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Ultraviolet activated persulfate based AOP for MTBE decomposition in aqueous solution

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ABSTRACT

The widespread use of MTBE facilitates its entrance to the environment. Therefore, it is necessary to achieve a suitable method for the elimination of this carcinogenic contaminant. This study was conducted to investigate the MTBE removal from aqueous solution by ultraviolet activated persulfate (PS) based AOP. The solutions containing MTBE were synthesized at the concentrations of 10, 50 and 100 mg/L, and the rate of the removal efficiency was evaluated under the process conditions including pH: 3, 5, 7 and 9, PS concentration: 5, 10 and 20 mg/L and retention time: 30, 60, 120, 200 and 300 min. The results of this study showed that MTBE removal in the UV/PS process was pH dependent and the performance was better in neutral and acidic conditions. It was also found that, with increasing the PS concentration and decreasing initial MTBE concentration, better removal efficiency was achieved. Moreover, MTBE decomposition followed the first-order equation and this process was successful in reducing organic carbon. In conclusion, the UV/PS system can be considered as an effective process for eliminating the resistant pollutants such as MTBE.

Keywords: MTBE; Decomposition; Fuel additive; Persulfate; AOPs; Aqueous solution

1. Introduction

Among various fuel additives, methyl-tert-butyl ether (MTBE) has worldwide been used as a common fuel oxygenate to improve engine combustion and reduce air emission pollutants [1,2]. Although MTBE is a xenobiotic oxygenate, it has been applied in gasoline at the concentrations up to 15% by volume because of its advantages such as low water solubility, good material compatibility and excellent anti-knock properties [3]. The use of MTBE has been banned in many countries due to environmental hazards. This compound is known as irritant, as well as a potential human carcinogen, which can accumulate in the blood stream [1]. Other health impacts include vomiting, headaches, fever, diarrhea, cough, muscle aches, disorientation, sleepiness, dizziness, and skin and so forth [4].

Despite the ban and harmful effects of MTBE, it is one of the most widely used fuel additives in many Asian countries such as Iran [5]. It is therefore not surprising that MTBE can find its way to the environment through accidental fuel leakage during storage or transportation [6]. This material has an affinity for both gasoline and water, as well as a low

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tendency for adsorption on soil. Some characteristics such as a low biodegradation rate and high solubility in water (42 g/L) make it one of the most mobile groundwater contaminants [7,8]. It was the second most frequently detected compound out of 60 urban areas in the United States in 1993–1994. MTBE has been detected up to a concentration of 185 mg/L owing to leakage and sudden discharge [6]. The U.S. Environmental Protection Agency has set a drinking water advisory level of 20–40 μg/L for it [9].

Common water purification methods such as adsorption processes may not be cost-effective for MTBE removal; also, the presence of other organic materials in water adversely affects the efficiency of adsorption processes [10,11]. Thus, biological and chemical methods can be employed as an alternative. Biological oxidation mostly gains little chance for water treatment related to fast chemical remediation methods [12]. Nowadays, advanced oxidation processes (AOPs), due to high efficiency and the lack of secondary pollution, are of interest [13–15]. AOPs, using chemical oxidants such as permanganate, ozone, ferrate, chlorine, chlorine dioxide, hydroxyl radical ($HO^{\bullet}$) and sulfate radical ($SO_4^{\bullet-}$), are effective in oxidative destruction of many micro-pollutants and gasoline aromatic hydrocarbons [16].

PS-based advanced oxidation is a promising method for destructive removal of pollutants from soil and water. This process involves the production of hydroxyl and selective PS radicals [17]. Due to the higher selective property of sulfate radical than hydroxyl ones for oxidation of the organics through electron transfer, oxidation by PS radical may offer advantages for some target species [18]. Stability in aqueous solution, lower consumption than hydroxyl radicals by natural organic compounds and activity over a wide range of pHs are some important advantages of PS radicals [19,20]. PS ($S_2O_8^{2-}$) is an oxidant with an oxidation potential of 2.01 V. Peroxodisulfate (PS) or peroxy mono-sulfate (PMS) is chemically stable; if it is activated by different methods such as heat, ultraviolet irradiation, carbon, phenol, transition metals or zero-valent iron, sulfate radicals ($SO_4^{\bullet-}$) with high redox potential (2.5–3.1 V), high reactive species could be generated [21]. PS is activated by thermal or ultraviolet irradiation, electrolysis and metal ions according to the following equations [22]:

$$S_2O_8^{2-} + \frac{\text{heat}}{\text{UV}} \rightarrow SO_4^{\bullet-} \tag{1}$$

$$S_2O_8^{2-} + e^- \rightarrow SO_4^{2-} + SO_4^{\bullet-} \tag{2}$$

$$S_2O_8^{2-} + M^{n+} \rightarrow SO_4^{2-} + SO_4^{\bullet-} + M^{(n+1)+} \tag{3}$$

