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Ozone Kinetics and Diesel Decomposition by Ozonation in Groundwater

Sung-Ho Kong[†], Choong-Il Kwon and Moo-Hoon Kim*

Dept. of Chemical Eng., Hanyang University, Seoul 133-791, Korea
*Samsung Engineering R & D Center, Suwon 449-844, Korea

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Abstract−The ozone kinetics (ozone auto-decomposition; effects of pH and solubility) and diesel/TCE/PCE decomposition (effects of hydroxyl radical scavenger, pH, and ozone/H_2O_2) by ozonation process were investigated in aqueous phase using deionized water, simulated groundwater, and actual groundwater. Reactions with deionized water and groundwater both showed the second-order reaction rates: the reaction rate was much higher in groundwater (half-life of 14.7 min) than in deionized water (half-life of 37.5 min). It was accelerated at high pH condition in both waters. The use of ozone showed high oxidation rates of TCE, PCE, and diesel. Hydroxyl radical scavengers acted as inhibitors for diesel decomposition, and high pH condition and addition of hydrogen peroxide could promote to degrade diesel in groundwater indicating ozone oxidation process could be effectively applied to treating diesel contaminated-groundwater.

Key words: Ozone, Groundwater, Diesel, Hydroxyl Radical, Hydrogen Peroxide

INTRODUCTION

Soil and groundwater contamination have been problems around the world. The US EPA reported that many volatile organic compounds (VOCs) such as trichloroethylene (TCE) and perchloroethylene (PCE) existed in many groundwater wells [Westrick et al., 1983]. In California alone, over 1500 organic compounds were detected such as chloroform, 1,1,1-trichlroethane and carbon tetrachloride, *etc.* [Mackay et al., 1990]. In Korea, extensive subsurface contamination by leakage from underground storage tanks (USTs) has been also reported, recently. To remediate contaminated soils and groundwater, various advanced oxidation processes (AOPs) have been developed such as Fenton, Fenton-like, ozone, ozone/UV, and ozone/H_2O_2 processes [Watts et al., 1999]. Although the Fenton reaction has been proven to be an effective method with hydroxyl radical formation, it has many problems with pH adjustment (pH 3-4) requiring neutralization and sludge production of $Fe(OH)_3$ [Oh et al., 1993; Bull et al., 1992].

Ozonation is considered to be an attractive method in chemical oxidation processes due to its high oxidant capacity (E^0=2.08 V) and electrophilic characteristics [Langlais et al., 1991]. Hydroxyl radicals can be also produced during ozonation that have even higher oxidation potential (E^0=3.06 V). However, few studies have been performed regarding ozone behavior and decomposition in groundwater since it consists of complex components and has site-specific characteristics [Freeze et al., 1979].

Diesel fuel, which is a complex mixture of intermediate distillates from crude oil, is composed of approximately 40% of *n*-alkanes, 40% of *iso*- and cycloalkanes, 20% of aromatic hydrocarbons, and a few percents of various mixtures. Among the components in diesel fuels, compounds of lower molecular mass will tend to evaporate and degrade more readily while other components remain in water. Ozone reactions with aromatic compounds in diesel fuel have been widely studied; however, little is known about the ozone reaction with saturated hydrocarbons that are major components in diesel fuel [Hamilton et al., 1968; Kuo et al., 1997]. In this study, ozone kinetics and diesel decomposition in groundwater by ozonation process were investigated for further pilot-scale application at the site.

MATERIALS AND METHODS

1. Experimental Apparatus and Methods

Fig. 1 is a schematic of the apparatus used in ozone oxidation

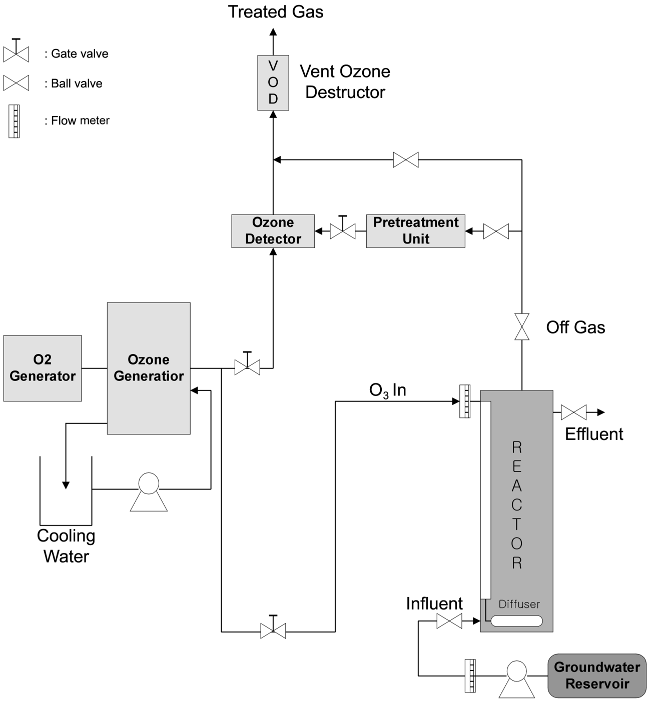

Fig. 1. Schematic of experimental diagram.

