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Pilot scale annular plug flow photoreactor by UV/H_2O_2 for the decolorization of azo dye wastewater

Hung-Yee Shu*, Ming-Chin Chang

Department of Environmental Engineering, Hungkuang University, No. 34 Chung-Chie Rd., Shalu, Taichung 433, Taiwan, ROC

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Abstract

A pilot scale annular plug flow photoreactor with thin gap size, which combines with UV irradiation and hydrogen peroxide, was employed to deal with colored dyeing wastewater treatment. In the experiment, a mono-azo dye acid orange 10 was the target compound. The experimental parameters such as flow rate, hydrogen peroxide dosage, UV input power, pH and dye initial concentrations in a pilot scale photoreactor with flow rate of 9.32 m³ day⁻¹ were investigated. Ultimately, the degradation rates were calculated and compared with a 100-l batch reactor. In our plug flow photoreactor design, the degradation rate of acid orange 10 was 233 times higher than that of 100-l annular batch reactor with same UV light source. The residence time needed for 99% decolorizing of 1001 of 20 mg l^{-1} acid orange 10 wastewater was 26.9 min for the thin gap plug flow reactor and was far shorter than that of batch reactor needed. © 2005 Elsevier B.V. All rights reserved.

Keywords: AOPs; UV/H2O2; Annular plug flow reactor; Azo dye

1. Introduction

Textile industry is one of the most important industries as well as contributed the Taiwan's economic development in last few decades. However, the environmental contamination, high level of color intensity and chemical oxygen demand (COD), is substantially produced from the effluent of textile dyeing and finishing industries as well as discharged into surface water body. Therefore, the stricter effluent standards have regulated by Taiwan EPA in order to enforce the decolorization and decontamination for the dye wastewater. Thus, decolorization technologies of dye wastewater become more and more important to implement the new standards.

In these two decades, the advanced oxidation processes (AOPs) have shown to be a promising method as well as received various full scale applications successfully in the wastewater treatment field. The oxidation process produces the free radicals (i.e. OH[•]), which attack and degrade the

fax: +886 4 26525245.

E-mail address: hyshu@sunrise.hk.edu.tw (H.-Y. Shu).

refractory organic compounds in wastewater unselectively. One of the AOPs, UV irradiated with hydrogen peroxide (UV/H_2O_2) , has efficiently decolorized the dyeing wastewater. In the UV/H₂O₂ photoreactors, the reaction rates were predominantly influenced by the UV light intensity, volume and pH of treated wastewater, dosage of hydrogen peroxide, and initial dye concentration. Additionally, most of investigations worked on lab-scale batch reactor, but little attention has been given to the usage of pilot or field scale reactor in the application of dyeing wastewater decolorization. Not until the last decade, some investigators have reported the successful applications of the UV/H2O2 photoreactor of dyeing wastewater treatment. Shu et al. [1] showed that the UV/H₂O₂ process had effectively decolorized acid red 1 (AR1) and acid yellow 23. They also found that initial hydrogen peroxide concentration was a very important operating parameter, which affected the pseudo first order reaction rate. An optimal molar ratio of hydrogen peroxide to dye concentration (H₂O₂/dye) of AR1 was about 416. Galindo and Kalt [2] observed the decolorization of acid orange 7 (AO7) was effective by the process. The optimal H₂O₂ initial concentration was about 100 mM for AO7 initial concentration of

^{*} Corresponding author. Tel.: +886 4 26318652 4111;

