



Electro-Fenton process based on sacrificial Iron electrode for Ponceau 4R removal

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Abstract Dyes are common pollutants that are found in industrial wastewater including food, pharmaceutical, and cosmetic industries. This study aimed to degrade Ponceau 4R (P4R) by the electro-Fenton (EF) process based on iron electrodes in the aqueous solutions. For the EF process, a 900 mL glass reactor containing two iron electrodes which connected to a power supply was used. The effects of five experimental variables namely applied voltage (5–20 V), electrolyte concentration (0–0.2 mol L⁻¹ Na₂SO₄), initial P4R concentration (50–250 mg L⁻¹), pH of the solution (2–6), and H₂O₂ content (0.033–0.100 %v/v) were investigated in four levels and optimized by Taguchi method (OA₁₆). The residual concentrations of P4R after the EF process were spectrophotometrically determined at 505 nm in the time range of 0–90 min with time intervals of 10 min and the removal efficiencies were obtained. Based on the results, the optimum conditions were obtained as pH=3, voltage=20 V, H₂O₂=0.033 % v/v, C_{Na₂SO₄}=0.1 M and the C_{P4R}=50 mg L⁻¹. Under the optimum conditions, about 100% of P4R was removed during 10 min. The

energy consumed in the EF process was calculated as 19.82 KWh/m³ during 10 min. The results obtained good removal efficiencies for the degradation of P4R in the real aqueous solutions introducing it as an efficient decolorization method for treatment of P4R polluted waters.

Keywords Dye · Electro-Fenton · Iron electrodes · Ponceau 4R · Removal · Electrochemical peroxidation

1 Introduction

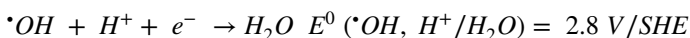
The chemical pollution of water resources is known as one of the most common environmental concern (Behrouzeh et al., 2022; Sarfo et al., 2023). Industrial and synthetic dyes are considered important sources of pollution that can change the chemical and physical properties of water and show adverse effects (Bektar et al., 2020). Ponceau 4R (P4R, also known as Cochineal Red A, C.I. Acid Red 18, Brilliant Scarlet 3R, Strawberry Red, New coccine, E124) (Ghalkhani et al., 2022; Santana et al., 2015) is a synthetic azo dye with N=N bond which has been broadly utilized in various industries including food, drinks, dairy and bakery products (Thiam et al., 2015), coloring material in medicines and cosmetic industries (Demirhan et al., 2021). The presence of N=N azo group and aromatic ring in its structure make it harmful to human health. P4R is currently not approved in some

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countries and it is listed as a banned dye by some authorities (Thiam et al., 2016). In general, the methods used for the treatment of industrial wastewaters containing P4R include physical, chemical and biological methods, which can be mentioned as examples of adsorption (Sojoudi et al., 2016; Toutouchi et al., 2020), biological methods (Masarbo et al., 2019), catalytic ozonation (Nguyen et al., 2023), advanced oxidation processes (Askarniya et al., 2022; Thiam et al., 2016), Fenton (Moghadam & Nori Kohbanan, 2018); photo-Fenton (Mossmann et al., 2019), electro-Fenton (Hafaiedh and Bellakhal., 2016), ozonation (Benincá et al., 2013), electro-coagulation and electro-Fenton (Kabir Suhan et al., 2020).

Nowadays, advanced oxidation processes (AOPs) are successfully used to treat such wastewaters, due to their high effectiveness, non-toxicity of reagents, simple operation, and their potential to degrade and remove organic pollutants (Januário et al., 2021; Rosales et al., 2012; Panizza & Cerisola, 2001; Anotai et al., 2006; Gholami Akerdi et al., 2017; Sohrabi et al., 2017; Yuan et al., 2009). AOPs are considered promising alternative technologies to degrade recalcitrant compounds through the in situ production of strong oxidants especially hydroxyl radicals ($\cdot\text{OH}$) (Le et al., 2022), whose average lifetime is estimated to be only a few nanoseconds. This radical is produced in situ in the reaction medium by chemical, photochemical, and electrochemical methods and can be applied to treat non-biodegradable organic compounds until their mineralization (Andreozzi et al., 1999). The hydroxyl radical is considered the strongest oxidizing agent (Eq. 1) after fluorine ($E^0(\text{F}_2/\text{HF}) = 3.05 \text{ V/SHE}$; $E^0(\text{F}_2/\text{F}^-) = 2.87 \text{ V/SHE}$) (Deng & Zhao, 2015).



This non-selective radical oxidizes most of the organic and organometallic pollutants and converts them to less complex and less harmful intermediate products including mono- and dehydroxylated derivatives. At sufficient reaction time and under proper conditions, the continued reaction of these compounds with $\cdot\text{OH}$ radicals lead to aromatic ring cleavage and to complete mineralization into harmless products including CO_2 , H_2O , and inorganic ions which are the end-product of chemical oxidation.

Therefore, the use of $\cdot\text{OH}$ radicals for the degradation of industrial pollutants seems very promising without bearing secondary pollution to the environment.

The Fenton process is the simplest AOP, in which the use of ferrous ions (Fe^{2+}) and hydrogen peroxide (H_2O_2) causes the production of a very active $\cdot\text{OH}$ radical. Fenton process has some advantages such as both iron and H_2O_2 are relatively inexpensive and safe, and the process is technologically simple. In the Fenton process, the Fe^{2+} is converted into Fe^{3+} after the reaction with H_2O_2 , and the produced trivalent iron can precipitate and produce sludge. So, in the conventional Fenton process, a high content of Fe^{2+} is needed and a large amount of iron sludge is produced that needs to be spent for disposal. To answer this issue, the electro-Fenton (EF) process, as an advanced form of the classic Fenton process, is used by a combination of two processes, Fenton and electrocoagulation, which aims to achieve greater efficiency by using an iron electrode and electricity source to supply the Fe^{2+} ions required for the reaction.

Over the years, several electrochemical advanced oxidation processes (EAOPs) have been used to treat water contaminated by organic pollutants (Thiam et al., 2015). The EF process is classified into four groups: In the first one, Fe^{2+} and H_2O_2 are produced electrically at the surface of the sacrificial anode and the cathode electrodes, respectively. In the second type, H_2O_2 is added to the reactor manually, while a sacrificial iron anode electrode is used as the electrical source of Fe^{2+} (Electrochemical peroxidation process). In the third type, ferrous iron is added to the reactor and H_2O_2 is produced through the diffusion of O_2 gas on the surface of the carbon cathode. In the fourth type, both Fenton's reagents (H_2O_2 and Fe^{2+})

are added externally to the reaction medium (Fered Fenton Process) (Umar et al., 2010).

