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# MTBE removal from contaminated water by the UV/$H_2O_2$ process

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## ABSTRACT

Advanced oxidation process, UV/$H_2O_2$, was investigated for MTBE degradation in MTBE-contaminated waters. Water solutions of MTBE were prepared in the laboratory with one to five ppm of MTBE. The photochemical oxidation was performed in a completely mixed-batch-photocatalytic reactor with a total volume of 500 mL. Two UV irradiation sources were employed individually; a low-pressure mercury vapor lamp and a high-pressure mercury lamp that is axially centered in the photoreactor. Neither UV nor hydrogen peroxide on its own was very effective for MTBE oxidation. While in combination (UV/$H_2O_2$), the degradation rate was enhanced by at least one-order of magnitude relative to direct photolysis. A complete MTBE degradation was achieved in less than 15 min. The calculated pseudo-first-order reaction rate constants were strongly dependent on the initial concentration of MTBE and on the hydrogen peroxide concentration. As indicated by the results, the MTBE degradation rate with the high-pressure UV source was highest ($k = 6.0\times10^{-3}\ s^{-1}$). After 10 min of irradiation, MTBE was completely degraded when using the 150 W TQ 150 mercury lamp. In addition, results showed that MTBE degradation rate is enhanced at a higher initial concentration of MTBE due to the higher initial $H_2O_2$ concentration when keeping the MTBE/$H_2O_2$ molar ratio constant.

*Keywords*: MTBE; Advanced oxidation processes; UV/$H_2O_2$; Kinetics

## 1. Introduction

Methyl tertiary butyl ether (MTBE) is found in gasoline and other petroleum fuels that is commonly stored in underground storage tanks. Although MTBE is relatively a new gasoline additive, it was found that out of 60 volatile organic chemicals analyzed, MTBE was the second most frequently detected chemical in samples of shallow ground water from urban areas that were collected as part of the US Geological Survey's National Water-Quality Assessment program [1]. The use of advanced oxidation processes (AOPs) for the treatment of aqueous pollutant has been under considerable research and commercial interest in the past 20 years [2–14]. AOPs are typically characterized by the activation of organic molecules which range from direct oxidation by absorption and dissociation to formation of organic free radicals or other reactive intermediates, such as the oxidation by generating hydroxyl radicals ($OH^{\bullet}$) at ambient temperatures by ultraviolet radiation alone or in the presence of an oxidant such as ozone, hydrogen peroxide and $TiO_2$ [10]. Several researchers have studied photocatalytic oxidation of MTBE; however, the photolysis kinetic, the radiation source effects, the degradation reaction mechanism, and the nature of the intermediate products are not well known [8–11]. The processes continue to be the subjects of intense research and improvements particularly in areas where waste oxidation rather than transfer to another phase is necessary.

