



In-situ production of ozone and ultraviolet light using a barrier discharge reactor for wastewater treatment

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Abstract: A dielectric barrier discharge (DBD) reactor consisting of water-filled dielectric tube electrodes was used for the treatment of wastewater. The inner dielectric tube, which acted as the discharging electrode, was filled with an aqueous electrolyte solution. The outer dielectric tube, which served as the other electrode, was in contact with the wastewater, which was grounded. The present reactor system was energy-efficient for the production of ozone, not only because the perfect contact between the aqueous electrode and the dielectric surface minimized the loss of the electrical energy, but also because the DBD reactor was cooled by the wastewater. In addition, the ultraviolet (UV) light produced in the DBD reactor was able to assist in the wastewater treatment since the quartz tube used as the dielectric material was UV-transparent. The performance of the present DBD system was evaluated using a synthetic wastewater formed from distilled water and an azo dye, amaranth. The experimental parameters were the concentration of the electrolyte in the aqueous electrode, the discharge power, the initial pH of the wastewater and the concentration of hydrogen peroxide added to the wastewater. The wastewater treatment system was found to be effective for achieving decomposition of the dye.

Key words: Dielectric barrier discharge (DBD), Aqueous electrode, Ozone, Ultraviolet (UV), Wastewater

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INTRODUCTION

There are many physical, chemical and physicochemical oxidation processes available for the treatment of wastewater, including Fenton's reaction (Gulkaya *et al.*, 2006), ultrasonification (Jiang *et al.*, 2002), photocatalysis (Daneshvar *et al.*, 2004), ozonation (Zhang *et al.*, 2004), ultraviolet (UV) irradiation (Behnajady *et al.*, 2004) and hydrogen peroxide methods (Deutsch, 1998). These processes can form highly oxidative radicals in wastewater, thereby leading to the decomposition of water contaminants. Furthermore, it has shown that two or more of these processes can be combined to enhance the rate of decomposition. For example, the combinations UV/ozonation, photocatalysis/ozonation and photocatalysis/ozonation/H₂O₂ show better waste-

water treatment performance than each of these processes alone (Getoff, 2001; Muruganandham and Swaminathan, 2004; Neamtu *et al.*, 2004; Mok and Jo, 2006). The main disadvantage of such combinations can be their installation and operating costs.

Recently, several electrical discharge methods such as corona discharge above a water surface, pulsed corona discharge in water, dielectric barrier discharge (DBD) with wetted wall, and gas-liquid gliding arc discharge have been applied to the decomposition of water contaminants (Lukes *et al.*, 2004; Dors *et al.*, 2006; Burlica *et al.*, 2006). When an electrical discharge occurs above a surface of water, chemically active species such as OH, O, O₃ and H₂O₂ produced in the gas phase can react with water contaminants at the gas-liquid interface. In this case, the area where chemical reactions occur is very limited, which lowers the wastewater treatment performance. An electrical discharge generated directly

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in water by submerging both high-voltage and ground electrodes may be more effective in terms of wastewater treatment performance, because chemically active species are formed in the water phase where the contaminants exist. Other physical effects such as UV light and overpressure shockwave can also be used for the decomposition of the contaminants (Lukes *et al.*, 2004). However, this kind of electrical discharge generated in water may not be suitable when the conductivity of wastewater is high.

