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Taguchi optimization for the removal of high concentrations of phenol from saline wastewater using electro-Fenton process

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ABSTRACT

The efficiency of the electro-Fenton process (EFP) was assessed for phenol removal from saline wastewater. The influence of operational parameters, such as current density (4–16 mA/cm^2), pH (2–8), H_2O_2 concentration (75–300 mg/L), reaction time (20–100 min), salinity (1–4%), and phenol concentration (250–2,000 mg/L), on the EFP were evaluated using an L25 orthogonal array design. The best conditions for removal of phenol were determined by the Taguchi method and obtained optimum condition as H_2O_2 = 150 mg/L, pH 3, current density = 8 mA/cm^2, reaction time = 20 min, salinity = 2%, and phenol concentration = 250 mg/L. These findings indicated that the electro-Fenton process can be used as an effective approach for removing and mineralizing phenol at high concentrations from saline wastewater.

Keywords: Phenol; Saline wastewater; Electro-Fenton; Taguchi

1. Introduction

Many industries such as leather, tannery, oil refinery, petrochemical, pesticide, and herbicide produce the effluents with high concentration of salts, organic compounds, and other toxic ingredients [1,2]. Phenol is one of the common organic compounds found in saline wastewater generated by refinery and petrochemical industries [3]. Phenol is very irritating to the skin, eyes, and mucous membranes in humans, and Environment Protection Agency (EPA) has classified it in Group D. It is also known as a toxic compound damaging the inner organs such as kidney, liver, and paralysis of the central nervous system. It has other chronic adverse effects on human health such as anorexia,

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progressive weight loss, diarrhea, vertigo, salivation, and a dark coloration of the urine [4]. Due to these negative effects, EPA has established the discharge limit of 0.1 mg/L of phenol in receiving waters. Therefore, it has to be removed from industrial wastewater before being discharged into the water bodies [5].

The efficiency of treatment processes in saline wastewaters containing a high concentration of inhibitory and toxic compounds has become a challenge in current years. The removal of phenol from saline wastewater can be achieved by different methods, such as biological [3], adsorption [6], and membrane processes [7]. Recently, advanced oxidation processes (AOPs) have been reported to be an effective method for the treatment of wastewaters containing high level of phenol. Compared to the conventional methods, AOPs are one of the best options for complete destruction of phenolic compounds [8]. AOP has been defined as those oxidation processes based primarily on the generation of the hydroxyl radical. Among the different AOPs, electro-Fenton process (EFP) has been reported as a promising treatment method for wastewater treatment [9]. In this process, in the presence of Fe^{2+}, hydroxyl radicals are produced according to the Eq. (1). In addition, in this step, H_2O_2 acts as a source for producing hydroxyl radicals. The produced radicals can degrade different pollutants such as phenol. Moreover, based on Eq. (2), during the process, Fe^{3+} are reduced Fe^{2+} and it can continually produce Fe^{2+} [10]:

$$\begin{aligned}&Fe^{2+} + H_2O_2 \longrightarrow Fe^{3+} + OH^- + {}^{\bullet}OH \\ &\quad K = 63 - 76\,l/mol\,s\end{aligned} \tag{1}$$

$$\begin{aligned}&H_2O_2 + Fe^{3+} \longrightarrow Fe^{2+} + HO_2^{\bullet} + H^+ \\ &\quad K = 0.01 - 0.02\,l/mol\,s\end{aligned} \tag{2}$$

Producing a huge amount of sludge is one the most important disadvantages in the Fenton process. Therefore, electro-Fenton (EFP) is a good alternative for solving this problem. In the EFP, Fe^{2+} is produced by electrical power (according to Eq. (3)):

$$Fe^{3+} + e^- \longrightarrow Fe^{2+} \tag{3}$$

EFP is classified in four categories: type (1) Fe^{2+} ion and H_2O_2 are generated electrochemically using a sacrificial cast iron anode and cathode electrodes, respectively; type (2) H_2O_2 is externally added while Fe^{2+} ion is generated by sacrificial cast iron anode; type (3) Fe^{2+} is externally added while H_2O_2 is provided by dispersion of oxygen on cathode surface; and type (4) Fenton reagents are used in this type of EFP [11,12]. Due to the high concentration of phenol, the EFP requires more generation of hydroxyl radicals. Therefore, second type of EFP was used for continuous production of Fe^{2+} and H_2O_2 during the process.

