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ORIGINAL PAPER

Modeling and optimizing Fenton and electro-Fenton processes for dairy wastewater treatment using response surface methodology

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Abstract

In this paper, dairy wastewater treatment was investigated by Fenton and electro-Fenton (EF) processes in respect of removal efficiencies of chemical oxygen demand (COD), orthophosphate, suspended solid (SS), and color. The response surface methodology (RSM) approach using Box–Behnken design was carried out to develop mathematical model and to optimize process parameters. Experimental data were analyzed by the analysis of variance (ANOVA) to identify the interaction mechanism between the process variables and the dependent variables. According to ANOVA results of Fenton process, COD removal increased with an increase in H_2O_2/COD ratio and reaction time but decreased with increased H_2O_2/Fe^{2+} ratio and initial pH. Opposing to that, in the EF process, COD removal increased with an increase in H_2O_2/Fe^{2+} ratio and reaction time but decreased with an increase in H_2O_2/COD ratio and initial pH. The COD removal efficiencies were 65.5 and 72% under the optimum conditions for Fenton (H_2O_2/COD ratio 1.9, H_2O_2/Fe^{2+} ratio 5, pH 4 and reaction time 10 min) and electro-Fenton (H_2O_2/COD ratio 2, current density 32 mA/cm^2, pH 2.4 and reaction time 45 min) processes, respectively. No significant removal differences for orthophosphate, SS and color were determined between the two processes because the removal efficiencies were over the 88% for each process configuration where *P* value was greater than $5.6 * 10^{-5}$ with 99% confidence level and greater than $1.7 * 10^{-3}$ with 95% confidence level for all responses for Fenton and EF processes, respectively).

Keywords Dairy wastewater · Fenton process · Electro-Fenton process · RSM · Optimization

Introduction

Dairy industries, similar to other agro-industries, generate enormous volume of wastewater up to 0.2–10 L per liter of processed milk (Vourch et al. 2008) primarily from the cleaning and washing operations in the milk-processing plants (Ramasamy et al. 2004), and the wastewater has been characterized by its non-stable pH characteristic due to the use of acid and alkaline cleaners and sanitizers in dairy industry (Baskaran et al. 2000), high COD, biochemical oxygen demand (BOD), and nutrients such as nitrogen, phosphorus, and potassium concentrations (Ayhan Şengin and Özacar 2006; Banu et al. 2008), and high levels of dissolved or suspended solids including fats, oils, and grease (Farizoglu and Uzuner 2011; Praneeth et al. 2014). Uncontrolled discharge of dairy wastewater along with high organic matter and nutrients causes serious pollution problems in water bodies and biodiversity such as algae and bacteria growth, resulting in oxygen depletion and, eventually, suffocating the rivers leading to the gradual disappearance of fish. Therefore, the need to treat highly polluted dairy effluents by various processes is indispensable (Perle et al. 1995; Banu et al. 2008; Deshpande et al. 2012).

The dairy wastewater can be treated using biological and physicochemical methods. Conventional anaerobic treatment requires high energy for aeration (Wheatley 1990), and aerobic treatment needs additional treatment to achieve discharge limits that are often used for

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treating such wastewater (Ayhan Şengin and Özacar 2006; Banu et al. 2008; Kushwaha et al. 2010a; Karadag et al. 2015; Dabrowski et al. 2017). On the other hand, physicochemical processes, such as coagulation–flocculation (Kushwaha et al. 2010a; Loloei et al. 2014), electrochemical treatment (Ayhan Şengin and Özacar 2006; Tchamango et al. 2010; Kushwaha et al. 2010b; Bazrafshan et al. 2013), nanofiltration (Turan 2004; Luo et al. 2012; Andrade et al. 2014; Chen et al. 2018), and reverse osmosis (Turan 2004; Balannec et al. 2005; Vourch et al. 2008), are used for removal of colloidals and SS in dairy wastewater. Fenton and EF processes are also alternative physicochemical treatment processes to treat dairy wastewater (Yavuz et al. 2011; Davarnejad and Nikseresht 2016).

In Fenton process, hydrogen peroxide is catalyzed by ferrous ions to produce hydroxyl radicals (OH·) where the OH· is involved in the breakdown of organic matters in the wastewater (Fenton 1896; Zhang et al. 2006; Bautista et al. 2008). Oxidation reactions initiated by hydroxyl radical lead to the ultimate decomposition of organic molecules into CO_2 and H_2O, which makes these processes “environmental-friendly” processes (Weast 1969; Ayoub et al. 2010; Cheng et al. 2016). The EF process is an indirect electrochemical oxidation that employs OH radical generated by the Fenton reaction to oxidize organic compounds. The process is based on the electrochemical in situ production of the Fenton’s reagent, either or both of H_2O_2 and Fe^{2+} (Oturan et al. 2000). The EF increases the degradation of organic matters in a highly strong wastewater (Qiang et al. 2003; Chang et al. 2004; Davarnejad and Nikseresht 2016). However, there are only a few studies which investigated the treatment of dairy wastewater using Fenton and EF processes so far. Yavuz et al. (2011) investigated treatment of dairy wastewater by electro-coagulation (EC) and EF processes and succeeded 79.2% COD removal at optimum conditions. On the other hand, Davarnejad and Nikseresht (2016) treated dairy wastewater by EF process results with 93.9% COD and 97.3% color removal efficiencies.

The present study investigates dairy wastewater treatment by Fenton and EF processes and aims: (1) to develop mathematical model and to optimize operating conditions on COD, orthophosphate, color and SS removal, (2) to evaluate the effects and interactions of process variables: H_2O_2/COD ratio, H_2O_2/Fe^{2+} ratio, initial pH, reaction time for Fenton process and H_2O_2/COD ratio, current density, initial pH, and reaction time for EF process. Optimizations of Fenton and EF processes were carried out by RSM approach using BBD to develop a mathematical model and to study the interactive effects of studied parameters.

Materials and methods

Dairy wastewater

The dairy wastewater was taken from wastewater treatment plant of a dairy factory in Istanbul where milk, yogurt, and butter are the main products. The characterization of raw dairy wastewater is given in Table 1.

Samples were stored in containers and kept at 4 °C until Fenton and EF applications. All dairy wastewater samples were preserved and analyzed according to the standard methods (APHA 2005).

Experimental setup and procedure

The schematic view of Fenton and EF systems are presented in Fig. 1. In Fenton oxidation process, 35% H_2O_2 solution with a density of 1.13 kg/L and 10 g/L stock solution of Fe^{2+} by dissolving $FeSO_4•7H_2O$ in pure water were prepared, and 500 mL of wastewater was used for each experimental test. In the first step of Fenton oxidation process, pH of dairy wastewater was adjusted to the desired value by addition of 6 N H_2SO_4 or 6 N NaOH. The necessary amount of the $FeSO_4•7H_2O$ was supplemented from the stock solution, and then desired volume of H_2O_2 solutions was added to initiate Fenton reaction. After this step, jar test apparatus was used for a rapid mix at 200 rpm for 5 min, and then samples were gently stirred at 20 rpm for a desired amount of reaction time. To improve sludge settling rates, pH was adjusted to 7.0 and around by adding 6 N NaOH solution, leading to the precipitation of residual Fe^{2+} ions. Then, samples were settled for 60 min in a graduated settling column, about 200 mL of supernatant was collected, and pH of supernatant samples was adjusted to 10 and mixed at 70 °C for 10 min to eliminate residual H_2O_2 to prevent any interference during COD measurements (Erkan and Apaydin 2015; Gotvajn et al. 2011). COD, orthophosphate, SS, and color were analyzed in final supernatant samples by only using analytical grade chemicals.

