



Aniline degradation from aqueous solution using electro/Fe²⁺/peroxydisulphate process

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ABSTRACT

Non-degradation compounds are nowadays not being disposed into the environment, or are removed before being disposed into the environment. The aim of this study was to assess the degradation of aniline from aqueous media by electro/Fe²⁺/peroxydisulphate (PDS) process. The effects of different parameters like pH (3–9), electric current (EC; 10–30 mA/cm²), dose of PDS (10–40 mM), Fe²⁺ dose (5–20 mM) and initial aniline concentration (20–100 mg/L) on the degradation of aniline were investigated in the laboratory reactor in the batch condition. The results showed that the removal efficiency of aniline decreased by increasing the initial pH and pollutant concentration; however, with increasing EC and PDS doses and ferrous concentration, the removal efficiency increased. The maximum of aniline degradation, equalling 83%, was obtained at 60 min of reaction time. Also, for reaction time of 60-min runs, PDS only, EC only, PDS + Fe, PDS + EC and PDS + Fe + Fe, respectively, have 8.9%, 53%, 43%, 65% and 83% of the removal efficiency. The degradation efficiency of aniline from synthetic wastewater was significantly improved when the combined processes PDS/electro-conduction/Fe²⁺ was applied.

Keywords: Aniline; Degradation; Sulphate radical; Iron; Peroxydisulphate

1. Introduction

The contamination of water resources by industrial development, urban development and population increase is known as one of the most important environmental crises in the world, especially in developing countries [1,2]. Some of the organic and inorganic contaminations are discharged into the receiving water from different industries [3]. Aniline

is one of the chemicals used to produce rubber accelerations and intermediate substances, manufacturing azo and plastic dyes, and, so, is widely released into the environment [4,5]. Aniline is also used for producing medicines, resins, polishes, perfumes, waxes, photographic chemicals, explosives, chemicals derived from petroleum refining, diphenylamine and phenols [6].

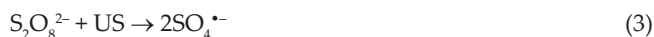
Aniline is toxic, dangerous and resistant to decomposition; its presence, even in very low densities, is dangerous for aquatic life [2,7]. Therefore, aniline should not be

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disposed into the environment or it should be removed before it enters the environment [8]. Conventional methods cannot completely remove aniline, and so, effective methods of treatment have to be developed to remove and decrease the toxic risks of aniline for the environment. Researchers are attempting to find appropriate methods for treating resistant and toxic pollutants [8,9]. Some of the methods for removing aniline are adsorption on solids, membrane processes, catalytic oxidation, biological refinement and adsorption by activated carbon [10]. Common methods—such as biological methods—do not have high efficiency for removing aniline from wastewater. On the other hand, in high capacities, these methods have high utilization and operating costs, especially for developing countries [11]. The adsorption process, by activated carbon, is also an expensive and time-consuming method: it simply condenses pollutants and the efficiency is not appropriate with the costs [7]. In recent decades, the use of advanced oxidation processes (AOPs)—as a promising method to eliminate resistant contaminants—has been broadly performed [12,13]. One of the AOPs is the use of sulphate radicals for degradation and oxidation of organic combinations [11,14].

Some researchers use sulphate radicals to remove different pollutants, for example, Guo et al. [15] for removing tetrabromobisphenol A from aqueous solution, Olmez-Hanci et al. [16] for removing bisphenol A from aqueous solution, Mendez-Diaz et al. [17] for removing sodium dodecylbenzenesulphonate from aqueous solution, Olmez-Hanci and Arslan-Alaton [18] for removing phenol from aqueous solution, Wang and Liang [19] for removing tetramethylammonium hydroxide and Khataee and Mirzajani [20] for removing C.I. Basic Blue 3. Accordingly, they have high efficiency in removing pollutants.