Few researchers have studied UV/$H_2O_2$ for the

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oxidation of MTBE in contaminated water. Wagler and Malley [4] explored UV/$H_2O_2$ to remove MTBE from model groundwater. They observed a minor pH effect and increasing reaction rates with higher hydrogen peroxide concentrations. Chang and Young [8] studied the removal of MTBE from aqueous solution by exposure to a low-pressure mercury lamp and hydrogen peroxide in a recirculation batch mode. Experiments were conducted at initial $H_2O_2$ to MTBE molar ratios of 4:1, 7:1 and 15:1. They reported no measurable degradation of MTBE using a low-pressure mercury lamp emitting an almost monochromatic radiation at 254 nm wavelength ($\lambda$). No direct photolysis of MTBE was observed because MTBE does not absorbs UV light when $\lambda$ was greater than 210 nm. In addition, hydrogen peroxide alone was not expected to oxidize MTBE. Conversely, in the presence of hydrogen peroxide, greater than 99.9% of MTBE was oxidized in water containing an initial concentration of 10 ppm with a moderately high first-order reaction rate constant of 3.9 (±0.73)×$10^9$ L/mol·s. Stefan et al. [9] investigated the degradation of MTBE by the UV/$H_2O_2$ process. A first-order reaction rate constant of 1.6×$10^9$ L/mol·s was reported for the disappearance of 0.92 mM MTBE aqueous solution. Liang et al. [10] conducted a series of pilot-scale tests to investigate the performance of different AOP arrangements such as UV/$H_2O_2$, ozone, and ozone/$H_2O_2$ for removing MTBE with concentration of greater than 2000 µg/L from surface water and groundwater. Results showed that hydroxyl radicals produced by UV/$H_2O_2$ and ozone/$H_2O_2$ processes were more effective in oxidizing MTBE than was UV photolysis or ozone alone. They also showed that groundwater which contained high level of nitrate had overall lower level of MTBE removal efficiency than the surface water for UV/$H_2O_2$ treatment systems. Because nitrate strongly absorbs UV light, thus decreasing the amount of hydroxyl radicals generated during the process. Bertelli and Selli [11] employed photocatalysis on $TiO_2$ and sonolysis with and without the presence of $H_2O_2$ to study the kinetic and efficiency of removal of MTBE from water under atmospheric conditions. They showed that the application of simultaneous of two oxidation techniques had additive effects. In addition, the highest degradation and mineralization efficiency was achieved under hydrogen peroxide photolysis at 254 nm.

Recently, Hong et al. [13] used anodic Fenton treatment to degrade MTBE and compared it with classical Fenton treatment. They showed that the anodic Fenton treatment is more effective than the classical method. Arana et al. [14] studied the effect of $TiO_2$ and Cu-$TiO_2$ on the MTBE photocatalytic degradation. The treatment of MTBE with Cu-$TiO_2$ was more efficient than that with $TiO_2$.

The efficiency of the oxidation of MTBE by UV/$H_2O_2$ is determined by two main factors: Attenuation of UV light by the matrix and OH radical scavenging by the natural organic matter and carbonate/bicarbonate. This study was conducted to assess the efficiency of MTBE removal from groundwater using advanced oxidation processes (AOPs) and to evaluate the kinetics of the process. Two UV irradiation sources were employed individually; a low-pressure mercury vapor lamp and a high-pressure mercury lamp that is axially centered in the photoreactor.

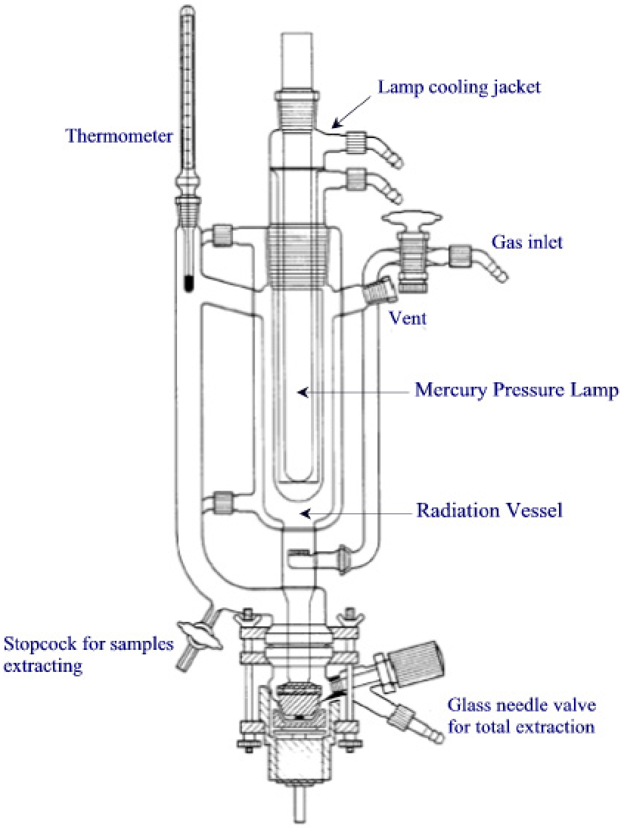
## 2. Experimental work

### 2.1. Materials

MTBE (99.9%, industrial grade) was acquired from a local producer and hydrogen peroxide (35%, analytical grade) was purchased from Sigma-Aldrich or Fluka AG. All were used as received. Compressed air, oxygen, helium, and nitrogen with better than 99.9% purity were obtained from local suppliers.