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 5.7×10^{-2} mM, and H₂O₂/dye molar ratio could be calculated as 1754. Similarly, Colonna et al. [3] expressed that the H₂O₂/dye molar ratio was about 400 for AR1. Thus, the critical effect of hydrogen peroxide in UV/H₂O₂ processes was intensively studied by several researchers [4-10]. Many researchers focused on discussing and observing the optimal hydrogen peroxide dosage as mentioned above, but use of the optimal hydrogen peroxide dosage only increased the observed rate constants up to one order of magnitude. Instead of hydrogen peroxide dosage, UV light intensity affects the rate constant significantly and linearly. Shu et al. [11] explored that the decolorization rate increase linearly with respect to UV input power applied to the reaction volume. With the limitation of equipment setup, very few researches have done on the effect of UV intensity on decolorization of dye wastewater [11-12]. Besides, a broad literature review by Venkatadri and Peters [13] stated the chemical oxidation technology by UV/H₂O₂ process, Fenton's reagent and UV/TiO₂, which generates hydroxyl free radicals •OH to enhance the degradation of refractory organics. The decomposition of BTEX (benzene, toluene, ethylbenzene, and pxylene) in aqueous solution was conducted efficiently by UV/H₂O₂ process [14]. The effluent from pulp bleach plant and cyanide from thermoelectric power station wastewater were all proved to be thoroughly treated by UV/H₂O₂ process [15–16].

UV light intensity, which affects the degradation of azo dyes in a UV/H₂O₂ photoreactor, is a major operating parameter. Theoretically, the higher the UV power, the faster is the formation of OH[•] free radicals. Thus, the higher UV power will lead to a higher degradation rate of azo dyes. However, the colored wastewater absorbs UV irradiation to result into parts of UV light left to irradiate hydrogen peroxide to reduce free radicals productions as well as reduction of decolorization rates. Consequently, the development of thin annular gap in UV/H2O2 photoreactors can overcome the disadvantage because of avoiding the block out of UV irradiation by colored wastewater [17]. On the other hand, UV/H₂O₂ photoreactors performed efficiently, yet an acceptable cost has not been fully established. Accordingly, the objective of this study was to evaluate the feasible treatment for azo dye wastewater by a pilot scale plug flow UV/H₂O₂ photoreactor. In the experiment, the reactor with thin annular gap was developed for the decolorization of acid orange 10. The effects of initial dye concentration, H₂O₂ concentration, UV light power, and flow rate, on the decolorization of azo dyes were studied in detail.

2. Experimental

2.1. Materials

Hydrogen peroxide was obtained from Fluka Chemical (35%, w/w). Acid orange 10 (molecular formula: $C_{16}H_{10}N_2Na_2O_7S_2$; molecular weight: 452.4; characteristic wavelength: 452 nm), obtained from Aldrich Chemical Co. without further purification.

2.2. Apparatus and analyses

Two reactor systems were used in this study. The experiments studied the effectiveness of UV light power and the effects of pH, initial azo dye concentration and hydrogen peroxide dosage in a plug flow reactor. Along the center of reactor, it was equipped with a medium pressure mercury UV lamp made by Canrad Hanovia, which was 25 in. (63.5 cm) arc length and major wavelength at 253.7 nm. This allowed a total input electric power of 5000 W applied to UV lamp and initiating the photochemical reaction in a 3000 ml quartz sleeve vessel. The inner diameter of outer quartz tube was 10.8 cm and outer diameter of inner quartz tube was 8.9 cm. The length of this quartz sleeve vessel was 105 cm. The bottom of both quartz tubes were all round in shape. On the other hand, the second reactor was a pilot scale stainless steel reactor with the dimension of 33 cm in diameter, 150 cm in height, 1001 in hold up volume. The UV lamp was located at the center of reactor with the same configuration as mentioned above. The dye solution was recirculated in and out the batch reactor to mix completely by a recycling pump. The pH was monitored by a pH controller and monitor device. The schematic diagrams of both reactor apparatus are shown in Fig. 1. Optical absorption spectrum of azo dye was determined by a Cray DMS-300 spectrophotometer so that the characterized wavelength was selected for quantitative analysis. The azo dyes were analyzed by a HPLC made by LDC/Millton. The conditions were C_{18} column of $6 \text{ mm} \times 75 \text{ mm}$ and packing particle size of 3 µm, mobile phase of 50% methanol and 50% water with 1 M acetic acid, obtained by the proposed method of Bruins et al. [18]. Hydrogen peroxide concentration was determined by spectrophotometer method based on the method proposed by Masschelein et al. [19].