In the first and second type of EF processes, the iron required for the EF process was supplied by the oxidation of the used iron electrode. With increasing iron production in the treated solution, the consumption of H_2O_2 increases. With the occurrence of these reactions, the production of hydroxyl radical increases that causes an increase in the efficiency of contaminant removal (Aaron et al., 2001).

According to the advantages of electrochemical processes, in this research, the EF process using two iron electrodes was used to degrade the P4R dye pollutant. The experimental variables influencing the removal efficiency (including applied voltage, electrolyte concentration, P4R concentration, pH of solution, and H₂O₂ content) were studied and optimized by Taguchi method and the amount of energy consumed in this method was investigated to develop a practical EF method for P4R removal with the minimum of experiments.

2 Experimental

2.1 Chemicals and reagents

The chemical reagents, including nitric acid (HNO₃, 65 wt. %), sodium hydroxide (NaOH), sodium sulfate (Na₂SO₄), hydrogen peroxide (H₂O₂, 30 wt. %) and Ponceau 4R (C₂₀H₁₁N₂O₁₀S₃Na₃, trisodium; 7-hydroxy-8-[(4-sulfonatophthalen-1-yl) diazenyl] naphthalene-1,3-disulfonate; Mw = 604.48 g mol⁻¹) were bought with high purity from Merck (Darmstadt, Germany). All of the working solutions were prepared using double distilled water (DW).

2.2 Instruments and equipment

Absorption studies were performed by an Agilent UV–Vis spectrophotometer (Cary 60, USA). A digital WTW pH meter (Inolab7110, Germany) with a combined glass electrode was applied to control the pH of the solutions. Iron sheets with dimensions of 80 mm × 63 mm × 2 mm were used as anode and cathode electrodes. The electrodes were connected to a DC power supply (PW-3034D, MICRO), providing 0–30 V applied voltage (0–3 A current). The sample solutions were agitated by a magnetic stirrer (LABINCO L81, Netherlands) to keep uniform concentration of P4R in the reactor during the EF process.

2.3 Electro-Fenton process for P4R removal

All of the batch EF oxidation experiments were done at room temperature (r.t.) in a 900 mL glass reactor as an undivided cell through two iron sheet electrodes (5 cm apart) that were connected to the DC power

supply and immersed in 300 mL solutions containing a certain amount of P4R (50–250 mg L⁻¹) and a magnetic bar. Prior to each EF process, the pHs of solutions were regulated to the proper value (pH = 3) by dropwise addition of HNO₃ (concentrated) or 0.1 M NaOH solutions. Before each EF process, the proper amount of Na₂SO₄ salt was added to the reactor to increase the conductivity of the solution. To start the oxidative degradation of P4R, an appropriate amount of H₂O₂ (1 mL H₂O₂ 10–30% equal to 0.033–0.100 % (v/v) H₂O₂ in the solution inside the reactor) as a source of ·OH was added manually to the reactor and the content was stirred using a magnetic stirrer to provide homogeneity in the reactor. At the time of the EF process, the ferrous ions were leached to the solution from the iron electrode. By applying the voltage between the iron electrodes, the EF process was started. To monitor the residual concentration of P4R during EF treatment, UV–Vis absorption measurements were done. Firstly, the UV–Vis absorption spectra of P4R solution (50 mg L⁻¹) was investigated in the wavelength range of 200–800 nm to find the best absorption wavelength of P4R. The UV–Vis spectra showed three absorption bands at 290, 333, and 505 nm that can be related to benzene rings, naphthalene groups and N=N azo bands, respectively (Benhsinat et al., 2017). According to the spectra, 505 nm (λ_{max}) was selected as the best wavelength for P4R determinations which was in agreement with the previous studies (Benhsinat et al., 2017; Shariati et al., 2020; Sojoudi et al., 2016).

For determination of P4R concentration, in the time range of 0–90 min with intervening period of 10 min, 3 mL of the solution in the reactor was drawn by pipette and then centrifuged for 3 min at 2900 rpm. Finally, the optical absorption of the residual P4R in the solutions was measured with the UV–Vis spectrophotometer at 505 nm. The P4R removal efficiency (RE %) or decolorization efficiency was calculated by the following equation (Eq. 2):

$$RE (\%) = \frac{A_0 - A_t}{A_0} \times 100 \quad (2)$$

Where, A₀ and A_t are the initial and equilibrium absorbance of P4R after treatment with the EF process, respectively. Figure 1 shows a schematic of the designed EF system.