Peroxydisulphate (PDS; $S_2O_8^{2-}$) is a strong oxidizer, widely used in the petrochemical industry for purification of hydraulic fluids. In treatment of hazardous wastewater by chemical oxidation in acidic or alkalinity pH range, oxidizing radicals are slowly produced in ambient temperature and, as a result, organic substances are slowly decomposed [21]. Therefore, increasing temperature, activation of the photochemical, ultrasonic waves and intermediate metals as activators can increase the production of radicals and accelerate the decomposition of resistant organic substances in chemical oxidation processes [22]. According to Eqs. (1)–(5), in the AOP using ($S_2O_8^{2-}$) ion, temperature increase, UV radiation, ultrasonic influences and intermediate metals like Fe, manganese, silver and copper increase the reaction velocity and decomposition of ($S_2O_8^{2-}$) ion to sulphate radical ($SO_4^{\bullet-}$) with 6.2 V oxidation potential [22].



According to Eqs. (4) and (5), Fe ion acts as an activator PDS and, on the other hand, it is considered as a problem in AOPs for deoxidizing Fe^{2+} to Fe^{3+} . In addition, Fe likewise can react with sulphate radicals and thereby reducing the process efficiency [14,23]. To solve this problem, they use some factors like sodium thiosulphate or integrating electrochemical processes to activate PDS and produce sulphate radical. In the integrating electrochemical processes (Eq. (6)) and activating PDS with Fe, which was used by Lin et al. [24] to remove bisphenol A, Fe^{2+} ions are revived in the cathode. Also, according to Eq. (7), sulphate radicals will be produced.



In previous studies, the efficiency of the disulphate process in the decomposition of resistant organic compounds was proven [9,24]. Nonetheless, existing reports demonstrate that this process is not used to remove aniline. Therefore, this study was aimed to remove aniline from aqueous solution through an AOP and activated electro-PDS with Fe ion in batch mode. In this respect, the effect of pH parameters, electric current, PDS concentration, Fe concentration and the initial concentration of aniline in different contact times on the removal efficiency was studied.

2. Experimental

2.1. Materials

Merck's analytical quality chemicals, such as aniline purity 99.5%, peroxy diammonium sulphate ($(NH_4)_2S_2O_8$), ferrous sulphate ($FeSO_4$), sodium sulphate (Na_2SO_4), sulphuric acid (H_2SO_4) and sodium hydroxide ($NaOH$), were used in the preparation of reagents. All other reagents were of analytical grade and all sample solutions were prepared using deionized water.

2.2. Experimental procedure

Fig. 1 shows a schematic of a reactor made of Plexiglass with 1-L capacity, including two steel plates as electrodes. It was installed at the reactor—8 cm in height and 4 cm in width and 2 mm in thickness. The copper wires, sealed with fast-drying epoxy coated with silicon sealant, were connected to the electrodes through the current collectors. The wires were connected directly to a programmable DC power supply (Micro Iran PS.303D). The pH was adjusted at the value desired via an initial injection of acid (H_2SO_4 ; 0.1 N) or alkaline ($NaOH$; 0.1 N) solution through a pH meters (PB.3001). At the end of specified amounts of dissolved iron sulphate, peroxy dioxide was added to the reactor. At all stages of the process, at specified intervals, the sampling (5 mL) was performed. To remove particles all samples, for 10 min, were centrifuged at 400 rpm.

In this work, the effects of pH value, electric current, Fe dose, initial concentration of aniline and reaction time in

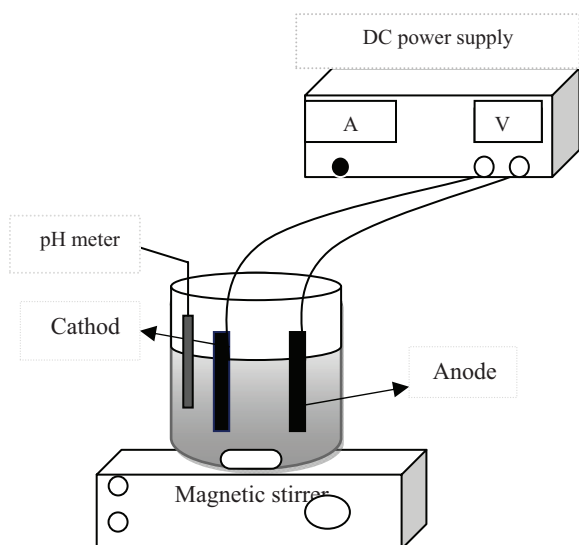


Fig. 1. Experimental setup of electro/ Fe^{2+} /peroxydisulphate.