Several key parameters, such as pH, reaction time, current density, peroxide dosage, phenol concentration, and electrolyte concentration, play an important role in EFP. As the number of factors and their levels increases, the use of statistical method is essential to optimize the process conditions. Taguchi methods are reported as statistical methods to reduce the number of test, required time, and experimental cost. Moreover, the contribution percentage of each parameter to the system can be determined by this method [13]. The aim of this study was to investigate and optimize phenol removal from saline wastewater using EFP by Taguchi statistical approach.

2. Materials and methods

2.1. Chemicals

All chemicals, including hydrogen peroxide, phenol with 99.5% purity, 4-aminoantipyrine, ferry potassium cyanide, buffer phosphate, and sulfuric acid, used for experiments were purchased from Merck Co. The synthetic wastewater was prepared by dissolving a certain volume of phenol, hydrogen peroxide, and NaCl in distilled water. Sulfuric acid (1 N) and sodium hydroxide (1 N) were used to adjust the pH of the solutions.

Analytical measurement of phenol concentration was performed using the colorimetric 4-aminoantipyrine method (5530D standard methods by an UV–visible spectrophotometer (HACH DR5000) at 500-nm wavelength [14].

2.2. Electro-Fenton reactor

All experiments were conducted at laboratory temperature and in a batch Plexiglas laboratory-scale electro-Fenton reactor with a volume of 1 L equipped with 6 cast iron electrodes connected to direct current power supply (PS-305D). The cathode and anode with the same dimensions (150 mm × 50 mm × 2 mm) in three pairs were used in this study. The distance maintained between plate electrodes was 1 cm. The solution was mixed continuously using magnetic stirrer at 200 rpm. The scheme of the experimental EFP is shown in Fig. 1.

2.3. Designation and optimization of electro-Fenton experiment

Minitab 16 Statistical software was used for the design of six key parameters: current density, initial

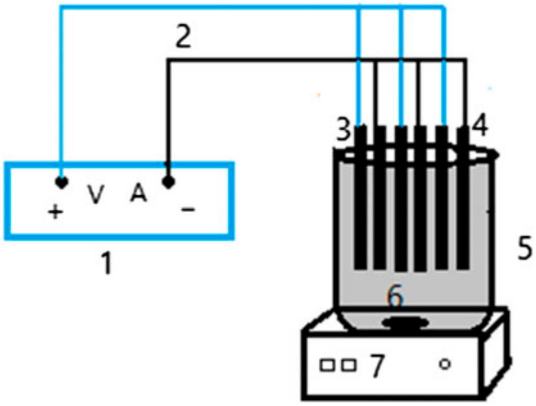

Fig. 1. Schematic EF setup.
Notes: (1) power supply, (2) weirs, (3) anode, (4) cathode, (5) electro-Fenton cell, (6) magnet, and (7) stirrer.

phenol concentration, salinity, pH, H_2O_2 dose, and contact time. These parameters were taken into account in the design of experiments based on the Taguchi method. Each parameter was configured at five levels (Table 1). All experiments were run in duplicate.

The signal-to-noise (S/N) ratio was applied to evaluate the experimental data. Among the three obtained values of S/N ratio, the highest value was selected as optimum condition (Eq. (4)) [13]:

$$\text{The signal-to noise } = -10\,\log_{10}\left[\frac{1}{n}\sum\left(\frac{1}{\mathrm{MRE}_i}\right)^2\right] \tag{4}$$

Maximum removal efficiency (MRE) (%) was defined as the phenol removal efficiency, and it was calculated according to the following equation [13].

$$\mathrm{MRE}\,(\%) = \frac{C_0 - C_\mathrm{e}}{C_0} \times 100 \tag{5}$$

where C_0 and C_e are initial and final concentrations of phenol, respectively.

To conclude the optimum conditions for the phenol removal experiments, relationship between each parameters and the percentage (%) contribution of their on the phenol removal in EFP, the analysis of mean (ANOM), and analysis of variance (ANOVA) were used.