2.3. Procedure

In the plug flow reactor experiments, the simulated wastewater was prepared in a tank reservoir. While changing dye concentration, hydrogen peroxide concentration, and pH. Dye wastewater was pumped through the plug flow reactor in various flow rates. While reaching steady state, wastewater was withdrawn and analyzed. In the batch reactor experiment, volume of 1001 of water was treated in the photo-oxidation reactor with hydrogen peroxide and UV irradiation, while various dye concentration and hydrogen peroxide dosage. Samples were withdrawn from the sampling port at regular time intervals for analysis.

3. Results and discussion

3.1. Effect of volumetric flow rate

Mean residence time (τ) of wastewater in this plug flow reactor was varied by adjusting flow rate through the reactor.



Fig. 1. The schematic diagrams of reactor apparatus used in this study. (a) Thin gap annular plug flow reactor and (b) 1001 recirculated batch reactor.



Fig. 2. The concentration ratio of effluent to influent (C_{eff}/C_{inf}) of AO10 as a function of flow rate for various hydrogen peroxide concentrations in the UV/H₂O₂ plug flow reactor. The initial dye concentration was 42.0 μ M (20.0 mg l⁻¹), hydrogen peroxide concentrations were from 0.116 to 3.722 mM, UV power input was 5000 W, and initial pH was varied from 5.3 to 5.8.

In the reactor, the space time or mean residence time is defined as dividing the reactor volume (V) by volumetric flow rate (v_0) entering the reactor as follows:

$$\tau = \frac{V}{v_0} \tag{1}$$

The color removal efficiency of acid orange 10 (AO10) was expressed by the concentration ratio of effluent to influent, $C_{\rm eff}/C_{\rm inf}$ versus flow rate while changing hydrogen peroxide concentrations are shown in Fig. 2. The volumetric flow rate was adjusted by a flow meter from 1.63 to 6.471 min^{-1} , which is in the range of $2.35-9.32 \text{ m}^3 \text{ day}^{-1}$. This range of treatment flow rate is surely a pilot scale process. In the experimental conditions of hydrogen peroxide concentrations, the higher color in effluent can be detected obviously while the higher flow rate applied. However, the color removal percentage of AO10 increased by decreasing the volumetric flow rate. In Fig. 3, $C_{\rm eff}/C_{\rm inf}$ versus mean residence time (τ) at various hydrogen peroxide concentrations was presented. Since the mean residence time is obtained by the ratio of reactor volume to initial volumetric flow rate, the longer residence time resulted into the higher color removal. The more hydrogen peroxide concentration performed higher color removal efficiency, too. For example, while hydrogen peroxide concentration of 0.465 mM and mean residence time of 0.98 and 1.83 min, the $C_{\rm eff}/C_{\rm inf}$ value were 0.092 and 0.012 so that the distinguished color removal efficiencies were obtained as 90.8 and 98.8%, respectively.



Fig. 3. The $C_{\rm eff}/C_{\rm inf}$ of AO10 as function of mean residence time under various hydrogen peroxide concentrations in the UV/H₂O₂ plug flow reactor. The operating conditions were the same as given in Fig. 2. The regression line was base on Eq. (3) by assuming pseudo first order reaction and irreversible reaction in a steady state plug flow reactor.

3.2. Calculation of kinetic rate constants

The decomposition of AO10 by UV/H_2O_2 process in continuous annular plug flow reactor was examined under various hydrogen peroxide concentrations and volumetric flow rates. Assuming the decomposition behavior of AO10 is pseudo first order and irreversible reaction in a steady-state plug flow reactor. Thus, the pseudo first order rate constant can be calculated as follows:

$$k_{\rm obs} = \frac{-\ln\left(\frac{C_{\rm eff}}{C_{\rm inf}}\right)}{\tau} \tag{2}$$

where $k_{\rm obs}$ denotes the pseudo first order rate constant, τ expresses mean residence time, $C_{\rm eff}$ and $C_{\rm inf}$ are effluent and influent concentrations of AO10, respectively.