oxidizing aniline from synthetic wastewater were evaluated. At each stage of experimental work, one factor was a variable parameter and the other variables were kept constant to determine their optimal value. Because of the importance of pH in chemical reactions, at the first stage of the experiments, the proper pH value in the range of 3–9 was determined. Then, at the optimum value of pH, the effect of EC at the range of 10–30 mA/cm was investigated when other variables were constants. The effects of other parameters—such as, Fe concentration, initial concentration of aniline and reaction time—were examined the same way when only one factor was a variable.

2.3. Methods of analysis

The pH value of the solutions was measured using a pH meter (PB.3001). The concentration of aniline in water was detected by UV–Vis spectrophotometer (PG Instrument Limited, UK) at the range of 263 nm from a calibration curve.

3. Results and discussion

3.1. The effect of pH

The effect of different pH values in the range of 3–9 on the removal of aniline using electro/PDS was studied. As the results show in Fig. 2, at its lowest, aniline removal efficiency was 33.7% at pH 9 and in a reaction time of 60 min. The results of this stage show that by increasing pH, the efficiency of aniline removal significantly decreases. Consequently, the highest removal efficiency was 65% than that obtained at pH 3. So pH 3 was selected as pH optimum for the next stages. As experimental data indicated, the pH of solution is one of the most effective factors for removing pollutants in chemical processes. In AOPs, pH has a direct effect on the production of hydroxyl and sulphate/radicals in solution [25,26]. The related researches have indicated that the highest performance for removing pollutants was obtained in electro/PDS in acidic condition (pH=2–4). If we use Fe^{2+} in the AOP, the pH level had effects on $\text{Fe}(\text{OH})_3$ in solution and also on the reactions of persulphate with pollutants

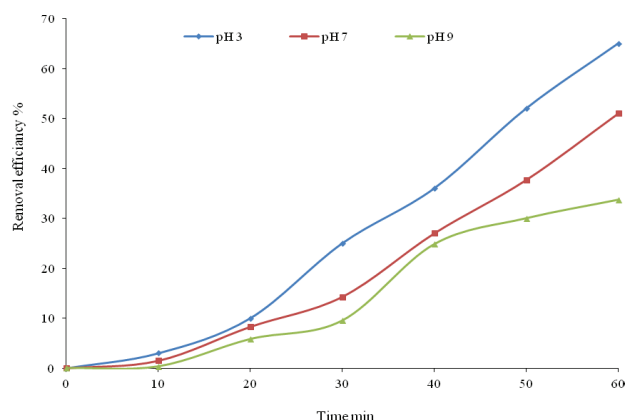


Fig. 2. The influence of solution initial pH on aniline removal in the electro/ Fe^{2+} / $\text{S}_2\text{O}_8^{2-}$ process (aniline = 40 mg/L, iron = 15 mM/L and persulphate = 30 mM/L, electric current = 30 mA/cm²).