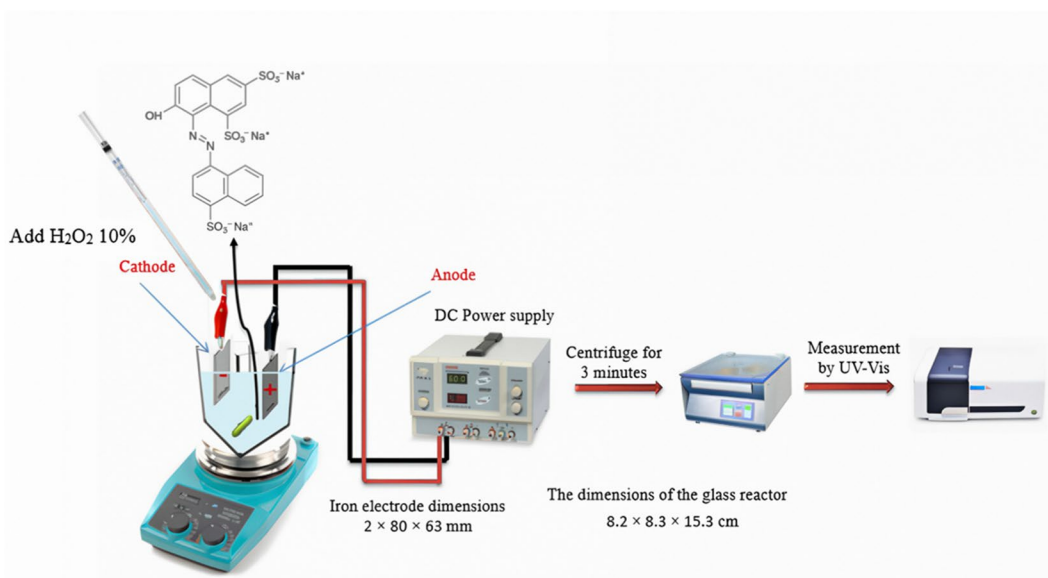


Fig. 1 Schematic of the experimental setup for EF process

2.4 Optimization of experimental variables using Taguchi orthogonal array design

In the present research, Taguchi method as a powerful optimization strategy was used to find the best decolorization efficiency with the minimum experiments (Kouchakinejad et al., 2022). To study the effect of the EF experimental factors and to determine the optimal conditions for obtaining maximum removal efficiencies, the influence of five effective variables in the P4R decolorization process including applied voltage (5, 10, 15, and 20 V), electrolyte concentration (0, 0.05, 0.1, and 0.2 mol L⁻¹), P4R concentration (50, 100, 200, and 250 mg L⁻¹), pH of the solution (2, 3, 4, and 6) and H₂O₂ content (1 mL of 10, 20, 25, and 30 % (v/v) H₂O₂) were investigated in four levels by Taguchi fractional factorial design (OA₁₆). The main advantage of this method is reducing the number of tests and as a result reducing the cost of consumables and time. All optimization experiments were performed using 300 mL solution containing P4R at r.t. According to the number of main factors, the (4⁵) matrix was planned by Minitab software (version 19), and 16 experiments were programmed for optimizing the EF variables. The desired factors and levels were selected according to our pre-tests that were conducted in various conditions. To escape any personal or random errors, the order of the

experiments was randomized. The removal efficiency for each experiment was calculated by measuring the UV–Vis absorbance of the residual solution using Eq. 2 (Table 1). The mean values for the four levels of each variable were calculated to show the removal efficiencies variate with the changing of the levels of each factor. The mean of P4R removal efficiencies for various levels of each variable at different times in the time range of 10–90 min were calculated and summarized in Table 2. From the main effect plots of the means, the levels producing larger removal efficiencies were chosen as the optimum value.

3 Results & Discussions

In this research, the EF based on the electrochemical peroxidation process was used. Iron electrode acts as the source of ferrous ions and H₂O₂ was manually added into the reactor as oxidant. In this method, Fe²⁺ ions are electrogenerated on the surface of the sacrificial iron anode (Eq. 3) as well as in the bulk of the solution (Eq. 5), respectively (Kurt et al., 2007). At pH ~3, the presence of low amounts of Fe²⁺ produces OH radicals in the bulk solution through homogeneous catalysis via Fenton reagent (Eq. 4) (Thiam et al., 2016). The •OH radicals resulting from the reaction of H₂O₂ and Fe²⁺ ions with the highest oxidation

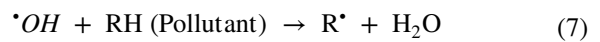
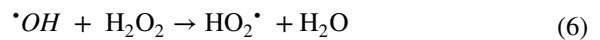
Table 1 OA₁₆ matrix for P4R removal experiments

Exp. Order	Run	Variables					RE (%)													
		H ₂ O ₂ (%)	pH	C _{P4R} (mg L ⁻¹)	Ionic Strength (mol L ⁻¹)	Voltage (V)	Time (min)													
							0	10	20	30	40	50	60	70	80	90				
1	12	25	6	100	0	15	0	13.9	19.5	24.5	30.2	34.6	38.9	43.6	46.3	49.7				
2	8	20	6	200	0.05	5	0	55.5	58.0	64.0	72.0	80.9	84.6	87.1	89.0	90.7				
3	5	20	2	100	0.1	20	0	96.0	99.2	99.1	99.3	99.4	98.6	99.3	99.2	98.8				
4	10	25	3	250	0.1	5	0	98.5	99.2	98.5	98.6	99.4	99.4	99.4	99.4	99.5				
5	4	10	6	250	0.2	20	0	44.1	97.9	98.3	98.5	98.6	98.5	98.8	98.7	98.6				
6	9	25	2	200	0.2	10	0	94.5	99.3	99.0	99.4	99.3	99.4	99.4	99.4	99.3				
7	2	10	3	100	0.05	10	0	98.7	98.5	98.8	98.9	99.0	99.1	99.0	99.1	99.1				
8	16	30	6	50	0.1	10	0	46.0	67.0	82.0	92.1	94.9	96.5	97.5	98.1	98.4				
9	11	25	4	50	0.05	20	0	96.4	100.0	100.3	100.3	100.4	100.4	100.3	100.3	99.8				
10	1	10	2	50	0	5	0	96.1	91.0	87.7	90.3	100.1	100.3	100.2	100.0	99.9				
11	13	30	2	250	0.05	15	0	97.4	99.9	100.1	100.1	100.1	100.0	100.1	100.2	100.1				
12	7	20	4	250	0	10	0	2.1	4.9	4.8	15.3	24.5	32.3	37.2	49.4	47.2				
13	6	20	3	50	0.2	15	0	99.9	100.0	100.0	100.3	99.9	100.0	100.1	100.3	100.0				
14	3	10	4	200	0.1	15	0	80.2	93.6	97.3	97.9	98.7	99.1	99.3	99.4	99.6				
15	14	30	3	200	0	20	0	99.4	99.8	99.9	100.0	100.0	100.0	100.0	99.9	99.9				
16	15	30	4	100	0.2	5	0	76.9	82.2	86.9	93.3	95.5	97.0	97.7	98.2	98.6				

Table 2 RE (%) for each level of experimental variables

Time (mi n)	RE (%) for each level of Variables																								
	H ₂ O ₂ (%)					pH					C _{P4R} (mg L ⁻¹)					Ionic Strength (mol L ⁻¹)					Voltage (V)				
	10	20	25	30	3	4	6	50	100	200	250	0	0.05	0.1	0.2	5	10	15	20						
10	79.7	63.4	75.8	79.9	96.0	99.1	63.9	39.9	84.6	71.4	82.4	60.5	52.8	87.0	80.2	78.8	81.7	60.3	72.8	84.0					
20	95.2	65.5	79.5	87.2	97.4	99.4	70.2	60.6	89.5	74.8	87.7	75.5	53.8	89.1	89.7	94.9	82.6	67.4	78.2	99.2					
30	95.6	67.0	80.6	92.2	96.5	99.3	72.3	67.2	92.5	77.3	90.1	75.5	54.3	90.8	94.2	96.1	84.3	71.2	80.5	99.4					
40	96.4	71.7	82.1	96.4	97.3	99.4	76.7	73.2	95.7	80.4	92.3	78.1	59.0	92.8	97.0	97.9	88.6	76.4	82.1	99.5					
50	99.1	76.2	83.4	97.6	99.7	99.6	79.8	77.3	98.8	82.1	94.7	80.7	64.8	95.1	98.1	98.4	94.0	79.4	83.3	99.6					
60	99.2	78.9	84.5	98.4	99.6	99.6	82.2	79.6	99.3	83.4	95.8	82.6	67.8	96.0	98.4	98.7	95.3	81.8	84.5	99.4					
70	99.3	80.9	85.7	98.8	99.7	99.6	83.6	81.7	99.5	84.9	96.4	83.9	70.2	96.6	98.9	99.0	96.1	83.3	85.8	99.6					
80	99.3	84.5	86.3	99.1	99.7	99.7	86.8	83.0	99.7	85.7	96.9	86.9	73.9	97.1	99.0	99.1	96.7	86.5	86.5	99.5					
90	99.3	84.2	87.1	99.3	99.5	99.6	86.3	84.4	99.5	86.5	97.4	86.3	74.2	97.4	99.1	99.1	97.1	86.0	87.4	99.3					

