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The photocatalytic degradation and kinetic analysis of BTEX components in polluted wastewater by UV/H_2O_2-based advanced oxidation

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ABSTRACT

The present work mainly deals with the UV-based advanced oxidation; UV/H_2O_2 were tested in batch reactor systems to evaluate the removal efficiencies and optimal conditions for the photodegradation of Benzene, Toluene, Ethyl Benzene, and Xylene (BTEX) in order to treat the wastewater. The influences of operational parameters such as: the initial concentration of H_2O_2, pH, temperature, initial concentration of BTEX, reaction time, and UV amount on the degradation of BTEX were studied. The obtained results showed that the highest degradation rate occurred during the first 30 min of the reaction time. The optimal conditions of the average and different BTEX concentrations with 0.421, 0.724, 1.11, 1.34, and 1.736 g/L initial concentrations of H_2O_2 and an acidic pH value of 3.1 were applied under three UV lights. Under the optimal conditions, for the average (550 mg/L) and the lowest (210 mg/L) concentration of BTEX, the chemical oxygen demand reduction reached about 90 and 98%, respectively for the UV/H_2O_2 system during the first period of 180 min. A kinetic analysis has been done which showed that a pseudo-first-order kinetic model with respect to BTEX concentration can be used to explain the BTEX degradation for UV/H_2O_2 system.

Keywords: Advanced Oxidation; BTEX; Photodegradation; UV/H_2O_2; Wastewater; COD removal

1. Introduction

In recent years, the petroleum industry has played an important role in the global economy. Oil, as well as its residues contains, mainly, aromatic and aliphatic compounds [1]. The wastewater created in most complexes contains aromatic compounds, including: Benzene, Toluene, Ethyl Benzene, and Xylene (BTEX); these compounds have been proven to be very harmful to the environment and human health. Recent studies have shown that the aromatic fractions are more toxic than the aliphatic fractions, especially, because aromatics (including BTEX components) are known to be more recalcitrant [2].

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There are several well-known conventional techniques that can be applied for the treatment of wastewater, and these divide into three main methods: physical, chemical, and mechanical. A few of the familiar processes include: centrifugation, gravitational separation, filtration, application of coagulants, flotation, and adsorption with activated coal [3]. These conventional treatments are usually combined with biological treatments for greater efficiency.

An advanced oxidation process (AOP) is a powerful technique for wastewater treatment [4,5]. This process utilizes advanced techniques for the decontamination of media containing dissolved recalcitrant organic substances, which would not be removed efficiently by traditional methods. This process is based on two steps: first, the production of strong oxidants (e.g. hydroxyl radicals) which are very reactive, and secondly, quick and nonselective reaction of these oxidants with organic compounds dissolved or dispersed in aquatic media. Some common methods for the production of oxidants are as follows: O_3/UV, H_2O_2/UV, H_2O_2/Fe^{2+}, TiO_2/UV, and TiO_2/H_2O_2/UV. These methods can either be applied individually or in combination. Hydroxyl radicals and ozone play the most important roles in the degradation of toxic organic pollutants.

Oturan et al. [6] used the AOP to remove herbicide diuron from aqueous medium. In order to enhance the oxidation power of Fenton's reagent, they assisted in photochemical and electrochemical oxidations and obtained high mineralization efficiencies for total organic carbon removal under optimal operating conditions.

Ghodbane and Hamdaoui [7] studied the decolorization of C.I. Acid Blue 25 by using methods, such as UV irradiation alone, UV/H_2O_2, and UV/Fe(II). The decolorization rate was increased with the presence of H_2O_2 and Fe(II) compared with UV irradiation alone. They found the best working condition and showed that the tested AOPs were effective for the decolorization of AB25 in aqueous solutions.

Aleboyeh et al. [8] have showed that the photolytic degradation of C.I. Acid Orange 7 azoic dyes in aqueous solution with combined UV and hydrogen peroxide is faster than its mineralization in a continuous circulated photo reactor.

Photodegradation of methyl tertiary butyl ether (MTBE) by UV/H_2O_2 and UV/TiO_2 have been investigated experimentally by Qinhai Hu et al. [9]. They obtained optimal conditions by testing different operational conditions for both systems of UV/H_2O_2 and UV/TiO_2. Their results showed that UV/H_2O_2 system can remove approximately, all the MTBE; however, the removal efficiency was lower for the second method.