In this work, we investigated a wastewater treatment method using both ozone and UV light produced by the DBD. The DBD reactor consisted of two coaxial quartz tubes with different diameters. The inner tube, which acted as the discharging electrode, was filled with an aqueous electrolyte solution, and was connected to alternating current (AC) high voltage. The DBD reactor was submerged in wastewater that was grounded. In this case, the wastewater acted not only as an electrode but also as the cooling medium of the DBD reactor. The main merit of this DBD system is in-situ production of reactive species like ozone and UV light available for the decomposition of water contaminants (Mok *et al.*, 2007). The performance of this DBD system was evaluated using a synthetic wastewater. The model water contaminant was an azo dye, amaranth. In this system, the contaminant can be decomposed by two pathways, i.e., DBD-induced ozonation and photolysis. The quartz tube used as the dielectric material is transparent to UV, and accordingly the wastewater can absorb the photons produced in the DBD reactor. The ozone-containing gas formed in the DBD reactor was transferred to the wastewater using a porous gas diffuser, greatly increasing the gas-liquid interfacial area. The electrolyte in the aqueous solution filling the inner quartz tube was sodium chloride. The effects of the electrolyte concentration on the discharge power and ozone generation were examined. The experimental parameters affecting the wastewater treatment performance were the voltage applied to the discharging electrode (or the discharge power), the initial pH of the wastewater, and the concentration of hydrogen peroxide added to the wastewater.

EXPERIMENTS

Fig.1 shows a schematic diagram of the ex-

perimental apparatus used for the treatment of wastewater. The DBD reactor was made from two quartz tubes with different diameters, which were arranged coaxially. A cross-sectional view of the DBD reactor is shown in Fig.2. The inner quartz tube (outer diameter: 13 mm, thickness: 0.7 mm) was filled with aqueous electrolyte (sodium chloride) solution to which AC high voltage was connected. The DBD reactor was submerged in the grounded wastewater, and the surface of the outer quartz tube (outer diameter: 23.7 mm, thickness: 1.4 mm) was in perfect contact with the wastewater. In this case, the wastewater functioned as the ground electrode while the aqueous electrolyte solution in the inner quartz tube acted as the discharging electrode. The concentration of sodium chloride used as the electrolyte was varied up to 50 g/L. In one experiment, a 9.5-mm copper rod instead of the aqueous electrolyte solution was used as the discharging electrode to compare the discharge characteristics.

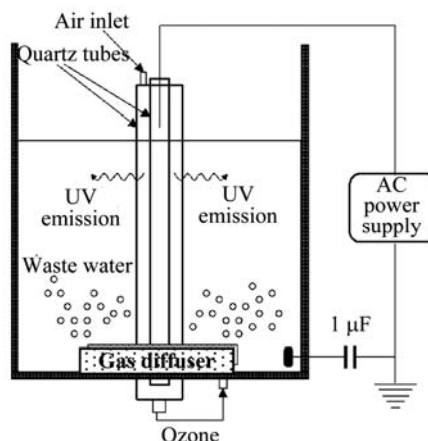


Fig.1 Schematic of the experimental apparatus

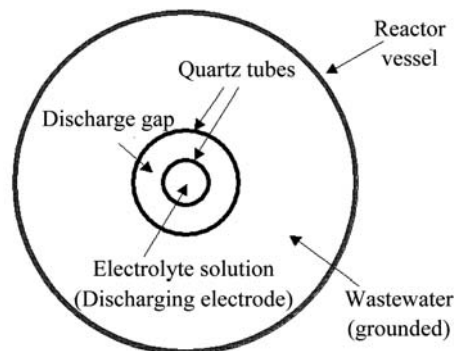


Fig.2 Cross-sectional view of the DBD reactor

Dry air of 6 L/min flowed through the space between the inner and outer quartz tubes, where the electrical discharge occurred when high voltage was applied. The electrical discharge produced various reactive species (mainly ozone) and UV light. The vessel containing the wastewater was 90 mm in diameter. The model water contaminant was amaranth ($C_{20}H_{11}N_2Na_3O_{10}S_3$, Sigma-Aldrich Co.), dissolved in distilled water. The molecular structure of amaranth is given in Fig.3. The volume of the synthetic wastewater prepared as above was 500 ml, and the concentration of the water contaminant was 25 mg/L. The ozone-containing gas from the DBD reactor was well dispersed in the wastewater using a porous gas diffuser. The depth of the synthetic wastewater in the vessel was 100 mm, but it increased slightly to 105 mm due to the gas bubbles produced when the ozone-containing gas was transferred to the synthetic wastewater. As the wastewater itself functioned as the ground electrode, the length over which the electrical discharge occurred was 105 mm. All experiments were carried out at room temperature (20 °C).