[†] To whom correspondence should be addressed.
E-mail: shkong@hanyang.ac.kr

[‡] This paper is dedicated to Professor Baik-Hyon Ha on the occasion of his retirement from Hanyang University.

experiments in this study. The reactor vessel is cylindrical, approximately 0.2 m in diameter by 1m in height with effective volume of 25 L. The height to diameter ratio of the vessel was about 5 : 1 to maintain good mixing of the solution by bubbling [Gurol et al., 1982]. A liquid sampling port was located approximately 5 cm from the bottom of reactor.

Ozone gas was provided by an ozone generator (model OZAT^{R} CFS-2A, Ozonia Co., USA). Gas-phase ozone measurements were made by an in-line ozone monitor (Afx series H1-S bench, IN-USA Co., USA). Excess ozone gas was passed through to an ozone destructor. The concentration of residual dissolved ozone concentration in water was followed by indigo procedure [Ciesceri et al., 1998].

To determine changes of diesel decomposition in aqueous phase, TOC (TOC analyzer, Sievers. Co., USA) was measured by varying ozone gas dosage. Analytical measurement for TCE (Junsei. Co., Japan) and PCE (Aldrich Co., USA) was done with a GC-ECD (HP 6890, Hewlett Packard Co., USA) after liquid-liquid extraction with n-hexane for 24 hr.

Deionized water was prepared as ultrapure condition (≥18.2 MΩ, Maxima Ultra Pure Water, Elga, USA), and simulated groundwater (SG) was prepared based on the major components in groundwater at the site. Ozone kinetics experiments were performed with deionized water and actual contaminated groundwater, and TCE, PCE and diesel decomposition experiments were performed with deionized water and SG. The pH was adjusted with hydrochloric acid (HCl) and sodium hydroxide (NaOH), and all experiments were performed in batch-type mode.

2. Groundwater Analysis

Groundwater for this experiment was collected from monitoring wells at the site where it was contaminated with diesel. Table 1 shows the results of groundwater analysis. The heavy metals, which are hazardous to public health, were below maximum concentration levels (MCLs) or not detected.

Table 1. Properties of groundwater at the site and groundwater quality standards

	Category	This groundwater	MCLs*
General contamination matters	pH	6.5-7.5	5.8-8.5
	COD	32	≤6 (mg/L)
	MPN/100 mL	140	≤5,000
	NO_3-N	7.6	≤20 (mg/L)
	Cl^-	51	≤250 (mg/L)
Specific contamination matters	Cd	0.094 μg/L	≤0.01 (mg/L)
	As	0.34 μg/L	≤0.05 (mg/L)
	CN^-	No detect	No detect
	Hg	No detect	No detect
	Pb	2.3 μg/L	≤0.1 (mg/L)
	Cr^{6+}	1.5 μg/L	≤0.05 (mg/L)
	Phenol	No detect	≤0.005 (mg/L)
	Organic phosphorous	No detect	No detect
	TCE	188 μg/L	≤0.03 (mg/L)
	PCE	79 μg/L	≤0.01 (mg/L)

*MCLs: Maximum contaminant levels

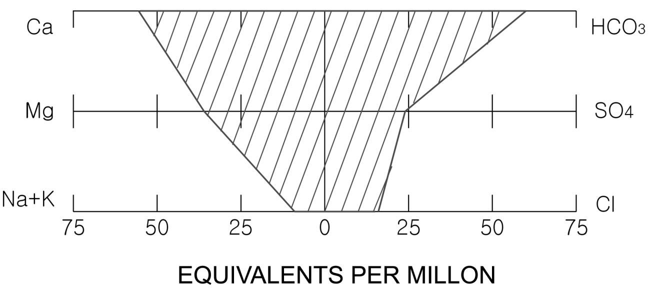

Fig. 2. Stiff diagram of groundwater.

Fig. 2 shows the major components in groundwater. Cations were analyzed with inductively coupled plasma mass spectrometry (ICP-MS, HP 503, Hewlett Packard Co., USA) and anions were measured with ion chromatography (Dionex, USA).

RESULTS AND DISCUSSION

1. Ozone Auto-decomposition

The decomposition of ozone in aqueous solution has been studied for several decades. In the literature regarding the kinetics of ozone decomposition, the most common observation is the reported disagreement among different researchers as to both the order of the decomposition reaction and the magnitude of the reaction rate constant. The range of conditions investigated by various researchers and their conclusions concerning the reaction order relative to ozone is summarized in Table 2 [Gurol et al., 1982].