Primarily, the mean of the S/N ratio ($M_{\mathrm{S/N}}$) for each factor at a certain level was determined (Eq. (6)). Eq. (6) [13]:

$$M_{\mathrm{S/N}} = M^{\text{Level}-i}_{\text{Factor}-I-_{\mathrm{S/N}}} = \frac{1}{n_{Ii}}\sum_{j-1}^{n_{Ji}}\left[\left(\frac{S}{N}\right)^{\text{Level}-i}_{\text{Factor}-I}\right]_j \tag{6}$$

Afterward, the highest value of $M_{\mathrm{S/N}}$ as better characteristics was selected as optimum conditions of each parameter that was assessed in Taguchi method. The percentage influence of each factor on the phenol elimination using EFP was found via substituting the factorial sum of squares ($\mathrm{SS_F}$) $\left(\mathrm{SS_F} = mn/\mathrm{L}\sum_{k-1}^{L}\left(\overline{\mathrm{MRE}}^{F}_{K} - \overline{\mathrm{MRE}_r}\right)^2\right)$, the total sum of square ($\mathrm{SS_T}$) $\left(\mathrm{SS_T} = \sum_{j=1}^{m}\left(\sum_{j=1}^{n}\mathrm{MRE}_i^2\right) - mn\left(\overline{\mathrm{MRE}_T}\right)^2\right)$, and the variance of error (V_E) $\left(\left(V_\mathrm{E} = \frac{\mathrm{SS}_\tau - \sum_{F-A}^{D}\mathrm{SS_F}}{m(n-1)}\right)\right)$ was calculated according to the following equation:

$$P\,(\%) = \frac{\mathrm{SS_F} - (\mathrm{DOF}_A \times V_\mathrm{E})}{\mathrm{SS_T}} \times 100 \tag{7}$$

3. Results and discussion

3.1. Optimum conditions

The efficiency of each test was calculated according to the Eqs. (1) and (2). Table 2 shows the S/N ratio of each experiment from different arrangements of factors with the boldface referring to the maximum value of S/N ratio among the 25 run. The best laboratory conditions for phenol removal are as follows: current

Table 1
Control factors and their levels used for design of experiments in this study

Designation	Description	Level 1	Level 2	Level 3	Level 4	Level 5
A	Current density (mA/cm^2)	0	4	8	12	16
B	Initial phenol concentration (mg/L)	250	500	1,000	1,500	2,000
C	Salinity (%)	0	1	2	3	4
D	pH	2	3	4	6	8
E	H_2O_2 dose (mg/L)	0	75	150	225	300
F	Contact time (min)	20	40	60	80	100

Table 2
The S/N ratio of each experiment from different arrangements of factors

Run	*A*	*B*	*C*	*D*	*E*	*F*	MRE (%) average	S/N
1	1	1	1	1	1	1	31.5	−3
2	1	2	2	2	2	2	47.5	2/8
3	1	3	3	3	3	3	41.5	1/2
4	1	4	4	4	4	4	28.5	−4/7
5	1	5	5	5	5	5	20.5	−23/2
6	2	1	2	3	4	5	36.5	−0.5
7	2	2	3	4	5	1	32.5	−2/5
8	2	3	4	5	1	2	29.5	−4/2
9	2	4	5	1	2	3	77.5	8/7
10	2	5	1	2	3	4	24	−9/5
11	3	2	4	1	2	4	60.5	5/7
12	**3**	**1**	**3**	**2**	**3**	**1**	**91**	**11/6**
13	3	3	5	2	4	5	59	5/4
5	3	4	1	3	5	2	33	−2
15	3	5	2	4	1	3	66	7
16	4	1	4	2	5	3	54.5	4/5
17	4	2	5	3	1	4	53.5	4/2
18	4	3	1	4	2	5	48.5	3/16
19	4	4	2	5	3	1	35.5	−1
20	4	5	3	1	4	2	51.5	4
21	5	1	5	4	3	2	56	5
22	5	2	1	5	4	3	20.5	−17
23	5	3	2	1	5	4	43.5	2
24	5	4	3	2	1	5	61.5	6
25	5	5	4	3	2	1	50	4

Note: Bold values indicate the optimum conditions.

