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Degradation of 2, 4, 6-trinitrotoluene (TNT) from aqueous solution by coupled electrocoagulation process with persulfate salt

Amir Mirshafiee [1,2] · Mohammad Darvish [1]

Abstract

Purpose The 2, 4, 6-trinitrotoluene (TNT) is the major explosive extensively applied in demolition of structure, military activity, and mining industry. This research was investigating the degradation rate of TNT in aqueous solution using electrochemical combined with persulfate anions.

Methods In this study, the effect of seven independent variables including pH, reaction time, current density, type (NaCl, Na_2SO_4, $NaNO_3$) and concentration of salts, initial concentration of TNT, scavenger type, and persulfate concentration were studied on TNT removal. High performance liquid chromatography (HPLC) was applied to measure TNT concentration.

Results The findings indicated that the optimum conditions for TNT reduction were pH = 5, time = 45 min, current density = 1.75 mA/cm^2, initial concentration of TNT = 25 mg/l, persulfate concentration = 0.3 mg/l, and Na_2SO_4 concentration = 1.75 mg/l which the removal rate of TNT was found to be $88.0 \pm 4.4\%$.

Conclusion According to the findings, electrocoagulation procedure is suggested as an applicable method in TNT reduction from aqueous solution.

Keywords TNT · Red water · Electrochemical · Persulfate

Introduction

The 2,4,6-trinitrotoluene is known as one of the most significant nitro-aromatic compounds which its industrial production is frequently related to civilian and military targets [1, 2]. There are some documents indicated applying numerous procedures to reduce nitro-aromatic compounds, such as advanced oxidation processes (AOPs), adsorption by activated carbon, and conventional biological processes (e.g., activated sludge) [3–10].

The physical-chemical techniques, such as adsorption [11–14] and distillation [15] have been as effective methods to reduce the refractory organics including nitro-aromatic compounds. Rajagopal et al. achieved high efficiency of granular activated carbon for TNT and 2,4-DNT adsorption.

Though, there are many critical problems when applying physical-chemical methods. The organic compounds cannot be degraded and they remain in the distillation residue or the absorbents, which will still threatening living beings [11, 16, 17].

The AOPs have attracted attention as a result of the capability to oxidize and eliminate organic materials in TNT red water. Various AOPs in TNT red water reduction have been introduced, for instance, wet oxidation [18], supercritical water oxidation [19], Fenton oxidation and zero-valent iron [20], and photocatalytic oxidation [21–23]. Though, the AOP by-products are very complex, accordingly limiting the use of these approaches [12, 24–27]. Most recently, there has been an increasing attention in applying electrochemical treatment for actual wastewater due to no production of any secondary pollutants and no harsh reaction conditions are required [28, 29]. In situ electrochemical reactors produce oxygen-based radicals, hypochlorite, nitrogen oxides, ozone, and other oxidizing agents [30–32]. Through such procedures, the organic compounds can be oxidized directly or indirectly. While electrochemical treatment demonstrates the capable potential to the efficient treatment of wastewater, there are limited investigations on using electrochemical method in TNT red water.
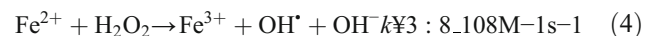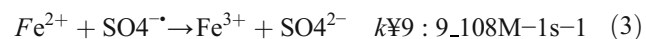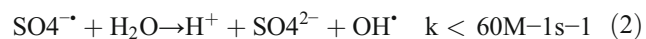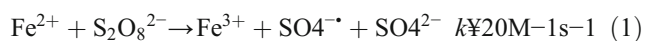
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Among AOPs methods, electrochemically activated persulfate (EC/PS) procedure has been considered as an efficient technique in wastewater treatment due to its high ability in degradation of various pollutants [29, 33, 34]. Though, the hybrid EC/PS methods is still not extensively recognized, more attention have been paid as it is comparatively low-cost, safe, and effective compared with other general activators such as heat, photo, hydrogen peroxide activated persulfate, phenols, metal, and chelated metal. In the hybrid EC/PS process, Fe^{2+} is released from a sacrificial iron electrode at a definite rate under direct current and electro-generation of hydrogen peroxide (H_2O_2) may happen in the EC systems. Consequently, both $OH^{\bullet}$ and $SO4^{-\bullet}$ are produced in the system based on the reaction Nos. 1 to 4 [35, 36]. $SO4^{-\bullet}$ is a strong aqueous oxidizing species with a redox potential estimated to be 2.6 V, similar to that of the $OH^{\bullet}$, 2.7 V [37, 38].