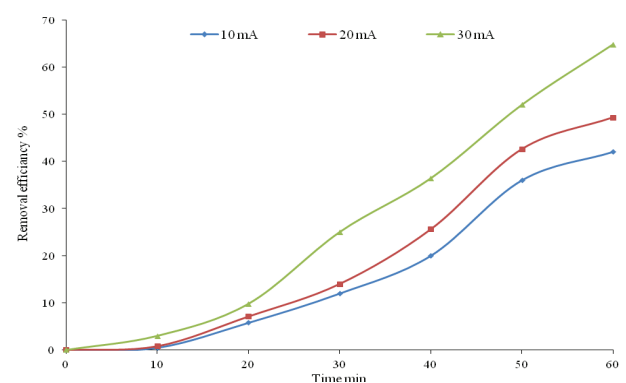
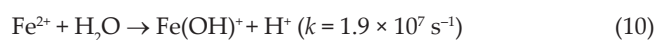
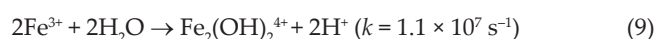
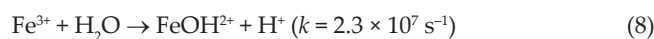


Fig. 3. The influence of electric current on aniline removal in the electro/ Fe^{2+} / $\text{S}_2\text{O}_8^{2-}$ process (aniline = 40 mg/L, iron = 15 mM/L, persulphate = 30 mM/L, pH = 3).

[6,27]. As the pH in solution gradually increases from acidic to alkaline, the percentage removal of aniline decreases, and these results are also similar to the studies of Zhang et al. [11] and Lin et al. [24], which focused on the removal of aniline and bisphenol A. This phenomenon is caused by precipitation of Fe ions at pH > 4. When the pH increases, Fe ions are converted from Fe^{2+} to Fe^{3+} and precipitation in the colloidal state and, according to Eqs. (8) and (9), the efficiency of reaction decreases [24]. Also, at pH > 4, Fe^{2+} ion could decrease resulting from the production of Fe^{2+} complexes, Eq. (10).



3.2. The effect of electric current

Fig. 3 shows the effect of different EC values (10, 20 and 30 mA/cm²) on the efficiency of the removal of aniline in the

electro/peroxydisulphate process (EPDSP). Increasing EC intensity causes increasing removal efficiency of aniline in EPDSP. The maximum removal efficiency was 65% while EC was 30 mA/cm² at the reaction time of 60 min. The decrease in EC to 10 mA/cm² results in the decrease in the removal efficiency of aniline to 42% at a reaction time of 60 min. According to Faraday's law, if the applied EC increases, the eroded metal ions from the anode also increase. If EC density increases, the amount of gas bubbles increase, and their diameters decrease. These two factors—and the increase in the amount of hydrogen gas bubbles—increase the efficiency of removing pollutants. These bubbles enmesh molecules of pollutants and they cause flock growth and thereby produce more sludge [9]. Wu et al. [27] showed that at the beginning of the reaction, EC has a slight effect on the decolorization of the acid orange 7, and when EC increases, the decolorization process of acid orange 7 will be carried out faster. They also reported that when there is Fe²⁺ ion, EC increases, and this enhancement can produce more sulphate radicals and, according to Eq. (11), increase electron transfer. The production of more sulphate radicals and decomposing and removing pollutants are its results. According to this study, when EC increases, removal efficiency increases as well.



3.3. The effect of peroxydisulphate

Fig. 4 shows the effect of different concentrations of PDS on the efficiency of removing aniline (40 mg/L) in the EPDSP at different reaction times. According to Fig. 3, when the amount of PDS—as a main factor for production of the sulphate radical—increases, the efficiency of removal increases significantly. So, when the concentrations are 10, 20, 30 and 40 mM, after 60 min, the efficiencies would be 27%, 47%, 65% and 73.6%. Increasing the dose of PDS increases the production of hydroxyl radicals and sulphate radicals, and subsequently increases the efficiency of aniline removal in aqueous solution. Hence, increasing the amount of PDS to a certain concentration can increase the production of sulphate radical and thereby increase the removal efficiency. Instead, Eq. (12) its excessive rise and consumption of the sulphate radical, can decrease the