For EF process, a laboratory-scale plexiglass reactor with 9 cm diameter and 13 cm height was manufactured. One

Table 1 Characteristics of raw dairy wastewater

Parameter	Value
COD (mg/L)	6055
TS (mg/L)	11,900
TSS (mg/L)	1320
TKN (mg/L)	90
Orthophosphate (mg/L)	94.57
pH	5.7
Conductivity (mS/cm) (20 °C)	6.0
Color (Pt–Co)	1700

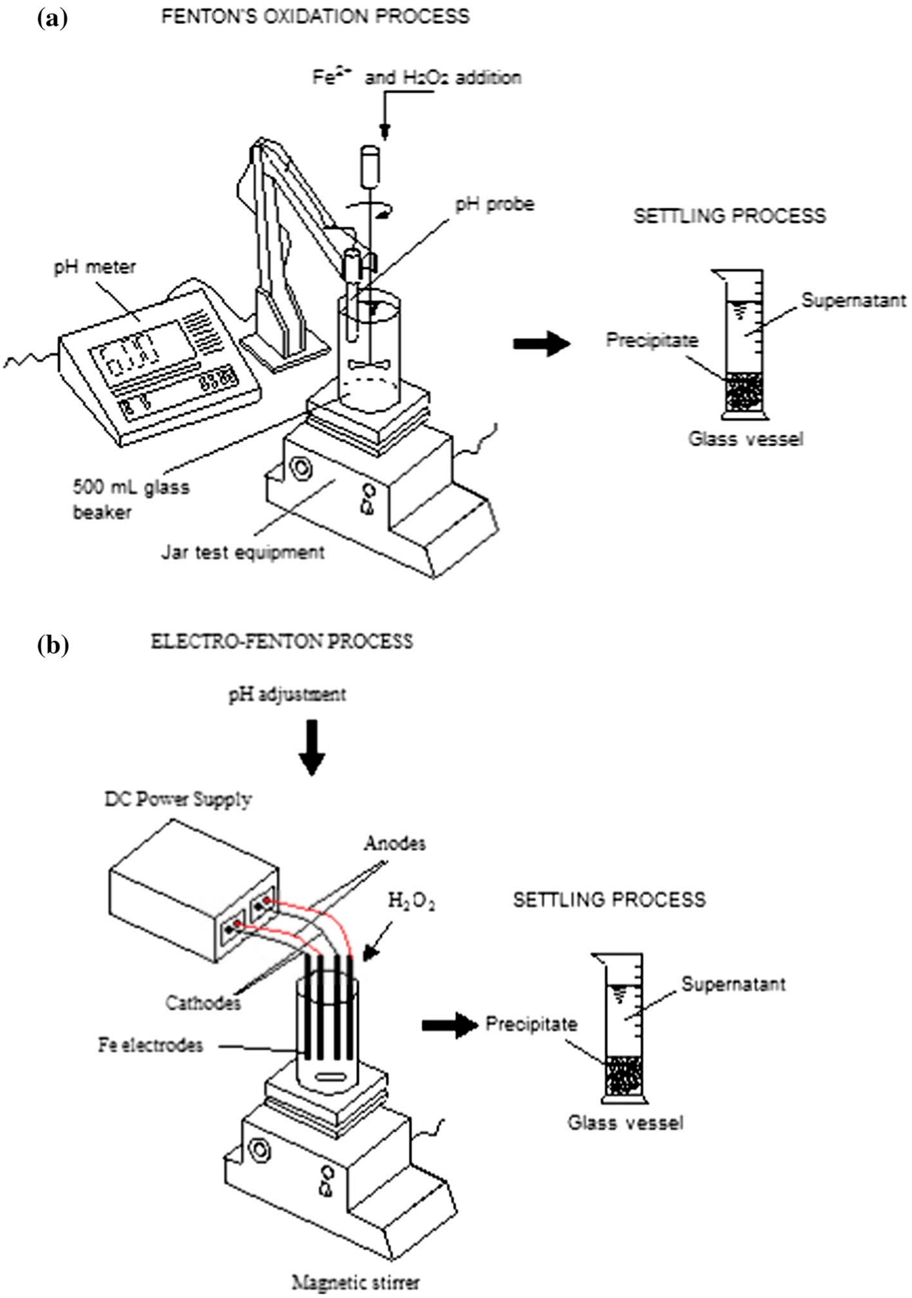


Fig. 1 Shematic diagrams of experimental systems for Fenton (**a**) and electro-Fenton (**b**) processes

anode and one cathode iron electrodes (comprised of two monopolar (MP) plates) with 6 cm width × 11.5 cm height, 0.1 cm thickness, and 46.2 cm^2 effective area were placed 2 cm apart from each other. A valve was installed at the bottom of the reactor to discharge the precipitated material through a sludge chamber. For each test, 500-mL wastewater sample was used. Before each run, electrodes were washed with acetone and the impurities on the aluminum electrode surfaces were removed by dipping in a solution freshly prepared by mixing 100 cm^3 35% HCl solution and 200 cm^3 2.8% hexamethylenetetramine aqueous solution for 5 min (Gengec et al. 2012). The EF experiments were initiated by supplying a current density between 4 and 32 mA/cm^2 by a DC power supply. At the end of each run, the floated and precipitated materials were collected, and the clarified effluent sample was pipetted out from the reactor and then allowed to settle for a few hours in a polyethylene flask. Finally, the clarified supernatant liquid was collected and preserved according to the Standard Methods (APHA 2005) and stored for characterization. All analyses were performed in accordance to the Standard Methods (APHA 2005). All chemicals used were analytical reagent grade.

Design of experiments and data analysis

In this study, the BBD based on RSM was used to design the set of experiments for Fenton and EF processes. RSM is fundamentally a particular set of mathematical and statistical methods for designing experiments, buildings models, determining the effect of variables, and investigating optimum operating conditions (Körbahti 2007). Statgraphics Centurion XVI.I software program was used for the statistical design of experiments and data analysis. The four operational parameters: H_2O_2/COD ratio (X_1), H_2O_2/Fe^{2+} ratio (X_2), initial wastewater pH (X_3), and reaction time (X_4) were optimized for Fenton process, whereas H_2O_2/COD ratio (X_1), current density (X_2), initial wastewater pH (X_3), and reaction time (X_4) were optimized for EF process in dairy wastewater treatment. Each independent factor was coded at three levels in the range of − 1 and + 1 determined by preliminary experiments (Table 2).

RSM makes possible to represent independent process parameters in quantitative form as:

$$Y = f\left(X_1, X_2, X_3, \dots, X_n\right) \pm \varepsilon \tag{1}$$

where Y is the response (dependent parameter), f is the response function, ε is the experimental error, and X_1, X_2, X_3,…, X_n are independent variables. In the optimization process, the responses can be related to independent factors by linear or quadratic models. A quadratic model which includes the linear model is given in Eq. (2).

$$\begin{aligned} Y = & \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 \\ & + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 \end{aligned} \tag{2}$$

where β is set of regression coefficients: the intercept (β_0), linear (β_1, β_2, β_3), interaction (β_{12}, β_{13}, β_{23}), and quadratic coefficients (β_{11}, β_{22}, β_{33}). The experiment sets are presented in Table 3 for Fenton and EF processes.

Analysis of variance (ANOVA) was used to obtain the interaction between the independent variables and the responses. The quality of the fit polynomial model was evaluated by R^2, and its statistical significance was checked by the Fisher *F* test in the same program. Model terms were utilized by the *P* value (probability) with 95% confidence level.

Table 2 Experimental range and levels of the independent variables in Fenton and electro-Fenton processes

	Variables	Symbol	− 1	0	1
Fenton	H_2O_2/COD ratio (w/w)	X_1	0.4	1.2	2
	H_2O_2/Fe^{2+} ratio (w/w)	X_2	5	15	25
	Initial pH	X_3	2	3	4
	Reaction time (min)	X_4	5	25	45
Electro-Fenton	H_2O_2/COD ratio (w/w)	X_1	0.4	1.2	2
	Current density (mA/cm^2)	X_2	4	18	32
	Initial pH	X_3	2	3	4
	Reaction time (min)	X_4	5	25	45

Results and discussion

Statistical analysis of Fenton and EF processes

The Fenton and EF batch experiments were designed using RSM in order to understand the effect of the variables (H_2O_2/COD ratio, H_2O_2/Fe^{2+} ratio or current density, pH and reaction time) on four important process responses (COD, orthophosphate, color, and SS). All the factors and responses are shown in Tables 2 and 3. The experimental results for all responses analyzed by ANOVA are summarized in Table 4. As can be seen in Table 4, R^2 values were over 91% indicating high coefficient of determination for actual and predicted values for COD, orthophosphate, SS, and color removal. For the Fenton process, *F* values of 10.91, 10.91, 12.06, and 20.52 implied significant models for COD, orthophosphate, SS, and color removal efficiencies, respectively. On the other hand, *F* values of COD, orthophosphate, SS, and color removal efficiencies were found 24.98, 9.31, 9.76, and 11.17 for EF process, respectively. The large *F* value represents a high significance of the corresponding term. The values of Prob. > *F* less than 0.05 imply that the model terms are significant, whereas the values greater than 0.1 indicate that the model terms are insignificant (Körbahti and Rauf 2008; Arslan-Alaton et al. 2009). Prob. > *F* values less than 0.0001 indicates that the terms are highly significant in all the models except orthophosphate removal for EF process.