Furthermore, $SO4^{-\bullet}$ have a longer half-life than $OH^{\bullet}$ due to their preference for electron transfer reaction, while $OH^{\bullet}$ can participate in a diversity of reaction with equal preference [33, 39]. Therefore, $SO4^{-\bullet}$ are more selective than $OH^{\bullet}$ for the oxidation of many organic pollutants [40].

$$Fe^{2+} + S_2O_8^{2-} \rightarrow Fe^{3+} + SO4^{-\bullet} + SO4^{2-} \quad k¥20M{-}1s{-}1 \quad (1)$$

$$SO4^{-\bullet} + H_2O \rightarrow H^+ + SO4^{2-} + OH^{\bullet} \quad k < 60M{-}1s{-}1 \quad (2)$$

$$Fe^{2+} + SO4^{-\bullet} \rightarrow Fe^{3+} + SO4^{2-} \quad k¥9:9\_108M{-}1s{-}1 \quad (3)$$

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^{\bullet} + OH^- k¥3:8\_108M{-}1s{-}1 \quad (4)$$

Regarding the growing attention of the processes of electrochemical advanced oxidation for organic pollutants treatment due to the high ability of electrochemical systems to work at ambient temperature and pressure as well as strong performance and pollution problem prevention, this study was aimed to introduce a hybrid system based on the electrochemical and persulfate for 2,4,6-trinitrotoluene degradation from aqueous environment.

Materials and methods

Chemicals

The applied chemicals including TNT (purity = 99%), $Na_2S_2O_8$ (purity = ≥99.5%) $NaNO_3$ (purity = 99%), Na_2SO_4 (purity = 99%), NaCl (purity = 99%), and humic acid (99%) were supplied from Sigma-Aldrich with analytical grade. In order to working solution preparation, the specified amounts of each chemical were dissolved in the deionized water. The solution of $Na_2S_2O_8$ was prepared immediately prior to usage.

Setup of electrocoagulation process

The schematic of electrocoagulation process is shown as Fig. 1. The batch reactor is consisted of reactor (10 cm × 10 cm × 15 cm) with operating volume 1 L and two pairs of AL/Fe electrodes with monopolar connection and working surface of 4 cm^2. Each pair of two electrodes were vertically situated and parallel to each other with 2 cm of distance between them. To establish the continuous current, direct current (CD) power supply was applied and also to obtain complete mixing during experiments, the magnetic stirrer was employed.

Experimental procedure

Prior to each experiment, the electrodes surfaces were washed by NaOH (100 mM) many times and then rinsed by double distilled water. One mL of TNT solution with the difinite concentration was prepared and discharged into the reactor. Then by adding a determined NaCl concentration as supporting electrolyte, experimentation started which then by altering the regulator of voltage, CD was established to the desired value. When removing the TNT by coupled electrocoagulation process with persulfate salt, samples were extracted at the desired interval, filtered via a filter paper (Whatman, Cat No. 1441–150) and then subjected to the TNT analysis. In this study, the effect of seven independent variables including pH, reaction time, current density, type and concentration of supporting electrolyte, initial concentration of TNT, scavenger type, and persulfate dose were investigated.