Table 3
Response table for $M_{S/N}$ ratios for the tested factors and corresponding levels

Level	*A*	*B*	*C*	*D*	*E*	*F*
1	−5.364	**4.614**	−5.756	1.934	0.48	**2.302**
2	1.08	1.804	1.974	**4.768**	1.048	−0.242
3	**2.13**	1.288	**2.808**	1.442	**4.784**	1.474
4	1.442	1.458	2.138	−2.69	0.696	1.362
5	1.314	−8.018	0	−4.308	−0.614	−3.74

Note: Bold values indicate the optimum conditions.

density: 8 mA/cm^2, initial concentration of phenol: 250 mg/L, electrolyte: 3%, the pH of the solution: 4, H_2O_2 concentration: 300 mg/L, and reaction time: 40 min. The S/N ratio is determined to be (or equals to) 11.6 under these conditions. Table 3 indicates the response of $M_{S/N}$ ratios for the studied factors and corresponding levels. The highest $M_{S/N}$ ratio was selected as the optimum condition for each parameter in Table 3. Different results were obtained at laboratory conditions compared to Taguchi method. Accordingly, pH, reaction time, H_2O_2 concentration decreases from test 12, and the value of S/N ratio was determined to be 14.2. These results showed that the higher efficiency of EFP obtained at lower reaction time, pH, and H_2O_2 concentration.

3.2. Current density

To appreciate the influence of current density on the efficiency of EFP in removing phenol from salinity wastewater, several current densities in the range 4–16 mA/cm^2 were applied. Fig. 2 illustrated the results based on the value of S/N ratio, which indicated a positive influence of current density on the process performance, especially at higher values. Based on illustrated data in Fig. 2, an increase in the current density from 4 to 8 mA/cm^2 led to an increase in the S/N ratio from 1.08 to 2.13, respectively. The higher phenol removal at the higher current density can be explained as follows: This increase can be attributed to the increase in electrical current between the Fe electrodes that enhanced anodic scarification and the generation of Fe^{2+} ions, which is a main element required to run the Fenton reaction (Eq. (3)). The reaction between Fe^{2+} and H_2O_2 is accelerated and increase the amount of Fe^{3+} available in solution, consequently. In turn, the higher generation of $^{\cdot}OH$ resulted in increased phenol removal. Then, the hydroxyl radical can degrade the phenol and improve the process efficiency.

Phenol removal efficiency was decreased in EFP with a further increase in current density. The decrease in process efficiency at higher current density can be attributed to the interfere reactions (Eqs. (8) and (9)) that both reactions exhibited an inhibitory effect on generation of hydroxyl radical [9,10]. These results are comparable with some researches showing the COD removal efficiency was 85.07% at 80 mA/cm^2 compared with 92.06% at 50 mA/cm^2 [15]:

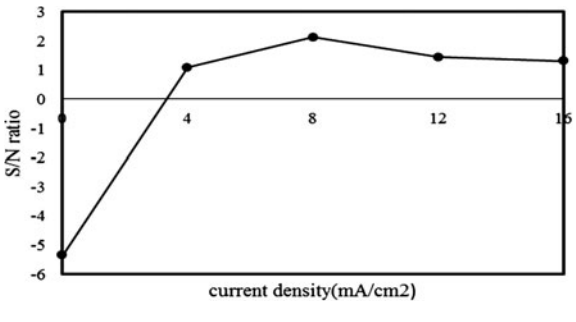

Fig. 2. The effect of current density on S/N ratio in phenol removal.

$$2\mathrm{H_2O} \longrightarrow 4\mathrm{H}^+ + \mathrm{O_2} + 4\mathrm{e}^- \quad (8)$$

$$2\mathrm{H}^+ + 2\mathrm{e}^- \longrightarrow \mathrm{H_2} \quad (9)$$

3.3. Initial phenol concentration

In EFP, the initial pollutant concentration is one of the important factors. As shown in Fig. 3, the S/N ratio for phenol concentration was decreased from 4.614 to −8.018 with the increase in concentration from 250 to 2,000 mg/L, respectively. This can be due to the formation of intermediates at high initial phenol concentration. These compounds act as hydroxyl radical scavengers which lower the EEP efficiency in term of phenol removal [16]. These data are comparable with other studies [17–19].