The approximating functions for Fenton and EF processes are presented in Eqs. 3–10. The first-order terms (X_1, X_2, X_3 or X_4) represent the effects of the linear main factor; the interaction effects terms (X_1X_2, X_1X_3, X_1X_4, X_2X_3, X_2X_4 or X_3X_4) and the second-order terms (X_1^2, X_2^2, X_3^2or X_4^2) represent the interaction between the two factors and the quadratic effects in these equations, respectively. The positive sign in front of the coefficients indicates a synergistic effect, whereas the negative sign indicates an antagonistic effect (Kim 2016).

$$\begin{aligned} Y_1 = & 74.7463 + 3.6683\,X_1 - 2.99835\,X_2 - 16.4535\,X_3 + 0.376445\,X_4 \\ & + 0.458016\,X_1^2 - 0.593164\,X_1X_2 + 4.55022\,X_1X_3 + 0.0147089\,X_1X_4 \\ & + 0.060255X_2^2 + 0.282597\,X_2X_3 + 0.00334847\,X_2X_4 + 2.19349X_3^2 \\ & - 0.206111\,X_3X_4 + 0.00155671\,X_4^2 \end{aligned} \tag{3}$$

Table 3 Removal efficiencies of the responses for Fenton and electro-Fenton processes

Run	X_1	X_2	X_3	X_4	Fenton process				Electro-Fenton process			
					COD (Y_1)	Orthophosphate (Y_2)	Color (Y_3)	SS (Y_4)	COD (Y_5)	Orthophosphate (Y_6)	Color (Y_7)	SS (Y_8)
1	− 1	0	0	− 1	26.67	98.03	91.47	88.39	52.35	99.39	94.76	93.79
2	1	0	0	1	45.00	98.96	99.95	86.36	62.63	99.99	97.88	93.78
3	1	0	0	− 1	43.68	94.27	98.88	92.24	40.18	99.42	98.06	91.39
4	0	1	0	− 1	29.64	98.70	96.06	91.67	48.06	99.61	98.65	91.82
5	− 1	0	1	0	23.86	97.24	95.88	92.27	51.28	99.48	95.82	95.00
6	0	− 1	− 1	0	56.59	98.82	82.76	89.42	37.64	99.71	99.76	92.91
7	0	0	− 1	1	32.62	98.92	90.06	93.06	61.30	99.99	99.12	94.39
8	0	0	1	− 1	50.34	99.76	76.59	95.52	50.66	99.85	99.35	94.24
9	0	0	0	0	33.11	99.75	98.53	89.52	54.99	99.90	98.06	89.18
10	1	0	− 1	0	38.07	96.57	99.21	90.91	49.13	99.78	98.71	89.69
11	1	1	0	0	33.86	98.37	99.99	89.24	66.97	99.90	97.82	93.39
12	0	1	0	1	28.98	99.99	99.99	92.12	58.46	99.59	97.41	93.73
13	0	− 1	1	0	49.79	99.84	98.94	88.91	36.58	99.70	98.53	98.94
14	0	0	− 1	− 1	34.76	98.56	97.29	86.67	39.29	99.28	98.06	92.69
15	− 1	0	− 1	0	29.81	99.21	94.41	86.73	53.46	99.74	93.82	94.69
16	− 1	− 1	0	0	38.56	98.61	94.18	89.85	41.25	99.51	97.41	94.54
17	0	0	0	0	35.92	99.93	98.00	88.79	54.04	99.90	98.94	88.76
18	0	0	0	0	32.78	99.97	98.82	89.40	52.21	99.92	98.47	89.03
19	0	0	1	1	31.71	99.65	99.99	90.00	57.19	99.69	97.06	94.48
20	1	0	1	0	46.68	98.96	96.23	89.18	59.78	99.99	97.18	96.21
21	0	− 1	0	− 1	50.08	98.61	90.06	89.85	33.74	99.10	98.35	96.36
22	1	− 1	0	0	48.80	99.66	96.71	88.48	32.65	99.33	97.35	93.94
23	− 1	1	0	0	28.90	99.49	99.97	89.24	50.78	99.34	91.88	92.12
24	0	1	− 1	0	30.55	99.98	99.99	87.76	57.52	99.79	98.59	93.48
25	0	1	1	0	35.06	99.81	90.59	93.41	62.54	99.90	96.65	94.51
26	0	− 1	0	1	46.74	99.51	97.94	90.15	43.85	99.76	99.41	95.76
27	− 1	0	0	1	27.05	96.89	99.93	93.51	54.25	99.90	97.18	94.00

Table 4 ANOVA results of the predicted response surface quadratic model

Process	Model	R^2	Adj. R^2	Sum of squares	Mean square	F value	Prob. > F
Fenton	COD	0.93	0.84	2081.90	138.27	10.91	$9.4*10^{-5}$
	Orthophosphate	0.92	0.84	44.60	2.95	10.91	$9.4*10^{-5}$
	Color	0.93	0.86	837.23	55.83	12.06	$5.6*10^{-5}$
	SS	0.96	0.91	132.21	9.06	20.52	$1.1*10^{-6}$
Electro-Fenton	COD	0.97	0.93	2314.64	159.85	24.98	$1*10^{-6}$
	Orthophosphate	0.91	0.82	1.63	0.01	9.31	$2.1*10^{-3}$
	Color	0.92	0.82	81.20	5.33	9.76	$1.7*10^{-3}$
	SS	0.93	0.85	144.74	9.60	11.17	$8.3*10^{-5}$

$$\begin{aligned}Y_2 = {} & 34.1395 + 6.18014X_1 + 2.69831X_2 + 28.3589X_3 - 0.4688X_4 \\ & + 2.5321X_1^2 - 0.211929X_1X_2 - 1.38916X_1X_3 - 0.115534X_1X_4 \\ & - 0.00752745X_2^2 - 0.639663X_2X_3 - 0.00494125X_2X_4 - 4.52198X_3^2 \\ & + 0.383045X_3X_4 - 0.00622484X_4^2\end{aligned} \tag{4}$$

$$\begin{aligned}Y_3 = {} & 77.1511 + 10.7206X_1 - 0.522648X_2 + 1.02041X_3 + 0.473837X_4 \\ & - 0.574871X_1^2 + 0.0842057X_1X_2 - 2.27272X_1X_3 - 0.171877X_1X_4 \\ & + 0.000769978X_2^2 + 0.15409X_2X_3 + 0.000189375X_2X_4 + 0.72423X_3^2 \\ & - 0.148864X_3X_4 + 0.00359654X_4^2\end{aligned} \tag{5}$$

$$\begin{aligned}Y_4 = {} & 98.6258 - 0.224435X_1 + 0.07259X_2 - 0.0760153X_3 + 0.0260836X_4 \\ & - 2.753X_1^2 - 0.00258057X_1X_2 + 1.36537X_1X_3 + 0.0911422X_1X_4 \\ & + 0.00122684X_2^2 - 0.0299075X_2X_3 + 0.000480125X_2X_4 - 0.117053X_3^2 \\ & - 0.00579875X_3X_4 - 0.00200579X_4^2\end{aligned} \tag{6}$$

$$\begin{aligned}Y_5 = {} & 43.6957 - 28.8275X_1 + 0.753469X_2 - 0.945905X_3 + 0.707873X_4 \\ & - 0.151094X_1^2 + 0.553335X_1X_2 + 4.01119X_1X_3 + 0.321014X_1X_4 \\ & - 0.0289842X_2^2 + 0.108486X_2X_3 + 0.000266071X_2X_4 \\ & + 0.109438X_3^2 - 0.193458X_3X_4 - 0.0042335X_4^2\end{aligned} \tag{7}$$

$$\begin{aligned}Y_6 = {} & 85.8009 + 10,941X_1 - 0.0901018X_2 + 3.00026X_3 + 0.187605X_4 \\ & - 3.06372X_1^2 + 0.133929X_1X_2 - 1.10294X_1X_3 - 0.0404406X_1X_4 \\ & - 0.0011129X_2^2 - 0.0126036X_2X_3 - 0.00204839X_2X_4 - 0.115217X_3^2 \\ & - 0.0419113X_3X_4 + 0.000539208X_4^2\end{aligned} \tag{8}$$

$$\begin{aligned}Y_7 = {} & 129.266 - 14.8922X_1 - 0.459656X_2 - 16.4831X_3 - 0.291844X_4 \\ & + 2.86025X_1^2 + 0.0419375X_1X_2 + 1.94128X_1X_3 + 0.0340922X_1X_4 \\ & + 0.0150446X_2^2 - 0.0893929X_2X_3 + 0.00224562X_2X_4 + 2.91843X_3^2 \\ & - 0.0181812X_3X_4 + 0.00578849X_4^2\end{aligned} \tag{9}$$

$$\begin{aligned}Y_8 = {} & 98.4464 - 0.145102X_1 + 0.0325376X_2 \\ & + 0.0479981X_3 + 0.072289X_4 - 0.214471X_1^2 \\ & + 0.016596X_1X_2 + 0.14792X_1X_3 \\ & + 0.00113864X_1X_4 + 0.001052X_2^2 \\ & + 0.00217679X_2X_3 - 0.000603188X_2X_4 \\ & + 0.00618162X_3^2 - 0.0110127X_3X_4 - 0.00040504X_4^2\end{aligned} \tag{10}$$

On the basis of the coefficients in Eq. (3), it can be said that COD removal efficiencies increase with an increase in H_2O_2/COD ratio and reaction time, but the COD removals decrease with increased H_2O_2/Fe^{2+} ratio and initial pH values in Fenton process. In the EF process (Eq. 7), the COD removal efficiencies increase with an increase in H_2O_2/Fe^{2+} ratio and reaction time but decrease with an increase in current density and initial pH. On the other hand, orthophosphate removal efficiencies in Fenton process increase with an increase in H_2O_2/COD ratio, H_2O_2/Fe^{2+} ratio, and initial pH, whereas the removals decrease with an increase in reaction time (Eq. 4). In the EF process, the orthophosphate removals increase with current density, initial pH, and the reaction time (Eq. 8). Color removal efficiencies increase with increase in H_2O_2/COD ratio, initial pH, and the reaction time in the Fenton process (Eq. 5). H_2O_2/Fe^{2+} ratio has a negative effect on color removal efficiencies in both processes.