power can react with excess H₂O₂ to produce HO₂[•] (Eq. 6). •OH radicals are highly reactive species without any selectivity that destroy the most pollutants and organic compounds until their mineralization to CO₂, H₂O, and inorganic ions (Eq. 7) (Eyüp, 2009).



In preliminary experiments, the chemical stability of P4R in the presence of H₂O₂ without applying the EF process was investigated (pH=3). The results showed addition of H₂O₂ without applying voltage, did not change the absorption spectra of P4R during stirring the solution for 60 min at r.t. The results confirmed that there is no significant P4R removal in the presence of H₂O₂ alone (Fig. S1).

3.1 Effect of pH value on the P4R removal efficiency

Solution pH is one of the main important and influential variables that limit the performance of Fenton and Fenton-related processes and can control the generation of •OH radicals. Previous studies showed the effect of the initial pH of the environment is very different depending on the types of used processes and pollutants. It affects the speciation of iron and the decomposition of H₂O₂. Before the optimization of the solution pH, to investigate the effect of this variable on the stability and the UV–Vis spectra of P4R, the solution pH was changed in the range of 2.0–6.0 and the results showed no variation in P4R UV–Vis spectrum with changing pH. This result confirmed the chemical stability of P4R in this range of pH (Fig. S1).

During the optimization step, to find the effect of solution pH on the removal efficiency of P4R in the EF process, the solution pHs in the acidic range of 2.0–6.0 were investigated (Table 2, Fig. 2).

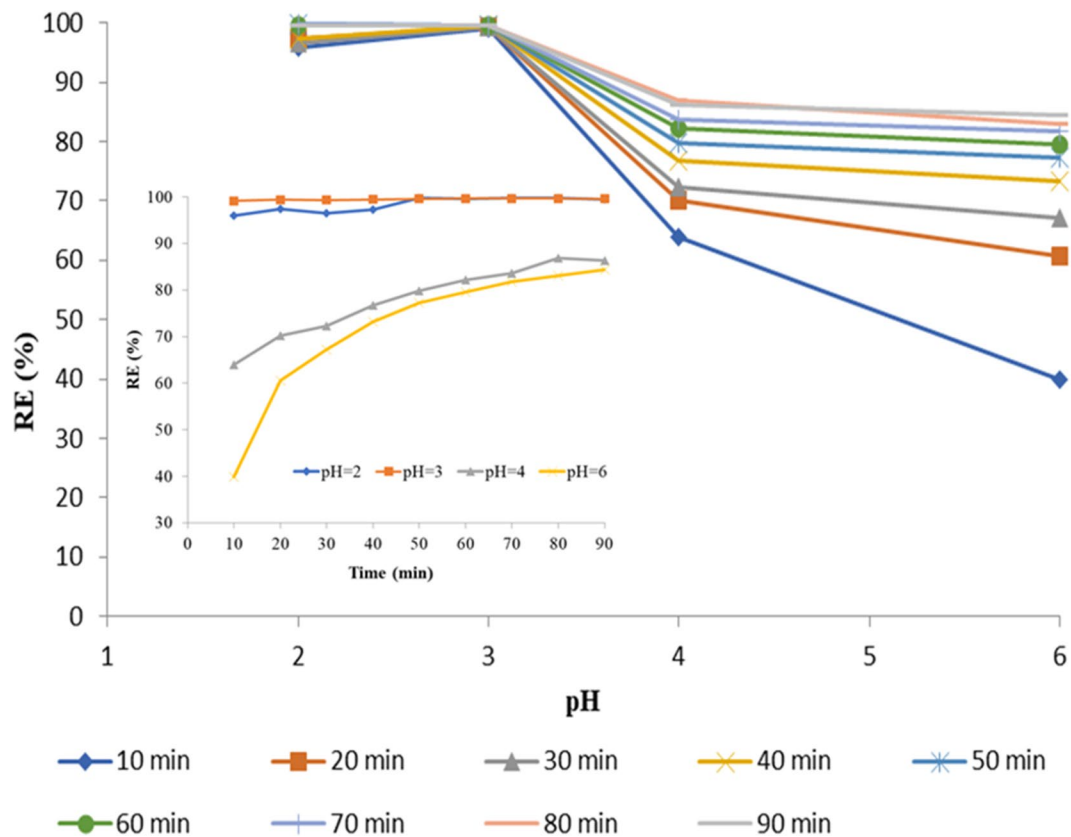


Fig. 2 The effect of solution pH on the P4R decolorization efficiency

The results indicated the highest P4R removal has been observed in the acidic pH conditions (pH=2 and 3) which are 99.6% in the initial times of EF process. Therefore, pH=3.0 was selected as the optimum pH. By increasing the pH to 4 and 6, P4R removal efficiency reduced to 63.9 and 39.9% after 10 min EF process, respectively. According to Eq. 4, decreasing the solution pH, enhances the $\bullet\text{OH}$ production. At higher pHs, the Fe^{2+} ions are deactivated by the formation of $\text{Fe}(\text{OH})_2$ and $\text{Fe}(\text{OH})_3$, resulting a reduction in $\bullet\text{OH}$ concentrations. Based on the results that are shown in Table 2 and Fig. 2 (insert), at pH=3 during 10 min EF process, more than 99% removal efficiency was obtainable whereas by increasing pH to 6, nearly 86.3% removal efficiency was obtained after 90 min. This finding is consistent with the results of other researchers regarding the removal of dyes with AOP processes at pH=3 (Mossmann et al., 2019; Thiam et al., 2016; Wang et al., 2022).

