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PII: S0043-1354(00)00120-2

Wat. Res. Vol. 34, No. 15, pp. 3882–3887, 2000
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Printed in Great Britain
0043-1354/00/$ - see front matter

RESEARCH NOTE

DESTRUCTION OF HUMIC ACID IN WATER BY UV LIGHT—CATALYZED OXIDATION WITH HYDROGEN PEROXIDE

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(First received 8 September 1999; accepted in revised form 24 December 1999)

Abstract—A batch reactor was used to evaluate the advanced oxidation process of the UV/H_2O_2 system for control of natural organic matter (NOM) in drinking water. The light sources used include a 450 W high-pressure mercury vapor lamp and sunlight. Both quartz and Pyrex filters were used to control the wavelength and energy of UV light applied to the aqueous systems. The results showed that NOM oxidation and H_2O_2 decomposition followed first-order and zero-order reaction kinetics, respectively. The optimum H_2O_2 dose was found to be 0.01% for the oxidation of humic acids in this study. Carbonate and bicarbonate ions inhibited the degradation of humic acids. © 2000 Elsevier Science Ltd. All rights reserved

Key words—humic acid, hydrogen peroxide (H_2O_2), advanced oxidation process (AOP)

INTRODUCTION

The presence of natural organic matter (NOM) in both surface and ground water supplies has received much public attention in recent years because toxic disinfection byproducts (DBPs) can result from chlorination procedures in the water treatment processes. The removal of NOM from raw water is largely achieved by chemical coagulation and flocculation, using aluminium sulfate or ferrous sulfate and lime in the presence of excess chlorine. However, the NOM removal in conventional drinking water treatment processes is quite low, between 10 and 50% (Jacangelo *et al.*, 1995).

Due to the presence of a wide variety of NOM and the necessity of the chemical disinfection process to protect the public health, many water utilities have to face the problem of DBPs formation. For the control of DBPs, some treatment alternatives have been proposed (Owen *et al.*, 1995). Among the proposed methods for the control of DBPs, advanced oxidation processes (AOP) deserve more study for potential applications in drinking water treatment. AOP can effectively mineralize many organic contaminants and have become attractive for the control of synthetic organic compounds in wastewater treatments (Kang and Lee, 1997). AOP have many advantages in water treatment processes and have been proposed as an alternative for the control of DBP precursors (Symons and Worley, 1995; Eggins *et al.*, 1997).

The UV/H_2O_2 process is an example of a homogeneous AOP. Generally, the effectiveness of homogeneous light-driven oxidation processes is associated with very reactive species, such as hydroxyl radicals, which are generated in the reaction mixture by the direct photolysis of H_2O_2 under UV irradiation:

$$H_2O_2 + h\nu \rightarrow 2OH^{\cdot}$$

The hydroxyl radicals attack organic compounds relatively non-selectively with rate constants ranging from 10^6 to 10^{10} M^{-1} s^{-1} (Buxton *et al.*, 1988), oxidizing them by hydrogen atom abstraction or by addition to double bonds. The UV/H_2O_2 process has been proven effective in treating waters containing a number of aliphatic and aromatic compounds (Beltrán *et al.*, 1993; Sundstrom *et al.*, 1986). In UV/H_2O_2 process, the optimum H_2O_2 dosage should be obtained and applied since the excess H_2O_2 dose can reduce the oxidation rate (Ku *et al.*,

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1998). The presence of Cu^{2+} will enhance the H_2O_2 decomposition and increase the rates of organic oxidation (Baxendale and Wilson, 1957), however, the carbonate/bicarbonate ions will inhibit the oxidation of organics due to its scavenging effect (Liao and Gurol, 1995). Despite the increasing interest in this process for water and wastewater treatment, there is very little information available regarding destruction of the NOM by the UV/H_2O_2 process (Symons and Worley, 1995). The aim of this paper is to examine the influence of light intensity, the initial concentration of humic acid, H_2O_2 dose, and the matrix of carbonate/bicarbonate ions on the photodegradation of humic acid in order to evaluate the applicability of UV/H_2O_2 technology to the remediation of DBP precursors in water.