Other researches have been conducted for the remediation of hydrocarbon pollution in groundwater [10–12]. The obtained results showed that H_2O_2 provides good removal efficiency in synthetic aqueous solutions. In real polluted groundwater, some salt contents and dissolved organic matters distract the treatment process, and TiO_2 has lower removal efficiency.

The previous investigations show clearly that AOPs can be applied effectively to remove hydrocarbon pollution in water. However, only a few studies have been done directly for BTEX components [13–15]. These researches have generally checked one of four components and a few operational conditions have been studied. Therefore, more detailed experimental investigations are required for a better understanding of the removal efficiency of these very harmful components.

In this study, photodegradation of BTEX compounds in wastewaters are assessed by evaluating the effects of H_2O_2 along with UV lamps in a batch reactor. The experiments are done separately by using H_2O_2, UV light, and a combination of UV/H_2O_2. The synthetic wastewater is made by mixing all the components of BTEX in the ranges that exist in many industrial wastewaters, especially in South Pars petrochemical complexes in Iran. The efficiencies of the methods are obtained by providing different numbers of UV lamps and by changing important parameters, such as pH, temperature, reaction time, initial concentrations of H_2O_2, and different initial concentrations of BTEX compounds. Finally, the photodegradation kinetics of BTEX has been studied at different hydrogen peroxide concentrations for UV/H_2O_2 systems.

2. Experimental

2.1. Chemicals

Benzene, Toluene, Ethyl Benzene, Xylene (99.9%, Merck, Germany), $K_2Cr_2O_4$, H_2SO_4, $HgSO_4$, NaOH, and Ag_2SO_4 (Extra Pure, Merck, Germany) are all analytical reagent grades. Each compound was obtained in the highest purity that is commercially available. A stock solution of all BTEX compounds were made in aqueous solution and stored at 5˚C in the freezer.

The synthetic solution of BTEX substances were prepared by solving of all BTEX components in distillated water to make different initial concentrations of BTEX as presented in Table 1.

Table 1
Initial concentration of BTEX components in synthetic wastewater

Benzene (mg/L)	Toluene (mg/L)	Ethylbenzene (mg/L)	Xylene (mg/L)	COD (mg/L)
2.89	2.93	2.91	2.87	210
3.47	3.52	3.48	3.45	380
4.33	4.37	4.35	4.31	550
5.78	5.82	5.80	5.75	640
8.67	8.71	8.70	8.62	860

For chemical oxygen demand (COD) tests, two standard methods can be used—closed reflux and open reflux. In this research, closed reflux method has been selected.

In a closed reflux method, two necessary synthetic solutions should be prepared with high accuracy as follows:

(1) A high range digestion solution can be prepared by the following procedure: 500 mL of distilled water was added to 10.216 g of $K_2Cr_2O_7$ (primary standard grade, previously dried at 150˚C for 2 h), 167 mL H_2SO_4, and 33.3 g $HgSO_4$. It must be dissolved, cooled to room temperature, and diluted to 1,000 mL.

(2) A sulfuric acid reagent should be made as a catalyst to increase the rate of reaction: Ag_2SO_4 (in reagent or technical grade, crystal or powder) was added to H_2SO_4 at the rate of 5.5 g Ag_2SO_4/kg H_2SO_4. It should stand for one to two days in order to dissolve and then mixed.

2.2. Photo reactor and reaction procedures

A schematic of the used batch reactor system is shown in Fig. 1. For the first section of the experiments, predetermined amounts of H_2O_2 in different ranges were used in the batch reactor in different ranges of pH. H_2SO_4 (0.1 N) and NaOH (0.1 N) solutions were used to set the pH of the solution. In the second section, an ultraviolet lamp (6 W power, LP Hg, 254 nm) was attached to the quartz glass tube in the center of the reactor (reactor volume = 1,000 mL), and two other lamps were attached outside the reactor. An 800 mL BTEX solution (adjusted to the desirable pH) was carefully poured into the Plexiglas tube with the predetermined amount of H_2O_2. Single experiments with only UV lamps were also done in the mentioned batch reactor. A magnetic stirrer, as shown in Fig. 1, was used to adjust the temperature and to ensure that the reactants were completely mixed. Since BTEX compounds are volatile in high temperatures, the reactor was tightly closed. The ultraviolet lamps were then turned-on to start the reaction. Temporal samples were typically taken after 30, 60, 90, 120, 150, and 180 min, from the sampling port at the top of the reactor.

