



Photocatalyzed destruction of organic dyes using microwave/UV/O₃/H₂O₂/TiO₂ oxidation system

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ABSTRACT

Photocatalytic decomposition characteristics of three different single-component organic dyes and their mixture using microwave/UV/O₃/H₂O₂/TiO₂ photocatalysts hybrid process system were investigated in this study. The decomposition rates of all organic dyes were higher in single-component experiments than in mixed solution experiments. The decomposition rates of all organic dyes increased with the ozone injection rate. The decomposition rate increased with increasing hydrogen peroxide concentration until it reached a maximum point beyond which it decreased with increasing hydrogen peroxide concentration. The result demonstrates that combination of elemental techniques that have little effect when used alone can make a synergy effect.

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1. Introduction

The treatment of wastewater containing dyes is difficult. Generally, adsorption using activated carbon and biological treatment using microorganisms are used to remove organic pollutants such as dyes contained in waste water. However, these methods do not easily remove the complex aromatic compounds with various substitutions contained in dye wastewater and causes generation of large amount of sludge and solid waste leading to high treatment cost. Oxidation has been widely used to convert toxic non-biodegradable materials into biodegradable forms. Conventional oxidation processes using ozone or hydrogen peroxide (H₂O₂), however, has limits in treating a number of different kinds of pollutants, calling for a more efficient oxidation process. Traditional methods (for example adsorption on activated carbons [1]) only transfer contaminations from one phase to another. The most promising way for removing dyes is photo-catalysis, because this process decomposes the end dyes to water and carbon dioxide [2]. Application of TiO₂ photocatalyst in water treatment has recently been investigated widely [3,4]. There are still many problems yet to be solved, however, in application of TiO₂ photocatalyst in the treatment of non-biodegradable materials. First, photocatalysis has

usually been used in air pollutants treatment because it is suitable for treatment of low-concentration pollutants. Concentrations of water pollutants, however, are much higher than those of air pollutants. Thus, their treatment by photocatalysis is difficult compared to that of air pollutants. Second, polluted water often contains mixture of hydrophilic and hydrophobic materials. Therefore, it is not easy for the pollutants to be adsorbed on the photocatalyst surface. Third, polluted water has high turbidity, hence low transparency, hindering activation of photocatalysts by ultraviolet (UV) rays. Fourth, some materials are not easily degraded by photocatalysis only. Fifth, the amount of oxygen available for photocatalytic oxidation is very low in water compared to in air. Due to these reasons, photocatalytic oxidation of water pollutants has not received large attention thus far. Recently, researches have been conducted actively to improve oxidative degradation performance by adding microwave irradiation as an effort to utilize TiO₂ photocatalyst in water treatment more efficiently [5–7].

In many photo-decomposition reaction systems, TiO₂ powders are often used as a photocatalyst [8]. However, powder photocatalysts have several problems, such as (1) difficulties in the separation of the catalyst from suspension after the reaction, (2) difficulties in the prevention of aggregation in high concentration suspensions. To avoid such agglomeration, suspension must be diluted. Then the overall reaction rate tends to be slow. On the one hand, these problems can be solved by the use of immobilized (i.e., coated) catalyst particles. However, the coated catalysts are easily detached