By rearranging the above equation, $C_{\text{eff}}/C_{\text{inf}}$ can be expressed as follows:

$$\frac{C_{\rm eff}}{C_{\rm inf}} = e^{-k_{\rm obs}\tau} \tag{3}$$

The nonlinear regression model was plotted by $C_{\rm eff}/C_{\rm inf}$ versus τ in Fig. 3. The regression model lines and the experimental data are in a very good agreement. That means the assumption of first order reaction is proved precisely.

3.3. Effect of hydrogen peroxide concentration

Hydrogen peroxide concentration is one of important parameter for the degradation of azo dyes in the UV/H_2O_2 reactor. UV light irradiates hydrogen peroxide to produce



Fig. 4. The observed first order rate constants and $C_{\rm eff}/C_{\rm inf}$ as functions of initial hydrogen peroxide concentration. The operating conditions were the same as given in Fig. 2. For $C_{\rm eff}/C_{\rm inf}$ versus initial hydrogen peroxide concentration experiments, the flow rate was $3.031 \,{\rm min^{-1}}$ and mean residence time was 0.98 min.

the hydroxyl free radical (OH[•]), which will theoretically produce more amounts by higher hydrogen peroxide concentration to increase the degradation rate of azo dye. When the OH[•] reaches equilibrium concentration, increase of hydrogen peroxide concentration produced no more OH[•] as shown in Fig. 4. Under the same UV light source, increasing hydrogen peroxide concentration from 0.116 to 3.722 mM (H₂O₂/AO10 ratio equals from 2.76 to 88.62), gave observable improvement by decrease of $C_{\text{eff}}/C_{\text{inf}}$. It was as low as 0.002 so that the color removal efficiencies was 99.8% while volumetric flow rate of 3.031 min⁻¹.

On the other hand, the observed pseudo first order rate constants (k_{obs}) of decolorization versus hydrogen peroxide dosage were also shown in Fig. 4 that the degradation rate enhances while increasing hydrogen peroxide concentration. Thus, the initial hydrogen peroxide concentration increase is capable to improve the decomposition rate of dye, though an optimum initial concentration exists. This is because that scavenging of hydroxyl radicals occurs at higher H₂O₂ concentration to limit the reaction of free radicals with AO10. In this work, the decreasing in pseudo first order rate constant was not seen in the experimental condition up to the highest hydrogen peroxide concentration of 3.722 mM. Thus, the optimal initial hydrogen peroxide concentration was observed as 3.722 mM for the operating conditions studied.

3.4. Effect of UV intensity

The effect of UV light power on the decolorization of AO10 was investigated as shown in Fig. 5, which shows significantly influence of AO10 color removal rate by UV power



Fig. 5. The $C_{\rm eff}/C_{\rm inf}$ of AO10 as a function of mean residence time for two UV light intensities. The initial dye concentration was 42.0 μ M, hydrogen peroxide concentration was 3.722 mM.

in the photochemical reactor. UV light irradiates hydrogen peroxide to decolorize AO10 in a relatively high reaction rate according to the hydroxyl radical generation. The more reactive hydroxyl radicals can react more readily with the dye. The wavelength of 254 nm of UV light employed in the experiment that light intensity can be adjusted by input power of 5000 and 3124 W as shown in Fig. 5. The color removal rate of AO10 increases while incrementing UV light power from 3124 to 5000 W. This is because that the increasing UV light power results into a faster photo activation of hydroxyl radicals substantially to perform a higher color removal rate. On the other hand, the mean residence time is also a very important operating parameter. For lower mean residence time, i.e. 0.5 min, the $C_{\text{eff}}/C_{\text{inf}}$ values are 0.15 and 0.04 for UV light power of 5000 and 3124 W, respectively. It is about 3.5 times difference. It implies that for a shorter residence time azo dye molecules cannot react with hydroxyl radicals sufficiently. Thus, in the system with larger UV input power, which provides higher hydroxyl radical concentration can degrade more azo dye molecules than that of system with lower UV input power. As for longer residence time, i.e. 1.5 min, the azo dye molecules have the chance to react with hydroxyl radicals efficiently. Therefore, for both 5000 and 3124 W UV system, the removal rate of azo dye can reach about 99.5% ($C_{\text{eff}}/C_{\text{inf}}$ is 0.004). Increasing UV light intensity from 3124 to 5000 W gives observable improvement in decolorization rate constant. UV intensity increases about 1.60 times from 3124 to 5000 W as well as the first order rate constant increases 1.69 times. It implies that relationship between k_{obs} and UV intensity is approximately linear and consistent with previous work [11]. The results are arranged in Table 1.