2.3. Sample analysis

For COD analysis using the closed reflux method, the following procedure and apparatus has been used.

First, a 2.5 mL sample was collected from the UV, H_2O_2, and UV/H_2O_2 systems. Then, the desired sample was prepared by adding the synthetic solutions made by the mentioned reagents in previous sections. The sample and reagent quantities for digestion vessels have been shown in Table 2.

When the desired samples were made with the above procedure in the sealed vials, and after about 10 min to reach equilibrium, the vials were put in the COD reactor (Hach Lange, Model: DRB 200, Made in Germany) and kept for 2 h in 150˚C to complete the required reactions. After this process, the vials were removed and cooled for 30 min. A spectrophotometer (Hach Lange, Model: DR 2800, Made in Germany) was used to analyze and read COD values under 600 nm of UV wave lengths.

3. Results and discussion

Some key parameters have been checked to determine the optimum conditions for BTEX removal from wastewater. In the first section of experiments, the optimal conditions, in terms of H_2O_2 dosage, pH,

Table 2
Sample and reagent quantities for digestion vessels

Digestion vessel (Culture tubes) (mL)	Sample (mL)	Digestion solution (mL)	Sulfuric acid reagent (mL)	Total final volume (mL)
16 × 100	2.5	1.5	3.5	7.5

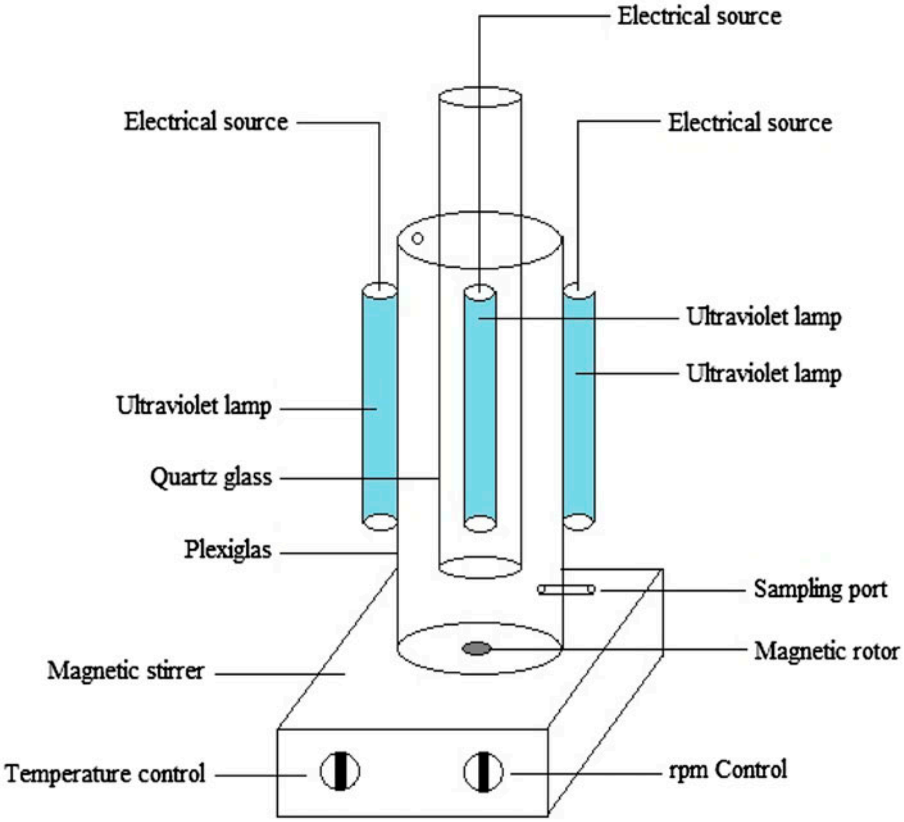

Fig. 1. Schematic diagram of batch reactor system using UV/H_2O_2.

time, and temperature, are obtained by H_2O_2 alone. In the second section, the UV irradiation lights along with the H_2O_2 solution are used in the optimal conditions obtained in the first section.