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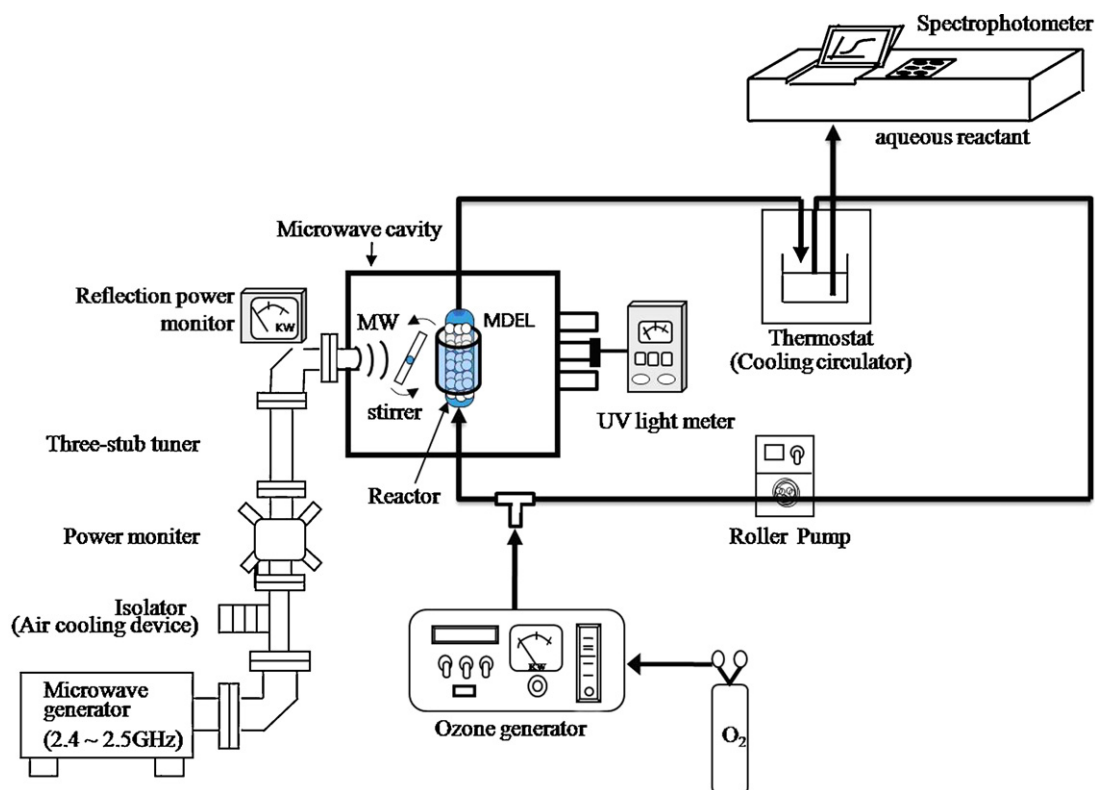


Fig. 1. Schematics of the microwave/UV/ozone/ H_2O_2 / TiO_2 photo-catalyst hybrid process system.

from the supports. To avoid these problems, TiO_2 thin films have been prepared by the sol-gel method [9], the sputter method [10] and the chemical vapor deposition (CVD) method [11,12]. Among these, CVD is considered as a promising method to prepare high-quality thin films over large surface area with a well-controlled composition and low defect density.

Microwave energy has been used more and more on synthetic organic chemistry because of its great ability to accelerate reactions and to improve yields and selectivity [13]. Some researchers have been conducted to investigate photocatalytic reactions assisted with microwave irradiation. Kataoka and co-workers found that the photocatalytic oxidation of ethylene proceeded faster (83.9%) in the presence of the microwave irradiation than in the absence [7]. Horiyoshi et al. proved by electron spin resonance (ESR) that about 20% more $\cdot\text{OH}$ radicals were generated by photocatalysis with microwave irradiation than photocatalysis alone [14]. Although microwave effectively accelerates photocatalytic degradation, traditional Hg lamp was not laid in microwave under microwave irradiation. The measures to avoid electrode spoilage would complicate equipment. The problem will be solved if microwave electrodeless lamp substitutes traditional lamp as light source. Moreover, microwave electrodeless lamp has some unique advantages, such as, good photochemical efficiency, long life, low cost, and simple photocatalytic equipment [15]. Horiyoshi et al. proved that photocatalysis with electrodeless lamp (a double quartz cylindrical plasma photoreactor) was about 10 times more efficient than the photocatalysis using traditional lamp [16].

In this study, a microwave/UV/ O_3 / H_2O_2 / TiO_2 photocatalyst hybrid process system, in which various techniques that have been used for water treatment are combined, is evaluated to develop an advanced technology to treat non-biodegradable water pollutants efficiently. The objective of this study is to develop a novel advanced oxidation process that overcomes the limitations of existing single-process water treatment methods by adding microwave irradiation

to maximize the formation of active intermediate products, e.g., OH radicals, with the aid of UV irradiation by MDEL, photocatalysts, and auxiliary oxidants. In particular, photocatalytic decomposition characteristics of three different single-component organic dyes and their mixture were investigated in this study.