3.2 Effect of applied voltage on the P4R removal efficiency

The applied voltage is an important variable influencing the efficiency of the EF process (Le et al., 2022). Using an undivided reactor as reaction medium causes less consumption of voltage for electrolysis due to the avoiding of voltage penalty of the separator that is presented in divided cells. The influence of studied levels of applied voltage (5–20 V) on the decolorization of the P4R solution during the EF process is shown in Fig. 3. Increasing the voltage to a certain extent will increase the concentration of ferrous ions (Wang et al., 2008). Enhanced decolorization efficiency was obtained with a rise in applied voltage from 60.3% at 10 V to 84.0% at 20 V for 10 min EF process. According to our findings, the increase in applied voltage enhances the decolorization efficiency. Instead of increasing voltage, enhancing the

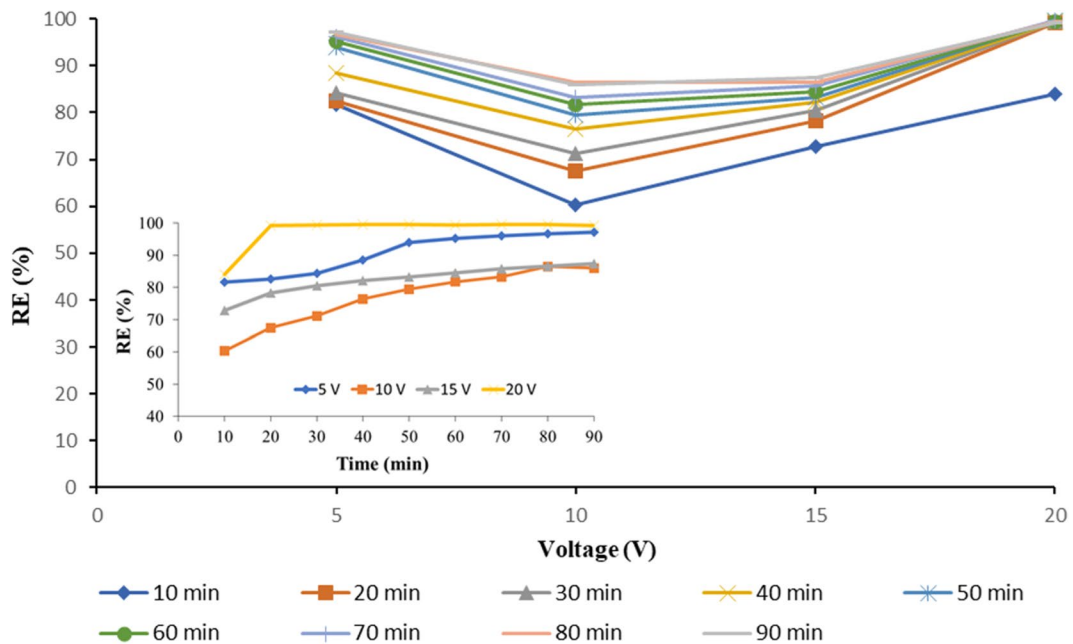


Fig. 3 The effect of applied voltage on the P4R decolorization efficiency

EF time can improve the decolorization efficiency. Based on the results, increasing time at 5 V applied voltage obtained 60.3, 71.2, 79.4, and 86.0% removal efficiency for 10-, 30-, 50-, and 90-min EF process, respectively (Fig. 3.). Therefore, 20 V was elected as the optimum voltage for the future experiments.

3.3 Effect of initial P4R concentration on the P4R removal efficiency

The effect of the initial P4R concentration on the removal efficiency was investigated by performing experiments with varying P4R concentrations in the range of 50–250 mg L⁻¹. The results are shown in Fig. 4. According to the results, at 10 min, the highest removal efficiency was obtained in 50 mg L⁻¹ of P4R, and for 250 mg L⁻¹ lowest removal efficiency was obtained. By increasing the P4R concentration in the solution, longer times are needed for the production and action of •OH radicals to decompose P4R molecules. This phenomenon is observed in Fig. 4 (insert) that shows at longer times, the P4R removal efficiencies are improved. At a constant time of the EF process, the dissolution of the iron anode to generate Fe²⁺ ions, is constant. Therefore, at higher concentrations of P4R, the availability of a sufficient amount of

•OH decreases and it is necessary to increase the time of the EF process.

3.4 Effect of electrolyte concentration on the P4R removal efficiency

To study the influence of electrolyte type on the P4R degradation efficiency, different electrolyte salts including NaNO₃, NaCl, NaHPO₄, and Na₂SO₄ were separately used in the EF process and the results showed Na₂SO₄ is more suitable electrolyte and after that NaCl, NaHPO₄, and NaNO₃, respectively. With salt addition, the P4R removal efficiency was improved due to the increase in the conductivity of the aqueous solution (Jinisha et al., 2018). Other experiments were done to investigate the effect of various concentrations of Na₂SO₄ salt (0, 0.05, 0.1, 0.2 mol L⁻¹) on the P4R removal process. The findings (Fig. 5) show more decolorization occurs in the presence of Na₂SO₄ salt and this trend is more evident in the longer times. According to the results, 0.1 M of Na₂SO₄ was sufficient and selected as the optimum value for P4R removal. This finding is in agreement with previous study on the electrochemical degradation of P4R by AOP that concluded 0.01 mol L⁻¹ Na₂SO₄ is sufficient for efficient discoloration (Thiam