3.4. Salinity

The effect of salinity (NaCl) percentage on phenol removal was investigated in the range of 0–4% in EFP. As shown in Fig. 4, when salinity is 2%, the S/N removal of phenol is 2.808. Lower S/N ratio was achieved when the salinity exceeded 2%. Therefore, salinity value of 2% selected as the optimum condition. The current density increased with the further presence of anions and cations at high concentration of NaCl. Accordingly, the higher production of electro-generated hydrogen peroxide results in the higher concentrations of radical hydroxyl, and consequently higher process efficiency in phenol removal. On the other hand, at NaCl concentration higher than 2%, the side reaction could consume hydrogen peroxide and trap hydroxyl radical according to the following reactions [16,17]:

$$\mathrm{Cl}^- + {}^{\bullet}\mathrm{OH} \longrightarrow {}^{\bullet}\mathrm{Cl} + \mathrm{H_2O} \quad (10)$$

$${}^{\bullet}\mathrm{Cl} + \mathrm{H_2O_2} \longrightarrow {}^{\bullet}\mathrm{OOH} + \mathrm{Cl}^- + \mathrm{H}^+ \quad (11)$$

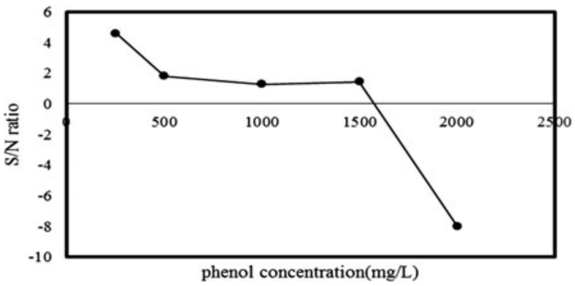

Fig. 3. The effect of phenol concentration on S/N ratio in phenol removal.

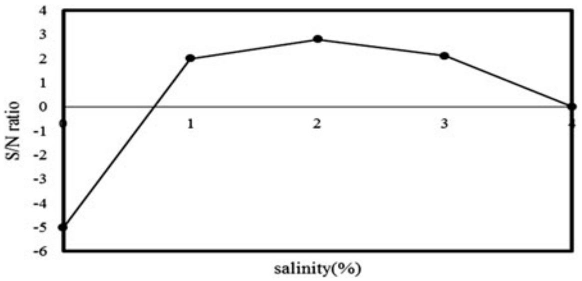

Fig. 4. The effect of salinity on S/N ratio in phenol removal.

3.5. Solution pH

Fig. 5 shows the effect of pH on EFP process. The results indicated that the maximum phenol removal was attained at pH 3 with S/N ratio of 4.768. At either lower or higher pH values, the EFP efficiency was decreased. Previous studies reported that EFP occurs at acidic conditions [3,9,16]. At pH of 3 and in the presence of ferrous ion as Fenton's reaction catalyst, hydroxyl radical and peroxide hydrogen are present. When pH increases, the stability of hydrogen peroxide decreases. Furthermore, the increase in the formation of Fe^{3+} under alkaline conditions and the reaction of $\mathrm{H_2O_2}$ and Fe^{3+} lead to more consumption of peroxide hydrogen (Eq. (12)). At pH > 3, creation of the hydroxyl radical was small [15]:

$$\mathrm{Fe}^{3+} + \mathrm{H_2O_2} \longrightarrow \mathrm{Fe}^{2+} + \mathrm{HO}_2^{\bullet} + \mathrm{H}^+ \quad (12)$$

The decay of EFP efficiency at pH < 3 can be explained by stability of $\mathrm{H_2O_2}$ during the formation of $\mathrm{H_3O_2^+}$ via reaction (Eq. (13)):

$$\mathrm{H_2O_2} + \mathrm{H}^+ \longrightarrow \mathrm{H_3O_2^+} \quad (13)$$

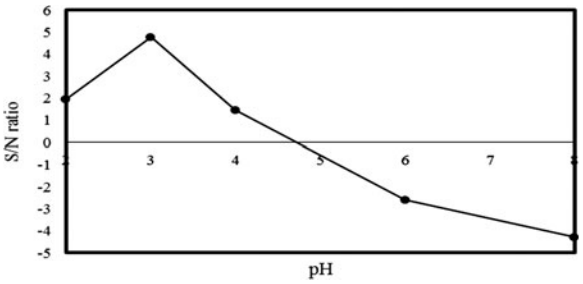

Fig. 5. The effect of pH on S/N ratio in phenol removal.

The efficiency decreases due to lower oxidation potential of *in situ* formed products such as $HO_2^{\bullet}$ compared to $OH^{\bullet}$ [12].