Table 2. Summary of the kinetics of ozone decomposition in water

pH	Temperature (°C)	Reaction order
2-4	0	2
5.3-8	0	2
acidic		3/2
basic		1
1-2.8	0-27	1
7.6-10.4	1.2-19.8	1
0-6.8	25	3/2
8-10	25	2
5.4-8.5	5-25	3/2
10-13	25	1
9.6-11.9	25	1
6	10-50	3/2-2
8	10-20	1
2-4	30-60	2
0.22-1.9	5-40	1 or 2
9	20	1
8.5-13.5	18-27	1
0.5-10.0	3.5-60	1
2.1-10.2	25	3/2
acidic	25	1-2
basic	25	1
in this study		
5.5-5.8 (deionized water)	15	2
6.8-7.3 (groundwater)	12-15	2

These major differences among the findings of the various researchers are believed to be due to different experimental conditions (pH, temperature, and ionic strength, *etc.*), different experimental systems, the presence of impurities in the reagents, and various data analysis methods. Additionally, most of the researchers studied the decomposition reaction in batch reactors and analyzed their data only by the integral method [Gurol et al., 1982].

Kuo et al. [1977] measured the ozone auto-decomposition rate at 25 °C in deionized water (pH 5.2-5.4), and the results showed reaction orders of 1 and 3/2 and half-life of 40 min each. Although the reaction order and reaction rate constants were different, the half-life (approximately 40 min) showed results similar to many previous studies.

Fig. 3 is the result of ozone auto-decomposition in deionized water (a) and in groundwater (b) at different initial concentrations. Two reactions showed the high correlation ($r^2>0.95$) with second-order reaction constants. In deionized water, second-order reaction rate constant (k) was $5.0*10^{-3}$, $2.9*10^{-3}$, and $1.7*10^{-3}$ L/mg min in each reaction, and half-life of ozone was averaged at 37.5 min. Although reaction order was different with that of Kuo et al. [1977], the half-life in this study showed similar results. On the other hand, the reaction rate constant in groundwater was $4.9*10^{-2}$, $1.4*10^{-2}$, and $0.65*10^{-2}$ L/mg min in each reaction with 14.7 min of half-life of ozone. The results shown in Fig. 3 demonstrate that the ozone auto-decomposition rate in groundwater was much higher than that in deionized water, indicating ozone could have been decomposed rapidly reacting with various organic species in groundwater.

2. The Effect of pH on Ozone Decomposition

Generally, it has been known that variables such as pH, temperature, natural organic matters (NOMs), ionic strength, and UV could affect ozone decomposition. Especially, pH has been known more effective on ozone decomposition than the other variables. At acidic condition, ozone is stable; however, ozone decomposition rate is accelerated at high pH (pH 9-12) [Langlais et al., 1991; Staehelin et al., 1985].

Fig. 4 shows the ozone auto-decomposition at different pH values (acidic, neutral, and basic conditions) showing second-order reaction rates in all conditions. Similar reaction rates were observed below neutral pH conditions, and higher reaction rate was examined with pH of 10.2 [Fig. 4(a)] indicating hydroxide ion (OH^-) acceler-

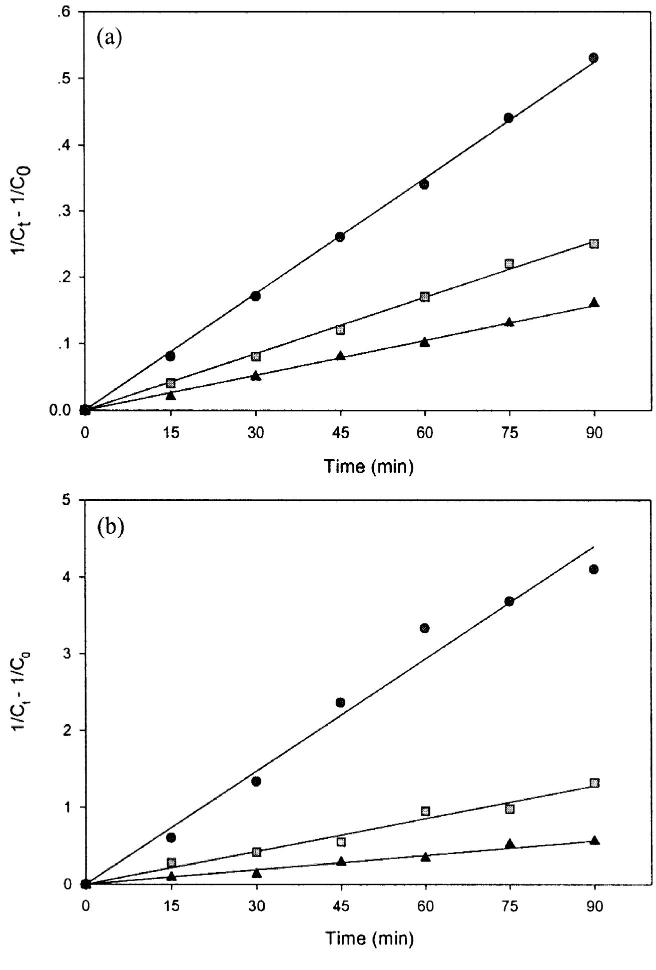

Fig. 3. Ozone auto-decomposition rate with second-order reaction at different initial concentration in water.
(a) Deionized water (pH: 5.5-5.8, Temp: 15 °C)
(b) Groundwater (pH: 6.8-7.3, Temp: 12-15 °C)
Initial ozone gas concentration (λ: 10 mg/L, ν: 20 mg/L, σ: 30 mg/L)