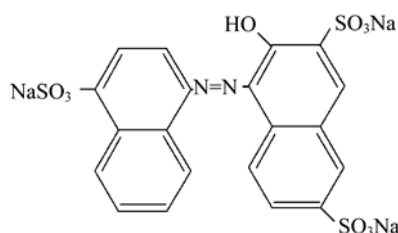


Fig. 3 Chemical structure of amaranth dye

The discharge power was changed by varying the voltage applied to the discharging electrode, which was estimated using the so-called Lissajous figure (voltage-charge plot) (Rosocha, 2005). A 1 μ F capacitor (Fig.1) was used to measure the charge stored in the DBD reactor. The voltage was measured using a high voltage probe with 1000:1 attenuation ratio (PVM-4, North Star Research, Co.) and a digital oscilloscope (TDS 3032, Tektronix). The initial pH of the synthetic wastewater was varied from 3.2 to 10.3 by adding 0.05 mol/L sulfuric acid solution or 0.1 mol/L sodium hydroxide solution dropwise until the desired pH was achieved. To take advantage of the UV emission from the DBD reactor for the decomposition of the water contaminant, hydrogen peroxide was added to the wastewater. The concentration of the

hydrogen peroxide was varied up to 23 mmol/L. During electrical discharge, the dry air fed to a DBD reactor can form nitrogen oxides that can affect the pH of the wastewater (Mok *et al.*, 2000). The concentrations of nitrogen oxides such as NO and NO₂ were analyzed using a chemiluminescent NO-NO₂-NO_x analyzer (Model 42C, Thermo Environmental Instruments, Inc.). Samples of the wastewater were taken at appropriate time intervals, and immediately analyzed using a UV/visible spectrophotometer (Model UV-2500, Labomed, Inc.). The decomposition efficiency of the water contaminant was determined by measuring the absorbance at 521 nm.

RESULTS AND DISCUSSION

Effect of discharging electrode material on the generation of ozone

The rate of ozone generation with the aqueous electrolyte solution or the copper rod as the discharging electrode was compared as a function of the discharge power (Fig.4). In both cases, the ground electrode was the wastewater. As observed, the rate of ozone generation with the aqueous electrolyte solution was higher than that with the copper rod. The contact between the aqueous electrolyte solution and the dielectric surface is almost perfect, thereby minimizing the loss of electrical energy, i.e., all of the energy is delivered to the gas flowing through the gap

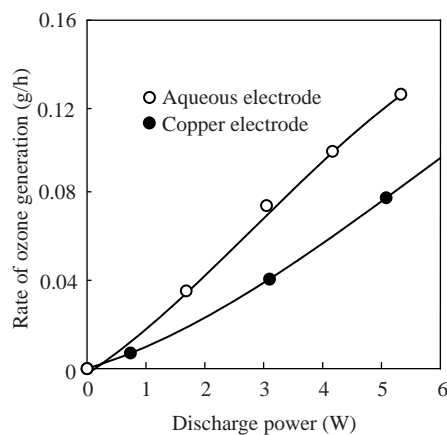


Fig. 4 Comparison of the rate of ozone generation between the aqueous electrolyte solution and the copper rod as the discharging electrode

between the inner and outer quartz tubes. The difference in the discharge mode may also explain the results in Fig.4. The barrier discharge occurred in a form of glow-like discharge when the aqueous electrolyte solution was used (Fig.5a). However, typical filamentary barrier discharge was observed with the copper electrode (Fig.5b). It is believed that uniformly distributing the energy over the discharge region is more favorable than transferring it to the scattered discharging filaments with respect to ozone generation. Yoshioka and Shoyama (2006) showed that the energy yield for the removal of NO was higher with a glow-like discharge mode than that with a filamentary discharge, which agrees with this study.