3.6. H_2O_2 concentration

Fig. 6 shows the effect of H_2O_2 concentration on S/N ratio in phenol removal. Results show the optimum H_2O_2 dosage to be 150 mg/l with S/N ratio of 4.784. Increased H_2O_2 concentration decreased the removal efficiency. One possible reason could be explained by considering the Eqs. (14) and (15). In these side reaction, hydroxyl radical act as scavenger matter of H_2O_2. Furthermore, consumption of H_2O_2 leading to its exit from reaction and production of radical hydroxyl decreased while the production amount of $HO_2^{\bullet}$ increased. It is well known that the hydroperoxyl radical with ($E^0 = 1.65$ eV) has lower oxidation potential than that of OH ($E^0 = 2.8$ eV) [9,20]. Therefore, in optimum conditions, the EFP was limited for removing phenol in high H_2O_2 dosage. This result is accordance with the finding of petroleum wastewater treatment conducted by Yavuz et al. [21].

$$H_2O_2 + {}^{\bullet}OH \longrightarrow HO_2^{\bullet} + H_2O \tag{14}$$

$${}^{\bullet}OH + HO_2^{\bullet} \longrightarrow O_2 + H_2O \tag{15}$$

3.7. Reaction time

Results of phenol degradation based on the S/N ratio and as a function of reaction time are plotted in Fig. 7. As can be seen in Fig. 7, after 20 min of reaction time, the S/N ratio was 2.302. By further increase in contact time, no significant improvement was observed on phenol removal. Under this condition, the percentage contribution of each factor is in suitable point. In other words, after the completion of 20-min oxidation reaction by increasing the reaction time and consumption of H_2O_2, the production of OH radical gradually slowed down [9,22]. These results are consistent with other studies reporting the maximum leachate treatment occurs in the first 30 min of reaction time and later it gradually slowed down [10].

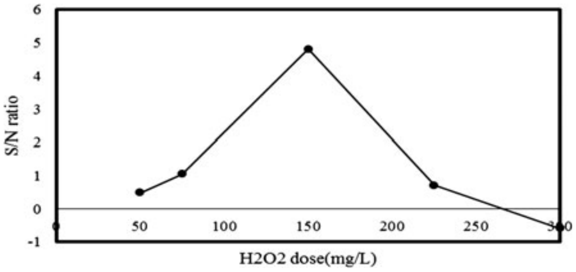

Fig. 6. The effect of H_2O_2 dose on S/N ratio in phenol removal.

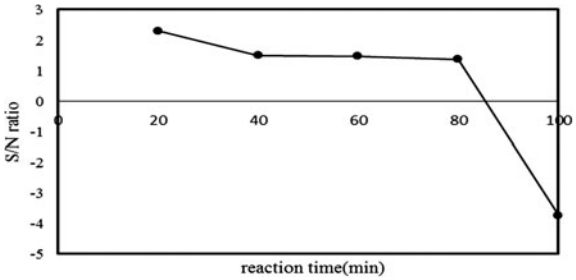

Fig. 7. The effect of reaction time on S/N ratio in phenol removal.

3.8. Percentage of contribution

The percentage contribution of each parameter on phenol removal from saline wastewater in EFP was evaluated using ANOVA analyses. The data from the ANOVA analyses are summarized in Table 4. Referring to Table 4, the percentage contribution of solution pH, reaction time, initial phenol concentration, current density, salinity, and hydrogen peroxide dose were 16.66, 3.08, 25.25, 29.12, 18.3, and 7.6%, respectively. Results indicated that the EFP efficiency in phenol removal from saline wastewater was considerably influenced via the current density in comparison with other selected parameters. On the other hand, the reaction time has the lowest effect on the EFP efficiency. Therefore, the EFP efficiency in phenol removal depends highly on the current density.

3.9. COD removal

To investigate the EFP efficiency in phenol mineralization, COD removal was also measured. Fig. 8 depicts the EFP efficiency in mineralization and degradation of phenol in the optimum conditions (current density 8 mA/cm^2, initial phenol of 250 mg/L, hydrogen peroxide of 150 mg/L, 2% salinity, pH 3, and reaction time 20 min). As shown in Fig. 8, the efficiency of phenol degradation was around 96% after 20 min, and at same time, COD removal (mineralization efficiency) was lower than 62%. Due to the fact that during phenol removal processes various organic intermediates are produced, they need longer time for degradation by EFP. Therefore, produced intermediates can reduce the EFP efficiency in terms of COD

Table 4
Determination of percentage contribution of each factor on phenol removal