3.1. Effects of H_2O_2 dosages at different levels of pH

Fig. 2 shows the COD reduction efficiency in the reactor containing 550 mg/L BTEX, which was considered as average-tested COD. In these series of experiments, H_2O_2 at various initial concentrations ($[H_2O_2]_0 = 0.2775$, 0.555, 0.8329, 1.11, 1.9425 g/L) are tested at different pH levels.

Fig. 2 shows that the removal efficiency of COD increases with increasing H_2O_2 concentrations from 0.2775 to 1.11 g/L for all used pH levels. However, a further increase in H_2O_2 concentration to 1.9425 g/L reduces both the rate and extent of COD removal.

Fig. 2 also reveals that the optimal concentration of hydrogen peroxide is about 1.11 g/L for the used initial BTEX concentration. This can be described by using UV/H_2O_2 reactions as shown below [16–19]:

$$H_2O_2 + h\upsilon \rightarrow 2HO^{\cdot} \quad (1)$$

$$H_2O_2 + HO^{\cdot} \rightarrow HO_2^{\cdot} + H_2O \quad (2)$$

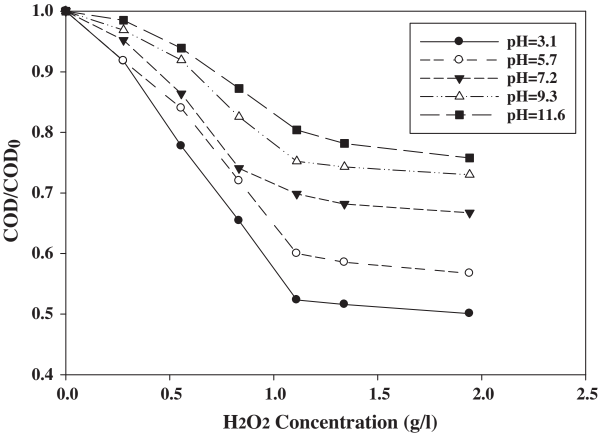

Fig. 2. COD reduction vs. H_2O_2 concentration in different pH levels.

Table 3
The optimal H_2O_2 values for different initial BTEX concentrations

Initial BTEX concentration (mg/L)	Optimal values of H_2O_2 (g/L)
210	0.421
380	0.721
640	1.340
840	1.736

In the aforementioned reactions, reaction 1 is rate limiting due to the slower rate of reaction compared with other reactions. Theoretically, in H_2O_2 processes, as well as in UV/H_2O_2 process, as the initial concentration of hydrogen peroxide increases, higher hydroxyl radicals are produced (Reaction 1) that decompose more organic compounds. However, an optimal concentration exists for hydrogen peroxide; the high concentrations of this oxidant would lead to the further reaction of excess hydrogen peroxide with hydroxyl radical and the formation of $HO_2^{\bullet}$, which reduces the concentration of hydroxyl radicals (Reaction 2).

In Table 3, the optimal concentrations of H_2O_2 at an acidic pH = 3.10 for other different initial BTEX concentrations are presented with the averages as well. The obtained results reveal that the optimum concentration is not a unique value and depends on the initial BTEX concentration. However, these results depict that a higher BTEX initial concentration needs a higher hydrogen peroxide concentration to reach optimum condition.

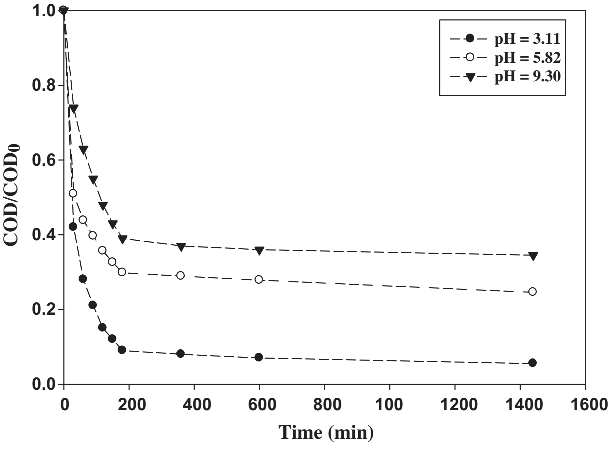

Fig. 3. COD reduction with time in H_2O_2 solution system at different levels of pH (H_2O_2 = 1.11 g/L).