2. Experimental

2.1. Microwave/UV- TiO_2 system

Fig. 1 shows the schematic of the Microwave/UV- TiO_2 experimental apparatus used in this study. Microwave radiation was carried out with a Microwave system manufactured by Korea microwave instrument Co. Ltd. It consisted of a microwave generator (frequency, 2.45 GHz; maximal power, 1 kW), a three-stub tuner, a power monitor, and a reaction cavity. Microwave radiation (actual power used, 200–600 W) used to irradiate the organic dye aqueous solution containing TiO_2 photocatalyst balls was delivered through a wave-guide. Microwave irradiation was continuous and the microwave intensity was adjusted by connection to a power monitor. Optimal low reflection of the microwave radiation was achieved using the three-stub tuner. The UV sensor and the microwave generator were located on the right side and left side of the microwave cavity, respectively. Both devices were set at 180° to each other as illustrated in Fig. 1. A stirrer was installed on the back side in the reaction cavity to enhance the transfer of microwave. As the microwave-irradiated reactant solution is heated steadily, it was not possible to carry out experiments at constant temperature without a cooling system. In this study, the reactant solution was stored in a stainless steel beaker installed in a constant-temperature equipment. A roller pump was used to circulate the heated reactant solution through a cooling system to keep the reaction temperature constant at 298 K. In this study, ozone was added as an auxiliary oxidant to increase the efficiencies of

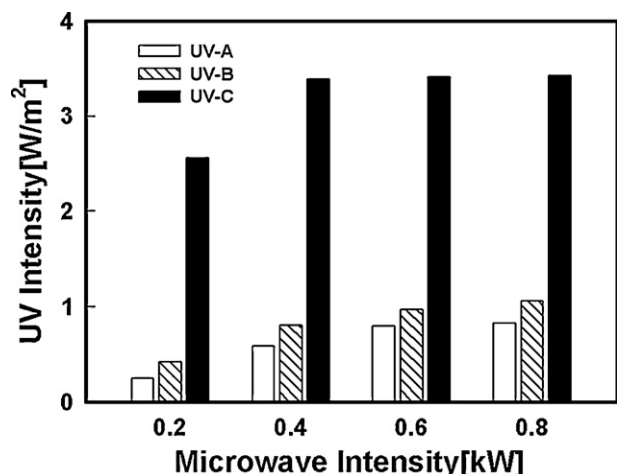


Fig. 2. Compares the UV intensities radiated at different microwave intensities.

the decomposition reactions of organic dye compounds. Ozone was produced by feeding oxygen gas with the flow rate of 500 cc/min to an ozone generator (Lab-1, Ozonotech Co. Ltd.) as is shown in Fig. 1. The ozone production rate was adjusted between 0.75 and 3.26 g/h by controlling the power consumption.

2.2. Microwave discharge electrodeless lamp

TiO₂ photo-catalysts are excited by UV light, producing strong oxidants that can degrade organic compounds. Therefore, provision of UV is essential for a use of TiO₂ photocatalysts. Typical UV lamps, however, have metal electrodes, which prevents them from being used in the microwave-irradiation equipment. Therefore, a double-tube type microwave discharge electrodeless lamp (170 mm length, 36 mm inner diameter, 55 mm outer diameter,

hereafter MDEL) that emits UV upon the irradiation of microwave was developed in this study. It was made of quartz to maximize the reaction efficiency. Small amount of mercury was doped between the tubes inside the double-tube UV lamp that was kept vacuumed. The lamp used in this study is UV-C type lamp although a little amount of UV-A and UV-B wavelength lights are emitted. Fig. 2 compares the UV intensities radiated at different microwave intensities. The sensor of the UV radiometer (HD2102-2, Delta OHM) was installed on the right-hand-side port of the microwave cavity. The distance between MDEL and the sensor was about 250 mm. The ranges of wavelength detected by UV-A, UV-B, and UV-C sensors are 315–400 nm, 280–315 nm, and 220–280 nm, respectively. At all microwave intensities tested in this study, UV-C exhibited much larger intensity than UV-A and UV-B. The UV-A and UV-B intensities increased with the microwave intensity, whereas the UV-C intensity showed little change at microwave intensity larger than 0.4 kW. Fig. 3 shows a photograph of the electrodeless UV lamp in the microwave cavity with (a) microwave off and (b) microwave on.

2.3. Preparation of TiO₂ photo-catalyst balls by chemical vapor deposition

TiO₂ photocatalyst balls used were prepared using a low pressure metal organic chemical vapor deposition (LPMOCVD) method. Titanium tetraisopropoxide (Ti(OC₃H₇)₄, TTIP) was used as the precursor to generate a TiO₂ film on alumina balls (Nikkto, HD-11) with 8 mm diameter. Details of the apparatus were described in our previous paper [11,12]. The CVD conditions used for the preparation of TiO₂ films are as follows; total flow rate of gas fed to the reactor 1500 sccm, oxygen concentration at the reactor inlet 50 mol%, operating pressure 1 torr, deposition temperature 773 K, and TTIP evaporator temperature 323 K. Fig. 4 shows the X-ray diffraction pattern and a SEM image of the prepared TiO₂ film. It was con-