EXPERIMENTAL

Humic acids extract, H_2O_2 (30% solution), sodium carbonate, and sodium bicarbonate used in this study were purchased from Nacalai Tesque (Kyoto, Japan). Milli-Q water (Millipore) was used to prepare the humic acid stock solution (which contained 1000 mg/l of non-purgeable organic carbon, NPOC). Before each experiment, aqueous solutions were prepared by diluting the humic acid stock solution and adding with known amounts of H_2O_2 and carbonate/bicarbonate.

A 10-l stainless-steel batch reactor (20 cm diameter × 30 cm depth) with a quartz window was used in this study for the photooxidation of humic acid in aqueous solutions (Hu and Yu, 1994). A 450 W high-pressure mercury-vapor lamp (Hanovia, Ace Glass Co., Vineland, NJ) was used as the light source. A UV lamp was inserted into the hollow quartz or Pyrex tube (55 mm od, 45 mm id) located at the center of the reactor. A water-cooling loop was used to prevent the lamp from overheating and to maintain the water at room temperature (25°C). The UV light was turned on ten minutes before performing the experiment. Eight liters of aqueous solution was then added into the reactor. The solution's mixing was achieved with a magnetic stirrer. Two aliquots of 30 ml were used to measure initial humic acid concentration; duplicate samples of 30 ml were withdrawn from the reactor at various time intervals for analysis. All of the experiments were conducted at pH = 7 except the ones to study the effects from carbonate ions, which were conducted at pH = 10. Control experiments were carried out under the same conditions in the dark.

The experiments were also carried out with direct sunlight. Four 2-l glass beakers, each filled with 1.5 l of water and then covered with a quartz plate, were put on the roof of a 15-story building (at 70 m elevation) in downtown Taipei between 11 am and 2 pm in July 1998. Unexposed controls were run with each batch of test samples to establish starting humic acid concentrations and also to monitor the consistency of the results. The control reactors were wrapped with aluminum foil to prevent UV exposure; they were set outside with the test samples.

Samples were filtered through a 0.45 μm syringe filter to remove particles and then acidified with two drops of 2 N HCl solution prior to TOC analysis. The NPOC concentration was measured by an organic carbon analyzer (Shimadzu TOC 5000A), and the UV absorbance was measured by a high-precision, double-beam spectrophotometer (Shimadzu UV 160A). The concentration of the hydrogen peroxide was determined by UV absorbance spectrometry at a wavelength of 260 nm (UV_{260}). For correction of the absorbance contributed from humic acid in the water, the UV_{260} from humic acids was deducted from the total absorbance.

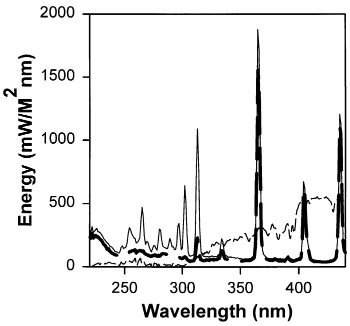

Fig. 1. Wavelength and energy distribution of the UV sources used in UV/H_2O_2 oxidation of humic acid. —: UV/quartz; – – –: UV/Pyrex; - - -: sunlight.

A UV radiation spectroradiometer (MSS 2040, Elekronik Gmbh, Bielefeld, Germany) was used to measure the UV spectrum and energy distribution of the light source. The spectroradiometer was placed outside the quartz or Pyrex window of the reactor to record the wavelength and energy distribution of the UV light. Figure 1 shows the UV spectra of the UV/quartz, UV/Pyrex, and sunlight that were used for the oxidation of humic acids in this study. The major spectral distribution of the UV lamp was at wavelengths of 254, 265, 297, 302, 312, 365, 405, and 435 nm, if the quartz tube was used as the cooling device. However, the UV light at 254, 265, 297, and 302 nm was filtered out when the Pyrex cooling tube was used. When sunlight was used as the light source, a continuous UV spectrum was observed at wavelengths longer than 300 nm. The light intensities for UV/quartz, UV/Pyrex, and sunlight were 275.8, 20.8, and 23.2 W/m^2, respectively.