3.2. Effects of reaction time on H_2O_2 system

Time consumption is an important factor for each experimental work in batch or semi-batch reactors. Fig. 3 shows COD reduction with time at different levels of pH and specified hydrogen peroxide concentration. As it is clear from this figure, for all ranges of pH, the curves have become a straight line after about 180 min, which means that the main BTEX removal occurs during this period of time. The same results were found for other levels of pH and hydrogen peroxide concentrations; as a result, this period of time (180 min) was considered as an optimal time for the rest of the experiments.

3.3. Effects of UV light

The effects of UV lights (without hydrogen peroxide) on the removal efficiency of BTEX are shown in Fig. 4. The amount of energy input for 1, 2, and 3 UV lights are 0.0001, 0.0002, and 0.0003 J/min mL, respectively in the batch reactor.

It is obvious from the figure that the BTEX photodegradation is much more effective when the number of UV lights is increased.

Increasing the amount of irradiation helps the improvement of mineralization reactions of BTEX compounds. It should be considered that this method does not have high efficiency alone without using hydrogen peroxide, a fact which is obvious from high amounts of COD remaining after 180 min.

3.4. Combination of UV light and H_2O_2

This process includes H_2O_2 injection and mixing followed by a reactor that is equipped with UV lights. During this process, ultraviolet radiation is

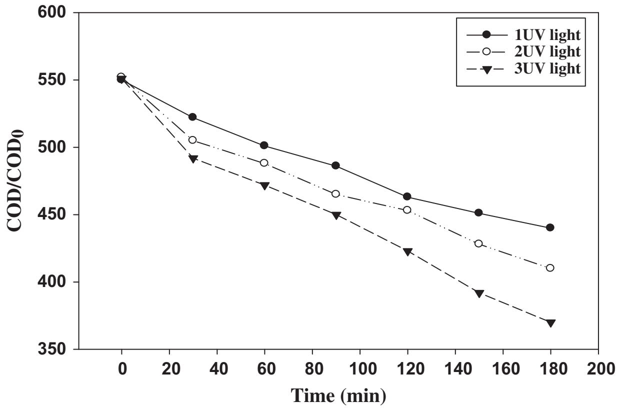

Fig. 4. COD reduction using different number of UV lights in optimal condition ($[BTEX]_0$ = 550 mg/L, pH = 3.11).

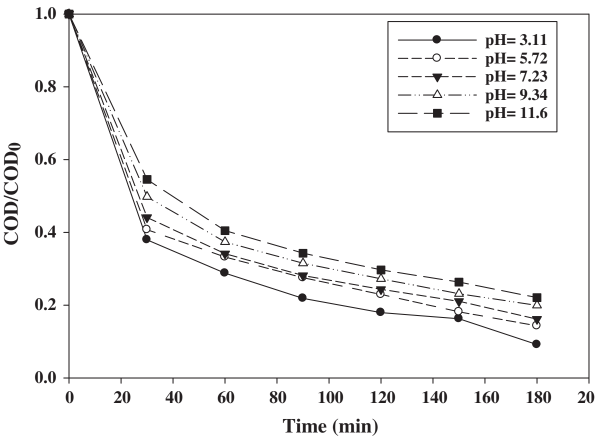

Fig. 5. COD reduction with time in various pH levels at $[H_2O_2] = 1.11$ g/L and three UV lights.

used to cleave the O–O bond of hydrogen peroxide and increase the generation of the hydroxyl radicals. These reactions were presented in Eqs. (1) and (2) [16–19].

3.4.1. Effects of pH

Another key parameter which affects the efficiency of AOP systems is pH. Therefore, some experiments were performed to obtain the COD reduction values vs. time in different levels of pH for obtaining average concentration of BTEX (550 mg/L) in a UV/H_2O_2 system.