	Flow rate $(l \min^{-1})$	Mean residence time (min)	[0,4-5]UV light intensity (W)		
			3124	5000	
Removal efficiency	3.03	0.98	98.9%	99.8%	
	6.47	0.46	85.3%	96.2%	
First order rate constant (min^{-1})			4.232	7.171	

The decolorization results of AO10 for two UV intensities (for AO 10 concentration of 42.0 µM and hydrogen peroxide concentration of 3.722 mM)

3.5. Effect of influent dye concentration

Table 1

Many researchers investigated the effect of initial concentration on the degradation of dyes in solution [1,2,11,12]. Mohey El-Dein et al. [12] reported that UV/H₂O₂ processes degraded azo dye reactive black 5 more efficiently at a lower dye concentration. We have the similar results as above in the decolorization of AO10 as depicted in Fig. 6, which plotting the $C_{\rm eff}/C_{\rm inf}$ versus mean residence time at various influent dye concentrations. The color removal rate of AO10 is faster while influent dye concentration of 42.0 μ M than 84.0 μ M. Three influent dye concentrations of 42.0, 63.0, and 84.0 μ M, respectively, were employed and the concentration ratio of hydrogen peroxide to dye ($C_{\rm H_2O_2}/C_{\rm inf}$) of 22.17 was fixed in the experiment while the first order rate constant obtained as 2.391, 2.352, and 2.178 min⁻¹, respectively, in Fig. 6a. The initial pH values of experiments were



Fig. 6. The effect of initial concentration on the decolorization of AO10. (a) The molar ratio of $C_{\text{H}_2\text{O}_2}/C_{\text{inf}}$ was fixed at 22.17 and (b) The $C_{\text{H}_2\text{O}_2}$ was fixed at 1.861 mM. The UV power input was 5000 W, and initial pH was varied from 5.3 to 5.8.

affected by various hydrogen peroxide and AO10 concentrations and varied from 5.3 to 5.8. The $C_{\rm eff}/C_{\rm inf}$ increases slightly by raising initial dye concentration as seen in figure. From Fig. 6b, the effect of influent dye concentration (C_{inf}) on the decolorization was shown by plotting the same influent dye concentrations and mean residence time as in Fig. 6a, while fixing the concentration of hydrogen peroxide at 1.861 mM. Under the same hydrogen peroxide concentration, the curve of $C_{\rm eff}/C_{\rm inf}$ increases significantly by raising initial dye concentration at 42.0, 63.0, and 84.0 µM that the first order rate constant can be obtained as 5.761, 2.645, and 2.178 min^{-1} , respectively. Therefore, our results were similar to the observations by other investigators that the declining pseudo first order rate constants with respect to increasing initial dye concentrations. This phenomenon is associated with the characteristic of UV-vis absorption spectrum of AO10, because that it will compete with photons under UV radiation. The high influent concentration substantially absorbs more UV light at 254 nm than that with low concentration, so that the photon numbers reduces by decreasing the UV light intensity to decrease the formation of OH[•] radicals. Accordingly, the color removal rate of AO10 decreases as the influent AO10 concentration increased. It seems that at a high influent concentration of AO10, high color removal fraction can also be achieved about 99% for sufficient mean residence time of 1.8 min. This illustrates that the plug flow reactor functions well at a high initial concentration of AO10.