Factors	DF	Seq. SS	Adj. SS	Adj. SS	*P* (%)
Current density (mA/cm^2)	4	4.9853	4.98530	4.98530	29.12
Phenol concentration (mg/L)	4	4.3231	4.32307	1.08077	25.25
Salinity (%)	4	3.1336	3.13358	0./78	18.3
pH	4	2.8515	2.85149	0.71287	16.66
H_2O_2 dose (mg/L)	4	1.3004	1.30040	0.32510	7.60
Reaction time (min)	4	0.5268	0.52683	0.52683	3.08

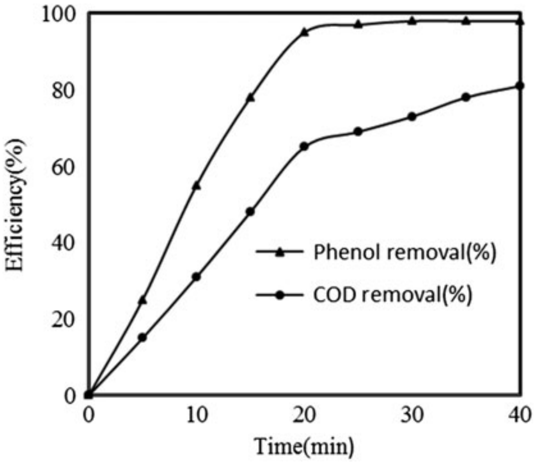

Fig. 8. COD and phenol removal in the optimum condition (current density 8 mA/cm^2, initial phenol concentration 250 mg/L, dose of hydrogen peroxide 150 mg/L, salinity 2%, pH 3).

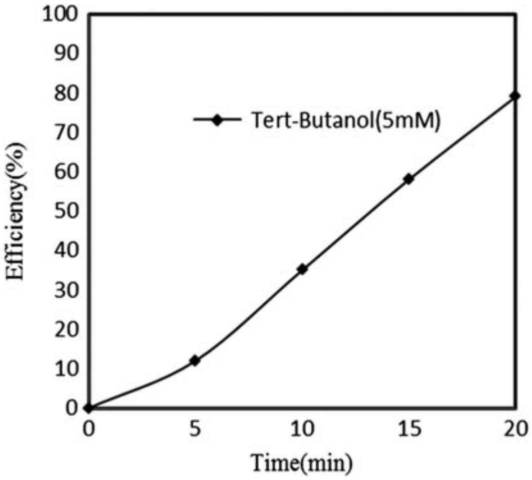

Fig. 9. The effect of the OH radical scavenger tert-butanol (current density 8 mA/cm^2, initial phenol concentration 250 mg/L, dose of hydrogen peroxide 150 mg/L, salinity 2%, pH 3).

removal [23]. Moreover, this result is consistent with the findings of Moussavi et al. study on formaldehyde removal at high concentrations using EFP [12].

3.10. Effect of tert-butanol

A radical scavenger is a chemical substance added to a reactor for removing impurities from the reactor. Moreover, it can react with the free radicals in the oxidation process and slow the oxidation rate. Therefore, radical scavenger can be applied as an indicator of hydroxyl radical reactions [24,25]. Tert-butanol reacts with hydroxyl radical, lowers the OH radical concentration, and consequently results in lower phenol removal. In this study, tert-butanol (5 mM) was used as a hydroxyl radical scavenger under optimum conditions. Results indicated that the 78% of phenol removal was obtained after 20 min in the presence of tert-butanol in Fig. 9. However, when tert-butanol is not present in electro-Fenton reactor, the removal efficiency increased to 92% at the same reaction time. This is due to the scavenging effect of hydroxyl radicals by tert-butanol which lowers the concentration of these radicals and consequently lowers the phenol removal. These data confirmed the key role of hydroxyl radical in EFP.

4. Conclusion

Taguchi method was applied to obtain the optimal experimental conditions for EFP in phenol removal at high concentrations from salinity wastewater. Optimum conditions for removing phenol were found on current density 8 mA/cm^2, reaction time 20 min, initial phenol concentration 250 mg/L, hydrogen peroxide dosage 150 mg/L, salinity 2%, and pH 3. According to optimization method, the current density had a significant influence on phenol removal in EFP (29.12%), and reaction time had lower contribution (3.08%). These data suggested that the optimization of EFP via Taguchi method could be useful in terms of cost and efficiency.

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