After activation, high reactive radicals capable of oxidizing a wide range of organic and inorganic compounds are produced. Because of disadvantages of other methods such as sludge production or difficulty of metal ion removal or high energy consumption [23], PS-based AOPs are of interest. Also, among activation alternatives, UV is a promising process providing high reactive species, gentle reaction conditions and no metal ion leakage [23]. Therefore, in the current research, the removal of MTBE by PS activated with ultraviolet light was investigated.

2. Materials and methods

2.1. Reagents and materials

Methanol, acetonitrile, acetone, tetrachloroethylene, carbon tetrachloride, chlorobenzene and MTBE were purchased from Merck (Germany). A stock standard solution of MTBE was prepared at a concentration of 1,000 mg/L. A series of solutions at the concentrations of 1, 25, 50 and 100 mg/L were prepared daily as the working standard solutions, by diluting the stock solution with distilled water. Nitrogen and hydrogen gases (99.999% purity) were purchased from Roham Gas Co. (Middle East Dubai, United Arab Emirates).

2.2. Experimental reactor

The experiments were performed in a Plexiglass cube with dimensions of 10 cm and useful volume of 400 mL. An 8 W Philips UV lamp (TUV 8W G8 T5 UVC, Philips Lighting Co., Netherlands) with dominant emitted wavelength at 254 nm was used as a light source. The UV lamp emits 2.4 W of UV-C radiation and was placed in a quartz sheath across the container. The solution was mixed thoroughly by a magnetic stirrer at 400 rpm.

2.3. Operational condition

The behaviors of the UV/PS process were determined in different experimental conditions: pHs (3, 5, 7 and 9), PS concentration (5, 10 and 20 mg/L), reaction time (30, 60, 120, 200 and 300 min) and initial MTBE concentration (10, 50 and 100 mg/L). At the end of the reaction, the MTBE concentration was measured and removal efficiency was calculated according to Eq. (4).

$$\text{MTBE Removal efficiency}\left(\%\right) = \frac{\left(\text{MTBE}_0 - \text{MTBE}_t\right)}{\text{MTBE}_0} \times 100 \tag{4}$$

MTBE_0: initial MTBE concentration, mg/L; MTBE_t: MTBE concentration at sampling time, mg/L.

2.4. Kinetic study

Kinetic studies were performed to determine the reaction speed of MTBE degradation by the UV/PS process. For this purpose, solutions with initial MTBE concentration equal to 10, 50 and 100 mg/L were prepared and adjusted at pH 7 and then under the UV/PS process. Sampling was done at 10 min intervals and the residual concentrations of MTBE were determined.

2.5. Analysis

At the end of the reactions, MTBE was extracted from aqueous solution based on dispersive liquid–liquid microextraction. Briefly, a 10 mL working solution containing 1 mL of tetrachloroethylene was placed in a 15 mL conical glass tube. Acetonitrile (500 μL), as dispersive solvent containing 25 μL tetrachloroethylene as extraction solvent, was injected rapidly into the sample solution by using a 5 mL syringe. The produced cloudy solution was centrifuged for 3 min at 5,000 rpm with a MIKRO 22R Hettich centrifuge (Germany).

After centrifuging, the dispersed fine droplets of carbon tetrachloride settled at the bottom of the test tube (20 ± 1 µL). The sediment was completely transferred to another test tube with a conical bottom using a 50 µL HPLC syringe, and 1.0 µL was injected into the detection system.

The measurements were performed using a GC system (6890N, Agilent Technologies, Wilmington, DE, USA) equipped with an FID and a HP-5 cemetery capillary column (60 m × 0.25 mm i.d., 0.25 µm film thickness, Restek, USA). Nitrogen gas was used as the carrier gas with the flow rate of 1 mL/min. The injector temperature was 200°C with a splitless mode and the FID temperature was 250°C. The oven temperature programming was as follows: the initial column temperature was maintained at 30°C for 1 min and then raised at 25°C/min to 120°C and held for 4 min. For online data collection and processing ChemStation software was used.