According to Fig. 5, changing the pH affects the COD reduction significantly. The figure reveals that the BTEX reduction is the highest at acidic pH and decreases when the pH is increased. The maximum COD reduction occurs at the acidic pH around three. When pH is decreased to lower than three, COD reduction is increased, respectively; however, it should be considered that working at lower pH levels use more acid to adjust the desired pH of solution and may not be practical in industrial applications. The reason for the lower BTEX removal at high levels of pH can be attributed to the instability of H_2O_2, since H_2O_2 decomposes rapidly at alkaline conditions. On the other hand, working at lower levels of pH ($pH < 4$) deactivates some intruder species, specifically carbonate and bicarbonate ions, which leads to a better degradation rate [20].

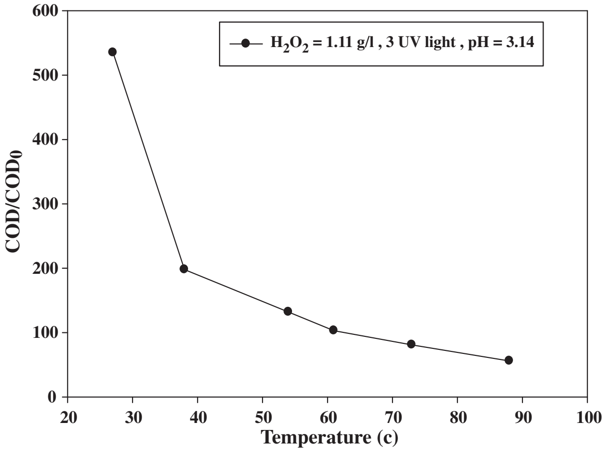

Fig. 6. COD reduction with temperature in optimal condition at various temperatures ($pH = 3.11$, $[H_2O_2] = 1.11$ g/L, $t = 30$ min), three UV lights.

3.4.2. Effects of temperature

A magnetic stirrer was used to adjust the temperature between 26 and 90˚C in the batch reactor. Previous research shows that for some organic pollutants, such as pesticides, increasing the temperature between 40 and 75˚C have no influence in the rate and extent of degradation of the pollutants [21]. On the other hand, a significant enhancement of COD reduction efficiency during the wastewater treatment was obtained when the temperature was increased from 40 to 60˚C [22]. Therefore, it seems that the effects of temperature are related to the nature of pollutants. The results of this research for UV/H_2O_2 system states that the BTEX degradation is increased considerably by increasing the temperature. The obtained data after 30 min for each temperature at average BTEX concentrations are plotted in Fig. 6.

Fig. 6 shows that the efficiency of BTEX removal increases by increasing the temperature from 27 to 86˚C. This proves the fact that increasing the temperature would increase the production rate of hydroxyl ions and consequently the extent of COD removal. However, it should be considered that by increasing the temperature, a certain level of energy and cost is needed which may not be practical at industrial scales.

3.4.3. Effects of different initial BTEX concentration on UV/H_2O_2 system

Fig. 7 shows the effects of initial BTEX concentrations ($[BTEX]_0$) on the removal efficiency of BTEX in selected UV/H_2O_2 system. Initial BTEX concentration is an important operational parameter because a large variation of BTEX concentrations exist in industrial wastewaters, especially in wastewaters of petrochemical complexes.

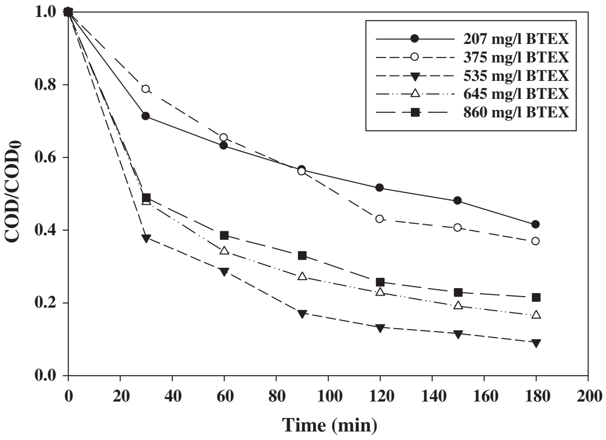

Fig. 7. Effects of initial BTEX concentrations on the removal efficiency of BTEX in UV/H_2O_2 system (1.1 g/L H_2O_2, pH = 3.12, three UV lights).