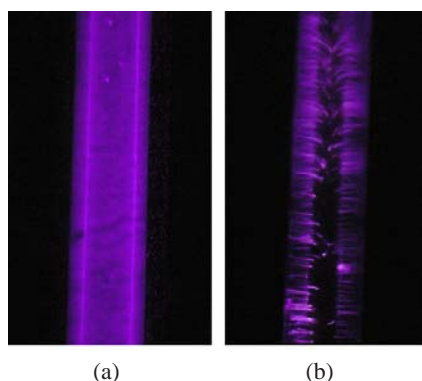


Fig.5 Photographs of the barrier discharge with the aqueous electrolyte solution electrode (a) and with the copper electrode (b)

Fig.6 shows the effect of the concentration of sodium chloride in the aqueous solution filling the inner quartz tube on the rate of ozone generation and the discharge power when the voltage was 29.5 kV. It is expected that the increase in the concentration of the electrolyte should decrease the electrical resistance of the aqueous solution. However, the change in the concentration of electrolyte in this experimental range did not affect the rate of ozone generation. This result may be attributed to the high electrical resistance of the discharge gap. Although the gas flowing through the DBD reactor is slightly conductive when high voltage is applied, the resistance of the discharge gap is much higher than that of the aqueous electrolyte solution, i.e., the overall resistance is dominated by that of the discharge gap. Thus, the change in the concentration of the electrolyte in the aqueous solution cannot significantly affect the rate of ozone generation.

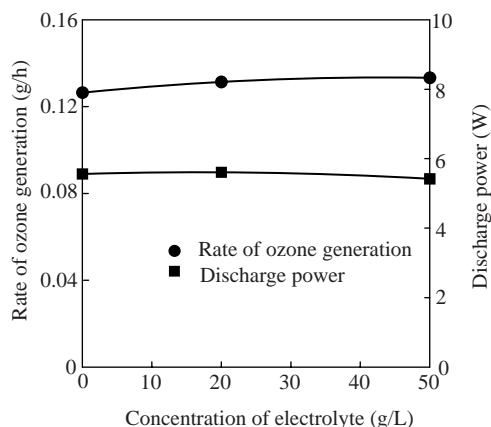


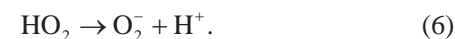
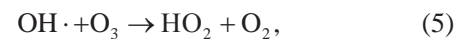
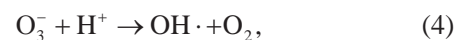
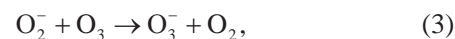
Fig.6 Effect of the concentration of sodium chloride in the aqueous electrolyte solution on the rate of ozone generation and the discharge power

Effect of discharge power on the decomposition of the contaminant

The rate of ozone generation mainly depends on the discharge power, which eventually affects the decomposition of the water contaminant (Mok and Jo, 2006). The relation between the decomposition efficiency and the discharge power is shown in Fig.7. The decomposition efficiency (DE) is defined as

$$DE = \frac{A_0 - A}{A_0} \times 100\%, \quad (1)$$

where A is the absorbance at 521 nm and the subscript 0 indicates the initial condition. In this experiment, the discharge power was changed from 1.7~5.3 W by varying the voltage applied to the discharging electrode (aqueous electrolyte solution). As shown in Fig.4, the rate of ozone generation was 0.035~0.126 g/h at 1.7~5.3 W. The ozone transferred to the wastewater can be decomposed to produce hydroxyl radicals that are a strong oxidative species. The reactions related to the decomposition of ozone dissolved in water may be summarized as follows (Staehelin *et al.*, 1984; Khraisheh, 2003):



The hydroxyl radical ($\text{OH}\cdot$) and perhydroxyl radicals (HO_2) formed by reactions Eqs.(2)~(6) can oxidize the water contaminant to inorganic compounds and smaller molecules. Also, the ozone fed to the wastewater can react directly with the water contaminant at the gas-liquid interface. The increase in the discharge power increased the decomposition efficiency (Fig.7). When the discharge power was 1.7 W, the decomposition efficiency was only 30% at 60 min, but when the discharge power was increased to 5.3 W, almost 100% decomposition efficiency was obtained in 30 min.