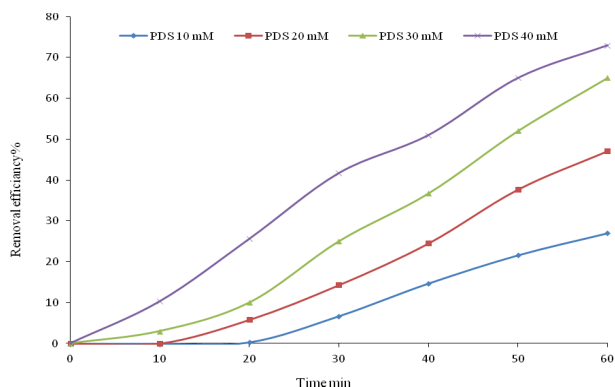


Fig. 4. The influence of persulphate concentration on aniline removal in the electro/Fe²⁺/S₂O₈²⁻ process (aniline = 40 mg/L, iron = 15 mM/L, electric current = 30 mA/cm², pH = 3).

efficiency of removal [8]. Note that in this study, the added dose of PDS was lower than the effective amount, maximum to act as a radical consumer, according to Eq. (12), and so, increasing its dose did not decrease the removal efficiency.



Cai et al. [9], Liang et al. [28] and also Astereki et al. [3] showed that the reason for this phenomenon is the increase in reactive reactants in the solution. Panbehkar Bishe and Ayati [21] showed that free increment in persulphate radicals, which is caused by radiation in the presence of water, can produce more hydroxyl radicals in the environment. By increasing the dose of PDS, the concentration of sulphate and hydroxyl radicals will increase. As a consequence, the removal efficiency increases.

3.4. The effect of iron ion

Fig. 5 shows the effect of FeSO₄ concentration—as a source for producing Fe²⁺ ions—on the removal efficiency of aniline, in the range of 5–20 mM. The results show that increasing the concentration of Fe ions can increase the efficiency of electro/PDS process. The highest removal efficiency (70.2%) was obtained in 20 mM of FeSO₄. Considering that in this range, the dose of used Fe²⁺ ions was not enough to limit the process, and, therefore, 20 mM of FeSO₄ ion was selected as the optimal dose for the next few steps. Fe²⁺ ions increase the amount of aniline removed from the aqueous solution, because, according to the reaction, four increments of Fe²⁺ concentration can accelerate conversion of PDS to sulphate radical [23,29]. A high concentration of Fe²⁺ ions improves decomposition of PDS into sulphate radicals. The other studies on removal of acid orange 7, furfural, acid red 14, and bisphenol A, including Yang et al. [30], Samarghandi et al. [26], Rasoulifard et al. [22] and Lin et al. [24] corresponded to the results of this study.

3.5. The effects of the initial concentration of aniline

Fig. 6 shows the effects of the initial concentration of aniline on the removal efficiency in electro/Fe²⁺/PDS process.

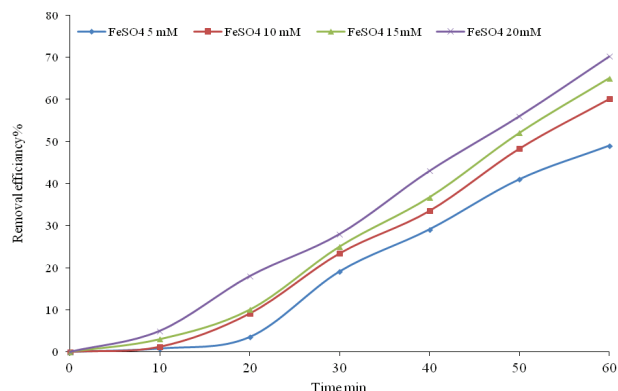


Fig. 5. The influence of iron ion concentration on aniline removal in the electro/Fe²⁺/S₂O₈²⁻ process (aniline = 40 mg/L, electric current 30 mA/cm², persulphate = 30 mM/L, pH = 3).