ANOVA results and response surface plots for responses

The ANOVA tables obtained from the response surface quadratic models for COD removal with both process are shown in Table 5. As can be seen from Table 5, the comparison of the ANOVA results showed that there are highly significant interaction ($P < 0.0001$) between X_1 (H_2O_2/COD ratio) and X_2 (H_2O_2/Fe^{2+} ratio) within the experimental range for COD removal in Fenton process. On the other hand, X_2 (current density) and X_4 (time) have highly significant effect for COD removal in EF process.

Response surface graphs were produced by varying two of the process variables within the experimental range while holding the other factors at their central values to visualize the effect of the response variables on the dependent ones. The response surface graphs for COD removal efficiencies are presented in Figs. 2 and 3. As can be seen from Fig. 2a, a slight increase in COD removal was observed with an increase in the H_2O_2/COD ratio at low H_2O_2/Fe^{2+} ratio in Fenton process. As an operational approach, low H_2O_2/Fe^{2+} ratio is suggested. It is remarkable that the influence of X_1 and X_2 were very significant. Figure 2b shows that the influence of H_2O_2/COD ratio and pH on COD removal efficiency was significant, and it was observed that high COD removal efficiencies were obtained at the low H_2O_2/Fe^{2+} ratio with an increase in pH. As higher pH values results with lower chemical consumption for pH adjustment, it can be said that pH about 4 could act more acceptable and much suitable. The main effective parameters were H_2O_2/COD ratio, H_2O_2/Fe^{2+} ratio, and pH. As it could be observed, time was not significant on any experimental set of Fenton process. In EF process, maximum COD removal efficiency was obtained high current density and long reaction time at low H_2O_2/COD ratio (Fig. 3a). The effect of pH was found similar to Fenton process. H_2O_2/COD ratio, current density, and time were found the main operating parameter in EF process. COD removal efficiencies varied between 23.9–56.6 and 32.6–67.0% for Fenton and EF processes, respectively.

The ANOVA results for orthophosphate removal are given in Table 6 for Fenton and EF processes. In terms of orthophosphate removal, the X_2X_3, X_3X_4 have highly significant effects on Fenton process ($P < 0.0001$), whereas the only X_4 has highly significant effect on orthophosphate removal in EF process ($P < 0.0001$).

Figures 4 and 5 show the effects of dependent variables on orthophosphate removal efficiencies. As seen from Fig. 4, H_2O_2/COD ratio was kept on a central value at high initial pH. On the other hand, orthophosphate removal efficiencies increased with an increase in reaction time, and H_2O_2/Fe^{2+} ratio did not have a remarkable influence on orthophosphate removal. In EF process, orthophosphate removal efficiencies increased with an increase of current

Table 5 ANOVA results for the response surface quadratic model for COD removal by Fenton and electro-Fenton processes

	Source	Sum of squares	Df	Mean square	*F* ratio	*P* value	Remark
Fenton	X_1	611.654	1	611.654	48.26	<0.0001	Highly significant
	X_2	988.628	1	988.628	78.00	<0.0001	Highly significant
	X_3	6.25401	1	6.25401	0.49	0.4958	Not significant
	X_4	33.0248	1	33.0248	2.61	0.1325	Not significant
	X_1X_1	0.428737	1	0.428737	0.03	0.8572	Not significant
	X_1X_2	57.3672	1	57.3672	4.53	0.0548	Not significant
	X_1X_3	53.0034	1	53.0034	4.18	0.0634	Not significant
	X_1X_4	0.221545	1	0.221545	0.02	0.8970	Not significant
	X_2X_2	179.76	1	179.76	14.18	0.0027	Significant
	X_2X_3	31.9444	1	31.9444	2.52	0.1384	Not significant
	X_2X_4	1.79396	1	1.79396	0.14	0.7133	Not significant
	X_3X_3	26.6191	1	26.6191	2.10	0.1729	Not significant
	X_3X_4	67.9706	1	67.9706	5.36	0.0391	Significant
	X_4X_4	2.14515	1	2.14515	0.17	0.6880	Not significant
	Total error	152.102	12	12.6751			
	Total (corr.)	2087.93	26				
Electro-Fenton	X_1	5.27602	1	5.27602	0.82	0.3818	Not significant
	X_2	1172.81	1	1172.81	183.25	<0.0001	Highly significant
	X_3	32.2929	1	32.2929	5.05	0.0443	Significant
	X_4	448.96	1	448.96	70.15	0.0000	Highly significant
	X_1X_1	0.0498714	1	0.0498714	0.01	0.9311	Not significant
	X_1X_2	153.629	1	153.629	24.00	0.0004	Significant
	X_1X_3	41.1894	1	41.1894	6.44	0.0261	Significant
	X_1X_4	105.523	1	105.523	16.49	0.0016	Significant
	X_2X_2	172.121	1	172.121	26.89	0.0002	Significant
	X_2X_3	9.22701	1	9.22701	1.44	0.2530	Not significant
	X_2X_4	0.022201	1	0.022201	0.00	0.9540	Not significant
	X_3X_3	0.063875	1	0.063875	0.01	0.9221	Not significant
	X_3X_4	59.8813	1	59.8813	9.36	0.0099	Significant
	X_4X_4	15.2939	1	15.2939	2.39	0.1481	Not significant
	Total error	76.7988	12	6.3999			
	Total (corr.)	2314.64	26				

density, pH and time, whereas the influence of H_2O_2/COD ratio was not significant, and it should be kept central point. Orthophosphate removal efficiencies were found higher than 94% for both processes.

For color removals, the ANOVA results showed that the X_1X_3, X_1X_4, and X_3X_4 have highly significant effects on color removal in Fenton process (Table 7), whereas X_1 and X_1^2 have highly significant effects on color removal in EF process.

The effect of operational parameters on color removal is shown in Figs. 6 and 7 for Fenton and EF processes, respectively. From Fig. 6, maximum color removal efficiency could be obtained at high H_2O_2/COD ratio and H_2O_2/Fe^{2+} ratio for Fenton process. The pH value could hold central value. The effect of time was not considerable after a specific time, and there was no need for longer experimental time. In EF process, best color removal efficiency could be seen for central point of H_2O_2/COD ratio and low pH value. Current density and reaction time were not remarkably effective on color removal.

SS removal is one of the most important parameters for chemical and electrochemical treatment. For this reason, SS removal efficiencies were modeled for each experimental run. As seen from the ANOVA result in Table 8, X_1^2 and X_1X_4 have high significant effect on SS removal efficiencies for the Fenton process ($P < 0.0001$), whereas X_2^2, X_3^2, and X_4^2 have high significant effect on the SS for EF process.

The effects of variables on SS removal efficiencies are given in Figs. 8 and 9.