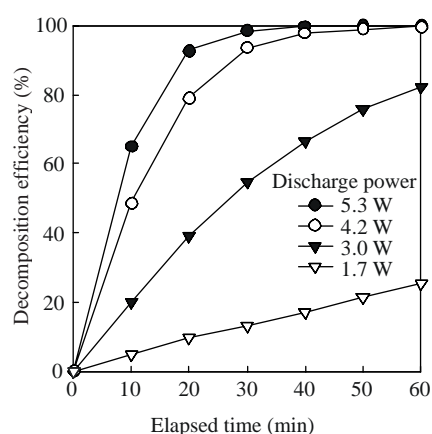


Fig.7 Effect of the discharge power on the decomposition of the water contaminant by ozone (initial pH of the wastewater: 3.2)

Effect of pH on the decomposition of the contaminant

As shown in reactions Eqs.(2)~(6), the pH value of the wastewater affects the formation of hydroxyl radicals resulting from the decomposition of ozone. Fig.8 shows the effect of the initial pH on the decomposition of the water contaminant. The initial pH was varied from 3.2~10.3 using sulfuric acid or sodium hydroxide. The concentration of ozone transferred to the wastewater was fixed at 0.126 g/h. According to (Stahelin *et al.*, 1984; Khraisheh, 2003), hydroxyl anions (OH^-) initiate the decomposition of ozone in water, and thus the higher the concentration of hydroxyl anions, the faster the rate of decomposition of organic compounds. The decomposition efficiency defined by Eq.(1) did not greatly depend on the initial pH in the range of 3.2~6.9 (Fig.8). However, the decomposition efficiency abruptly increased when the initial pH of the wastewater was increased to basic

levels, i.e., 8.2 or 10.3. This result is obviously caused by the increase in the concentration of hydroxyl anions that initiate the decomposition of ozone.

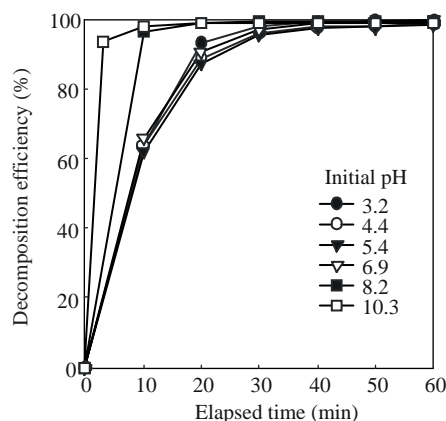
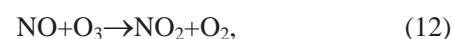


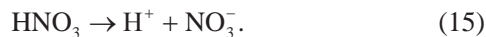
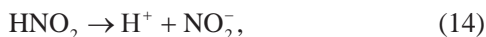
Fig.8 Effect of the initial pH on the decomposition of the water contaminant by ozone (discharge power: 5.3 W)

Fig.9 presents the variation in the pH value with elapsed time, which was measured under the same experimental conditions as in Fig.8. As observed, the pH value decreased to some extent for the first 10 min of operation and then stabilized. The decrease in the pH value may be explained by the formation of nitrogen oxides in the DBD reactor. When dry air is used as the feed gas for the generation of ozone, the following reactions occur to produce nitrogen oxides (Sathiamoorthy *et al.*, 1999; Mok *et al.*, 2000):



where e denotes high-energy electrons produced by the electrical discharge. In Fig.10, the concentrations of nitrogen oxides such as NO and NO_2 measured at the outlet of the DBD reactor are given as a function of the discharge power. Since reactions Eqs.(11) and (12) are fast, NO formed by reactions Eqs.(8)~(10) are easily oxidized to NO_2 . As shown, the concentration of NO was negligible, and the concentration of NO_2 increased with the discharge power. When the gas from the DBD reactor is transferred to the wastewater, NO_2 coexisting with ozone is dissolved

to form nitric acid and nitrous acid:



Reactions Eqs.(13)~(15) decrease the pH value of the wastewater.

