

Decolorization of C.I. reactive red 198 in UV/oxidant and UV/TiO₂/oxidant systems

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Abstract In this study, UV/oxidant and UV/TiO₂/oxidant systems were employed to treat textile wastewater. The parent compound was C.I. reactive red 198 (RR198). The selected oxidants were H₂O₂, Na₂S₂O₈, NaBrO₃, and NaIO₄. The effects of oxidant dosage (1–24 mM), wavelength of UV (254 and 365 nm) and radical scavenger addition (C₂H₅OH) were determined in UV/oxidant systems. The experimental results revealed that all oxidants effectively decolorized RR198 under 254 nm irradiation; however, only Na₂S₂O₈ and NaIO₄ can decolorize RR198 under 365 nm irradiation. The decolorization rates fit a pseudo-first order reaction model. Under 254 nm irradiation and 6 mM oxidant addition, the decolorization rate constants (*k*) of H₂O₂, Na₂S₂O₈, NaBrO₃, and NaIO₄ for RR198 were 10.24, 17.93, 13.37, and 11.90 h⁻¹. Under 365 nm irradiation, 1 g/L TiO₂ and 1 mM NaIO₄ addition, the *k* values of the UV/TiO₂, UV/NaIO₄, and UV/TiO₂/NaIO₄ systems were 0.50, 0.52, and 11.67 h⁻¹. The inhibition of RR198 decolorization by the addition ethanol indicates that the primary decolorization pathway involves hydroxyl radicals in UV/H₂O₂ and UV/Na₂S₂O₈ systems, and that oxidation by other radicals is probably important in UV/NaBrO₃ and UV/NaIO₄ systems.

Keywords Decolorization · TiO₂ · H₂O₂ · Na₂S₂O₈ · NaBrO₃ · NaIO₄

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Introduction

Wastewater from the textile dyeing industry has a high or low pH, a high temperature and a high concentration of coloring material. Several dyes are environmental hazards because they are toxic. Removing color from wastewater is important because small amounts of dye are clearly visible and adversely affect water quality. Colored wastewater is subject to strict environmental legislation because it has a negative effect on photosynthetic activity all over the world. Therefore, the decolorization of dye effluents is attracting an attention. C.I. reactive red 198 (RR198), a dye that contains two of the most commonly used anchors—monochlorotriazine and vinyl sulfone groups—was adopted as the parent compound in this study.

Conventional treatments of dye effluents include biological oxidation and adsorption. Although less expensive than other approaches, biological treatment is ineffective for decolorization because the dyes are toxic. Adsorption onto activated carbon transfers most of the contaminant from the wastewater to the solid phase. This method therefore requires further disposal of the sludge. Advanced oxidation processes (AOPs) are alternative procedures for decolorizing and reducing problematic wastewater loads from textile companies. AOPs are based on the formation of hydroxyl radicals in water, which are highly reactive and nonselective oxidants that can oxidize organic compounds. In a heterogeneous photocatalytic reaction, molecular oxygen acts as an electron acceptor to prevent the recombination of electrons and holes. To improve the photocatalytic efficiency of semiconductors, Serpone et al. [1] proposed an interparticle electron transfer process, which couples two semiconductors with different redox energy levels to increase charge separation for the corresponding conduction and valance bands. Coupled semiconductors improved charge separation and were responsible for the enhancement in the rate of organics photocatalytic degradation [1–4]. Degradation levels could also be improved using various oxidants as electron scavengers. Such inorganic oxidants as IO_4^- , $\text{S}_2\text{O}_8^{2-}$, BrO_3^- , ClO_3^- , and H_2O_2 can be utilized as additives instead of oxygen to enhance the photodegradation rates of organic substrates by quenching the conduction band electrons and form reactive radical intermediates [5–7]. These oxidants increase the effectiveness of UV/photocatalyst in degrading organic substrates by capturing the electrons that are ejected from the photocatalyst, to reduce the probability of recombination of photogenerated electrons and photogenerated holes: the available number and survival time of the photogenerated holes is thus increased, promoting their effective reaction with organic substrates. The effect of the oxidants in the UV/ TiO_2 system on the degradation rate of 4-chloro-2-methylphenol were found to follow the order $\text{IO}_4^- > \text{BrO}_3^- > \text{H}_2\text{O}_2 > \text{O}_2 > \text{ClO}_3^-$ [6]. Syoufian and Nakashima [8] indicated that the effectiveness of the oxidants in the UV/ TiO_2 system followed the order $\text{S}_2\text{O}_8^{2-} > \text{IO}_4^- > \text{BrO}_3^- > \text{H}_2\text{O}_2 > \text{ClO}_3^-$. Selvam et al. [9] found the order of enhancement, $\text{UV}/\text{TiO}_2/\text{IO}_4^- > \text{UV}/\text{TiO}_2/\text{BrO}_3^- > \text{UV}/\text{TiO}_2/\text{S}_2\text{O}_8^{2-} > \text{UV}/\text{TiO}_2/\text{H}_2\text{O}_2 > \text{UV}/\text{TiO}_2/\text{ClO}_3^-$. Yu et al. [7] demonstrated that in UV/ TiO_2 /oxidant systems, decolorization rate constants of C.I. reactive black 5 (RB5) varied with oxidant following the order $\text{NaIO}_4 > \text{Na}_2\text{S}_2\text{O}_8 > \text{NaBrO}_3 > \text{H}_2\text{O}_2 > \text{absence of}$

oxidant. Although several studies have examined the effects of adding oxidant to UV/photocatalyst systems, few data are available on the degradation ability of UV/oxidant systems. Hence, the decolorization performance of RR198 by H_2O_2 , $\text{Na}_2\text{S}_2\text{O}_8$, NaBrO_3 , and NaIO_4 under UV irradiation was evaluated. The goals of this study were to (i) determine the influences of oxidant dosage; (ii) compare the efficiencies of 254 and 365 nm irradiation, (iii) evaluate the inhibitions of adding ethanol to UV/oxidant systems, and (iv) calculate the synergistic effects of NaIO_4 addition in UV/ TiO_2 system.