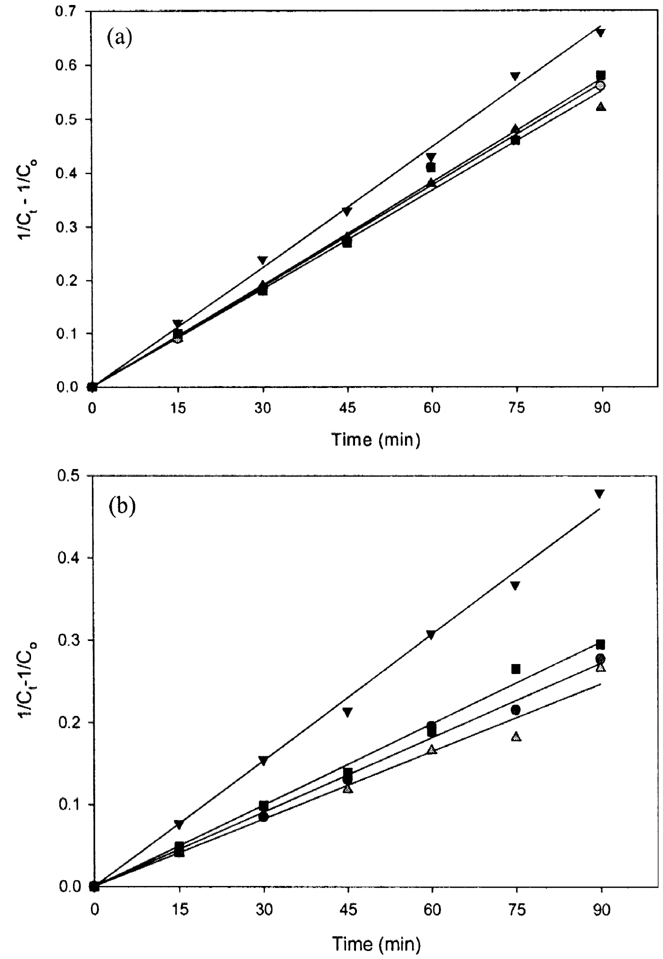

Fig. 4. The pH effect on ozone auto-decomposition at different pHs.
(a) Deionized water; σ (pH=3.9), λ (pH=5.8), ν (pH=7.5), τ (pH=10.2)
(b) Groundwater; σ (pH=3.5), λ (pH=5.6), ν (pH=7.1), τ (pH=10.8)

ated the ozone decomposition [Eq. (1); Gurol et al., 1982; Staehelin et al., 1982; Kuo et al., 1999].

$$O_3+OH^- \rightarrow O_2^- + HO_2\cdot \quad (1)$$

In Fig. 4(b) similar results with Fig. 4(a) are shown indicating ozone decomposition rate at high pH (pH=10.8) was faster than that of below neutral pH (pH=7.1) in groundwater. However, compared with Figs. 4(a) and 4(b), the differences of reaction rate constants between neutral pH and basic pH in groundwater were smaller than those in deionized water, indicating that ozone decomposition might not only be accelerated by hydroxide ion but also by various organic and inorganic matters in groundwater. The reaction rate constants above each reaction are summarized in Table 3. Reaction rate constants were much higher in groundwater than in deionized water, implying that effects of organic and inorganic matters could be stronger than those of pH in groundwater.

3. Ozone Transfer Efficiency

Generally, ozone solubility in aqueous phase is determined by ozone concentration, temperature, and pH. It could be expressed as Bunsen adsorption coefficient (β) or solubility ratio (S), which means ratio of ozone concentration in water to ozone concentration in gas at a certain temperature. Ozone solubility ratio could also be calculated with the following equation [Langlais et al., 1991].