### 2.2. Photochemical reactor

The batch experiments were carried out in the new NORMAG® tabular photoreactor with forced liquid circulation (NORMAG Laboratory and Process Technology GmbH, Ilmenau, Germany) as shown in Fig. 1. The photoreactor consist of radiation vessel, cooling



Fig, 1. The photoreactor with forced liquid circulation complete as shown in drawing: volume 500 ml, cooling tube and immersion tube were made of quartz.

tube, immersion tube, Hostaflon® pump circulator, and thermometer. The radiation vessel has a total volume of 500 mL. The cooling tube and immersion tube were made of quartz. The pump that was fixed below the reaction vessel circulated the liquid very effectively. The light source most suitable for any specific photochemical reaction depends on the effective spectrum of the substance or system and on the light output. MTBE does not absorb UV light with a wavelength above 210 nm and MTBE oxidation was expected to be exclusively by hydroxyl radical attacks. Hence, the irradiation in the photoreactor was obtained by a QVF low-pressure mercury lamp (TNN 15/32) of 15-W consumption. The light source was made of quartz glass, which emitted very intense radiation of the 254 nm mercury resonance line that may be described as practically monochromatic. To account for the light intensity effects on the degradation rate, other experiments were performed with a QVF high-pressure mercury lamp (TQ 150) of 150 W output. It was made of quartz glass, which emitted the characteristic mercury line system that extended from the short-wave UV range of about 240 nm to well into the visible (polychromatic).

### 2.3. Experimental procedure

Experiments were performed with MTBE-contaminated water using ultraviolet photolysis, hydrogen peroxide oxidation, and ultraviolet photolysis combined with hydrogen peroxide oxidation. MTBE stock solution was prepared by adding an appropriate amount, to make the desired concentrations, to deionized and distilled water. Since MTBE is highly volatile, samples are kept at refrigerator temperatures, 4°C, during storage. Mixtures were prepared at room temperature of 20°C and placed on wet ice or into a refrigerator. Water was distilled and deionized (Barnstead NANO Pure, USA). All chemicals in this study were used as reagent grade. The hydrogen peroxide was from Fluka Co.

The solution (500 mL) for photolysis was prepared by adding of appropriate amounts of $H_2O_2$ stock (35%) to the photoreactor. The solution was stirred and circulated at about 300-rpm for 5 min to insure sufficient mixing, and then turned on the lamps and the circulation pump. The pH was adjusted by slow addition of perchloric acid (1.0 M) or sodium bicarbonate (1.0 M). Temperature and pH of the solution were measured using thermometer and pH meter, respectively. The detection limits were 0.005, and the precision was better than 1% for both. Experiments were conducted with various initial MTBE concentrations that range between 1 and 5 ppm (0.01–0.06 mM). The molar ratio of $H_2O_2$ to the MTBE also was varied between 30 and 100. The experiments were performed at neutral pH (i.e. 6–7) except to a few runs to test for pH effects on the reaction rates. Periodically, samples were withdrawn through the sampling port then stored immediately into screw-cap glass vials for analyses. Before sampling, temperature and pH measurement was taken.

### 2.4. Analytical procedure

A Varian 3400cx model Gas Chromatograph (GC) equipped with a Photo-Ionization Detector (PID), Varian, Inc. Palo Alto, CA 94304-1030, USA, was used for the analyswas of MTBE. Tekmar 3100 Purge and Trap (P&T) technique was used for sample introduction. A 30 m long, 0.53 mm i.d., 1.5 μm thickness DB-1 fused silica capillary column from J&W Scientific, was used for the separation purpose. The oven temperature was programmed from 35°C (4 min) to 200°C (3 min) at a ramp rate of 20°C per min. The detector temperature was set at 250°C. Helium gas was used as a carrier at a flow rate of 3 mL and a makeup of 27 mL to have a total of 30 mL per min. Five (5) mL sample was purged by P&T unit and then directly injected into the GC. The P&T method used in this study was based on USEPA 5030. Total organic carbon was measured by a Shimadzu TOC analyzer (TOC-VE), Shimadzu EMIT CO., LTD., Kanda-ishikicho 2-chome, Chiyoda-ku Tokyo 101-0054 JAPAN. The analyzer used the 680°C combustion technique with newly designed high-sensitivity Non-dispersive Infrared (NDIR) detection. The TOC analyser was capable of TOC-trace aqueous sample analysis ranging from 0 to 20,000 μg for both TC and IC.