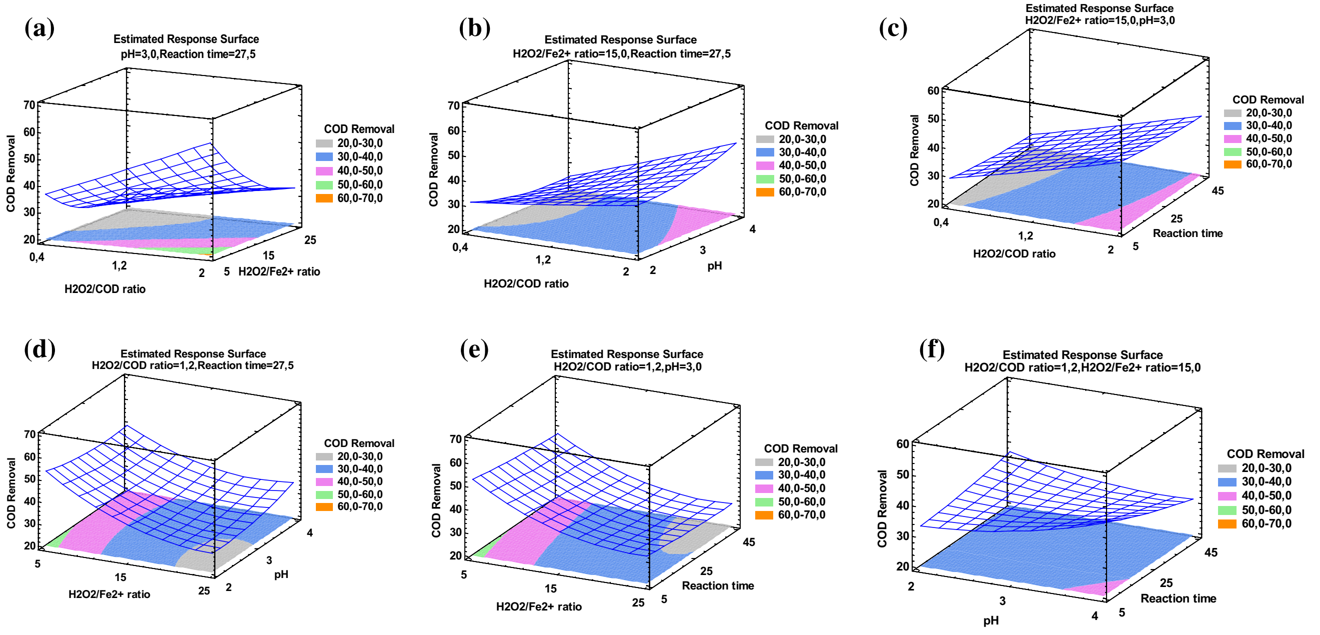


Fig. 2 Response surface graphs for the Fenton process for COD removal efficiency **a** H_2O_2/COD ratio vs. H_2O_2/Fe^{2+} ratio, **b** H_2O_2/COD ratio vs. pH, **c** H_2O_2/COD ratio vs. time, **d** H_2O_2/Fe^{2+} ratio vs. pH, **e** H_2O_2/Fe^{2+} ratio vs. time, **f** pH vs. time

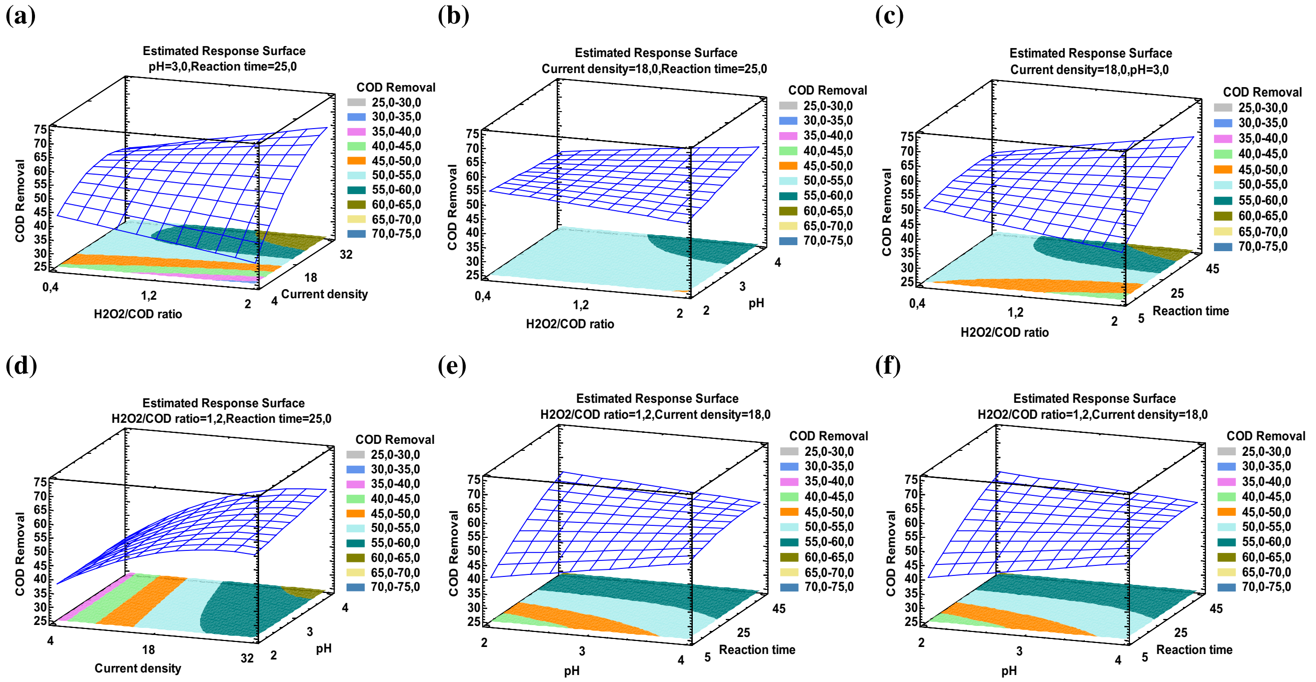


Fig. 3 Response surface graphs for the electro-Fenton process for COD removal efficiency **a** H_2O_2/COD ratio vs. current density, **b** H_2O_2/COD ratio vs. pH, **c** H_2O_2/COD ratio vs. time, **d** current density vs. pH, **e** current density vs. time, **f** pH vs. time

Process optimization

The optimum operating conditions obtained from ANOVA tests are presented in Table 9. Under the optimum conditions, the optimum COD removal efficiencies of model prediction were 70 and 75% for Fenton and EF, respectively. In order to control the optimization, the actual experimental processes were conducted at these optimum operating conditions, and

Table 6 ANOVA results for the response surface quadratic model for orthophosphate removal by Fenton and electro-Fenton processes

	Source	Sum of squares	Df	Mean square	*F* ratio	*P* value	Remark
Fenton	X_1	18.6602	1	18.6602	4.03	0.0677	Not significant
	X_2	28.4523	1	28.4523	6.15	0.0290	Significant
	X_3	2.85189	1	2.85189	0.62	0.4477	Not significant
	X_4	66.2677	1	66.2677	14.31	0.0026	Significant
	X_1X_1	13.1036	1	13.1036	2.83	0.1183	Not significant
	X_1X_2	7.32312	1	7.32312	1.58	0.2324	Not significant
	X_1X_3	4.94017	1	4.94017	1.07	0.3220	Not significant
	X_1X_4	13.6685	1	13.6685	2.95	0.1114	Not significant
	X_2X_2	2.80545	1	2.80545	0.61	0.4514	Not significant
	X_2X_3	163.667	1	163.667	35.35	<0.0001	Highly significant
	X_2X_4	3.90655	1	3.90655	0.84	0.3764	Not significant
	X_3X_3	113.13	1	113.13	24.44	0.0003	Significant
	X_3X_4	234.758	1	234.758	50.71	<0.0001	Highly significant
	X_4X_4	34.3002	1	34.3002	7.41	0.0185	Significant
	Total error	55.552	12	4.62933			
	Total (corr.)	837.232	26				
Electro-Fenton	X_1	0.0948307	1	0.0948307	8.25	0.0140	Significant
	X_2	0.0855597	1	0.0855597	7.44	0.0183	Significant
	X_3	0.00839867	1	0.00839867	0.73	0.4095	Not significant
	X_4	0.433915	1	0.433915	37.74	<0.0001	Highly significant
	X_1X_1	0.100484	1	0.100484	8.74	0.0120	Significant
	X_1X_2	0.138198	1	0.138198	12.02	0.0047	Significant
	X_1X_3	0.0560134	1	0.0560134	4.87	0.0475	Significant
	X_1X_4	0.00132763	1	0.00132763	0.12	0.7399	Not significant
	X_2X_2	0.226748	1	0.226748	19.72	0.0008	Significant
	X_2X_3	0.0037149	1	0.0037149	0.32	0.5802	Not significant
	X_2X_4	0.114099	1	0.114099	9.92	0.0084	Significant
	X_3X_3	0.000203799	1	0.000203799	0.02	0.8963	Not significant
	X_3X_4	0.194048	1	0.194048	16.88	0.0015	Significant
	X_4X_4	0.139995	1	0.139995	12.18	0.0045	Significant
	Total error	0.137981	12	0.0114985			
	Total (corr.)	1.63685	26				

COD removal efficiencies were obtained as 65.5 and 72% for Fenton and EF, respectively. The prediction of model for COD removal was in the confidence interval. The optimum orthophosphate, color, and SS removal efficiencies of model prediction were 100% for both Fenton and EF processes. At the end of the laboratory experiments, orthophosphate removal efficiencies were 90 and 88%, color removal efficiencies were 92.5 and 93%, and SS removal efficiencies were 95 and 92% for Fenton and EF process, respectively. The experimental results for orthophosphate, color, and SS removals were consistent with the predicted values.