RESULTS AND DISCUSSION

Direct photolysis of hydrogen peroxide

The photolysis of H_2O_2 in pure water was studied extensively by various groups of researchers in the 1950s (Baxendale and Wilson, 1957; Weeks and Matheson, 1956; Hunt and Taube, 1952) in order to elucidate the reaction mechanism and to determine the primary and overall quantum yields. The direct photolysis of H_2O_2 generates the very highly oxidizing and reactive $^{\cdot}OH$ radicals, which then participate in the steps leading to the degradation

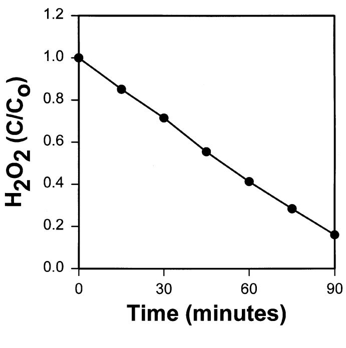

Fig. 2. Decomposition of hydrogen peroxide in the UV/H_2O_2 processes. [H_2O_2] = 0.1%; UV source: UV/quartz.

of organic pollutants (Volman, 1949). Figure 2 shows the experimental data obtained during the irradiation of a 0.1% hydrogen peroxide aqueous solution, with a rate of 0.30 mM min^{-1}. Although first-order decomposition was observed in literature (Liao and Gurol, 1995), the initial photolysis of hydrogen peroxide follows zero-order kinetics within the H_2O_2 addition tested in this study (0.01–0.5%).

Degradation of humic acid sensitized by hydrogen peroxide under UV light

The production of hydroxyl radicals from hydrogen peroxide requires a large dissociation energy (213 kJ mole^{-1}) in order to cleave the O–O bond, which means that short wave UV-C energy (wavelengths of 200–280 nm) is necessary to lead to useful radical yields (Atkins, 1990).

Both hydrogen peroxide and humic acids are weakly absorbing compounds in the UV range (Fig. 3). In the case of H_2O_2, the absorption increases as the wavelength decreases. When the solutions was irradiated by UV light, H_2O_2 absorb much more light than humic acids at wavelength less than 300 nm, as shown in Fig. 3. Therefore, in dilute aqueous solutions of humic acid (~10 mg/l) in the presence of various concentrations of hydrogen peroxide (0.001–0.5%), H_2O_2 is the principal absorber of UV light (Beltran *et al.*, 1997).

Figure 4 compares the destruction rate of humic acid irradiated by three UV light sources with an initial peroxide concentration of 0.1%. Approximately 90% of the humic acid was oxidized within 1 h of irradiation when UV/quartz was used. However, oxidation of only 20% of humic acid was observed after 2 h irradiation when UV/Pyrex or sunlight was used as the light source. The lamp with the quartz filter has the highest intensity in the UVC region, which favors its use for the activation of hydrogen peroxide. The employment of such a UV source will result in higher OH radical yields from the increased emission of UVC compared with the other sources, UV/Pyrex and sunlight. Of the three different light sources, the rate of humic acid decay with UV/quartz was the greatest. With UV/quartz, the humic-acid decay follows first-order kinetics with an observed rate constant of $k = 0.037\ \text{min}^{-1}$, while that of hydrogen peroxide obeys zero-order kinetics with a rate of 0.30 mM min^{-1}. Humic acid decomposed at a slower rate with UV/Pyrex ($k = 0.0025\ \text{min}^{-1}$) and sunlight ($k = 0.0007\ \text{min}^{-1}$), which have minor UVC emission and comparable total UV light intensity compared to UV/quartz. Because the primary quantum yield of hydrogen peroxide at 254 nm is very high, $\phi_{H_2O_2} = 0.5$ molecule/photon (Baxendale and Wilson, 1957), with UV/quartz, H_2O_2 could undergo photolysis and oxidation of humic acid would be due mainly to the UV/H_2O_2 process. The degradation of humic acid with UV/Pyrex or sunlight could result from both direct photolysis of humic acid and the UV/ H_2O_2 process (Wang *et al.*, 1997).