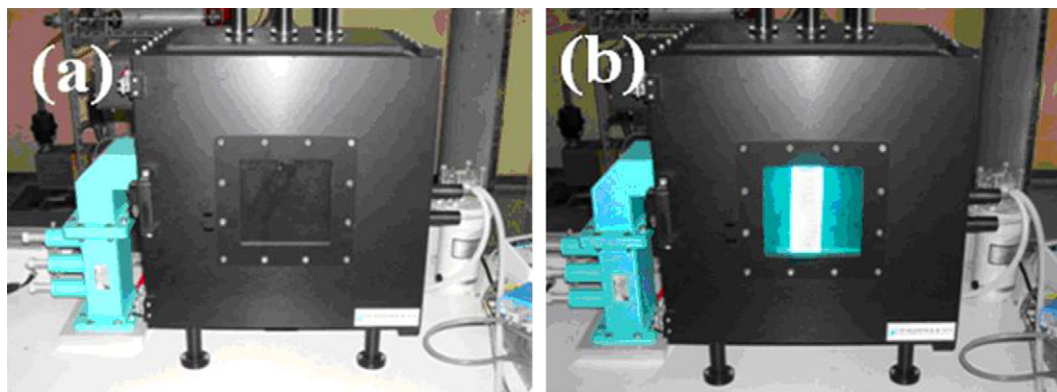


Fig. 3. The electrodeless UV lamp in the microwave oven with (a) microwave off and (b) microwave on.

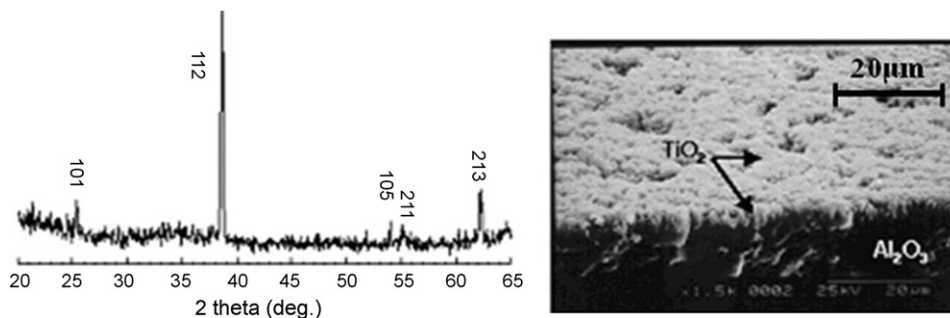


Fig. 4. X-ray diffraction pattern and SEM image of the TiO₂ film prepared by CVD method at 1 Torr and 773 K.

Table 1
Conditions of organic dyes employed during decomposition reaction.

Solution	Component	Initial concentration (mol/l)	Amount of solution (ml)	Wavelength (nm)	Absorbance
Single component	BTB	15.33	750	420	0.35
	RB	10.05	750	550	1.00
	MB	40.83	750	665	1.00
Three component mixtures	BTB	15.33	250	420	0.124 ^a
	RB	10.05	250	550	0.342 ^a
	MB	40.83	250	665	0.332 ^a

^a Absorbance was measured in three component mixtures solution.

firmed from the diffraction pattern that 1 1 2 face oriented anatase crystal structure TiO₂ film was prepared. The SEM image showed that the TiO₂ film was prepared on the substrate, the alumina balls, with a thickness of 3–5 μm .

2.4. Evaluation of photo-catalytic reaction activity

In a series of experiments carried out in this study, a hybrid photocatalysis system supplemented by microwave, ozone, and hydrogen peroxide was used to degrade three different organic dyes, bromothymol blue (referred to BTB hereafter), Rhodamine B (referred to RB hereafter), and methylene blue (referred to MB hereafter), and their mixture. The experimental conditions are summarized in Table 1. In the single-component experiments, 750 ml of water solutions of each organic dye were prepared. The concentrations of the dyes were adjusted so that the maximum absorbance became 1 for RB and MB and 0.35 for BTB, respectively. The wavelength of maximum absorbance was 420, 550, and 665 nm for BTB, RB, and MB, respectively. In addition, 750 ml of mixed organic dyes solution was prepared by mixing 250 ml of each single-component organic dye solution described above. The decomposition experiments were carried out for these four different solutions. The absorbance measured for the organic dye solutions are shown in Fig. 5. The absorbances measured for the mixed solution at maximum-absorbance wavelengths are shown to be about one third of those for single-component solutions (also see Table 1), which indicates that BTB, RB, and MB exist independently without interaction in the mixed solution. Therefore, the decomposition of each component in the mixed solution was evaluated from the changes in the absorbance measured at the maximum-absorbance wavelength of each organic dye. A spectrophotometer (UV-1601, Shimadzu) was used to measure the absorbance at various wavelengths. The measured absorbances were converted into organic dye concentrations, which were then used to calculate the decomposition rates. The reactant solutions were thermally equi-