Table 3. Reaction rate constants with second-order reaction at different pH in deionized water and groundwater

	pH	k (Reaction rate constant; L/mg·min)	Half-life (min)
Deionized water	3.9	0.0027	38.6
	5.8	0.0030	35.8
	7.5	0.0033	31.9
	10.2	0.0051	21.3
Groundwater	3.5	0.0061	17.6
	5.6	0.0063	16.9
	7.1	0.0064	16.6
	10.8	0.0075	14.1

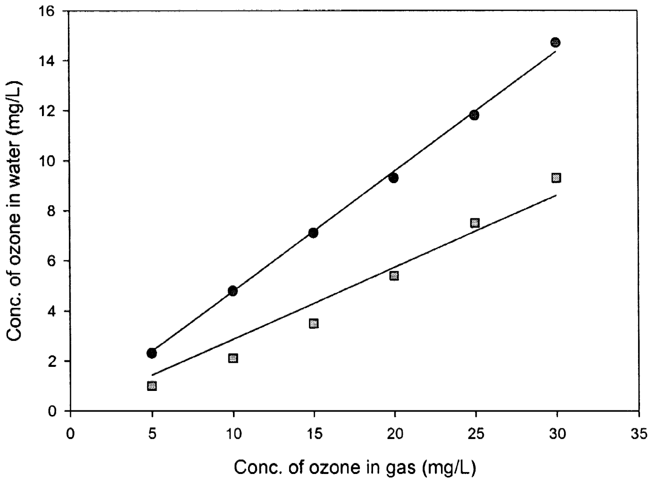

Fig. 5. Residual ozone concentration in aqueous phase (λ: Deionized water, v: Groundwater).

$$\text{Ln } S = -0.45 - 0.043\, t \quad (2)$$

where, S=solubility ratio as mg/L in water to mg/L in gas
t=temperature (°C)

Fig. 5 shows the residual ozone concentration in water. When the experiment was performed with deionized water at 15 °C, ozone solubility S could be calculated as 0.34 by using Eq. (2). However, S was observed at 0.48 in this study, which was much higher than the theoretical value. This result shows that ozone might have been supersaturated in water due to better contacting of ozone gas with water, and a large amount of ozone could react with organic matters in aqueous phase. The residual ozone concentration in deionized water (S=0.48) was higher than that in groundwater (S=0.28), indicating ozone was decomposed quickly by reacting with organic matters in groundwater (Fig. 5).

4. TCE and PCE Degradation

Groundwater analysis showed that TCE and PCE, which are known as carcinogens, were detected at the level of 6-8 times higher than MCLs. Fig. 6 shows the removal efficiencies of TCE and PCE

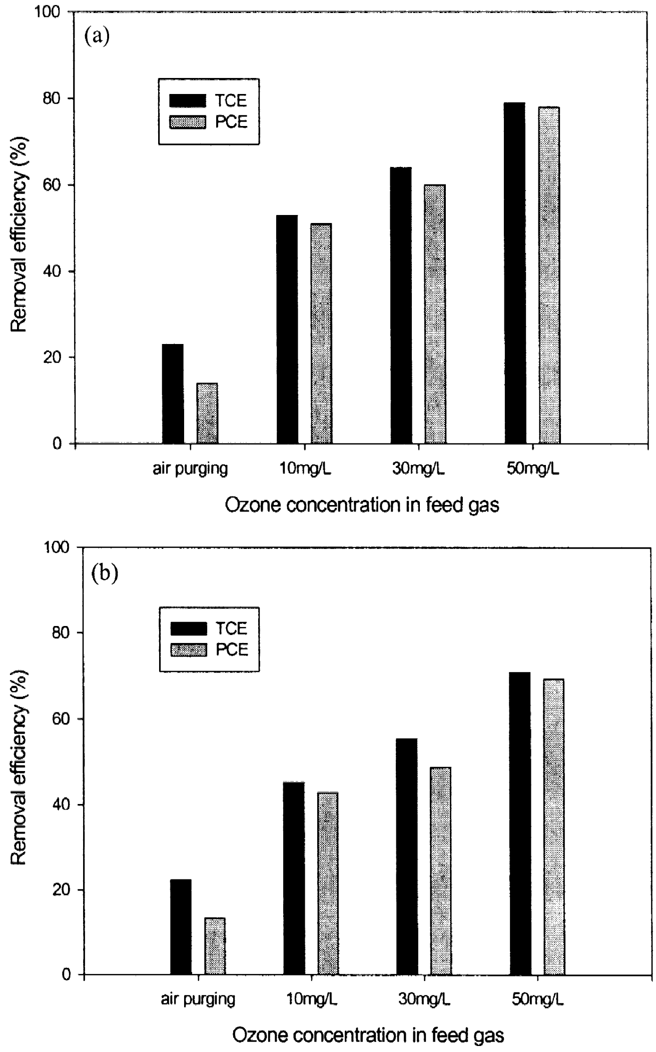

Fig. 6. TCE and PCE decomposition with varying ozone dosage after 3 hr ozone reaction.

(a) Deionized water (b) Simulated groundwater in deionized water [Fig. 6(a)] and SG [Fig. 6(b)]. Trichloroethylene and PCE evaporate easily since TCE (Henry's constant, $9.1*10^{-3}$ atm-m^3/mole at 25 °C) and PCE (0.0153 atm-m^3/mole) have low Henry's constants [Watts, 1998]. The removal efficiency by ozonation was much higher at low ozone concentration compared to air purging itself indicating ozone oxidation could be applied to eliminate TCE and PCE from groundwater efficiently.