Analytical procedures

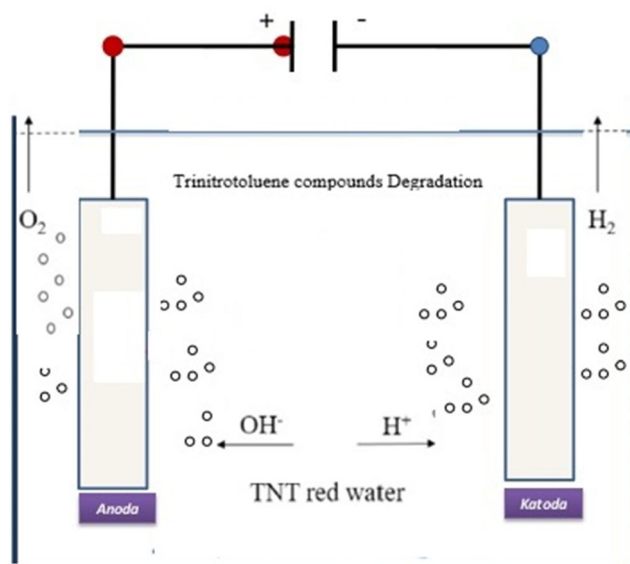The residual concentration of TNT was measured by high performance liquid chromatography (HPLC-Hatch USA).

Fig. 1 Schematic of electrocoagulation process setup

Through HPLC running, 20 mL solution sample was injected and flowed at 1.0 mL/min of rate.

Result and discussion

Effect of pH value

Figure 2 displays the TNT reduction in the coupled electrocoagulation process with persulfate salt at different initial pH levels.

As depicted in Fig. 2, the TNT degradation in the coupled electrocoagulation process with persulfate salt was pH dependent. With growing the pH solution from 3 to 5, the removal rate of TNT improved from $33.33 \pm 1.67\%$ to $42.93 \pm 2.15\%$ and after that promptly declined and reached to $22.67 \pm 1.13\%$. It is obvious that the degradation rate of TNT declines meaningfully with rising pH amounts [41]. The observation may be clarified as Eqs. (5) and (6).

$$S_2O_8^{2-} + H^+ \rightarrow HS_2O_8^- \tag{5}$$

$$HS_2O8^- \rightarrow SO4^{-\bullet} + SO4^{2-} + H^+ \tag{6}$$

The sulfate radical's generation rate converted from persulfate anions via Eq. (5) could be enhanced by rising the wastewater acidity. Yu et al. [42] investigated electrochemical activated persulfate for degradation of diuron and reported that when the initial pH reached alkaline condition, $FeOH^{2+}$ and $Fe(OH)_3$ occupy a main part instead of Fe^{2+}, and the formation of $FeOH^{2+}$ and $Fe(OH)_3$ reduced the free Fe^{2+}concentration, therefore falling the activation of sulfate radicals. In addition, the regeneration of Fe^{2+} from ferric ions at the cathode was also withdrawn, additional decelerating the diuron degradation rate. Therefore, it can be concluded that the EC/PS process can be effectively used in acidic conditions compared to in alkaline conditions.
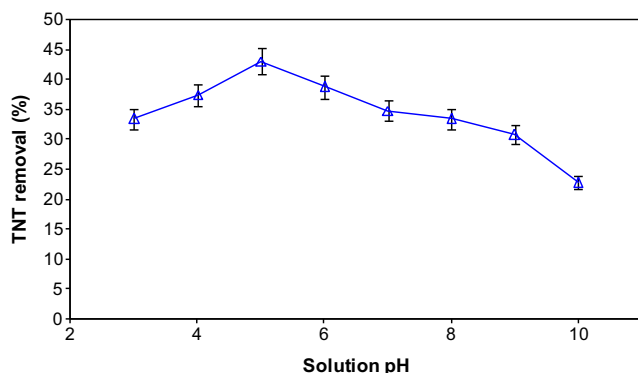
Effect of electrolysis time