brated at 298 K in the cooling water bath and then were circulated into the reactor using a roller pump. Microwave and UV were irradiated simultaneously by the electrodeless UV lamp installed in the vicinity of the reactor leading to photocatalytic degradation reactions. The decomposition rate was evaluated from the change of dye concentration at the reactor outlet as a function of irradiation time. The concentration of dye was measured by the absorbance using a spectrophotometer (UV-1601, Shimadzu). The degradation reaction condition was as follows. Circulation rate of reactant solutions was 300 ml/min, reactor volume 0.176 m³, amount of reactant solution 750 ml, TiO₂ photocatalyst balls volume 0.048 m³.

3. Results and discussion

3.1. Photocatalytic of degradation of organic dyes

Fig. 6 shows the changes in organic dye concentrations due to microwave-assisted TiO₂ photocatalytic decomposition by MDEL. In this figure, filled symbols represent the results of single-component experiments, whereas hollow symbols represent the results for BTB, RB, and MB in the mixed solution. Among the three organic dyes, MB was degraded the fastest both in single-component experiments and in mixed solution experiments. The decomposition rates of all organic dyes were higher in single-component experiments than in mixed solution experiments. This result can be attributed to a couple of reasons. First, the concentration of each organic dye component in the mixed solution was one third of that in the single-component counterpart. At a lower concentration, the reaction rate tends to be diffusion-limited rather than reaction-limited, which leads to a lower reaction rate. Second, the mutual inhibitory effect may have counted against the decomposition of the dye components. The existence of pollutants in a mixture is an important issue because the decomposition of one component may be inhibited by other components in the mixture.

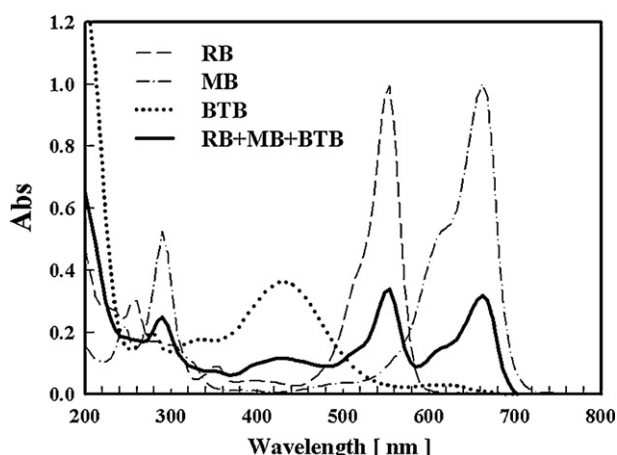


Fig. 5. UV-Vis spectral patterns of organic dye solution using in this study.

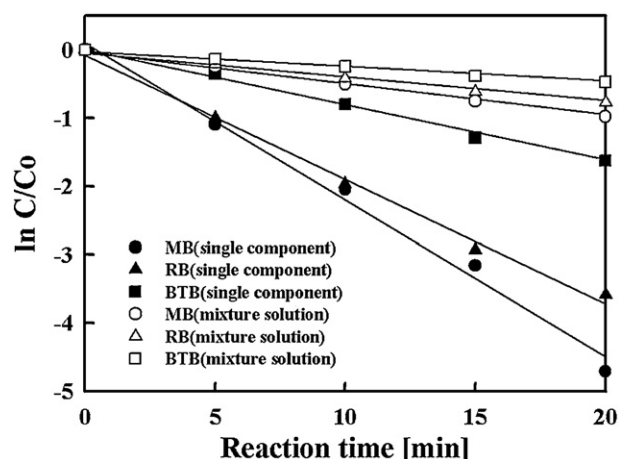


Fig. 6. Photo-catalytic decomposition of organic dyes using MDEL.