Considering those results, TCE ($k_{OH\cdot}=4.0*10^9\ M^{-1}S^{-1}$) and PCE ($k_{OH\cdot}=2.8*10^9\ M^{-1}S^{-1}$) have high reaction rate constants with hydroxyl radical [Watts, 1998], whereas they have very low reactivity with ozone (TCE $k_{O3}=1.7*10^1\ M^{-1}S^{-1}$, PCE $k_{O3}=<10^{-1}\ M^{-1}S^{-1}$) [Hoigne et al., 1983]. Therefore, it could be assumed that most of them were degraded by hydroxyl radicals produced by ozone decomposition. Comparing Figs. 6(a) and (b), removal efficiency in deionized water was higher than that in SG by 10%, indicating hydroxyl radicals were quenched by scavengers such as carbonate ($k_{OH\cdot}=4.2*10^8\ M^{-1}S^{-1}$) and bicarbonate ($k_{OH\cdot}=1.5*10^7\ M^{-1}S^{-1}$) in groundwater [Watts, 1998].

5. Diesel Decomposition

In aqueous solution, ozone may act on various organic compounds by direct reaction with the molecular ozone and by indirect reaction with the radical species formed by ozone decomposition process in water [Staehelin et al., 1982; Nelson et al., 1994; Legube et al., 1999].

Fig. 7 shows the remaining TOC in diesel saturated-deionized water [Fig. 7(a)] and SG [Fig. 7(b)] after ozone oxidation. Air purging itself could not lower TOC values. TOC values were only lowered by the ozonation process, and applying higher ozone concentration showed higher removal of TOC. The removal of TOC was greater in deionized water than in SG coinciding with the results of Fig. 6. In Fig. 7, the TOC values increased initially and afterwards decreased, indicating that the large molecular organic matters might have been broken down into smaller molecular matters as reacting with ozone in aqueous solution. Especially, it is reported that the hydroxyl radical reaction is probably the only reaction capable of degrading the saturated aliphatic molecules since ozone has very low reactivity with saturated hydrocarbons [Nelson et al., 1994; Legube et al., 1999]; therefore, it could be assumed that ozone reacted with unsaturated hydrocarbons and aromatic compounds at the initial reaction.

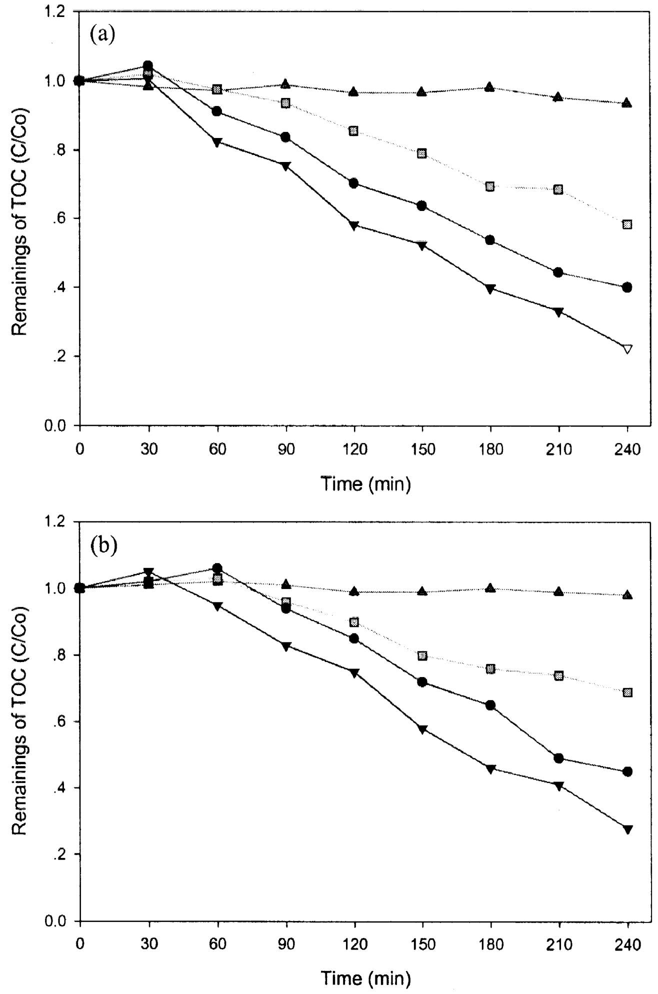

Fig. 7. TOC removal in diesel-saturated water with varying ozone dosage.
(a) Deionized water (b) Simulated groundwater
(σ: Air purging, ν: 10 mg/L, λ: 30 mg/L, τ: 50 mg/L in feed ozone gas)

Fig. 8 shows the effect of hydroxyl radical scavenger in SG. As a result of groundwater analysis, bicarbonate was a main component in scavengers. As previously mentioned, bicarbonate has high reactivity with hydroxyl radicals, and it has been proven to be an effective hydroxyl radical scavenger. When bicarbonate concentration increased, TOC removal decreased (Fig. 8), explaining the results of Figs. 6 and 7 that the removal efficiencies were lower in SG than in deionized water. Therefore, the removal of bicarbonate by softening or demineralizing would enhance the rate of oxidation of organic micropollutants.