## 3. Results and discussion

Different parameters were investigated to evaluate their effects on the removal efficiency of MTBE including initial concentration of MTBE and $H_2O_2$, and UV intensity. Direct UV photolysis of aqueous solutions contaminated with MTBE were performed at atmospheric pressure and room temperature of 25±2°C for 30 min at neutral pH 7±0.5 with continues stirring. In all cases, no appreciable photooxidation was observed. The results of one experimental setting for each of the UV sources are shown in Fig. 2. The initial MTBE concentration was 1 ppm. Sample analysis showed no appreciable reduction in MTBE initial concentration. Only 20% degradation occurred over 30-min period under continual irradiation due to direct UV photolysis, or hydroxyl radical attacks that were formed in-situ, i.e. from water. Based on these results, the reaction time was rather long which would drive-up energy costs. MTBE was treated with hydrogen peroxide only (no UV irradiation) at atmospheric pressure and room temperature of 25±2°C for more than one hour at different pH levels with continuous stirring, but no appreciable oxidation was observed. GC and TOC analysis for collected samples show less than 5% degradation of MTBE. Initial MTBE concentration was 1 ppm and $H_2O_2$/MTBE molar ratio was around 50. As it was observed, neither

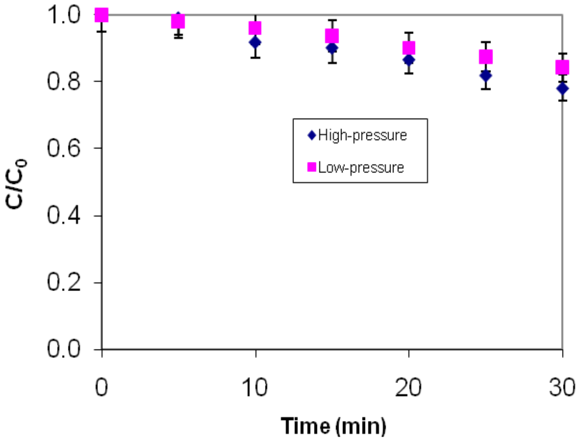


Fig. 2. MTBE oxidation by Low- and High-pressure UV, no hydrogen peroxide, and $C_0 = 1$ ppm.

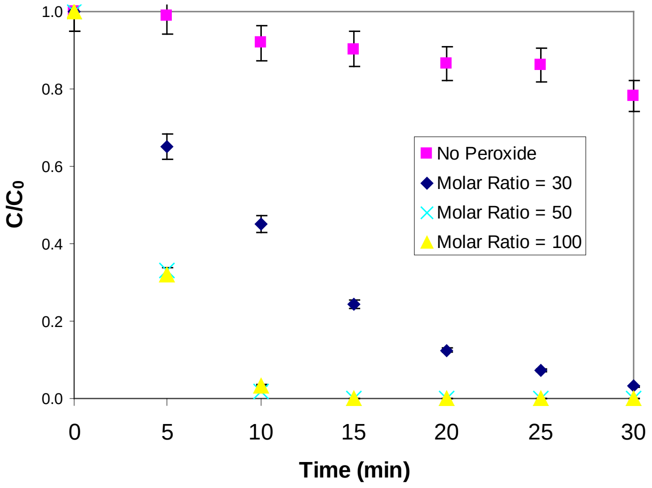


Fig. 3. UV/$H_2O_2$ oxidation of MTBE with the low-pressure UV source at various $H_2O_2$/MTBE ratio and constant initial MTBE concentration (1 ppm) at ambient conditions.

ultraviolet radiation nor hydrogen peroxide on its own was very effective for MTBE oxidation. Conversely, UV irradiation combined with hydrogen peroxide has achieved most remarkable results. Hydrogen peroxide combined with ultraviolet radiation enhanced the degradation rate by at least one fold relative to the two previously discussed treatments.