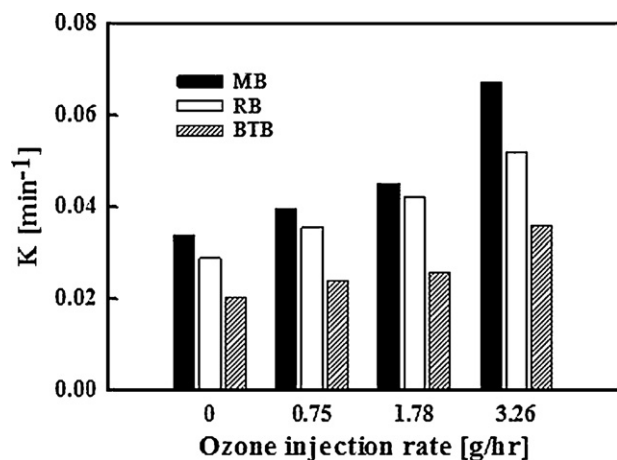


Fig. 7. Effect of ozone injection rate on the photocatalytic activities for decomposition of organic dyes.

It has been reported that decomposition of a pollutant in a mixture can be significantly affected by other pollutants existing in the mixture [17,18]. More experimental data are needed to quantify the mutual inhibitory effect, which is beyond the scope of this study. Fig. 6 shows the concentrations of the organic dyes as a function of degradation reaction time at 0.4 kW of microwave intensity. The decomposition rate was evaluated from the rate of the change in dye concentration as a function of irradiation time. All the organic dyes were degraded well by microwave-assisted photocatalytic reactions. The results shown in Fig. 6 indicate that decomposition of dyes by photocatalytic reaction in presence of TiO_2 photocatalyst is approximated by a pseudo first-order reaction model:

$$C/C_0 = \exp(-Kt) \quad (1)$$

where C is the organic dye concentration at time t , C_0 the initial concentration, and K the over-all rate constant. Over-all rate constant K is determined as the slope of the line in Fig. 6 by regression analysis.

3.2. Effects of ozone

Ozone, a strong oxidant with the electric potential difference of 2.07 V, has widely been used in water treatment because it can effectively remove taste, odor, and precursors of trihalomethanes. However, the direct ozone reaction is relatively selective in oxidation of organic compounds because ozone has very low reactivity on single-bond chemicals and aromatic compounds with specific functional groups such as $-\text{COOH}$ and $-\text{NO}_2$. On the contrary, the hydroxyl radical ($\bullet\text{OH}$), which has a higher oxidation potential (2.80 V) than ozone and reacts with organic compounds unselectively, can be applied to oxidation treatment effectively. Therefore, large attention is being given to the advanced oxidation processes (AOPs), in which the organic compounds are decomposed using OH radicals. The microwave/UV/ $\text{O}_3/\text{H}_2\text{O}_2/\text{TiO}_2$ photocatalyst hybrid process used in this study is an AOP that can overcome the limitations of the single-process ozone water treatment by using microwave and UV irradiations and resulting activation of photocatalysts to maximize the formation of OH radicals.

Fig. 7 compares the results of the decomposition of mixed organic dye solution obtained with different experimental conditions. Three different levels of ozone addition was tested; 0.75, 1.78, and 3.26 g/h. The circulation flow rate of the solution and the microwave irradiation intensity were 300 ml/min and 0.4 kW, respectively, when they were applied. The decomposition rates of all organic dyes increased with the ozone injection rate. At all ozone

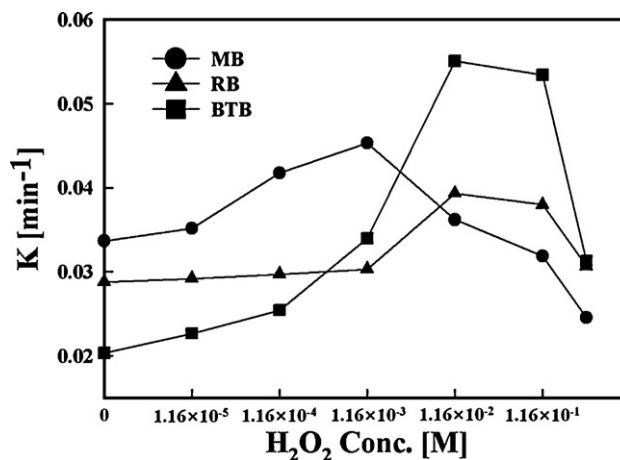


Fig. 8. Effect of addition H_2O_2 for decomposition of organic dye in aqueous solutions.

injection rates tested, the decomposition rate of MB was the highest, while that of BTB was the lowest. With a high ozone injection rate, the reaction finished too fast. Therefore, the ozone injection rate of 1.78 g/h was used in the follow-up experiments, in which various combinations of each elemental technique were evaluated, so that the decomposition rate can be measured more easily.