6. The Effects of pH on Diesel Decomposition

Legube et al. [1999] reported basic solution improved the efficiency of treating contaminated water by ozone. In Fig. 4, it was shown previously that ozone decomposition rate was accelerated at high pH in both deionized water and SG. Fig. 9 shows the results of TOC removals at different pHs in deionized water and SG. Diesel

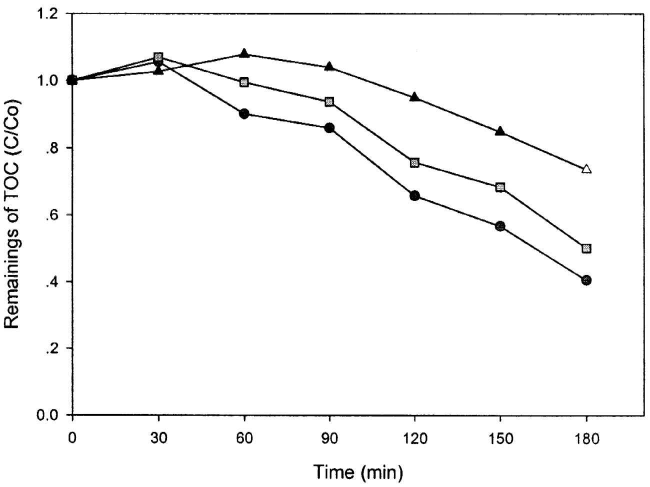

Fig. 8. The effects of hydroxyl radical scavengers in diesel decomposition.
Ozone concentration: 50 mg/L, HCO_3^- concentration, λ: 0, ν: 381.25 mg/L, σ: 762.5 mg/L in solution

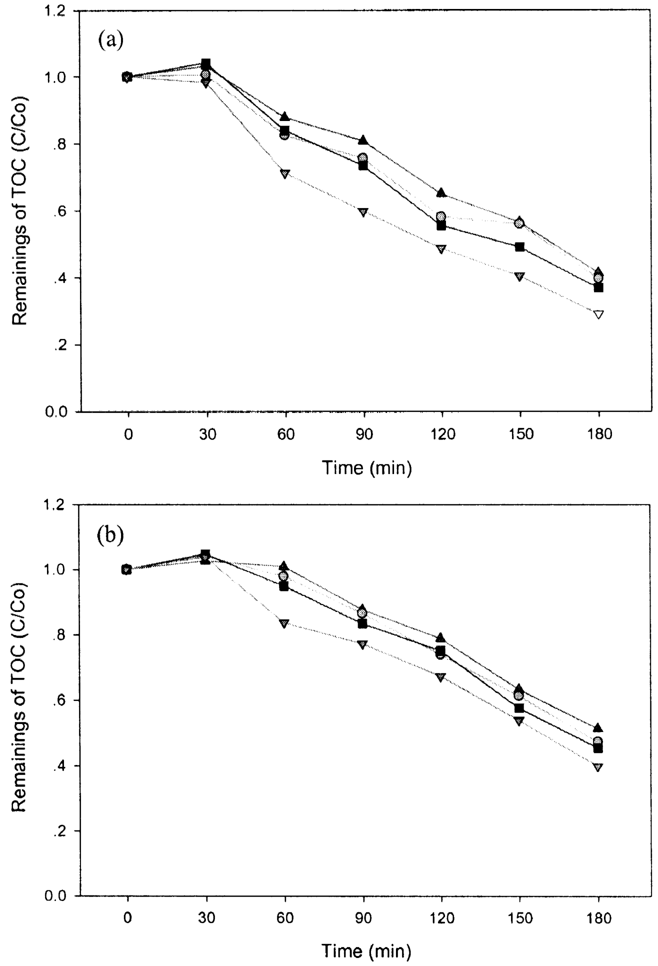

Fig. 9. TOC removal in diesel-saturated water at different pHs.
Ozone gas concentration: 50 mg/L
(a) Deionized water (*σ*: pH=3.2, *λ*: pH=5.8, *ν*: pH=7.5, *τ*: pH=10.1)
(b) Simulated groundwater (*σ*: pH=3.5, *λ*: pH=5.7, *ν*: pH=7.2, *τ*: pH=10.2)