Effect of H_2O_2 concentration

Although H_2O_2 does not oxidize humic acid at all, as observed in this work, when combined with UV radiation the rate of humic acid oxidation increases extraordinarily compared to that of direct photolysis. Data showing the effect of H_2O_2 concentration on the pseudo-first-order rate constants for humic acid degradation are plotted in Fig. 5. The initial H_2O_2 concentration varied from zero to 0.5% (147 mM), the UV dose rate was 275.8 W/m^2 for each run, and the initial concentration of humic acid was set at 5 mg/l. The destruction rate of humic acid increased with the increase of hydrogen peroxide concentration up to 0.01% and then decreased with further increases of H_2O_2 concentration. Similar experimental results have been reported by Ku *et al.* (1998). In this process, hydroxyl radicals generated from the direct photolysis of H_2O_2 were the main responsible species for humic acid elimination. However, H_2O_2 also reacted with these radicals and hence acted as an

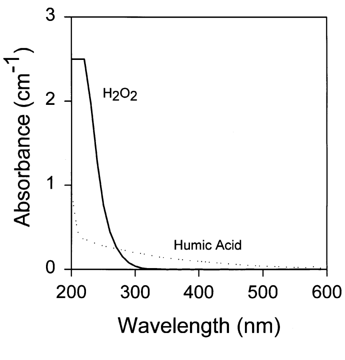

Fig. 3. Absorption spectra of H_2O_2 and humic acid (Nacalai Tesque) in water. (—) H_2O_2; (…) Humic acid; UV source: UV/quartz.

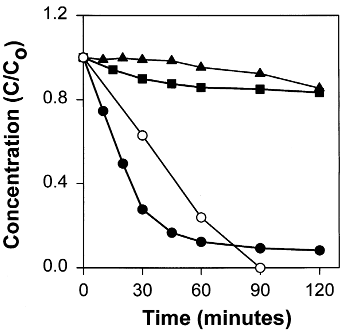

Fig. 4. UV/ H_2O_2 oxidation of humic acid with various UV sources. [humic acid]$_0$=5 mg/l; [H_2O_2]$_0$=0.1%; (●) UV/quartz; (■) UV/Pyrex; (▲) sunlight; solid symbol for humic acid and hollow symbol for H_2O_2.

inhibiting agent of humic acid oxidation. In addition, H_2O_2 itself absorb lights in the system and hence the light intensity available for humic acids is reduced at higher H_2O_2 concentrations. As can be deduced from Fig. 5, when the concentration of H_2O_2 is higher than 0.01% its OH radical scavenging effect becomes of great importance and the humic acid oxidation rate decreases. It has to be noted, however, that elimination of humic acid due to hydroxyl radicals continues to be the main pathway even at the high H_2O_2 concentration of 0.5% for which the oxidation rate is still higher than that obtained from direct photolysis (Fig. 5, $[H_2O_2]_0 = 0$).

Effect of initial humic acid concentration

At an H_2O_2 concentration of 0.1% (29.4 mM), the initial rate constants for humic acid degradation were similar with the initial humic acid concentration of 3 and 5 mg/l but slightly decreased with the initial humic-acid concentration of 8 mg/l (Fig. 6). At H_2O_2 concentrations of 0.3 and 0.5%, the initial rate constants of humic acids showed no significant difference among 3, 5, and 8 mg/l of initial humic acid concentration. At short irradiation times, where humic acids and hydrogen peroxide can be considered the only effective OH radical scavengers, a steady-state kinetic analysis can be considered on the basis of the following simple reaction scheme (Brezonik and Fulkerson-Brekken, 1998):

$$H_2O_2 + h\nu \rightarrow 2^{\cdot}OH \quad \phi_{OH}FG_0/V \tag{1}$$

$$^{\cdot}OH + \text{humic acid} \rightarrow \text{humic acid radical} + H_2O$$
$$k_{HA} = 2.3 \times 10^4 \ (\text{mg of C/l})^{-1}\ s^{-1} \tag{2}$$

$$^{\cdot}OH + H_2O_2 \rightarrow HO_2^{\cdot} + H_2O$$
$$k_{H2O2} = 2.7 \times 10^7\ M^{-1}\ s^{-1} \tag{3}$$