3. Results

3.1. Effect of pH

The effect of pHs (3, 5, 7 and 9) on the removal efficiency was studied at MTBE concentration = 50 mg/L, PS = 20 mg/L, UV light intensity = 0.007 W/cm^2 and reaction time = 2 h. Fig. 1 shows the variations of the MTBE removal efficiency in the UV/PS process with the elevation of pH from 3 to 9. However, MTBE removal in UV/PS system was pH dependent, based on Fig. 1, the removal efficiency in the pH range of 3–7 remained constant and it was higher in neutral and acidic conditions than in alkaline conditions. Based on the previous studies, solution pH plays an important role in the type of radicals produced during the UV/PS process [24]. In general, during this process SO_4 radicals are the most frequent radicals produced under acidic conditions, and $^{\bullet}OH$ radicals are the major radicals produced under alkaline conditions. Because, with increasing OH^- ions, they react with SO_4 radicals and, in turn, $^{\bullet}OH$ is produced. In terms of redox potential, SO_4^- radical is much stronger than $^{\bullet}OH$ radical. While the $^{\bullet}OH$ radicals are converted to O^- radicals, with a lower redox potential, under alkaline conditions. All reactions are listed in Eqs. (5)–(8) [25].

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

$$HS_2O_8^- \rightarrow SO_4^{\bullet-} + SO_4^{2-} + H^+ \tag{6}$$

$$SO_4^{\bullet-} + OH^- \rightarrow SO_4^{2-} + OH^{\bullet} \tag{7}$$

$$OH^{\bullet} + OH^- \rightarrow O^{\bullet-} + H_2O \tag{8}$$

According to the above-mentioned equations, it can be concluded that under alkaline conditions, the efficiency of the UV/PS process decreased due to the formation of radicals with low redox potentials [25,26]. Also, in removal of MTBE and benzene contamination from groundwater with PS, similar results have shown that MTBE was oxidized much slower under alkaline conditions [27,28].

To study the pH changes at different reaction times, solutions with initial pH of 3, 5, 7 and 9 were prepared and the samples were taken every 10 min during the experiment until it reached constant values. As shown in Fig. 2, after 60 min, the pH values were reduced and fixed after 60 min. Solutions with the initial pH of 3, 5, 7 and 9 decreased after 60 min to 2.6, 2.8, 3.15 and 4, respectively, and then remained constant. In a previous study by Liang et al. [27], PS was used to remove MTBE contaminated groundwater. They showed that solution pH at different reaction times declined after 30 min and the pH values decreased to 3 and it was independent on initial adjusted pHs. The decrease in pH values can be attributed to the production of sulfate and subsequent sulfuric acid during the reaction [27,28]; other studies have reported the same trend [29]. The effect of pH on MTBE, methyl paraben and chloramphenicol removal in the study of the UV/PS oxidation showed the successful application of PS over a wide range of pHs and due to cost-effectiveness of experiments under neutral condition (without any pH adjustment), the pH value of 6–7 was found as the optimum value; this observation is consistent with the results of the present study [25,27,30]. In the current study, there was not any significant difference between removal efficiency at pH 3, 5 and 7, thus, further experiments were conducted at natural condition (without pH adjustment).

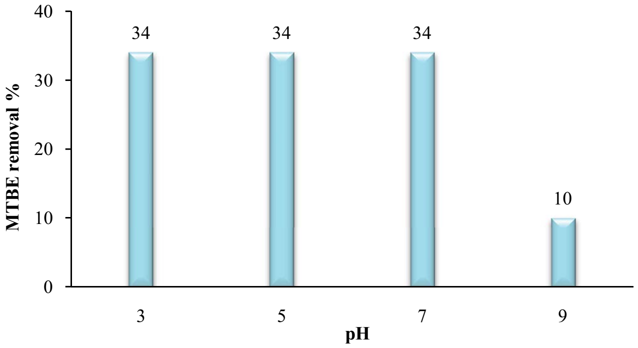

Fig. 1. Effect of pH on MTBE removal (experiment conditions: MTBE = 50 mg/L, PS = 20 mg/L, UV light intensity = 0.007 W/cm^2, time = 120 min, pH = 3, 5, 7, and 9).