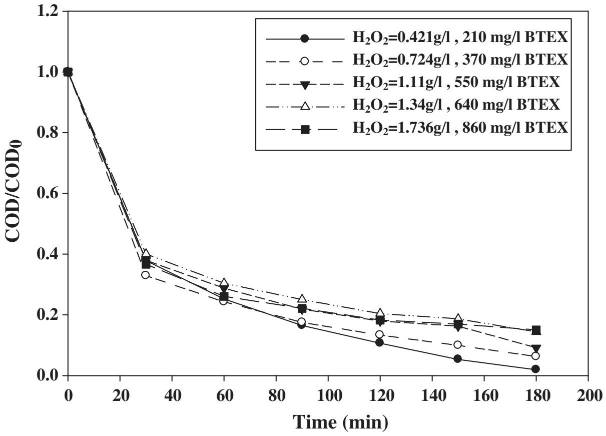

Fig. 8. COD reduction for UV/H_2O_2 system in different ranges of $[BTEX]_0$ (3 UV light = 18 W, pH = 3.10 and optimal H_2O_2 concentrations).

As it is obvious in Fig. 7 for BTEX concentrations in the range of 0.2, 0.267, and 0.40, the elapsed time shows that when the final COD values are decreased; it should be considered that by increasing the BTEX concentration for the mentioned concentrations, the efficiency of the UV/H_2O_2 system decreases, respectively. However, the figure reveals that at lower initial concentrations of BTEX, contrary to the expectations, the final value of COD (after 180 min) is increased in comparison with a higher BTEX concentration (550 mg/L) in the mentioned conditions. One major factor responsible for this result is the high dosage of H_2O_2 used for 210 and 380 mg/L of BTEX concentrations as discussed in Fig. 2. Therefore, the initial dosage of H_2O_2 must be carefully selected to avoid such conditions.

Fig. 8 depicts the results obtained by the combined method (3UV light/H_2O_2) for different values of $[BTEX]_0$ = 210, 380, 550, 640, and 860 mg/L. According to these figures, the UV/H_2O_2 process is efficient for mineralizing BTEX pollutants. As it is shown in Fig. 8, by applying three UV lights along with other optimal conditions, the highest rate of BTEX degradation and the respective values of COD reduction are observed.

Its removal efficiencies are about 82, 87, 90, 94, and 98% in $[BTEX]_0$ = 860, 640, 550, 380, 210 mg/L. This high efficiency in comparison with H_2O_2 alone and UV alone can be described by the fact that irradiation of UV on H_2O_2 produces more hydroxyl radicals, and therefore, the remediation rate of COD increases. Another advantage for using a combined UV and H_2O_2 system is that hydrogen peroxide is completely soluble in water, and therefore, there is no mass-transfer limitation (in comparison with solid adsorbent or other AOPs such as ozonation) or need for a separation process after treatment [23,24].

3.5. Kinetic analysis

The photodegradation kinetics of BTEX has been studied at different hydrogen peroxide concentrations. The required data from Figs. 7 and 8 such as half-life

Table 4
Summary of pseudo-first-order rate constant and half-life obtained from different initial conditions in UV/H_2O_2 systems (pH 3.0 and 3 UV lights)

COD (mg/L)	H_2O_2 (g/L)	k (min^{-1})	$t_{1/2}$ (min)	$[H_2O_2]_0/[COD]_0$
550	1.110	0.0351	19.74	17.90
640	1.110	0.0210	33.01	13.41
860	1.110	0.0150	46.21	08.95
210	0.421	0.0194	35.72	10.07
380	0.721	0.0281	24.67	14.56
640	1.340	0.0312	22.21	16.18
860	1.736	0.0231	30.01	14.00

