سازی شدند. استفاده از این روش تعداد آزمایش‌ها را به حداقل رساند. شرایط بهینه برای حذف ٪98/29 COD در آزمایشگاه تحت شرایط pH ۲/۴۳، چگالی جریان mA/cm^2 ۲۳/۰۸، نسبت مولی H_2O_2/Fe^{2+} ۲/۶۹، نسبت حجمی $H_2O_2/PhW(ml/l)$ ۱/۸۴ و زمان واکنش ۲۸/۰۸ دقیقه به دست آمد.

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