Since pH leads to the production of hydroxyl radicals (•OH) and the concentration of ferrous ions in the solution, it plays a vital role in both processes (Mohajeri et al. 2010). During the processes, H_2O_2 is catalyzed by ferrous ions to produce •OH for degradation of organic matters in the wastewater (Zhang et al. 2007; Mohajeri et al. 2010). On the other hand, the electrical current causes the dissolution of metal electrodes into wastewater in EF process. The dissolved metal ions, at an appropriate pH, can form wide ranges of coagulated species and metal hydroxides that destabilize and aggregate the suspended particles or precipitate and adsorb dissolved contaminants (Bazrafshan et al. 2013). In the literature, there are several studies related to dairy wastewater treatment by EC process using aluminum and iron electrode (Sengil and Ozacar 2006; Tchamango et al. 2010; Kushwaha et al. 2010b; Bazrafshan et al. 2013). As aforementioned, however, there are only a few studies for the treatment of dairy wastewater using Fenton and EF processes. Yavuz et al. (2011) investigated treatment of dairy wastewater by

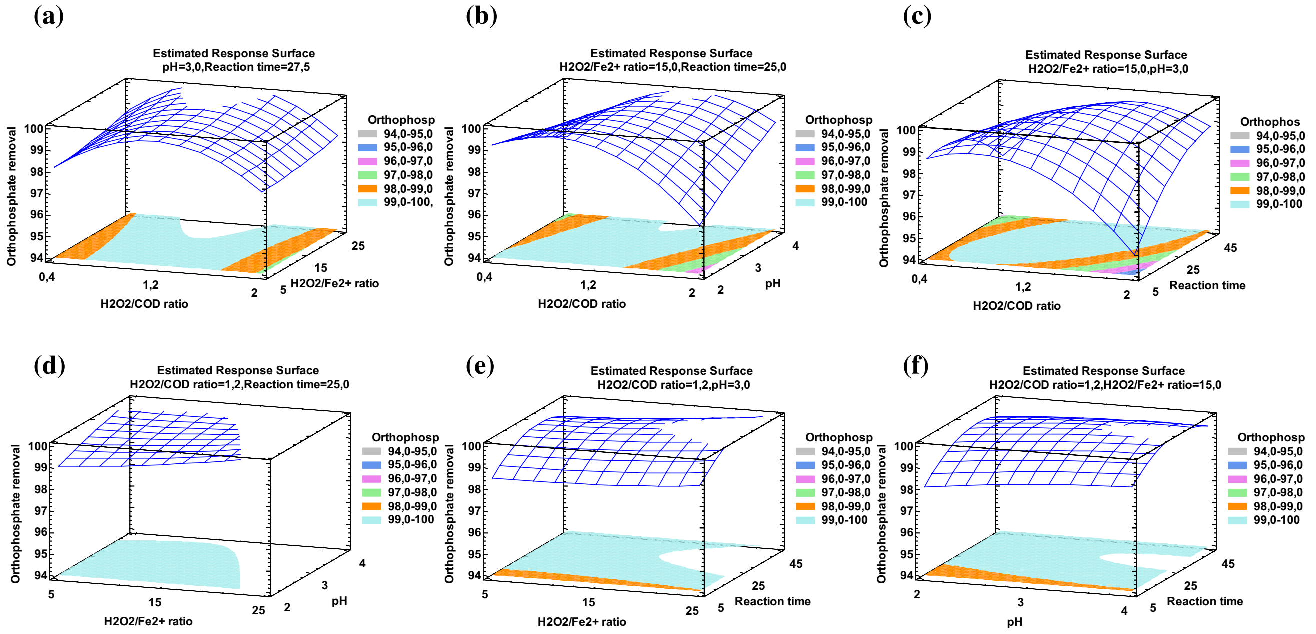


Fig. 4 Response surface graphs for the Fenton process for orthophosphate removal efficiency **a** H_2O_2/COD ratio vs. H_2O_2/Fe^{2+} ratio, **b** H_2O_2/COD ratio vs. pH, **c** H_2O_2/COD ratio vs. time, **d** H_2O_2/Fe^{2+} ratio vs. pH, **e** H_2O_2/Fe^{2+} ratio vs. time, **f** pH vs. time

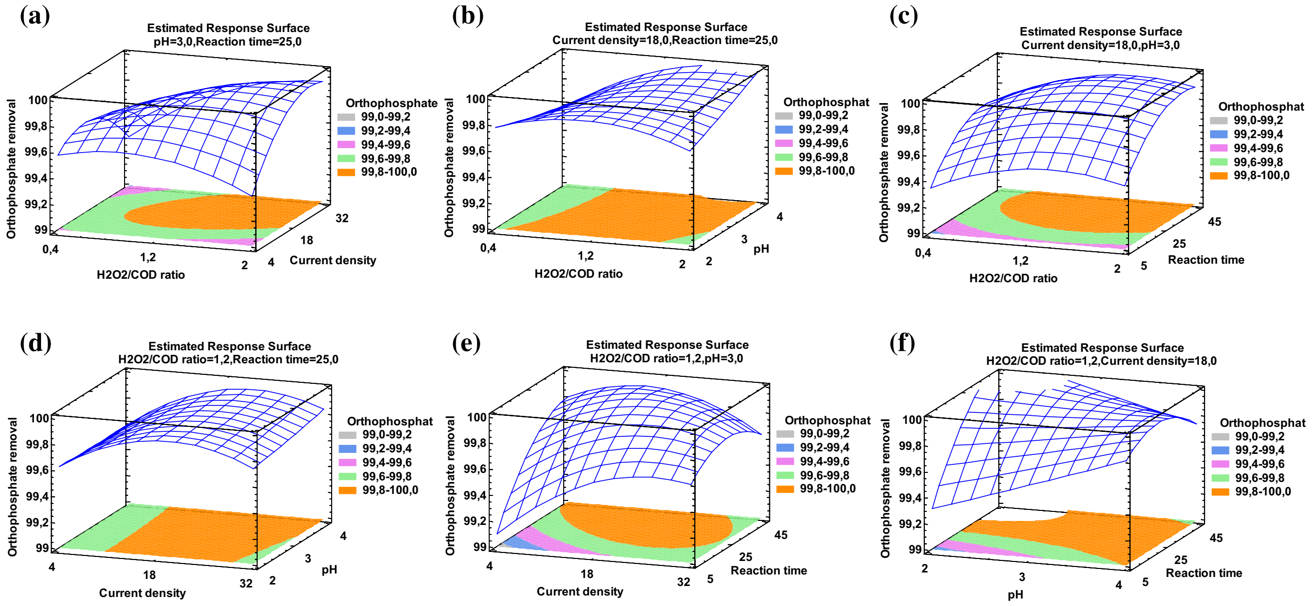


Fig. 5 Response surface graphs for the electro-Fenton process for orthophosphate removal efficiency **a** H_2O_2/COD ratio vs. current density, **b** H_2O_2/COD ratio vs. pH, **c** H_2O_2/COD ratio vs. time, **d** current density vs. pH, **e** current density vs. time, **f** pH vs. time

EC and EF processes and found 79.2% COD removal efficiency at current density of 15 mA/cm^2, natural pH (6–7) and 3000 mg/L H_2O_2 concentration. On the other hand, Davarnejad and Nikseresht (2016) studied dairy wastewater treatment using EF process and 93.9% COD removal efficiency and 97.3% color removal efficiency were achieved

Table 7 ANOVA results for the response surface quadratic model for color removal by Fenton and electro-Fenton processes

	Source	Sum of squares	Df	Mean square	*F* ratio	*P* value	Remark
Fenton	X_1	5.49582	1	5.49582	12.44	0.0042	Significant
	X_2	5.07591	1	5.07591	11.49	0.0054	Significant
	X_3	8.40156	1	8.40156	19.02	0.0009	Significant
	X_4	1.98018	1	1.98018	4.48	0.0558	Not significant
	X_1X_1	0.675413	1	0.675413	1.53	0.2400	Not significant
	X_1X_2	1.1561	1	1.1561	2.62	0.1317	Not significant
	X_1X_3	13.223	1	13.223	29.93	0.0001	Highly significant
	X_1X_4	30.2506	1	30.2506	68.47	<0.0001	Highly significant
	X_2X_2	0.0293537	1	0.0293537	0.07	0.8010	Not significant
	X_2X_3	9.49749	1	9.49749	21.50	0.0006	Significant
	X_2X_4	0.00573806	1	0.00573806	0.01	0.9112	Not significant
	X_3X_3	2.90185	1	2.90185	6.57	0.0249	Significant
	X_3X_4	35.4567	1	35.4567	80.25	<0.0001	Highly significant
	X_4X_4	11.4502	1	11.4502	25.92	0.0003	Significant
	Total error	5.30165	12	0.441804			
	Total (corr.)	132.208	26				
Electro-Fenton	X_1	21.6481	1	21.6481	39.65	<0.0001	Highly significant
	X_2	8.0416	1	8.0416	14.73	0.0024	Significant
	X_3	1.0037	1	1.0037	1.84	0.2001	Not significant
	X_4	0.0565401	1	0.0565401	0.10	0.7531	Not significant
	X_1X_1	20.5048	1	20.5048	37.56	0.0001	Highly significant
	X_1X_2	9.0	1	9.0	16.48	0.0016	Significant
	X_1X_3	3.11417	1	3.11417	5.70	0.0342	Significant
	X_1X_4	1.67469	1	1.67469	3.07	0.1054	Not significant
	X_2X_2	0.253762	1	0.253762	0.46	0.5083	Not significant
	X_2X_3	0.124538	1	0.124538	0.23	0.6415	Not significant
	X_2X_4	1.31584	1	1.31584	2.41	0.1465	Not significant
	X_3X_3	0.0707994	1	0.0707994	0.13	0.7250	Not significant
	X_3X_4	2.81048	1	2.81048	5.15	0.0425	Significant
	X_4X_4	0.248103	1	0.248103	0.45	0.5130	Not significant
	Total error	6.5518	12	0.545983			
	Total (corr.)	81.1978	26				