3.3. Effect of addition H_2O_2

The effect of H_2O_2 has been investigated in numerous studies and it was reported that it increases the photocatalytic degradation rate of organic pollutants [19]. The enhancement of the degradation rate with addition of H_2O_2 can be rationalized in terms of several reasons. First, it increases the rate by removing the surface-trapped electrons, hence by lowering the electron-hole recombination rate and increasing the efficiency of hole utilization for reactions such as $(\text{OH}^- + \text{h}^+ \rightarrow \text{OH}^\bullet)$. Second, H_2O_2 may split photo-catalytically to produce hydroxyl radicals directly, as a cited in studies of homogeneous photo-oxidation using UV /($\text{H}_2\text{O}_2 + \text{O}_2$). Because H_2O_2 seems to be an efficient electron acceptor in TiO_2 photocatalytic systems, its effect on photocatalytic degradation reactions was tested [20]. Fig. 8 shows how the photocatalytic degradation rate of the mixed organic dye solution is affected by the addition of H_2O_2 in the microwave-assisted photocatalytic system. The H_2O_2 addition to reactant solution increases the photocatalytic degradation rate to a maximum, but further addition of H_2O_2 above this level decreases the efficiency [21]. H_2O_2 is known to form a surface complex on TiO_2 [22]. The reduced photocatalytic degradation rate in the presence of excess H_2O_2 can be ascribed to both the blocking of surface sites by H_2O_2 and the OH radical scavenging by H_2O_2 ($\text{H}_2\text{O}_2 + \bullet\text{OH} \rightarrow \text{HO}_2^\bullet + \text{H}_2\text{O}$). The effect of hydrogen peroxide addition was the most prominent for BTB. For all organic dyes, the decomposition rate increased with increasing hydrogen peroxide concentration until it reached a maximum point beyond which it decreased with increasing hydrogen peroxide concentration. BTB and RB showed the maximum decomposition rate at the hydrogen peroxide concentration of 1.16×10^{-2} M, whereas MB showed the maximum decomposition rate at a lower hydrogen peroxide concentration (1.16×10^{-3} M). The hydrogen peroxide concentration of 1.16×10^{-2} M was used in the follow-up experiments. The reason why MB showed the maximum decomposition rate at a much lower H_2O_2 concentration than BTB and RB could not be inferred from the limited experimental results of this study. To obtain scientific explanation for this, a detailed chemical analysis using more precise analytical instruments is required.

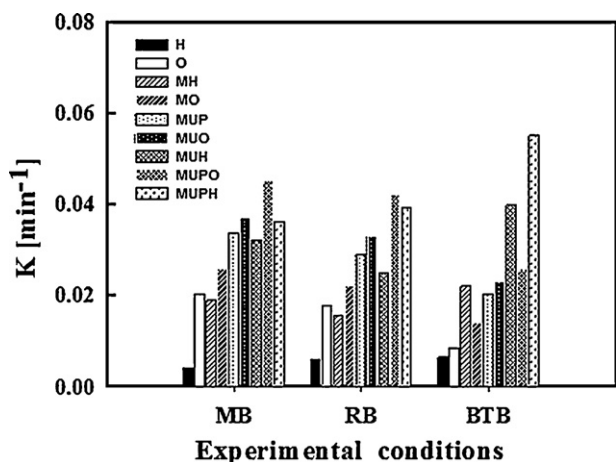


Fig. 9. Degradation reaction rate constants obtained under different experimental conditions.

3.4. Comparison of the effects of the constituent techniques

This study was aimed at enhancing the photocatalytic decomposition efficiency of organic dyes by adding microwave, ozone, and hydrogen peroxide. In order to find the optimal condition for decomposition of organic dyes, different combinations of each element techniques, i.e., microwave, ozone, hydrogen peroxide, UV, and photocatalyst, were evaluated.