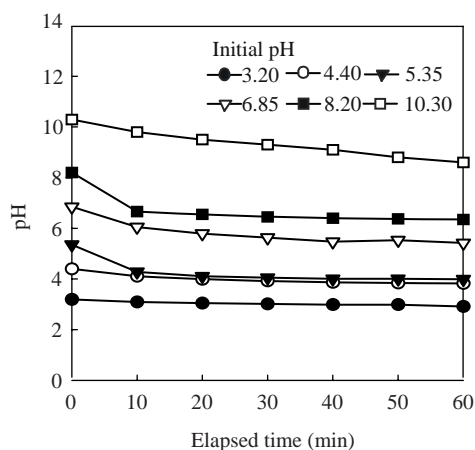


Fig.9 Variations of the pH values with the elapsed time (discharge power: 5.3 W)

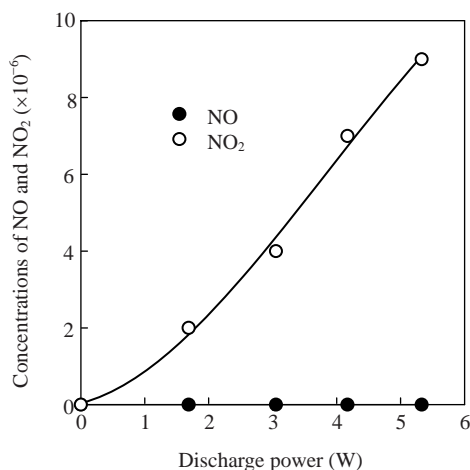


Fig.10 Concentrations of NO and NO₂ formed in the DBD reactor

Decomposition of the contaminant in the presence of hydrogen peroxide

The decomposition efficiencies obtained with (1) UV alone, (2) UV+H₂O₂, (3) O₃ alone, and (4) UV+H₂O₂+O₃ were compared (Fig.11). In the cases of UV alone and UV+H₂O₂, the ozone-containing gas from the DBD reactor was not fed to the wastewater.

Instead, dry air of 6 L/min was fed directly to the wastewater through the porous gas diffuser. A negligible amount of the water contaminant was decomposed with UV alone (Fig.11). The effect of the UV emission on the decomposition of the water contaminant can be enhanced in the presence of hydrogen peroxide, because UV can form hydroxyl radicals by cleaving the bond HO-OH as follows (Behnajady *et al.*, 2004; Muruganandham and Swaminathan, 2004):



As expected, the UV light in the presence of hydrogen peroxide (concentration: 1 mmol/L) was able to degrade the water contaminant, which obviously resulted from reaction Eq.(16). Furthermore, the effect of the UV emission can be maximized by introducing a photocatalyst that can promote the formation of hydroxyl radicals (Mok *et al.*, 2007). In the case of O₃ alone, the rate of the decomposition was very fast, compared to the cases of UV alone and UV+H₂O₂, indicating that ozone fed to the wastewater controlled the overall decomposition rate. When hydrogen peroxide of 1 mmol/L was used together with ozone (case (4)), further enhancement of decomposition was observed.