Experiments were conducted at various initial MTBE concentrations and hydrogen peroxide dosages. Fig. 3 shows four runs that were conducted with the low-pressure UV source at similar initial MTBE concentration of 1±0.05 ppm (0.0113 mM). Hydrogen peroxide doses varied between zero and 1.13 mM to determine the effects of $H_2O_2$/MTBE molar ratio. Experiments conducted spanned a wide ratio range (up to 100). Results indicate that hydrogen peroxide enhanced the photolytic degradation of MTBE greatly relative to direct photolysis. The amount of hydroxyl radicals increases with an increasing concentration of $H_2O_2$ and consequently the rate of the MTBE disappearance increases. However, as it was observed in previous studies as well as reported by other investigators, $H_2O_2$ levels had two opposing effects on the reaction rate [6,15,16]. Increasing initial hydrogen peroxide concentration enhances the oxidation process up to a certain level at which hydrogen peroxide inhibits the photolytic degradation. Because at higher hydrogen peroxide concentration, hydrogen peroxide acts as a free radicals scavenger itself, thereby decreasing the hydroxyl radicals concentration. Hence, there is an optimum $H_2O_2$ dosage, i.e., molar ratio of $H_2O_2$ to the contaminant. A significant reduction in the degradation rate is expected at much higher $H_2O_2$ concentrations. This dependence on hydrogen peroxide presence indicates that oxidation by radical attacks (i.e. $OH^{\bullet}$ and $^{\bullet}OOH$) is the major mechanism by which MTBE is degraded. Moreover, direct UV radiation oxidation and hydrogen peroxide oxidation is less significant and negligible. At the operating wavelength and light pressure, $H_2O_2$ levels did not show inhibitory effects on the photolytic degradation rate. Results showed that at $H_2O_2$/MTBE molar ratio of 100, a complete oxidation of MTBE occurred in less than 15 min (Fig. 3). The oxidation rate of MTBE was influenced by the initial concentration of both MTBE and $H_2O_2$ and the light intensity. Fig. 4 shows the results of MTBE treatment by the two UV sources at $H_2O_2$/MTBE molar ratio of 30 and 1 ppm of MTBE.

The results indicated that at low $H_2O_2$/MTBE molar ratio of 30, the rate of MTBE decay was much faster with the high-pressure UV lamp. A closer look at the results indicated that for the first 5 min, both setup were pro-

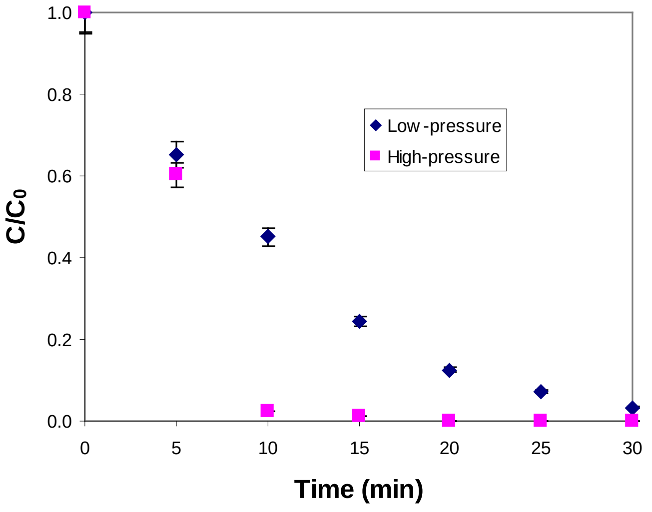


Fig. 4. UV/$H_2O_2$ oxidation of MTBE oxidation by low- and high-pressure UV, $C_0 = 1$ ppm and $H_2O_2$/MTBE molar ratio = 30.