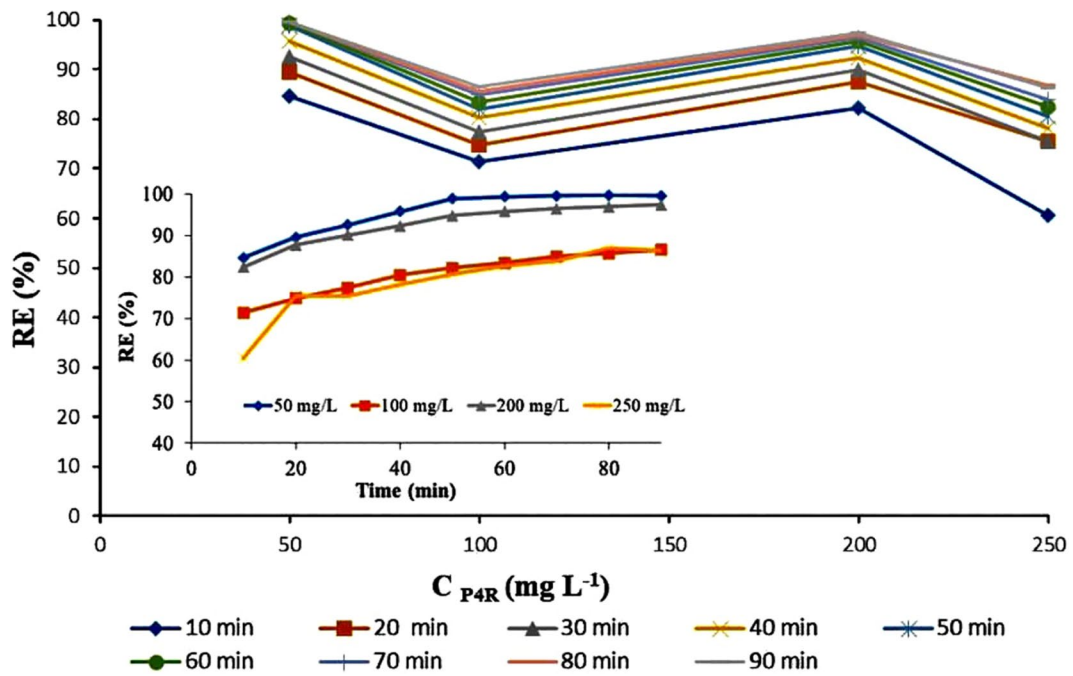


Fig. 4 The effect of initial P4R concentration on the P4R decolorization efficiency

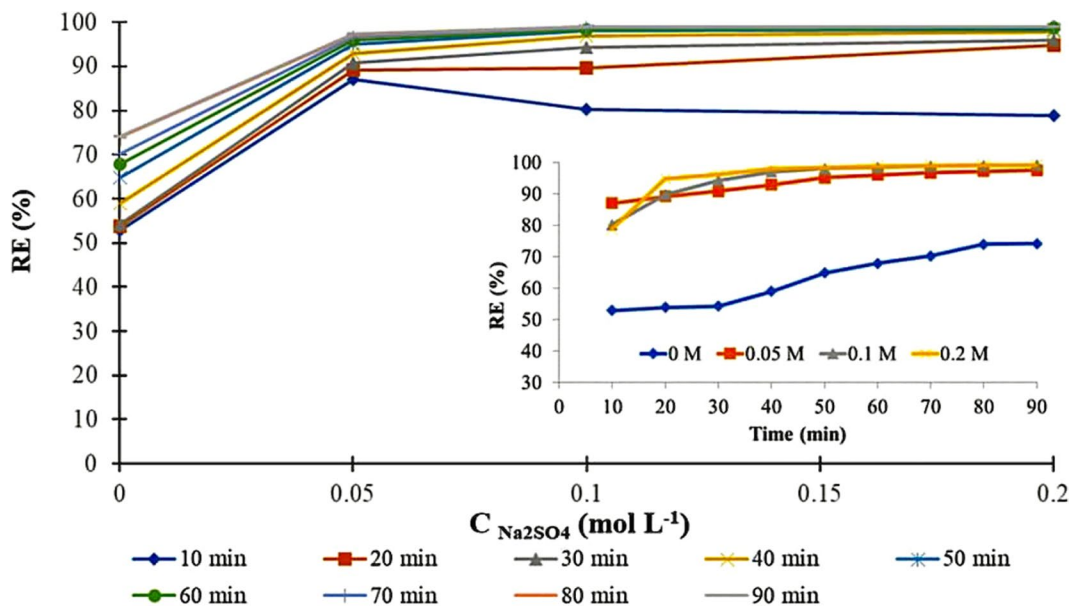
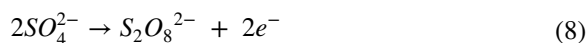


Fig. 5 The effect of Na_2SO_4 concentration on the P4R decolorization efficiency

et al., 2016). The generation of more active oxidant agents by Na_2SO_4 can justify the greater decoloration. Na_2SO_4 in an electrochemical reactor produces

peroxydisulfate ions ($S_2O_8^{2-}$) at the anode's surface, which are useful for oxidizing P4R molecules (Eq.8) (Escalona-Duran et al., 2020).



At higher amounts of SO_4^{2-} ions, the specific conductivity of solution increases which promotes the transport of the negatively charged P4R molecules towards the anode surface.

3.5 Effect of hydrogen peroxide on the P4R removal efficiency

To determine the optimal amount of H_2O_2 concentration and also to study the effect of its content on the EF process, the addition of 1 mL H_2O_2 solutions in the concentrations of 10, 20, 25, and 30% were investigated and the findings are presented in Fig. 6. These H_2O_2 concentrations equal to 0.033, 0.067, 0.083 and 0.100% (v/v) in reactor regarding the 300 mL solution volume, respectively. According to the plot, with increasing H_2O_2 content, the P4R removal efficiency decreased and it was concluded that the highest amount of P4R removal was obtained with 1 mL addition of 10% concentration of H_2O_2 (equals to 0.033% (v/v) H_2O_2 in the solution). As Fig. 6 (insert) shows, at longer reaction times from 10 to 20 min, the removal efficiency of P4R is enhanced and reaches its maximum value of 95.3%. As the reaction time

increases to more than 20 min, the P4R removal efficiency does not change significantly. In this study, due to the consumption of H_2O_2 in initial times, the removal efficiency does not change significantly after 20 min with increasing reaction time.

3.6 The P4R removal efficiency at the optimum conditions

In this study, P4R removal efficiency in the optimal conditions, before and after pH adjustment and salt addition, in the absence of H_2O_2 and without applying voltage were investigated. According to the results, addition of H_2O_2 at pH=3 without performing the EF process, did not change the absorption spectra of P4R during stirring the solution for 60 min at r.t (Fig. S2). Also, the amounts of P4R removal in the optimal conditions (pH=3, voltage=20 V, % H_2O_2 =1 mL of 10% (v/v) solution, $C_{\text{Na}_2\text{SO}_4}$ =0.1 mol L^{-1}) after 10, 20, 30, 40, and 50 min for five replicate decolorization experiments were investigated at three P4R concentration levels of 50, 100 and 250 mg L^{-1} and REs % were obtained as 100.00%, 99.92%, and 99.87%, respectively that confirmed the complete decolorization of P4R from aqueous samples. According to the previous studies, it is assumed that $\bullet\text{OH}$ radicals firstly attack to the N=N azo bands that are presented