Figure 3 indicates the variation of TNT reduction rate by electrolysis time when other variables were considered at constant.
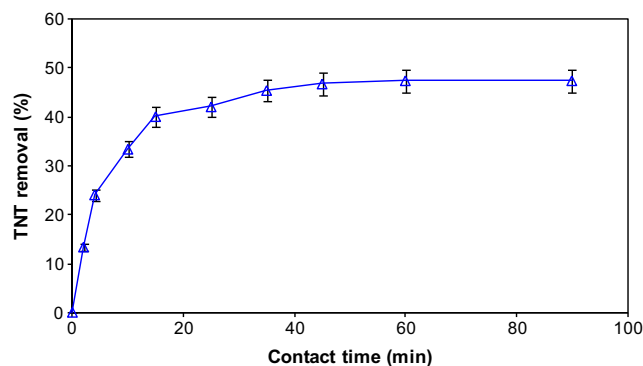
As can be seen in Fig. 3, with growing the reaction time from 5 min to 45 min, TNT removal percentage sharply increased from 13.3% to 46.7% and after that TNT removal percentage not significantly improved. This could be associated to the concentration variations and organic pollutants in TNT red water through electrochemical treatment. Murati et al. [43] applied electro-fenton to eliminate TNT which revealed that by increasing the electrolysis time from 5 min to 30 min, 75%, 86% and 95% of the initial TNT content is reduced, respectively for 60, 100 and 250 mA current intensity values.
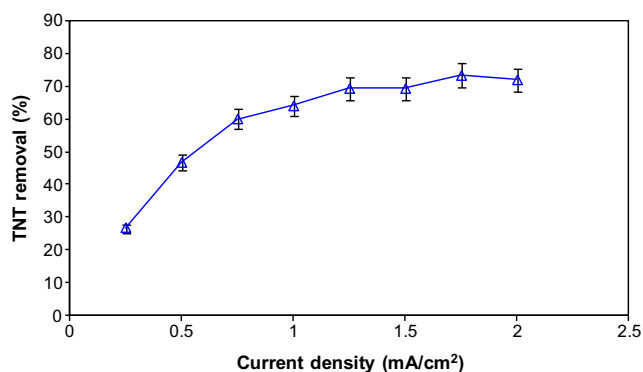
Effect of applied current

The removal efficiency of TNT by the coupled electrocoagulation process with persulfate salt at different applied currents ranging from 0.25 mA/cm^2 to 2 mA/cm^2 are shown in Fig. 4.

The results showed that the TNT degradation in the coupled electrocoagulation process with persulfate salt was current density dependent. The TNT degradation rate is found to be initially increasing linearly with current density and reaches its highest value. As the applied current increased from 0.25 mA/cm^2 to 1.75 mA/cm^2, the reduction rate of TNT significantly improved from 26.67 ± 1.33 to $73.33 \pm 3.67\%$. After this point, the TNT removal drops to $72.0 \pm 3.6\%$. This behavior could be due to the exacerbation of the oxygen evolution reaction as current density rises. At this time, the energy consumption could be enhanced while current density is too high.

The intensification of current density will rise the driving force of charged particles in the solution and lead to an increase in the OH$^\bullet$ groups concentration, which rise the probability of interaction between organic matter and charge. This

Fig. 2 Effect of pH value on TNT degradation by coupled electrocoagulation process with persulfate salt (TNT concentration: 75 mg/L, reaction time: 25 min, NaCl concentration: 0.5 g/L, and current density: 0.5 mA/cm^2)

Fig. 3 Effect of reaction time on TNT degradation (TNT concentration: 75 mg/L, pH: 5, NaCl concentration: 0.5 g/L, current density: 0.5 mA/cm^2)

Fig. 4 Effect of Applied Current on TNT degradation (TNT concentration: 75 mg/L, pH: 5, NaCl concentration: 0.5 g/L, current density: 0.5 mA/cm^2, and reaction time: 45 min)

could benefit the oxidation of organic pollutants and expand the effect of degradation.

Doppalapudi et al. [44] and Palaniswamy et al. [45] surveyed the TNT electrochemical degradation and conducted numerous experiments to evaluate the efficiency progression. Undeniably, they exhibited that the electrochemical developments can be successfully applied for TNT degradation and readily taking place under both anoxic and oxic conditions. The kinetic rates improved with the enhancement in current, but at higher density currents (0.79 mA/cm^2 and 0.96 mA/cm^2), mass transfer rates dominated the reduction rates under both anoxic and oxic conditions.