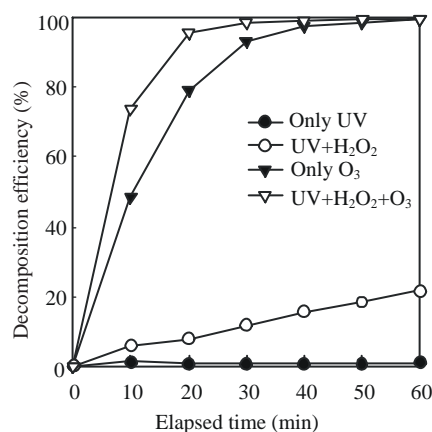
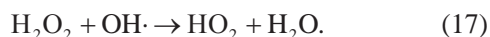


Fig.11 Comparison of the decomposition efficiencies obtained with (1) UV alone, (2) UV+H₂O₂, (3) O₃ alone, and (4) UV+H₂O₂+O₃ (discharge power: 4.2 W, initial pH of the wastewater: 3.2)

As described above, the addition of hydrogen peroxide can enhance the decomposition of the water contaminant by forming hydroxyl radicals. However, it should be noted that excess hydrogen peroxide may

act as a scavenger of hydroxyl radicals, i.e., there exists an optimal hydrogen peroxide concentration in terms of the decomposition efficiency. For example, the reaction of hydrogen peroxide with hydroxyl radicals forms perhydroxyl radicals as follows (Zhang *et al.*, 2003; Pekakis *et al.*, 2006):



Perhydroxyl radicals formed in reaction Eq.(17) are much less reactive than hydroxyl radicals. The oxidation potentials of hydroxyl radicals and perhydroxyl radicals are 2.8 V and 1.7 V, respectively (Sun *et al.*, 1997; Lawton and Robertson, 1999). Thus, the conversion of hydroxyl radicals into perhydroxyl radicals by reaction Eq.(17) decreases the rate of the decomposition. The optimal concentration of hydrogen peroxide may depend on several parameters, including the UV intensity, the concentration of the water contaminant, the chemical structure of the water contaminant, etc. Fig.12 shows the effect of the concentration of hydrogen peroxide on decomposition efficiency. For this experiment, the discharge power was fixed at 4.2 W, and at this discharge power the rate of ozone generation was 0.0983 g/h. As shown, there was an optimal concentration of hydrogen peroxide for the decomposition of the water contaminant. The increase in the concentration of hydrogen peroxide up to 1 mmol/L enhanced decomposition efficiency, but further increase brought about a contrary effect.

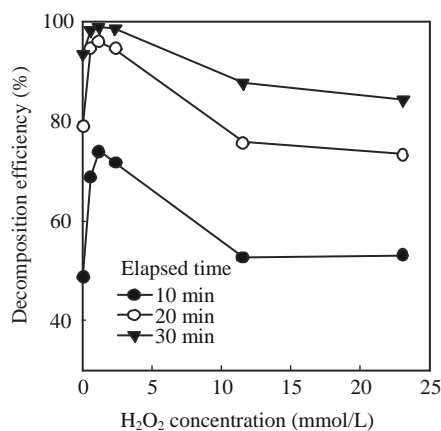


Fig.12 Effect of the concentration of hydrogen peroxide added to the wastewater on the decomposition of the water contaminant by UV plus O₃ (discharge power: 4.2 W, initial pH of the wastewater: 3.2)

CONCLUSION

The present study investigated the in-situ production of ozone and UV light using a DBD reactor consisting of water-filled dielectric tube electrodes. The DBD reactor was energy-efficient for the production of ozone, not only because the loss of the electrical energy was minimized by the perfect contact between the aqueous electrode and the dielectric surface, but also because the wastewater used as the ground electrode acted as the cooling medium of the DBD reactor. The present wastewater treatment system is capable of decomposing the contaminant in two pathways, i.e., the DBD-induced ozonation and UV emission, and was found to be a very effective method. The decomposition efficiency of the contaminant amaranth was higher in a basic than that in an acidic pH range. The pH value of the wastewater tended to decrease with the operation time due to the formation of nitrogen oxides in the DBD reactor. The addition of hydrogen peroxide to the wastewater enhanced the decomposition of the water contaminant, but excess hydrogen peroxide resulted in an adverse effect on decomposition.

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