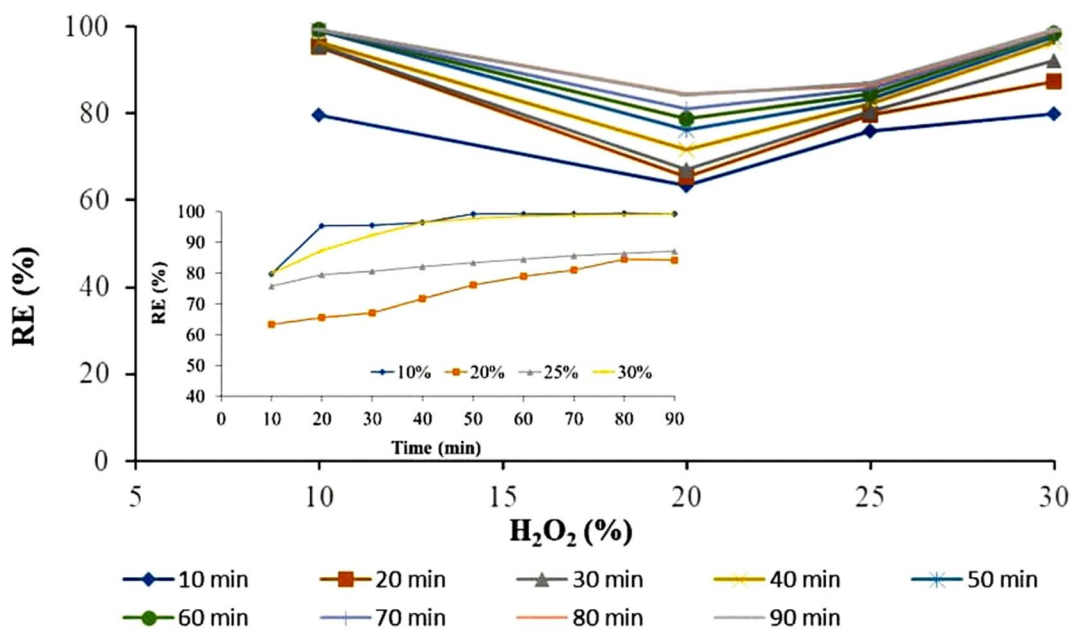


Fig. 6 The effect of H_2O_2 content on the P4R decolorization efficiency

in 505 nm in UV–Vis spectra. After that, naphthalene group (presented at 339 nm) and finally the benzene rings (occurred at 290 nm) are affected by $\bullet\text{OH}$ radicals (Benhsinat et al., 2017). The absence of absorption bands at 505, 333 and 290 nm after EF process at optimum conditions (Fig. S2), confirmed the probably oxidation of the P4R molecules via disruption of the N=N azo bands and opening the aromatic rings to give aliphatic compounds which may be subsequently mineralized.

3.7 Comparison between the EF and Ozone-based removal processes

Ozone (O_3) is considered as an electrophilic molecule and a selective oxidant with an oxidation potential of 2.07 V, and its molecular reaction rate constants with different groups of organic compounds have a wide range. In this study, to compare the P4R removal efficiency between the EF and O_3 -based removal processes with combined application of them (O_3 +EF processes), O_3 gas was produced using an ozone generator and purged with a constant flow into the reactor. For this purpose, firstly O_3 was purged into the solution without performing EF process at optimal conditions (pH=3, voltage=0 V, $C_{\text{P4R}}=50 \text{ mg L}^{-1}$, $C_{\text{Na}_2\text{SO}_4}=0.1 \text{ mol L}^{-1}$). The results showed 32.6, 49.3, 51.8, 60.5, and 98.0% decolorization efficiency after purging O_3 into the solution for 10, 20, 30, 40, and 50 min, respectively. According to these results, purging O_3 without applying voltage cannot completely decolorize P4R in times lower than 50 min whereas utilizing the EF

process at the optimum conditions (pH=3, voltage=20 V, $\% \text{H}_2\text{O}_2=1 \text{ mL}$ of 10% (v/v) solution, $C_{\text{P4R}}=50 \text{ mg L}^{-1}$, $C_{\text{Na}_2\text{SO}_4}=0.1 \text{ mol L}^{-1}$) obtained relatively complete P4R decolorization (100%). The combination of the EF process by purging O_3 showed complete decolorization even at 10 min (Fig. S3). Due to the complete P4R decolorization during the EF process without O_3 purging, future experiments were carried out by EF process alone.

3.8 Energy consumption of EF process

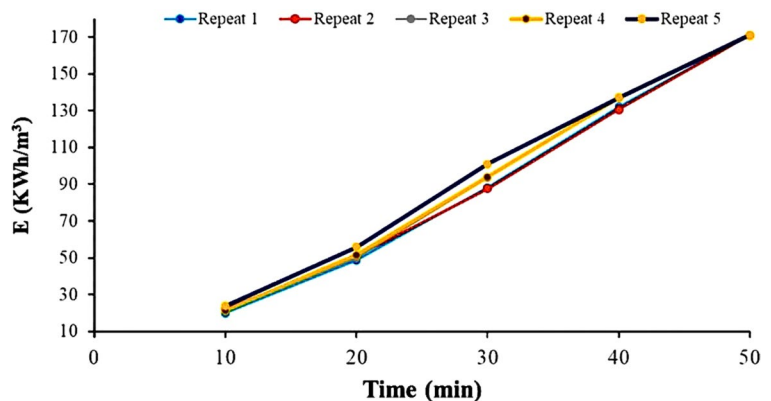
Energy consumption (EC) (KWh/m^3) is one of the main affecting factors in the electrochemical treatment, which shows a great impact on the justification of processes. In this study, the amount of energy consumed during the EF process was assessed according to Eq. 9 (Guvenc et al., 2019):

$$EC = \frac{U \times I \times t}{V} \quad (9)$$

where U denotes cell voltage (V), I is the applied current (A), t is the time of EF process (h), and V is the sample volume (m^3).

The effect of EF process time on the amount of energy consumed is shown in Fig. 7. According to the relationship mentioned for the calculation of energy consumption, it is obvious that with time increase in the EF process, the amount of energy consumption also increases. Based on the results, in the optimal P4R decolorization condition at 10 min, the amount of energy consumed was obtained as 19.817 (KWh/m^3).

Fig. 7 Energy consumption during the proposed EF process



3.9 Investigating the EF process for decolorization of P4R from real samples

In order to investigate the capability of the proposed EF process for P4R decolorization, six real wastewater samples including carpet cleaning, medical, dairy, food wastes and two concrete wastewaters were studied. These wastewaters were provided from Rasht Industrial Town (Guilan Province, Iran). For this purpose, each real sample was studied spectrophotometrically at 505 nm to investigate the initial absorbance of the sample. To investigate the effect of various matrices for P4R decolorization, 300 mL of each real sample was spiked by P4R standard solution to prepare 50 mg L⁻¹ P4R solution. The spiked solution was subjected to the EF process under the optimum conditions for 10, 20, 30, 40, and 50 min. Each real sample underwent three repetitive EF processes and the mean of REs % was considered. The results of the removal efficiency of P4R in real samples are summarized in Table 3.