where ϕ_{OH} is the primary quantum yield if $^{\cdot}OH$ radical generation, F is the fraction of UV light absorbed by H_2O_2, G_0 is the total incident UV is the photon flux and V is the total irradiated volume. In the steady-state approximation, $d[^{\cdot}OH]/dt = 0$, and the final kinetic expression extrapolated to time $t = 0$ can be written as:

$$-\frac{d[HA]}{dt}\bigg|_{t=0} = \frac{k_{HA}[HA]_0\phi_{OH}FG_0/V}{k_{HA}[HA]_0 + k_{H_2O_2}[H_2O_2]_0} \tag{4}$$

where $\{-d[HA]/dt\}\,|_{t=0}$ denotes the initial rate of humic acid degradation. The concentration of H_2O_2 is much larger than that of humic acid in this study; therefore equation (4) can be simplified to $\{-d[HA]/dt\}\,|_{t=0} = K[HA]_0$ when the H_2O_2 concentration and the light intensity are constant.

From Fig. 6 the initial humic acid concentration did not affect the apparent first order rate constant, therefore the rate of humic acid destruction appears to be truly first order in the range of 3–8 mg/l NPOC. However, at higher humic acids concentration, it should be noted that the scavenging effect of humic acids may influence the initial rate constant of itself when the concentration of H_2O_2 is low (Liao and Gurol, 1995), as shown in Fig. 6 (top).

Effect of bicarbonate/carbonate concentration

The effect of HCO_3^-/CO_3^{-2} on the degradation of humic acid in the UV/ H_2O_2 process was examined with the initial carbonate concentration of 100–400 mg/l as $CaCO_3$ (Fig. 7). A 22 and 70% reduction of the initial rate constants for humic acid removal (0.029 and 0.011 min^{-1}, respectively) were

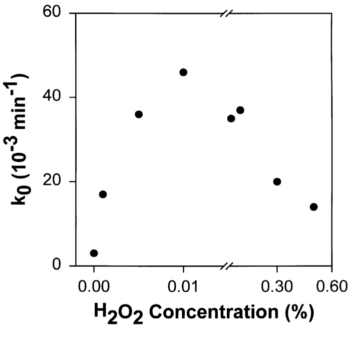

Fig. 5. Rate constant for humic acid degradation with various H_2O_2 concentrations. $[\text{Humic acid}]_0 = 5$ mg/l; UV source: UV/quartz.

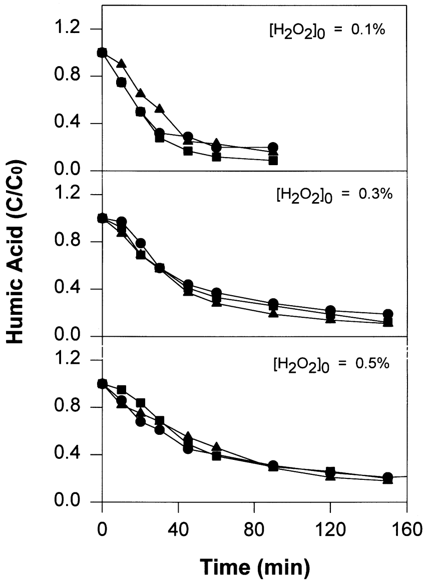

Fig. 6. Influence of initial humic acid concentration in UV/H_2O_2 systems. (●) $[\text{humic acid}]_0 = 3$ mg/l; (■) $[\text{humic acid}]_0 = 5$ mg/l; (▲) $[\text{humic acid}]_0 = 8$ mg/l; UV source: UV/quartz.

observed with initial bicarbonate and carbonate concentrations of 96 and 124 mg/l.