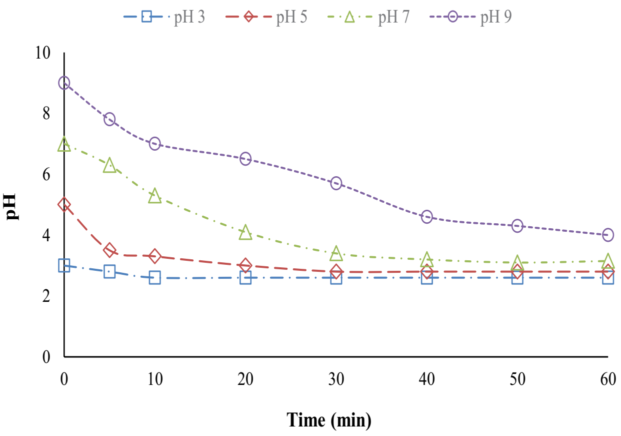

Fig. 2. pH changes at different reaction times in UV/PS process (experiment conditions: MTBE = 50, PS = 20 mg/L, UV light intensity = 0.007 W/cm^2, time = 10–60 min, pH = 3, 5, 7, and 9).

3.2. Effect of PS concentrations on MTBE removal in the UV/PS process

In order to study the effect of PS concentration on MTBE removal in UV/PS process, the experiments have been conducted at PS concentration = 5, 10 and 20 mg/L, time = 120 min, UV light intensity = 0.007 W/cm^2 and MTBE concentration = 50 mg/L. As can be seen in Fig. 3, the MTBE removal increased from 88 to 96% with increasing the PS concentration from 5 to 20 mg/L, respectively. Enhancing MTBE removal with increasing PS concentration in the UV/PS process can be attributed to the increase of SO_4 radicals in the reaction chamber [20,30].

It was reported by Dong et al. [31] that MTBE is the most resistant to PS oxidation in SO_4 based AOPs, compared with benzene, toluene, ethyl-benzene, xylenes (BTEX); they showed that the increase in PS concentration from 0.6 to 30 g/L led to an increase in degradation efficiency of MTBE. Also, the maximum level of MTBE removal was 48%, which is lower than that obtained in the current study [31].

The study of PS concentration on antibiotics removal in the UV/PS system showed that an increase in PS concentration led to more degradation of antibiotics [32,33]. But further increase in PS concentration led to saturation and scavenging effect, and consequently a decrease in the performance of the UV/PS system in antibiotic degradation [34,35]. The comparison of the amount of PS used in this study with others showed that the PS concentration range was selected appropriately (according to the high efficiency attained) and no scavenging effect of excess PS was observed. Also, in this study, the produced sulfate ion content in the solution used as an indicator of PS over-use. The results showed that after the process, the sulfate concentration was 95 mg/L, which did not exceed the national standards for effluent discharge. The increase in sulfate ions is attributed to the effect of radical capturing during the process, due to the presence of well-known radical scavengers, carbonates and bicarbonates [20,25,30].

3.3. Effect of initial MTBE concentration on the UV/PS process

To study the effect of MTBE initial concentration on its removal efficiency in UV/PS system, the experiments were conducted with MTBE concentration = 10, 50 and 100 mg/L, PS concentration = 20 mg/L, UV light intensity = 0.007 W/cm^2 and time = 30–300 min. As shown in Fig. 4, an increase in MTBE initial concentration led to a decrease in MTBE degradation. In MTBE concentration of 10 mg/L after 30 min 100% removal was obtained but in MTBE concentrations of 50 and 100 mg/L after 120 min 92% and 94% removal was obtained, respectively (Fig. 4). Also, based on the study previously conducted to determine the efficiency of UV/PS for removal of methyl paraben, with the increased initial concentration from 19.9 to 65.7 µM, the removal efficiency dropped from 100% to 60%, which is consistent with the results of the present study [25]. A decrease in removal efficiency in UV/PS system is because of the high concentration of MTBE resulting in a decrease in penetration of photons into the solution, inducing inner filter effect and inhabitation of the photolysis efficiency [30]. However, the method of PS activation may provide different efficiencies of contaminants removal; the study of microwave-activated PS oxidation for the removal of Azo dye acid orange 7 showed that the an increase in initial concentration of the target contaminant led to a decrease in degradation [36,37].