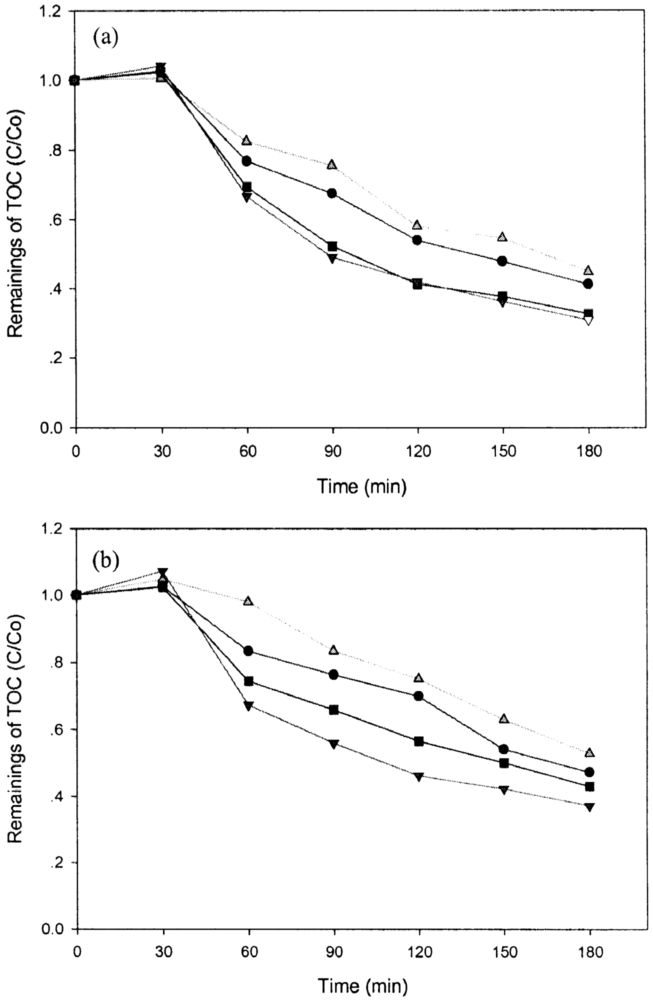

Fig. 10. Diesel decomposition with ozone/H_2O_2 oxidation.
H_2O_2/O_3 molar ratio (R), *σ*: no H_2O_2, *λ*: R=0.25, *ν*: R=0.5, *τ*: R=1.0
(a) Deionized water (b) Simulated groundwater

decomposition was not affected by pH conditions below neutral but removal efficiency was higher at basic conditions. It was probably due to hydroxyl radical formation through ozone decomposition by hydroxide ions.

7. The Diesel Decomposition with Ozone/H_2O_2

As for treatment of industrial wastewater, many studies have led to further improvement in efficiency of ozonation for various applications; ozone in alkaline solution (O_3+OH^-), photolysis of ozone (O_3+UV), and perozone (O_3+H_2O_2) are the principal existing AOPs known as the most promising processes for industrial effluents depollution [Beltran et al., 1998; Bellamy et al., 1991; Benitez et al., 1999; Legube et al., 1999]. Fig. 10 shows the results of H_2O_2/O_3 oxidation in diesel decomposition.

As a result of experiment in deionized water, the H_2O_2/O_3 system was far more effective than O_3 alone system, and the optimal H_2O_2/O_3 molar ratio was 0.5 coinciding with a previous study [Bellamy et al., 1991]. However, it was 1.0 in SG due to various inorganic matters existing in SG. Therefore, it may be necessary to optimize the H_2O_2/O_3 ratio regarding the site characteristics when applied at the site.

The mechanism of hydrogen peroxide in ozone solution is as follows:

$$H_2O_2+H_2O \rightarrow HO_2^-+H_3O^+ \qquad (3)$$

$$O_3+HO_2^- \rightarrow OH\cdot+O_2^-+O_2 \qquad (4)$$

When hydrogen peroxide is combined with water, it partially dissociates into hydroperoxide ion (HO_2^-) [Eq. (3)]. Also, hydroperoxide ion is likely to induce decomposition of ozone in water generating highly reactive hydroxyl radicals [Eq. (4)]. Comparing Figs. 9 and 10, addition of hydrogen peroxide introduced a higher efficiency than pH adjustments, indicating hydroperoxide ion was better than hydroxide ion (OH^-) in hydroxyl radical formation.

CONCLUSION

In groundwater, the ozone auto-decomposition rate showed second-order reaction, and residual ozone concentration in groundwater was lower than in deionized water indicating scavenging effects of ozone by organic and inorganic matters in SG. The high pH condition was more effective in ozone decomposition in both aqueous phases. Ozone oxidation showed good removal efficiencies of TCE, PCE, and diesel in aqueous phase. These contaminants were degraded by direct and indirect reactions of ozone. The higher ozone concentration showed the greater diesel TOC removal, and high pH and addition of hydrogen peroxide to ozone accelerated diesel decomposition, suggesting ozone oxidation process could be applied to diesel-contaminated groundwater site remediation with proper adjustment of pH and addition of H_2O_2.

ACKNOWLEDGMENTS

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