3.6. Effect of pH

The pH effect on AO10 decolorization was investigated as shown in Fig. 7, while influent dye concentration of 84.0 µM and hydrogen peroxide concentration of 3.722 mM in a plug flow UV/H₂O₂ reactor under various pH conditions. The decolorization rate constant was 3.211 and 4.237 min^{-1} for pH 5.8 and 3.6 of original pH without adjustment, respectively. That meant at the same concentration of AO10 and hydrogen peroxide, reducing pH from 5.8 to 3.6 conducts fair improvement in decrease of $C_{\rm eff}/C_{\rm inf}$. While volumetric flow rate of 3.031 min^{-1} (mean residence time of 0.98 min) and pH at 5.8 and 3.6, the $C_{\rm eff}/C_{\rm inf}$ values were 0.129 and 0.069, so that the color removal efficiencies were obtained as 87.1 and 93.1%, respectively. Similar effect of pH on decolorization by UV/H2O2 process was reported by Galindo and Kalt [20] for acid orange 5. They observed the rise of the kinetic constant of acid orange 5 and increase of bulk acidity



Fig. 7. The $C_{\rm eff}/C_{\rm inf}$ of AO10 as a function of mean residence time for two pH values. The initial dye concentration was 84.0 μ M (40.0 mg l⁻¹), hydrogen peroxide concentration was 3.722 mM, and UV power input was 5000 W.

during the decolorization test. It was concluded that reaction rate increased drastically when pH of solution was closed to the pK_a of azo dye. The pH of highest rate constant was located at pH 2 for acid orange 5 and then decreased sharply while increasing solution pH up to 12. The results of this study were in good agreement with Galindo and Kalt's work.

3.7. Comparison with pilot batch reactor

An attempt was made for the comparison of AO10 degradation rates with a 100-1 recirculated batch reactor with 33.0 cm in diameter and the plug flow reactor designed in this experiment under the same UV irradiation power. The AO10 degradation rate constant of latter one was 5.761 min^{-1} , which indicated about 233 times higher than the former one of $0.0247 \,\mathrm{min}^{-1}$, while initial dye concentration of $20.0 \text{ mg} \text{ } 1^{-1}$ (42.0 μ M) and hydrogen peroxide concentration of 1.861 mM. Fig. 8 shows the color removal of AO10 by recirculated batch UV/H2O2 process. Additionally, to decolorize 1001 of 42.0 µM AO10 wastewater, the thin gap plug flow reactor takes only 26.9 min to decolorize about 99.0% of color removal, while the rerciculated batch reactor with larger annular gap takes about 186.4 min to obtain less than 1.0% of residual color in the same condition. Consequently from the above, the light penetration is a very important factor for enhancement of degradation rate of AO10 in UV/H₂O₂ system. Meanwhile, the larger volume of thick gap of batch reactor resulted into ineffective volume of decolorization. Thus, the advantage of taking the total reactor volume effectively for photo-oxidation of pollutants by reducing the gap between reactor wall and UV lamp could be understood.



Fig. 8. The normalized concentrations (C/C_0) of AO10 as functions of residence time in the 100-1 UV/H₂O₂ batch reactor. Initial dye concentration was 42.0 μ M and hydrogen peroxide concentration was 1.861 mM at initial pH of 5.3, and UV power input was 5000 W.

4. Conclusion

The results showed that the thin gap plug flow UV/H_2O_2 process can almost completely decolorize the synthesized azo dye wastewater under various operating conditions. The decolorization rates of AO10 were affected significantly by the hydrogen peroxide concentration, UV intensity, and initial dye concentrations. For the flow rate from 1.63 to 6.471 min⁻¹, 90% decolorization efficiency of AO10 reaches with the hydrogen peroxide concentration of 1.861 mM. Reducing the flow rate to 1.631 min^{-1} , the 90% decolorization goal can be reached under lower hydrogen peroxide concentration of 0.233 mM. Besides, the decolorization rate constant by using plug flow reactor was 233 times larger than that of a recirculated batch reactor. The residence time demand for decolorizing 99% of 1001 of AO10 wastewater by the plug flow reactor was 26.9 min, which was seven times less than that of the recirculated batch reactor.

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