3.4. Degradation of MTBE in UV/$S_2O_8$2 system in different concentrations of MTBE

In order to determine the behavior of the UV/S_2O_8 process in degradation of MTBE as a function of different initial MTBE concentrations, kinetics of the process was determined according to Eq. (9).

$$\frac{dC}{dt} = -KC \tag{9}$$

where C (mg/L) is the concentration of MTBE at time t (min) and K is the apparent degradation rate constant (min^{-1}). Integrating Eq. (1) gives Eq. (10).

$$\ln\frac{C}{C_0} = -k_{\text{app}}t \tag{10}$$

where C_0 and C are initial concentrations of MTBE and at time t (min), k_{app} is the pseudo-first-order rate constant, which is acquired from the slope of the regression line [33].

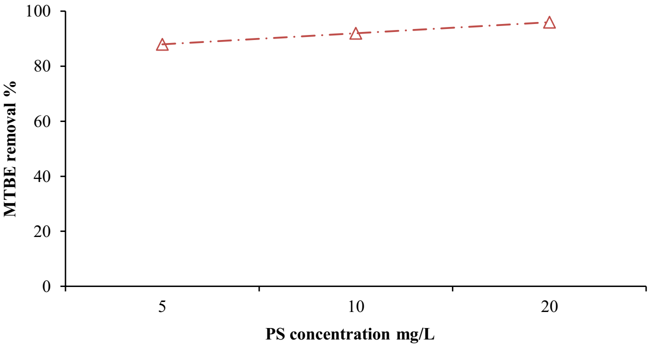

Fig. 3. Effect of PS concentration on MTBE removal (experiment conditions: MTBE = 50 mg/L, UV light intensity = 0.007 W/cm^2, time = 120 min, pH = no adjustment, PS = 5, 10, and 20 mg/L).

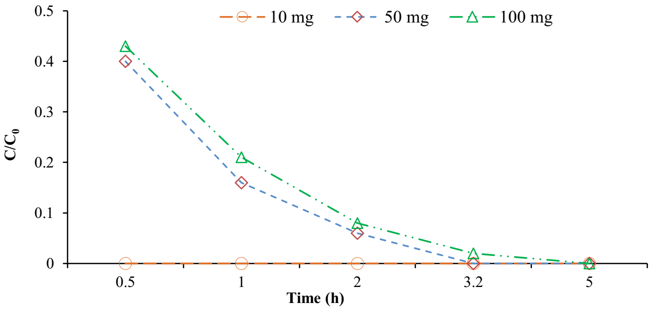

Fig. 4. Effect of initial MTBE concentration on MTBE removal (experiment conditions: MTBE = 10, 50, and 100 mg/L, UV light intensity = 0.007 W/cm^2, PS = 20 mg/L, time = 30–300 min, pH = no adjustment).

According to Fig. 5, the degradation followed the pseudo-first-order degradation rate. Table 1 presents the kinetic rate constants (k_{obs}) of MTBE degradation by UV/PS under different concentrations of MTBE. As can be seen from Table 1, with increasing initial MTBE concentration, the degradation rate constants decreased. Comparing the acquired rate constant with other researchers studied MTBE removal in heat-assisted PS oxidation system showed that UV/PS obtained lower rate constant [38] but related to the UV/TiO_2 process, it showed a lower rate constant (0.035 vs. 6.4×10^{-2} min^{-1}). It should be mentioned that in the removal of MTBE by the UV/TiO_2 process the MTBE concentration was 10.570 µg/L, which is much lower than that attained in the current study. Overall, some characteristics such as MTBE biological and chemical stability and solubility in water have led to need to longer time for degradation even in a strong oxidation system such as the UV/PS system [39].