Fig. 9 shows the results of the decomposition reaction of the mixed solution described in Table 1 by the microwave/UV/TiO₂/ozone/H₂O₂ hybrid process system. Ozone injection rate of 1.78 g/hr, hydrogen peroxide concentration of 1.16×10^{-2} M, and the microwave power of 0.4 kw were used in all experiments reported hereafter. In Fig. 9, H (H₂O₂) refers to the addition of hydrogen peroxide only, O (ozone) the ozone injection only, MH (microwave + H₂O₂) the combination of microwave irradiation and hydrogen peroxide addition, MO (microwave + ozone) the combination of microwave irradiation and ozone injection, MUP (microwave + UV + photo-catalyst) the UV irradiation driven by MDEL with the reactor filled with photo-catalyst balls, MUO (microwave + UV + ozone) the combination of UV irradiation driven by MDEL and ozone injection, MUH (microwave + UV + H₂O₂) the combination of UV irradiation driven by MDEL and hydrogen peroxide addition, MUPO (microwave + UV + photocatalyst + ozone) the combination of UV irradiation driven by MDEL and ozone injection with the reactor filled with photocatalyst balls, and MUPH (microwave + UV + photocatalyst + H₂O₂) the combination of UV irradiation driven by MDEL and hydrogen peroxide addition with the reactor filled with photocatalyst balls.

As can be seen in Fig. 9, three organic dyes showed similar decomposition characteristics except that the effect of hydrogen peroxide addition was remarkable for BTB. When only microwave was irradiated, organic dyes were not degraded at all probably because the solution temperature was maintained at 298 K in this study to exclude the thermal effect. When microwave was irradiated without MDEL with the reactor filled with TiO₂ photocatalyst balls, organic dyes were not degraded at all, either. When microwave was irradiated on top of ozone injection (MO) or hydrogen peroxide addition (MH), however, the decomposition rate of all organic dyes increased significantly compared to the cases of ozone injection only (O) or hydrogen peroxide addition only (H). Furthermore, the decomposition rate became even higher when microwave-assisted UV irradiation was added to MO (MUO) or to MH (MUH) and became the highest when all the elemental techniques were combined (MUPO or MUPH). In particular, a large synergy effect was observed for BTB when hydrogen peroxide addi-

tion was combined with other elemental techniques. As was shown in Fig. 8, the addition of hydrogen peroxide with its concentration of 1.16×10^{-2} M resulted in maximum decomposition rate for BTB and RB, whereas it led to little enhancement in decomposition rate of MB owing to the scavenging effect. When the hydrogen peroxide addition was combined with other elemental techniques, however, a significant increase in the decomposition rate of MB due to hydrogen peroxide addition was observed. This result demonstrates that combination of elemental techniques that have little effect when used alone can make a synergy effect.

In this study, microwave irradiation was added to the conventional photocatalysis system to activate the pollutants and photocatalysts and hence to accelerate the decomposition reaction. In addition, ozone and hydrogen peroxide were added as auxiliary oxidants to enhance the decomposition efficiency. When microwave irradiation was used alone or on top of photocatalyst, the effect was not significant. When microwave irradiation was used together with the addition of an auxiliary oxidant, i.e., ozone or hydrogen peroxide, however, the decomposition rate increased significantly. This result suggests that microwave activates the auxiliary oxidants leading to production of active species. This hypothesis, however, cannot be proven with the limited experimental data obtained in this study. More precise and quantitative analyses on the elemental techniques tested in this study are required in the future for more reliable evaluation and application of them.

4. Conclusions

The following conclusions were inferred from the results of photocatalytic degradation of three different single-component organic dyes and their mixture using microwave/UV/O₃/H₂O₂/TiO₂ photocatalysts hybrid process system:

- (1) Among the three organic dyes, MB was degraded the fastest both in single-component experiments and in mixed solution experiments. The decomposition rates of all organic dyes were higher in single-component experiments than in mixed solution experiments.
- (2) The decomposition rates of all organic dyes increased with the ozone injection rate. At all ozone injection rates tested, the decomposition rate of MB was the highest, while that of BTB was the lowest.
- (3) The effect of hydrogen peroxide addition was the most prominent for BTB. For all organic dyes, the decomposition rate increased with increasing hydrogen peroxide concentration until it reached a maximum point beyond which it decreased with increasing hydrogen peroxide concentration.
- (4) When microwave was irradiated on top of ozone injection (MO) or hydrogen peroxide addition (MH), however, the decomposition rate of all organic dyes increased significantly compared to the cases of ozone injection only (O) or hydrogen peroxide addition only (H). Furthermore, the decomposition rate became even higher when microwave-assisted UV irradiation was added to MO (MUO) or to MH (MUH) and became the highest when all the elemental techniques were combined (MUPO or MUPH).

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