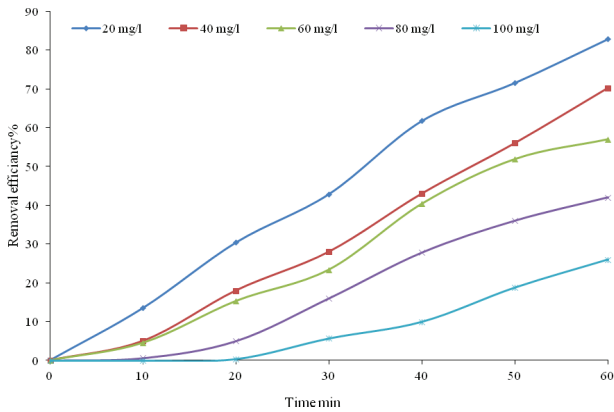


Fig. 6. The influence of initial aniline concentration on this removal in the electro/Fe²⁺/S₂O₈²⁻ process (persulphate = 40 mM/L, electric current = 30 mA/cm², iron = 20 mM/L, pH = 3).

According to the results, when the initial concentration of aniline increases, the efficiency of the process decreases. So, when the concentration is 100 mg/L and the reaction time is 60 min, just 26% of aniline is removed. However, at the same time, when the concentration is 20 mg/L, the efficiency is 82%. Owing to this practical point, checking the effect of the initial concentration of pollutants on the rate of removal is an important parameter. As we noted before, when the concentration of pollutants increases, the removal efficiency decreases. This is because of the proportion of aniline molecules to the sulphate radicals and also the use of intermediate substances that use free radicals. Intermediate substances like nitrobenzene, aminodiphenyl and diphenylhydrazine may produce the degradation of aniline at the present PDS [24,27], and following that, the removal efficiency decreases at higher concentrations. These results corresponded to the studies of Devi et al. [31] and Lin et al. [24].

3.6. The effects of reaction time

The results of the removal efficiency of aniline with optimal values of variables in the electro/Fe²⁺/PDS process are shown in Fig. 7, which implies that the constant removal rate for 20 and 40 mg/L aniline is more in accordance with the first-order kinetics model. According to the results, the review of the experimental data gained over time shows that the reaction velocity follows the pseudo-first-order kinetics. And the decomposition reactions of aniline are shown in Eqs. (13) and (14):

$$-r_{\text{aniline}} = k_{\text{overall}} [\text{aniline}] \tag{13}$$

$$-\ln(C_t/C_0) = k_{\text{overall}} \cdot t \tag{14}$$

where C_t is the aniline concentration at time t (mg/L), C₀ is the initial concentration (mg/L) and k_{overall} is the constant of rate of the pseudo-first-order reaction (1/min) obtained from the slope of the plotted -ln(C_t/C₀) vs. time.

3.7. The effects of synergies of variables

To determine the effect of the synergies of different variables on the removal efficiency of aniline in electro/Fe²⁺/PDS

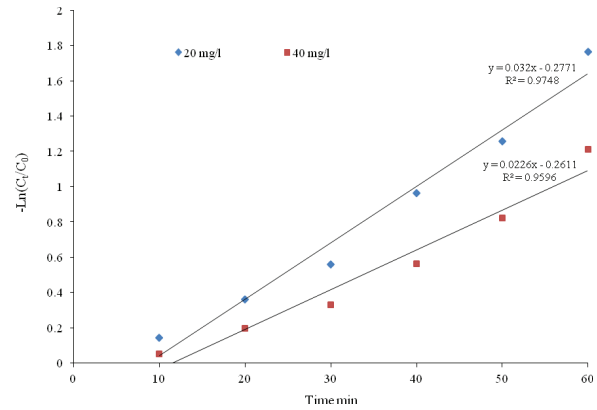


Fig. 7. The diagram of electro/Fe²⁺/S₂O₈²⁻ process kinetic (persulphate = 20 and 40 mM/L, iron = 20 mM/L, electric current = 30 mA/cm², pH = 3).