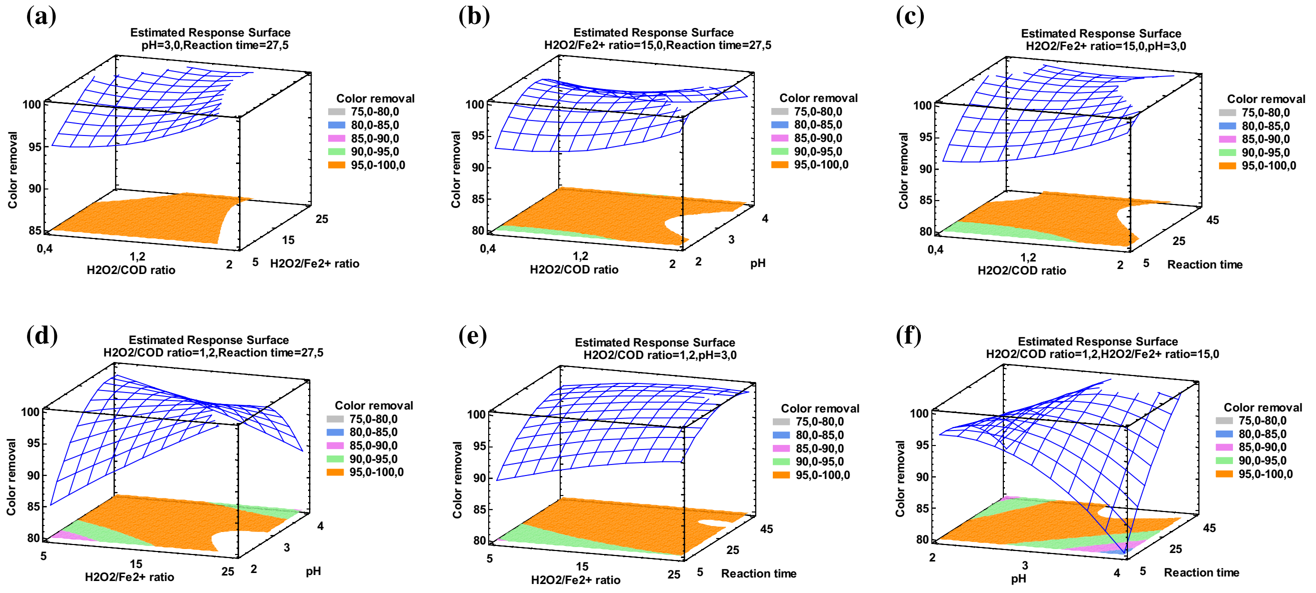


Fig. 6 Response surface graphs for the Fenton process for color removal efficiency **a** H_2O_2/COD ratio vs. H_2O_2/Fe^{2+} ratio, **b** H_2O_2/COD ratio vs. pH, **c** H_2O_2/COD ratio vs. time, **d** H_2O_2/Fe^{2+} ratio vs. pH, **e** H_2O_2/Fe^{2+} ratio vs. time, **f** pH vs. time

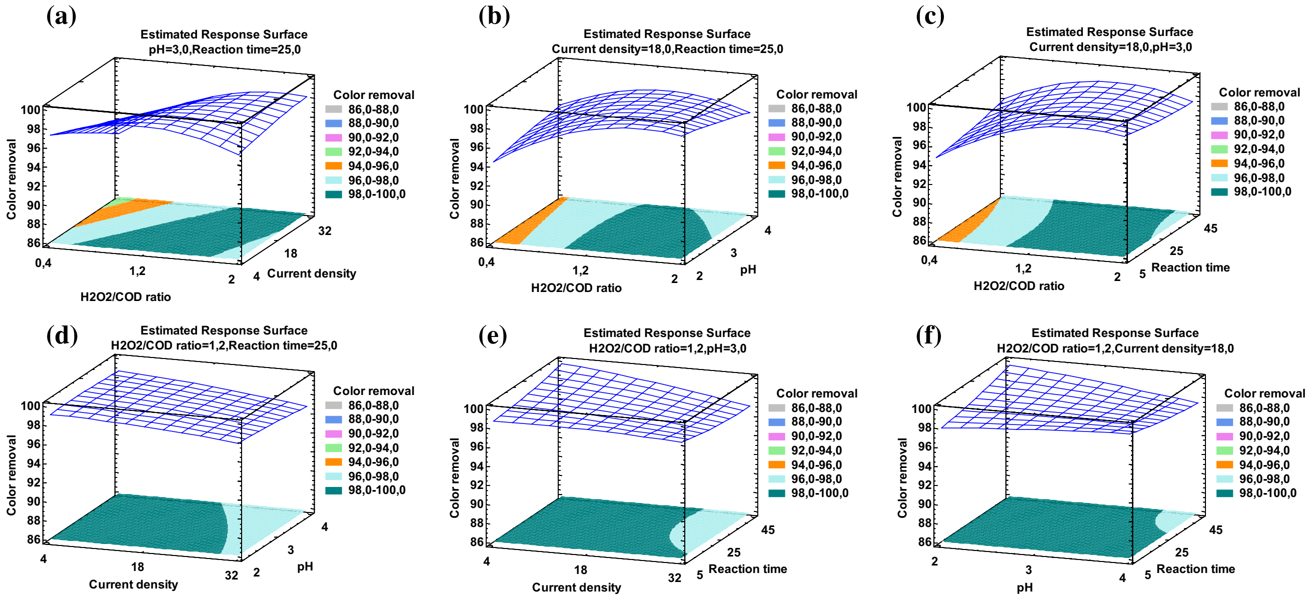
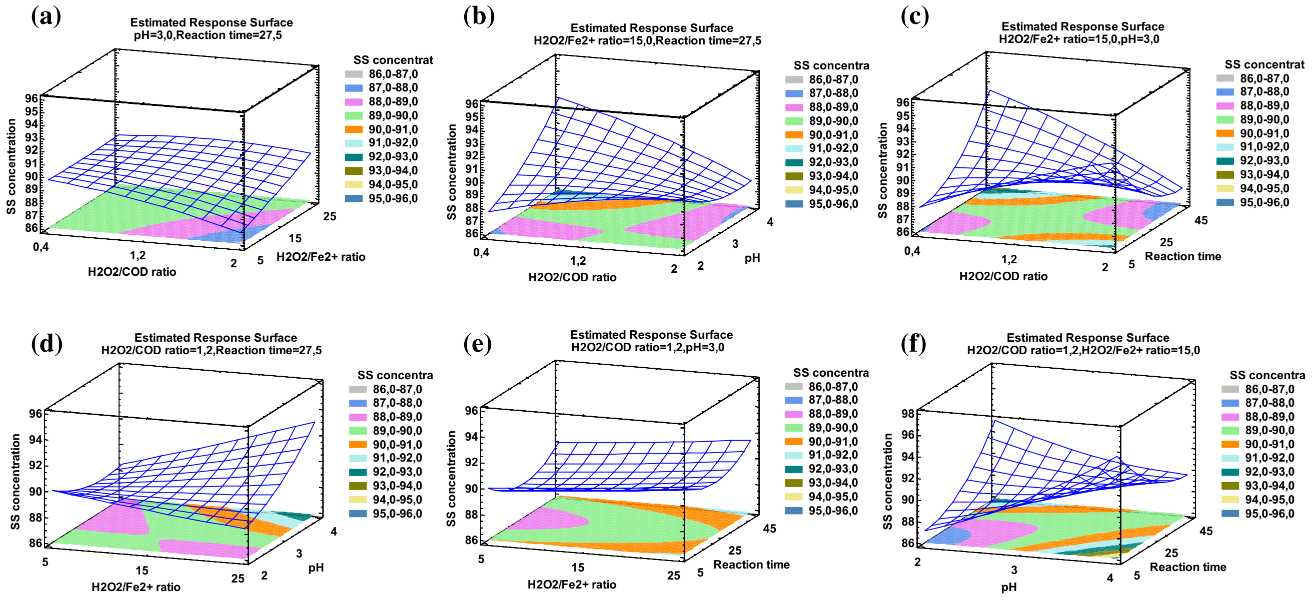


Fig. 7 Response surface graphs for the electro-Fenton process for color removal efficiency **a** H_2O_2/COD ratio vs. current density, **b** H_2O_2/COD ratio vs. pH, **c** H_2O_2/COD ratio vs. time, **d** current density vs. pH, **e** current density vs. time, **f** pH vs. time

Table 8 ANOVA results for the response surface quadratic model for SS removal by Fenton and electro-Fenton processes