Table 1
Pseudo-first-order degradation rate constants (k_{obs}) of MTBE degradation by UV/PS under different concentrations of MTBE

MTBE concentration mg/L	K (min^{-1})	R^2	$T_{1/2}$ (min)
10	0.035	0.9388	19.8
50	0.025	0.9684	27.72
100	0.0204	0.9833	33.97

3.5. Mineralization in terms of TOC removal

To investigate the effect of the UV/PS process on the mineralization of the target pollutant, total carbon, total organic carbon (TOC) and inorganic carbon were analyzed under the optimum conditions of MTBE = 50 mg/L, PS = 20 mg/L, time = 60 min, UV light intensity = 0.007 W/cm^2 and no pH adjustment. Given that mineralization is an important principle in AOPs, the results obtained in this regard were promising. As shown in Fig. 6, UV/PS was successful in reducing organic carbon and mineralization. Based on Fig. 6, over time in line with an increase in the decomposition efficiency, the amount of organic carbon decreased from 18.47 to 4.86 mg/L. Also, inorganic carbon to carbon ratio enhanced at the end of the reaction time related to the beginning of the reaction. The study of mineralization of β-lactam antibiotics by the activation of H_2O_2 and $Na_2S_2O_8$ under UV irradiation (254 nm) showed that, even with 588 min UV irradiation, a good TOC removal was rarely achieved and mineralization rates were 15.3% and 13.9% in UV/H_2O_2 and 25.5% and 33.0% in UV/PS, respectively, for cephalothin and ampicillin antibiotics [40]. In the present study, the removal rates of TOC were 54% and 73.6% at reaction times of 60 and 200 min, respectively. Mehrjouei et al. [41] surveyed the removal of 1 mM fuel oxygenates from water using photocatalytic ozonation (TiO_2/UVA/O_3); they reported that TOC level of MTBE, ETBE and tert-amyl ethyl ether decreased approximately 70%–80% after 1 h of reaction time [41].

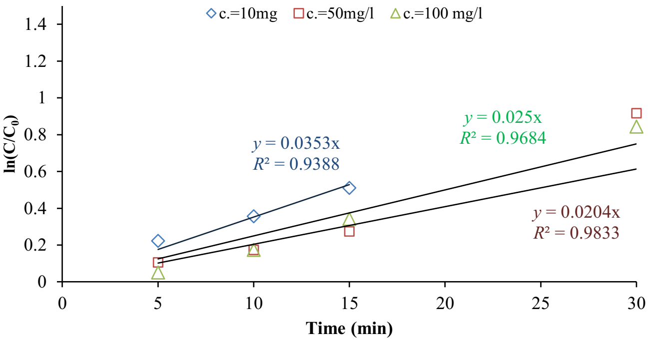

Fig. 5. Effect of initial MTBE concentration on degradation by PDS (experiment conditions: MTBE = 10, 50, and 100 mg/L, UV light intensity = 0.007 W/cm^2, PS = 20 mg/L, time = 5–30 min, pH = no adjustment).

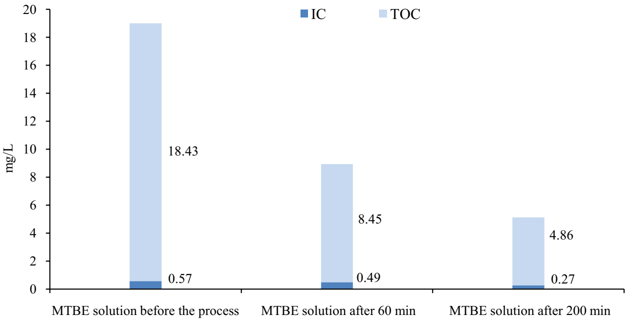

Fig. 6. Mineralization in terms of TOC removal (experiment conditions: MTBE = 50 mg/L, UV light intensity = 0.007 W/cm^2, PS = 20 mg/L, time = 60, 200 min, pH = no adjustment).

4. Conclusion

MTBE, as a common fuel oxygenate especially in Asian countries, finds its way to water resources. It is necessary to purify water contaminated with this compound. Therefore, this study aimed to investigate the removal of MTBE in aqueous solution by UV/PS-based AOP. Based on the results, it was found that the MTBE removal in the UV/PS system was pH dependent and the MTBE removal efficiency was better in neutral and acidic conditions than the alkaline conditions. The findings also showed that with increasing the concentration of PS and decreasing the initial MTBE concentration, the removal rate increased. Further, MTBE decomposition followed the pseudo-first-order kinetic model. Based on the TOC analysis results, this process was successful in reducing organic carbon. Finally, this research suggests that the UV/PS system can be considered as an effective and safe way for eliminating the resistant pollutants such as MTBE because it produces the least by products and maximum mineralization as well.

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