ceeding at approximately the same degradation rate. As time elapsed, the oxidation rate with the low-pressure UV source started slowing. This may be due to the lower concentration of the pollutants and the radicals. At low levels, the photocatalysis reaction rate is first order, and the overall reaction is rate controlled according to the Langmuir-Hinshelwood reaction kinetics mechanism, for dilute solution (< $10^{-3}$ M) [17]. To further explain this behavior, it is important to recall the fact that the rate of photolytic reactions depends on the intensity of the light. Therefore, it follows that the rate varies proportionally to the light intensity. Photolytic dissociation of hydrogen peroxide to hydroxyl radicals can theoretically occur up to a wavelength of 560 nm. This is the dissociation threshold wavelength. However, hydrogen peroxide does not absorb significantly above a wavelength of 310 nm. This wavelength is referred to as the absorption threshold. The maximum absorption of UV radiation by hydrogen peroxide occurs at about 200 nm, and it decreases as the wavelength increases [18]. The extinction coefficient (or molar absorptivity) of hydrogen peroxide at 254 nm is approximately 19.6 $M^{-1}\cdot cm^{-1}$ [19]. This low extinction coefficient translates into low absorption rates. Hence, increasing the UV light intensity will increase the reaction rate, until the mass-transfer (the diffusion of photons) limit is reached. The absorbance (*A*), also called solution optical density (*OD*), is directly proportional to the light-absorbing species concentration in the media. The extinction coefficient (ε) is a measure of the rate of the reduction of transmitted light through a substance; commonly has the units $l.mol^{-1}.cm^{-1}$. As light travels through the solution, the incident power magnitude that reaches each infinitesimal portion (layer) of the total volume decreases. Furthermore, the decrease in power is proportional to the power incident upon that section, which explains the logarithmic relation between transmittance and concentration [20]. The values of *A* and ε depend on the light source wavelength, and the larger the proportionality coefficient (ε), the greater is *A*. Hence, at low intensities, the quantum efficiency is independent of light intensity and remains constant.

The utilized low-pressure mercury lamp has a dominant emission wavelength at about 254 nm. With these types of lamps, a high dosing rate of hydrogen peroxide must be added to the system to generate the required quantity of hydroxyl radicals. This was obvious when the hydrogen peroxide concentration was increased (Fig. 5). Other experiments were performed at higher initial MTBE concentration to study the impact of the initial MTBE concentration on the removal of MTBE with UV/H2O2 processes. The testing was done at initial MTBE concentration of 5 ppm and keeping the initial molar ratio between MTBE and $H_2O_2$ constant (Fig. 6). Results indicate that a high concentration of MTBE may retard the degradation of MTBE due to a larger competition for the hydroxyl radicals. As expected, it was also observed that the rate of MTBE decay was much faster with the high-pressure UV lamp. This significant reduction of the decay process at high initial concentration of MTBE may be due to the higher concentration of MTBE and reaction intermediates. These intermediates are also reactive towards hydroxyl radicals and may consequently compete with MTBE for the hydroxyl radicals and retard the removal of MTBE.

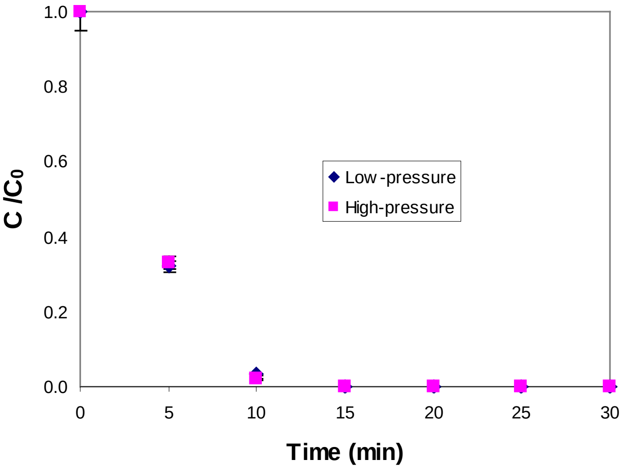


Fig. 5. UV/$H_2O_2$ oxidation of MTBE oxidation by low- and high-pressure UV, MTBE $C_0$ = 1 ppm, and $H_2O_2$/MTBE molar ratio of 50.