	Source	Sum of squares	Df	Mean square	*F* ratio	*P* value	Remark
Fenton	X_1	0.445303	1	0.445303	1.64	0.2239	Not significant
	X_2	0.946144	1	0.946144	3.49	0.0862	Not significant
	X_3	0.728137	1	0.728137	2.69	0.1270	Not significant
	X_4	0.943662	1	0.943662	3.49	0.0865	Not significant
	X_1X_1	15.4896	1	15.4896	57.21	<0.0001	Highly significant
	X_1X_2	0.00108579	1	0.00108579	0.00	0.9506	Not significant
	X_1X_3	4.77248	1	4.77248	17.63	0.0012	Significant
	X_1X_4	8.50626	1	8.50626	31.42	0.0001	Highly significant
	X_2X_2	0.0745217	1	0.0745217	0.28	0.6094	Not significant
	X_2X_3	0.357783	1	0.357783	1.32	0.2727	Not significant
	X_2X_4	0.0368832	1	0.0368832	0.14	0.7185	Not significant
	X_3X_3	0.0758034	1	0.0758034	0.28	0.6064	Not significant
	X_3X_4	0.0538008	1	0.0538008	0.20	0.6637	Not significant
	X_4X_4	3.56133	1	3.56133	13.15	0.0035	Significant
	Total error	3.24924	12	0.27077			
	Total (corr.)	44.6016	26				
Electro-Fenton	X_1	2.73359	1	2.73359	3.18	0.0998	Not significant
	X_2	14.9636	1	14.9636	17.41	0.0013	Significant
	X_3	20.0756	1	20.0756	23.35	0.0004	Significant
	X_4	2.85041	1	2.85041	3.32	0.0936	Not significant
	X_1X_1	17.8717	1	17.8717	20.79	0.0007	Significant
	X_1X_2	0.882472	1	0.882472	1.03	0.3310	Not significant
	X_1X_3	9.64755	1	9.64755	11.22	0.0058	Significant
	X_1X_4	1.19017	1	1.19017	1.38	0.2621	Not significant
	X_2X_2	46.3739	1	46.3739	53.95	<0.0001	Highly significant
	X_2X_3	6.26501	1	6.26501	7.29	0.0193	Significant
	X_2X_4	1.58143	1	1.58143	1.84	0.2000	Not significant
	X_3X_3	45.4253	1	45.4253	52.84	<0.0001	Highly significant
	X_3X_4	0.528893	1	0.528893	0.62	0.4480	Not significant
	X_4X_4	28.5923	1	28.5923	33.26	0.0001	Highly significant
	Total error	10.3155	12	0.859623			
	Total (corr.)	144.745	26				


(a) Estimated Response Surface pH=3,0,Reaction time=27,5

(b) Estimated Response Surface H2O2/Fe2+ ratio=15,0,Reaction time=27,5

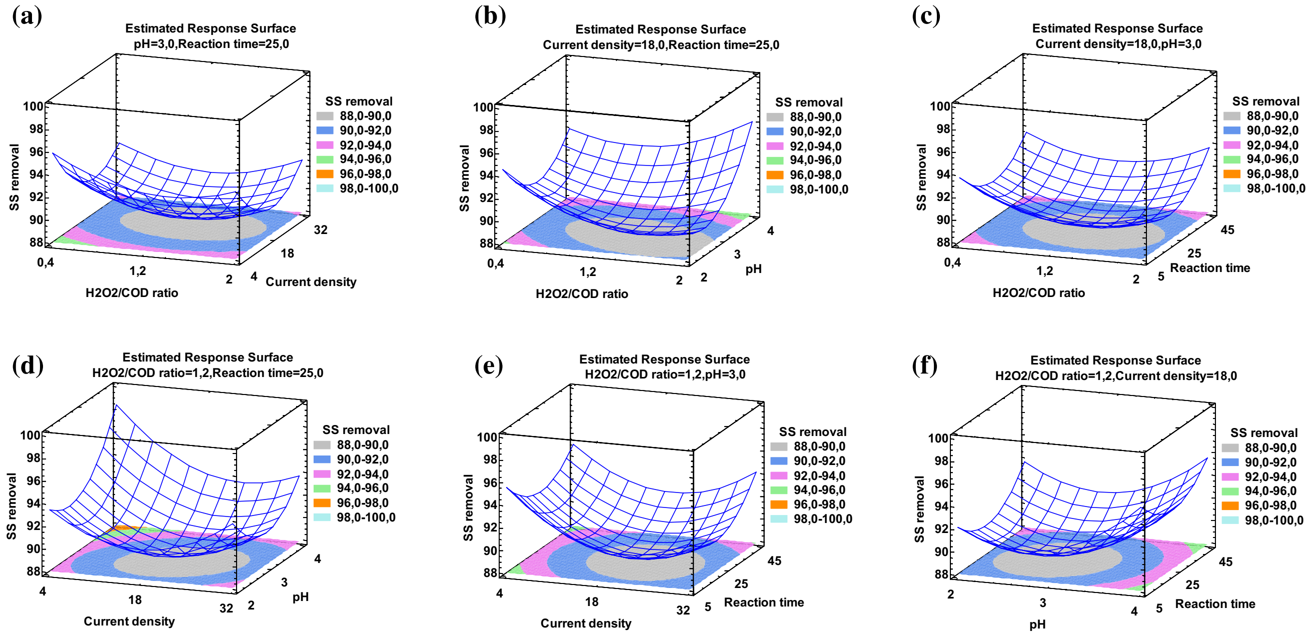
(c) Estimated Response Surface H2O2/Fe2+ ratio=15,0,pH=3,0

(d) Estimated Response Surface H2O2/COD ratio=1,2,Reaction time=27,5

(e) Estimated Response Surface H2O2/COD ratio=1,2,pH=3,0

(f) Estimated Response Surface H2O2/COD ratio=1,2,H2O2/Fe2+ ratio=15,0

Fig. 8 Response surface graphs for the Fenton process for SS removal efficiency **a** H_2O_2/COD ratio vs. H_2O_2/Fe^{2+} ratio, **b** H_2O_2/COD ratio vs. pH, **c** H_2O_2/COD ratio vs. time, **d** H_2O_2/Fe^{2+} ratio vs. pH, **e** H_2O_2/Fe^{2+} ratio vs. time, **f** pH vs. time


(a) Estimated Response Surface pH=3,0,Reaction time=25,0

(b) Estimated Response Surface Current density=18,0,Reaction time=25,0

(c) Estimated Response Surface Current density=18,0,pH=3,0

(d) Estimated Response Surface H2O2/COD ratio=1,2,Reaction time=25,0

(e) Estimated Response Surface H2O2/COD ratio=1,2,pH=3,0

(f) Estimated Response Surface H2O2/COD ratio=1,2,Current density=18,0

Fig. 9 Response surface graphs for the electro-Fenton process for SS removal efficiency **a** H_2O_2/COD ratio vs. current density, **b** H_2O_2/COD ratio vs. pH, **c** H_2O_2/COD ratio vs. time, **d** current density vs. pH, **e** current density vs. time, **f** pH vs. time

Table 9 Optimum operating conditions and model prediction and experimental results at optimum conditions

Process				
Factor	COD	Orthophosphate	Color	SS
Fenton				
H_2O_2/COD ratio (w/w)	1.9	1	2	2
H_2O_2/Fe^{2+} ratio (w/w)	5	25	25	25
Initial pH	4	2	2	4
Reaction time (min)	10	30	5.3	5
Model prediction results (%)	70	100	100	100
Experimental results (%)	65.5	92.5	90	95
Electro-Fenton				
H_2O_2/COD ratio (w/w)	2	0.9	1.2	2
Current density (mA/cm^2)	32	12.6	4.1	4.3
Initial pH	2.4	2	2	3.9
Reaction time (min)	45	45	45	5
Model prediction results (%)	75	100	100	100
Experimental results (%)	72	93	88	92

at 55.1 mA/cm^2 current density, 7.48 pH, 0.907 mL/L H_2O_2/Fe^{2+} molar ratio, and 86 min reaction time.

Conclusion

In this study, BBD and RSM were adopted to model and to optimize the performance of Fenton and EF processes, and COD, orthophosphate, SS, and color removal efficiencies by Fenton and EF process were determined. The RSM approach was also applied to find the optimum operating parameters for these responses. According to the ANOVA results, the RSM could be used to navigate the design space with high regression coefficient value above 91% for all the responses. COD removal efficiencies were 65.5 and 72% at the optimum operating conditions for Fenton and EF processes. The orthophosphate, SS, and color removal efficiencies were over 88, 92, and 92.5% for both processes, respectively. According to the overall results, it can be concluded that the EF process was found as a much suitable treatment method for dairy wastewater. Contrary to Fenton process, extra consumption of chemicals can be avoided by EF process. The results also indicated that the RSM was a powerful technique for optimizing the operational conditions of Fenton and EF processes for the removal of COD, orthophosphate, SS, and color from dairy industry wastewater.

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