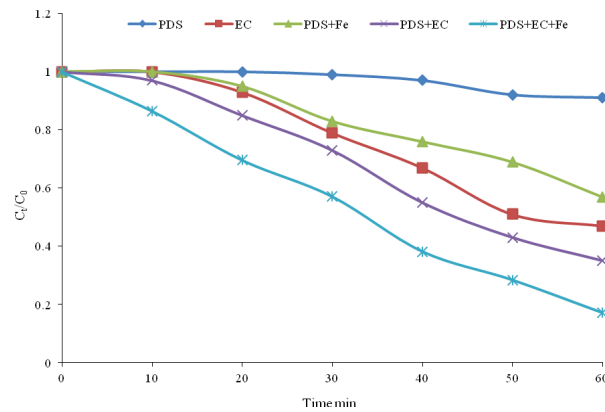


Fig. 8. Decomposition change the aniline removal amount by electro/Fe²⁺/S₂O₈²⁻ process in the presence of optimum amounts of variables types (aniline = 20 mg/L, persulphate = 40 mM/L, iron = 20 mM/L, electric current = 30 mA/cm², pH = 3).

process, the effect of each factor, individually and in combination, with the optimal values, have been investigated at reaction times of 10–60 min. As shown in Fig. 8, these separate processes of PDS and EC individually were able to remove 8.9% and 53% of aniline in aqueous solution at 60 min, respectively. Also, the PDS + Fe and PDS + EC compound process were able to remove 43% and 65% of aniline in aqueous solution, respectively. However, the compound process of PDS + EC + Fe can remove aniline up to 83%, showing the positive impact of a combination of parameters in this process.

The effects of different variables on removing aniline, which have efficient values, were studied individually and in a consolidated manner. When PDS is used individually, the removal efficiency was the lowest. Zhao et al. [32] believed that the limited oxidation power of S₂O₈²⁻ (E⁰ = 2.01 V) is related to these results, and it is not able to convert to free radicals in the solution. Anodic oxidation has singly removed about 53% of aniline, which is due to the production of hydroxyl radicals and water oxidation in the anode, but since the dose of these hydroxyls was not enough, the reaction was not done completely in 60 min [27]. By adding Fe to PDS (PDS/Fe²⁺ process),

the removal efficiency increased up to 34%. This improvement in removal efficiency shows that Fe reacts with PDS and, following that, produces sulphate radicals. Hence, the pollutant oxidation mostly occurs in the beginning of the process [33]. Combining both systems, $S_2O_8^{2-}$ and EC (PDS/EC process) in the reactor, improves the removal aniline efficiency up to 65%, which is caused by higher production of sulphate radicals due to electricity flowing through an electron transfer reaction [27]. When the electrochemical process was conducted in the presence of all the variables (PDS/EC/ Fe^{2+}), sulphate radical production with frequent reduction of Fe ions and with electrochemical production increased the removal efficiency up to 83%. It had the highest efficiency in this system. On the other hand, the increase of EC creates a synergistic condition in combining the PDS/ Fe^{2+} process, which increases the removal efficiency [27].

For determining the effect of different radicals (hydroxyl and sulphate) on the removal efficiency of aniline in electro/ Fe^{2+} /PDS process, *tert*-butanol can diminish the effect of hydroxyl radical on the aniline degradation. Generally, as a scavenger radical, it has a high solubility and reacts very fast with free $\bullet OH$ [3,25].

In the present study, *tert*-butanol has been added into the reactor in the presence of the optimal amount of variables. Results showed that sulphate radical as main factor on the aniline degradation was domination while hydroxyl radical has negligible effect (2.64%). Same observations have been reported by Zhao et al. [32] and Samarghandi et al. [26].

4. Conclusion

This study showed that the combination of PDS, EC and Fe^{2+} is effective in eliminating aniline. pH, PDS dosage, Fe^{2+} concentration and EC can increase the efficiency of the removal of aniline in the selected process. The optimal conditions for efficient aniline elimination were pH 3, PDS concentration 40 mM, Fe concentration 20 mM, EC 30 mA/cm² and aniline concentration 20 mg/L. Under the above conditions, the removal efficiency was 90% within 60 min. Therefore, using this integrated process for the removal of resistant pollutants is efficient, especially in industrial sewages.

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