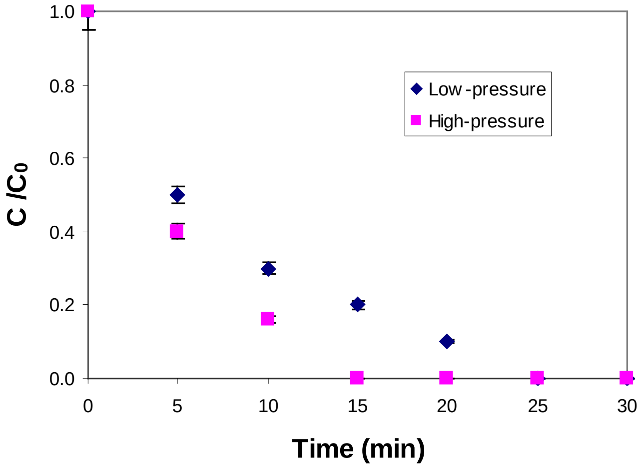


Fig. 6. UV/$H_2O_2$ oxidation of MTBE oxidation by low- and high-pressure UV, MTBE $C_0$ = 5 ppm, and $H_2O_2$/MTBE molar ratio of 50.

Based on the results, hydrogen peroxide was the only main species that effectively absorb the incident radiation emitted from the low-pressure mercury vapor lamp at 254 nm. Direct photolysis of any substance present in the system other than hydrogen peroxide, whether starting compound or an intermediate was neglected in the kinetic study. The kinetic of this AOP type is dependent on initial concentrations of starting organics (MTBE) and the oxidant ($H_2O_2$). As presented in this study, experimintal results showed that the initial rates of MTBE oxidation depend on hydrogen peroxide concentraion as well as the initial MTBE concentration. To determine the pseudo-first-order degradation rate constants, the degradation curves were plotted as natural logarithmic values as a function of irradiation time and straight lines were obtained (Figs. 7–9). The initial pseudo-first-order degradation rate constants were then calculated and the constants obtained are presented in Table 1. The calculated pseudo-first-order reaction rate constants were strongly dependent on the initial concentration of MTBE and on the hydrogen peroxide concentration. According to these results, the MTBE degradation rate with the high-pressure UV source was highest

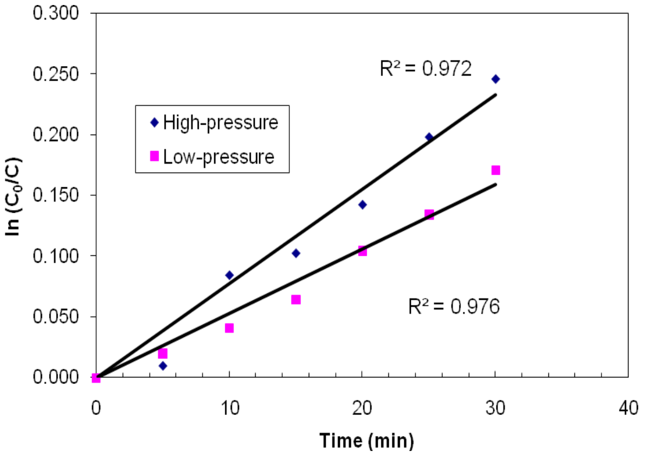


Fig. 7. Pseudo-first order rate for the degradation of MTBE in water with low- and high-pressure UV, no hydrogen peroxide, MTBE $C_0$ = 1 ppm.