Consistent with our results, Jiang et al. [46] indicated the same interpretation when applied electrochemical treatment for TNT red water. They reported 100 mA/cm2 of current density as the largest anode efficiency. The difference in results is due to the variation in working conditions applied (TNT concentration, reaction time, current density, etc).

Effect of type and concentration of electrolyte

A series of experiments by coupled electrocoagulation process with persulfate salt were carried out to evaluate effect of electrolyte type and concentration on process performance (Fig. 7).

As seen in Fig. 5, the TNT removal efficiency by coupled electrocoagulation process with persulfate salt with application of different type of electrolyte is summarized in the descending order as shown below.

Na_2SO_4(78.67 ± 3.93%) > NaCl(73.33 ± 3.67%) > $NaNO_3$(68.0 ± 3.41%)
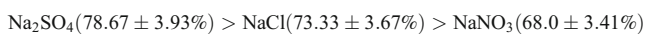
As expected, the incresing of electrolyte concentration has an posetive effect on degradation effieiciency of TNT by coupled electrocoagulation process with persulfate salt. When electrolyte concentration incresed from 0.5 to 1.75 g/L, the reduction rate enhanced from 78.67 ± 3.93% to 87.33 ± 4.3 and further incresing of electrolyte concentration lead to

degradation efficincy diminishing to 84.27 ± 4.21% at 2.5 g/L of electolyte concentration. In order to flow the electric current in the electrochemical process, presence of minimal conductivity in the solution is critical, which is obtained by the addition of salts such as NaCl and Na_2SO_4 [47].

The presence of Na_2SO_4 in optimum concentration in aqueous media lead to increasing of electrical conductivity, electrical energy reduction, and accelerates the production of hydrogen around the cathode [48]. At a constant current density (or electrical potential), the increseing electrolyte concentration caused electrical conductivity inceerasing too. Thus, the electric current flows more intensely, which improve Fe^{2+} production and $SO_4^{\cdot-}$ radicals generation and, ultimately, resulting in enhanced degradtion efficiency [49]. In line with our study, Palaniswamyet al. [45] evaluated the effect of electrolyte type, molar concentration of the electrolyte on the decline kinetics of TNT. Their findings indicated that the reduction rate of TNT enhanced with an improving in molar concentration of the electrolyte (6 and 9 mM).

Effect of Persulfate dosage

In order to enhancing reduction rate of TNT, the coupled electrocoagulation process with persulfate salt with optimal concentration of PS is necessary to the treatment of wastewater. Figure 6 demonstrates the effect of PS dosage on the coupled electrocoagulation process with persulfate salt behavior.

It clearly indicates that the TNT elimination rate displays a growing trend with an enhancement of persulfate concentration. As, approximately 90% of TNT was reduced when the concentration of persulfate was elevated to 0.3 mmol/L. However, the removal trend was reached to a constant value when the PS concentration was improved more than 0.3 mmol/L. The maximum reduction rate was found in the coupled electrocoagulation process with persulfate salt system because Fe^{2+} generated on the iron electrode can activate persulfate to produce $OH^\cdot$ and $SO4^{-\cdot}$. The findings indicated that combination of EC and PS can meaningfully boost the reduction rate of TNT.