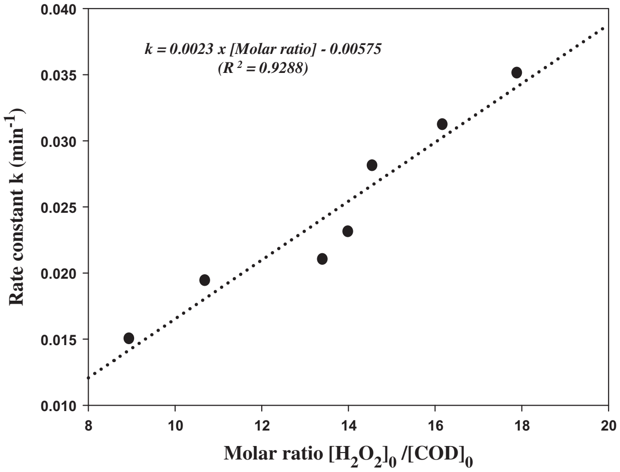

Fig. 9. Relationship between the pseudo-first-order rate constants (*k*) and the molar ratio of the initial concentrations of H_2O_2 to BTEX in UV/H_2O_2 system.

time with their own concentrations ($[H_2O_2]_0$ and $[BTEX]_0$) and also, the computed rate constants are summarized in Table 2 for UV/H_2O_2 systems.

According to Table 4, by decreasing the $[BTEX]_0$ (at constant $[H_2O_2]$) and increasing the $[H_2O_2]_0$ (at constant [BTEX]), the pseudo-first-order rate constants (*k*) increased, respectively.

Fig. 9 depicts the regression analysis by using the relation of the rate constant to the molar ratio of $[H_2O_2]_0$ to $[BTEX]_0$. This figure indicates that a satisfactorily linear relationship exists for this system. At all the initial conditions, a pseudo-first-order model follows the experimental data with an acceptable regression coefficient showing that the BTEX photodegradation follows a pseudo-first-order kinetic model with regards to BTEX concentrations.

In Fig. 10, the rate constants (*k*) vs. the initial hydrogen peroxide concentration are plotted. It is obvious from the figure that the rate of BTEX degradation is accelerated by increasing the H_2O_2 concentration to 1.11 g/L; however, a further increase in H_2O_2 concentration decreases the destruction rate. This result can be explained based on the existence of an optimal concentration for hydrogen peroxide as discussed in section 3.1.

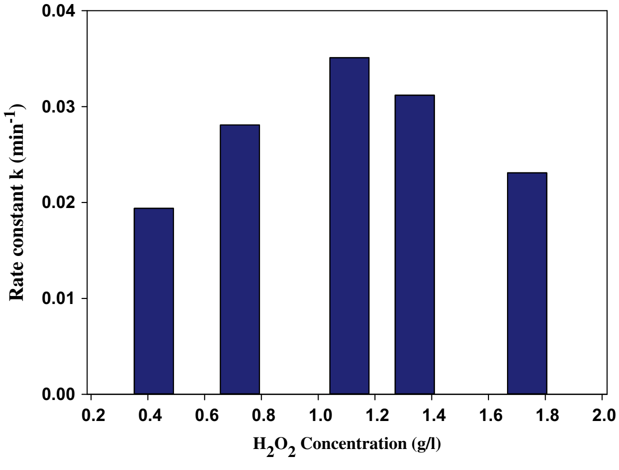

Fig. 10. Effect of initial concentration of H_2O_2 on photodegradation of BTEX ($[COD]_0 = 550$ (mg/L), pH = 3.12).

4. Conclusions

The treatment of BTEX-polluted wastewater was investigated in a synthetic solution by using the method of AOP (UV/H_2O_2). The efficiency of the method is checked by providing different number of UV lamps and different process conditions, such as pH, reaction time, initial concentration of H_2O_2, and initial concentration of BTEX compounds. The thermal condition was also investigated and the results showed that the COD rate of reduction increased by raising the temperature. This study demonstrated that the UV/H_2O_2 system degraded the BTEX in different initial concentrations of BTEX effectively. COD reduction efficiency was increased by raising the dosage of H_2O_2 to its optimum value for used initial concentrations of BTEX. Very alkaline pH and high concentrations of H_2O_2 had adverse results and reduced BTEX photodegradation. Under the optimal conditions for the average (550 mg/L) and the lowest (210 mg/L) concentrations of BTEX, the COD reduction during the first period of 180 min reached about 90 and 98%, respectively for the UV/H_2O_2 system which shows that the method can be effective for BTEX removal in industrial applications. Kinetic analysis showed that a pseudo-first-order kinetic model with respect to BTEX can be used to explain the BTEX degradation for UV/H_2O_2 system.

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