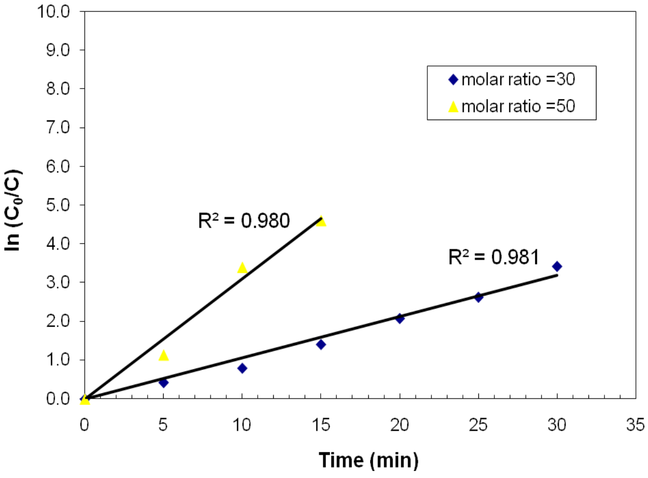


Fig. 8. Initial pseudo-first order rate constants for the degradation of MTBE in water with low-pressure UV, different molar ratio, MTBE $C_0$ = 1 ppm.

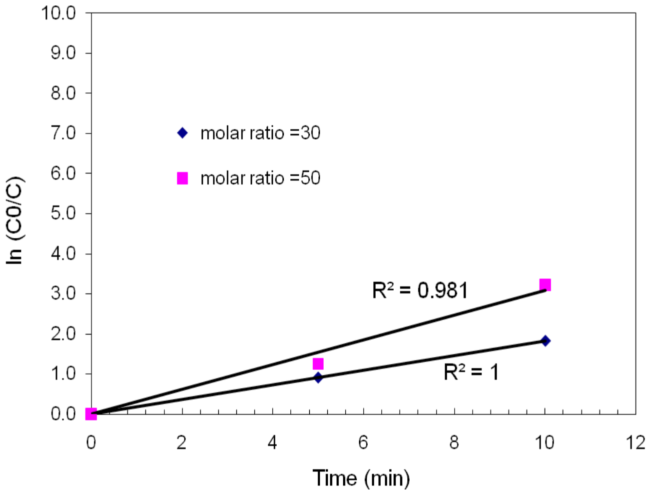


Fig. 9. Pseudo-first order rate constants for the degradation of MTBE in water with high-pressure UV, different molar ratio, MTBE $C_0$ = 1 ppm.

Table 1
Pseudo-first order rate constants for the degradation of MTBE in water with low- and high-pressure UV/$H_2O_2$

| Initial MTBE concentration (ppm) | Initial molar ratio MTBE/$H_2O_2$ | Pseudo-first order $k$ ($s^{-1}$) | |
|---|---|---|---|
| | | Low pressure | High pressure |
| 1 | No peroxide | $8.8\times10^{-5}$ | $1.3\times10^{-4}$ |
| 1 | 1:30 | $1.8\times10^{-3}$ | $5.1\times10^{-3}$ |
| 1 | 1:50 | $5.2\times10^{-3}$ | $6.0\times10^{-3}$ |
| 5 | 1:50 | $3.1\times10^{-3}$ | $5.1\times10^{-3}$ |

## 4. Conclusion

Neither ultraviolet radiation nor hydrogen peroxide on its own was very effective for MTBE removal from water. The UV irradiation combined with hydrogen peroxide process was very effective in removing up to 5 ppm of MTBE in less than 15 min in a batch reactor under ambient conditions. The results were represented by a pseudo-first order kinetics. According to these results, the MTBE degradation rate with the high-pressure UV source was highest. After 10 min of irradiation, MTBE was completely degraded when using the 150 W TQ 150 mercury lamp. A high concentration of MTBE may retard the degradation of MTBE due to a larger competition for the hydroxyl radicals. Nonetheless, results showed that MTBE degradation rate is enhanced at a higher initial concentration of MTBE due to the higher initial $H_2O_2$ concentration when keeping the MTBE/$H_2O_2$ molar ratio constant. A more complex reaction kinetics study is ongoing.

## Acknowledgement

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