3.10 Comparison between the proposed EF process with other studies

In previous studies, different water treatment methods including adsorption, Fenton, electro-Fenton, photocatalytic, and electro-oxidation processes or a combination of them were investigated and reported for P4R removal. In Table 4, the obtained removal efficiencies of the proposed EF method were compared with the reported studies. This table shows the proposed EF process has good removal efficiency and can completely remove the P4R from aqueous solutions. In 2016, Hafaiedh et al. used the EF process for mineralization of P4R in a synthetic solution using a graphite-felt cathode and a platinum anode. The

analysis of total organic carbon indicates that 23.5% of mineralization yield was achieved after 4 h treatment (Hafaiedh et al., 2016). In 2019, Mossmann et al. used polyethylene-supported zero-valent iron as a photo-Fenton buoyant catalyst to remove P4R from aqueous solution under visible light irradiation. The results showed that this substance has a satisfactory catalytic activity and in 15 min of reaction, the complete decolorization of P4R was achieved (Mossmann and et al., 2019). In other study, Thiam et al. (2016) studied the electrochemical degradation of the artificial food azo-color P4R by advanced oxidation processes, and the results showed that it became colorless and totally mineralized after 50 and 240 min (Thiam and et.al., 2016). The comparison of the aforementioned methods with the present study shows the proper performance of this process in removing P4R, taking into account the reusability of spent iron electrodes compared to consumed iron ions and their lower cost.

4 Conclusion

The present study aimed to assess the EF process for the P4R decolorization in aquatic environments. The results obtained optimal removal in the pH value of 3, the EF process time of 10 min, the P4R concentration of 50 mg L⁻¹, applied voltage 20 V, Na₂SO₄ concentration of 0.1 M, and H₂O₂ content of 0.033% (addition of 1 mL of 10% H₂O₂ solution). In these circumstances, the results showed that the EF process allows for a complete decolorization efficiency of 100% after 10 min treatment. The results of this study illustrate that the EF process with iron electrodes can completely decolorize P4R dye in a minimum reaction time through •OH radical reactions. The Fenton

Table 3 Results of EF process for P4R decolorization in real samples in the optimal conditions (pH = 3, voltage = 20 V, H₂O₂ = 1 mL of 10% (v/v) solution, C_{P4R} = 50 mg L⁻¹, C_{Na2SO4} = 0.1 mol L⁻¹)

Sample	Absorption of P4R solution	Absorption after EF process (10 min)	Removal Efficiency (%)
Carpet cleaning waste water	1.5531	0.0093	99.4
Medical waste water	1.6716	0.0059	99.6
Food waste water	1.4072	0.0054	99.6
Dairy waste water	1.3323	0.0019	99.9
Concrete waste water 1	1.4883	0.0019	99.9
Concrete waste waters 2	1.4735	0.0023	99.8

Table 4 Various advanced oxidation processes method comparisons for P4R removal

Pollutant	Method	Details	RE (%)	Ref
P4R	Solar-mediated heterogeneous photocatalytic ozonation	—	100%	(Quiñones-Murillo et al., 2019)
P4R	EF	cathode: Graphite-felt anode: platinum	100%	(Hafaiedh et al., 2016)
P4R	Photo-Fenton process	—	100%	(Mossmann et al., 2019)
P4R	Electro-oxidation with or without H ₂ O ₂ production, electro-Fenton and photoelectro-Fenton	Cathode: carbon-PTFE air-diffusion electrode Anode: Pt sheet or a BDD thin-film electrode	100%	(Thiam et al., 2015)
Tartrazine, P4R, Coomassie Brilliant Blue	Hybrid processes of hydrodynamic cavitation (HC) with potassium persulfate (KPS) and hydrogen peroxide (H ₂ O ₂)	—	50.1% (for Tartazine) 42.3% (for P4R) 92.3% (for Coomassie Brilliant Blue)	(Askarniya et al., 2022)
P4R	Cobalt (II)/NaHCO ₃ /H ₂ O ₂ system	—	96.3%	(Macías-Quiroga et al., 2020)
P4R	Magnetic ordered mesoporous carbon Nanocomposite	Sorption process	99%	(Toutouchi et al., 2020)
P4R	Amine Functionalized Kit-6 Mesoporous Magnetite Nanocomposite	Sorption process	98%	(Sojoudi et al., 2016)
Rhodamine B	EF process using iron-doped mesoporous silica as a heterogeneous catalyst	Graphite plates	97.7%	(Jinisha et al., 2018)
Remazol black B	Electro-coagulation and electro-Fenton	Two stainless steels	> 90%	(Kabir Suhan et al., 2020)
Reactive red 195	EF process in a cell with oxygen sparged fixed bed electrodes	Cathode: carbon cylinders Anode: a horizontal array of closely packed steel screens	100%	(Elbatea et al., 2021)
Ammoniacal nitrogen	EF assisted sonication	Graphite	65.5%	(Menon et al., 2021)
Reactive Blue 19 Dye	Fenton	—	70%	(Moghadam & Nori Kohbannan, 2018)
Neutral Red Dye	EF (homogenous EF, heterogeneous EF using HZC, FeZC nanocatalysts)	Graphite	85.9% (homogenous EF) 90.3% (heterogeneous EF using HZC) 92.4% (FeZC nanocatalysts)	(Ebratkhahan et al., 2021)
Aniline	EF processes utilizing carbon nanotube 3D morphology modification of an Ag-loaded copper foam cathode	Cathodes: The CF, Ag@Cu, CNT@Cu and CNT-Ag@Cu Anode: two platinum plates	99.2%	(Ma et al., 2021)
P4R	EF process	EF process using two iron electrodes	100%	This study

agent was successfully produced electrochemically in the treated solution, which led to the effective production of $\cdot\text{OH}$ radicals and complete elimination of P4R pollutant. In this method, electricity is utilized and the overall process does not generate secondary pollutants. The optimized EF process has been successfully applied to treat several real wastewaters and the results confirmed its efficiency for P4R removal. The compatibility with the environment, safety, versatility and high efficiency are the benefits of the developed EF method.

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Data Availability The data that support the findings of this study are available from the corresponding author upon reasonable request.

Declarations

Conflict of interests The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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