Yu et, al. indicated that the diuron concentrations remain constant when treated by persulfate alone, and the diuron reduction rate was approximately negligible owing to the low oxidation ability of PS. Equally, only 15% of diuron was decrease in the EC procedure. The removed part was undoubtedly attributed to the surface chelation or electrostatic attraction of Fe (OH)n precipitates generated in the system. Though, the EC/PS system expressively promoted the diuron degradation rate, and above 77% of diuron was reduced within 15 min in the samples with 0.5 mM initial PS dosage [42].
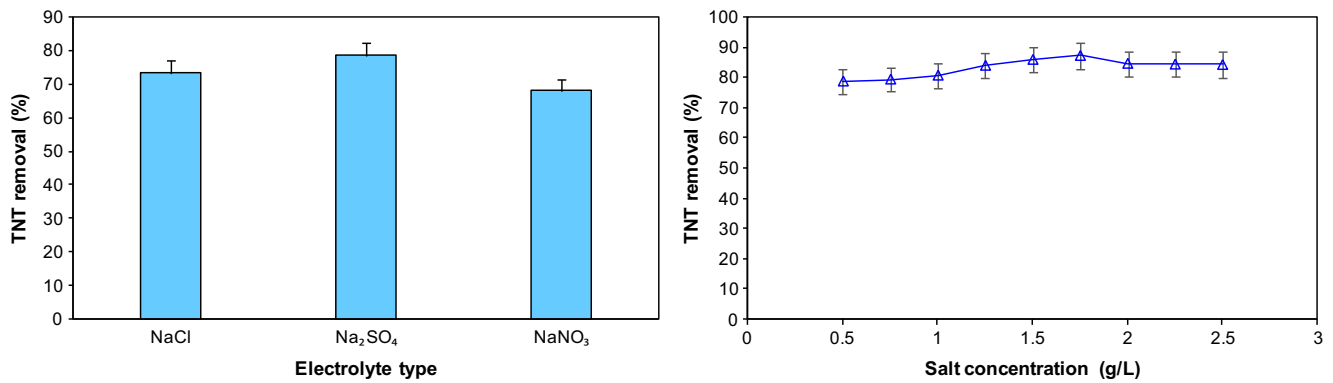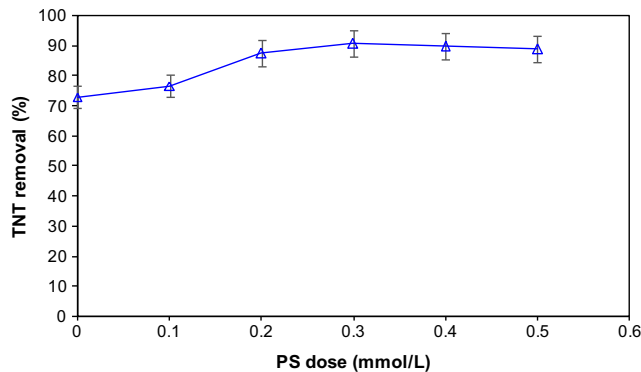
Fig. 5 Effect of type and concentration of electrolyte (TNT concentration: 75 mg/L, pH: 5, current density: 0.5 mA/cm^2, reaction time: 45 min, and current density: 0.5 mA/cm^2)

Effect of initial TNT concentration

To find the variation of degradation efficiency of TNT by coupled electrocoagulation process with persulfate salt with changing initial concentration of TNT from 25 to 150 mg/L, the experiments were conducted and exhibited in Fig. 7.

As can be seen, as the initial TNT concentration elevated from 25 to 150 mg/L, the performance of TNT degradation by coupled electrocoagulation process with persulfate salt declined $88.0 \pm 4.4\%$ to $59.33 \pm 2.97\%$, and similar to reported observation in the literature [50, 51]. At constant oxidant content, the increasing concentration of the organic contaminant lead to minimizing available oxidation potential and as a results, the performance of AOPs process is decreased [49]. In addition, at high concentrations of contaminants, the formation of intermediate compounds in solution may consume oxidizing agents, so the removal efficiency of AOPs is reduced [49, 52]. Sepyani et al. [51] investigated activation of PS by continuously electro-generated magnetic nanoparticle for degradation of amoxicillin and indicated that the reduction rate of amoxicillin decreased with growing the initial concentration of amoxicillin.
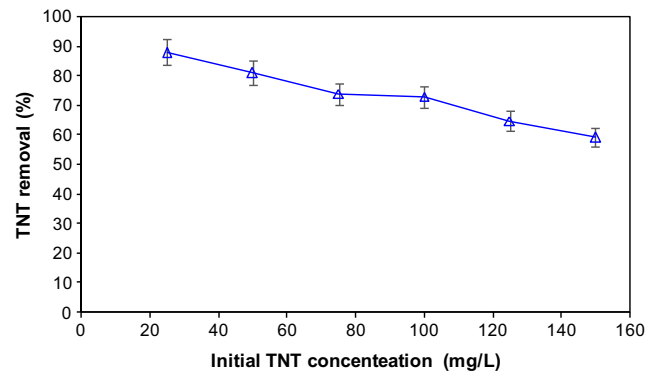
Effect of radical quenchers