In reality, there may be a significant amount of HCO_3^-/CO_3^{-2} in natural water and it may compete with organic matter and peroxide for reaction with OH radicals. Equations (5) and (6) indicate that an increase of bicarbonate/carbonate concentration will lower the OH radical concentration, so the destruction rate of target organics will decrease with an increase in bicarbonate/carbonate concentration. The effect of bicarbonate/carbonate on the oxidation rate will be significant, particularly when the concentration of the target organic is low (Ligrini *et al.*, 1993; Buxton *et al.*, 1988; Staehelin and Hoigne, 1985):

$$HCO_3^- + {}^{\cdot}OH \rightarrow CO_3^{-\cdot} + H_2O \qquad k = 1.5 \times 10^7\ (M^{-1}\ s^{-1}) \tag{5}$$

$$CO_3^{2-} + {}^{\cdot}OH \rightarrow CO_3^{-\cdot} + OH^- \qquad k = 4.2 \times 10^8\ (M^{-1}\ s^{-1}) \tag{6}$$

Beltrán *et al.* (1996) reported that the presence of bicarbonate had no effect on the direct photolysis of deethylatrazine and deisopropylatrazine, but it caused a significant inhibition of oxidation in the UV/H_2O_2 system. The hydroxyl radical scavenging character of bicarbonate and carbonate (equations (5) and (6)) can explain these inhibition effects. Although bicarbonate and carbonate do not adsorb UV light, they react readily with hydroxyl radicals (Buxton *et al.*, 1988; Staehelin and Hoigne, 1985) which are the primary oxidizing species in the UV/H_2O_2 process. Thus OH radical scavenging is suggested to account for the observed inhibition effect of bicarbonate and carbonate. Although the generated carbonate radical anion has been shown to be an oxidant itself, its oxidation potential is less positive than that of the OH radical (Ligrini *et al.*, 1993).

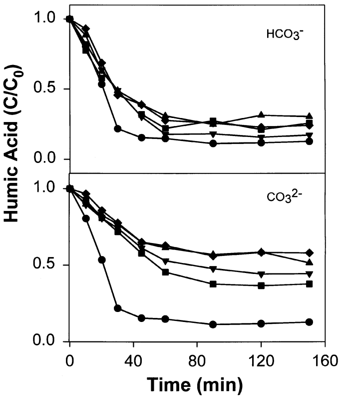

Fig. 7. Degradation of humic acid in UV/H_2O_2 systems with various HCO_3^- and CO_3^{-2} concentrations. [humic acid]$_0$ = 5 mg/l; [H_2O_2]$_0$ = 0.005%; UV source: UV/quartz; [HCO_3^-/CO_3^{-2}]: (●) = 0, 0 mg/l; (▼) 96, 124 mg/l; (■) 190, 186 mg/l; (◆) 242, 331 mg/l; (▲) 345, 412 mg/l.

Glaze et al (1995) and Liao and Gurol (1995) studied the effect of bicarbonate (0.1–2 mM) on benzene (initial concentration 0.64 mM) decomposition at a peroxide concentration of 3 mM. It was found that bicarbonate affects benzene decomposition insignificantly in the range of 0.5–2 mM. The lack of an effect of bicarbonate is due to the fact that the concentration of benzene is relatively high and the reaction rate constant of benzene with an OH radical is several hundred times greater than that of bicarbonate. The effect of bicarbonate/carbonate can be calculated approximately using the rate constant values of OH radicals with humic acid, peroxide, and bicarbonate/carbonate ion.

CONCLUSIONS

The rate of humic acid oxidation is greatly increased in the combined UV/H_2O_2 system but the presence of bicarbonate/carbonate species has a negative effect due to the scavenging of hydroxyl radicals, especially when its concentration is high. At the experimental conditions evaluated in this study, the humic-acid decay follows first-order kinetics with a rate constant of 0.037 min^{-1} when the UV/quartz was used as the light source, while that of hydrogen peroxide obeys zero-order kinetics at 0.30 mM min^{-1}. Humic acid decomposed at a much slower rate with UV/Pyrex (0.0025 min^{-1}) and sunlight (0.0007 min^{-1}), which have minor UVC emission and comparable total UV light intensity compared to UV/quartz. Hydrogen peroxide acts as both an initiating and scavenging agent of hydroxyl radicals. The former effect predominates when the initial hydrogen peroxide concentration is lower than 0.01% (2.94 mM), the latter at higher concentrations. However, even at high H_2O_2 concentrations, oxidation of humic acids is due to hydroxyl radical attack because H_2O_2 absorbs most of the light.

Acknowledgements—This research was supported by National Science Council, Taiwan, Republic of China, under Grant No. NSC 86-2211-E002-015. The authors thanks the reviewers for valuable comments on the preparation of this paper.

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