The formation of activated radicals including $SO_4^{-\bullet}$ and $OH^\bullet$ as well as the oxidative intermediate species by coupled electrocoagulation process with persulfate salt has been indirectly examined with the use of scavengers of these species. In these experiments, an evaluation is made between the original TNT degradation efficiency values with those obtained after addition of glucose as a quencher in the initial solution under otherwise identical conditions.

As revealed in Fig. 8, the serious inhibition of TNT removal efficiency is observed with increasing glucose concentration in experiment. The control experiment showed the TNT removal efficiency of 88.0 4.4% and reduced to $11.6 \pm 0.58\%$ at 100 mg/L of glucose concentration. When glucose was added, the $OH^\bullet$ and $SO_4^{-\bullet}$ radicals were consumed, leading to deterioration and degradation of TNT efficiency. Our findings were similar to the study conducted by Liang and Su who used a chemical probe method on thermally activated persulfate oxidation process to identify the oxidizing species, and determined that $OH^\bullet$ was the major radical at solution at pH of 10 [53].

Fig. 6 Effect of persulfate dosage on TNT degradation (TNT concentration: 75 mg/L, pH: 5, current density: 0.5 mA/cm^2, current density: 0.5 mA/cm^2, and reaction time: 45 min)
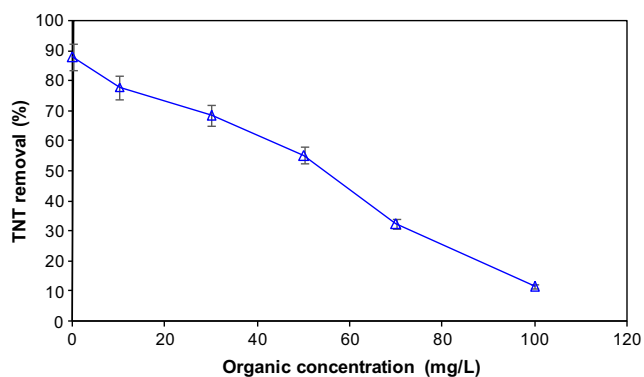

Fig. 7 Effect of initial TNT concentration on degradation efficiency (pH: 5, current density: 0.5 mA/cm^2, Na2SO4 concentration: 1.75 g/L, current density: 0.5 mA/cm^2, and reaction time: 45 min)

Fig. 8 Effect of persulfate dosage on TNT degradation (TNT concentration: 25 mg/L, pH: 5, current density: 0.5 mA/cm², current density: 0.5 mA/cm², and reaction time: 45 min)

Conclusion

This study showed effects of persulfate addition to electrocoagulation for TNT removal from aqueous solution. The influence of operating variables comprises solution pH, reaction time, initial TNT concentration, PS dose was assessed. Based on the obtained data, the subsequent results can be concluded as:

- The optimal pH solution for TNT reduction by coupled electrocoagulation process with persulfate salt was 5.
- The Na_2SO_4 was the best electrolyte type for TNT removal by coupled electrocoagulation process with persulfate salt.
- The optimum reaction time and persulfate dose were 45 min and 0.3 mM, respectively.

Declarations

This research was approved by the Ethics Committee, Environmental Health Research Committee of Baqiyatallah University of Medical Sciences (IR.BMSU.REC.1398.330) and project number # 98000370.

Conflict of interest The authors announce that they have no conflict of interests.

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