Experimental

Materials

The parent compound, RR198, was obtained from Aldrich Chemical Company, and was used without further purification. The formula, molecular weight and maximum wavelength of light absorption of RR198 were $\text{C}_{27}\text{H}_{18}\text{ClN}_7\text{Na}_4\text{O}_{15}\text{S}_5$, 967.5 g/mol and 520 nm, respectively. Fig. 1 shows the chemical structure of RR198. TiO_2 (Degussa P-25) was used directly without treatment. The crystal phases of Degussa P-25 were anatase/rutile at a ratio of 3/1. The specific surface area, average particle size and pH of zero point of charge (pH_{zpc}) of Degussa P-25 were $50 \text{ m}^2/\text{g}$, 30 nm and 6.6 [10]. H_2O_2 , $\text{Na}_2\text{S}_2\text{O}_8$, NaBrO_3 , and NaIO_4 were applied as oxidants to evaluate the decolorization efficiency. The hydroxyl radical scavenger was ethanol ($\text{C}_2\text{H}_5\text{OH}$). The pH of the solution was controlled by adding HNO_3 and NaOH via an automatic titrator. HNO_3 , NaOH , H_2O_2 , $\text{Na}_2\text{S}_2\text{O}_8$, NaBrO_3 , NaIO_4 , and ethanol were obtained from Merck. All reagents were of analytical grade and used as purchased.

Decolorization experiments

The RR198 concentration was 20 mg/L in all experiments. The dosage of TiO_2 in UV/ TiO_2 and UV/ TiO_2 / NaIO_4 systems was 1 g/L. The solution was maintained at pH 7 during the reaction. Decolorization experiments were conducted in a 3 L hollow cylindrical glass reactor. The inner tube was made of quartz, and an 8 W,

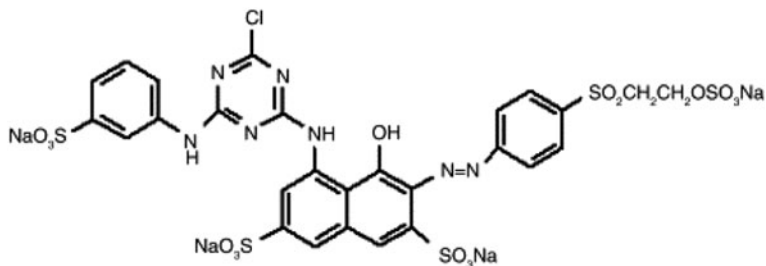


Fig. 1 Chemical structure of RR198

254 nm or 365 nm UV-lamp (Philips) was placed inside the tube as the source of irradiation. In the ethanol addition experiments, 1,200 mg/L ethanol was added. All of the systems were stirred continuously at 300 rpm and the temperature was controlled at 25 °C. A 15-mL aliquot was withdrawn from the photoreactor at pre-specified intervals. The RR198 concentration was measured using a spectrophotometer (Hitachi U-2001) at 520 nm. The decolorization efficiency was calculated from the difference between the dye concentrations before and after the experiment.

Results and discussion

Effects of oxidant dosage

Figs. 2 and 3 plot the effects of $\text{Na}_2\text{S}_2\text{O}_8$ dosage in a UV/ $\text{Na}_2\text{S}_2\text{O}_8$ system under 254 and 365 nm irradiation. For both wavelengths, 254 and 365 nm, the experimental results show that the decolorization rate constant (k) increased with the dose of $\text{Na}_2\text{S}_2\text{O}_8$ (Table 1). The k values of RR198 in the UV/oxidant systems followed pseudo-first order kinetics, and various works have found that dye decolorization rates can generally be approximated using pseudo-first order kinetics [7, 11–15]. Ivanov et al. [16] indicated that persulfate ions undergo photolysis under light irradiation, forming sulfate ion radicals (Eq. 1). Sulfate ion radicals react with water molecules to generate hydroxyl radicals (HO^\bullet) (Eq. 2) [17]. According to Eqs. 1 and 2, increasing the amounts of $\text{S}_2\text{O}_8^{2-}$ increased the additional sulfate ion radicals and HO^\bullet , accelerating decolorization. Since the k values increased with the dose of $\text{Na}_2\text{S}_2\text{O}_8$, the effects of HO^\bullet scavenging by $\text{S}_2\text{O}_8^{2-}$ were not observed herein (Eq. 3). Peternel et al. [18] indicated the optimal operating conditions of UV/ $\text{S}_2\text{O}_8^{2-}$ to decolorize C.I. reactive red 45 were pH 5 to pH 7 and $[\text{S}_2\text{O}_8^{2-}] = 15$ mM.

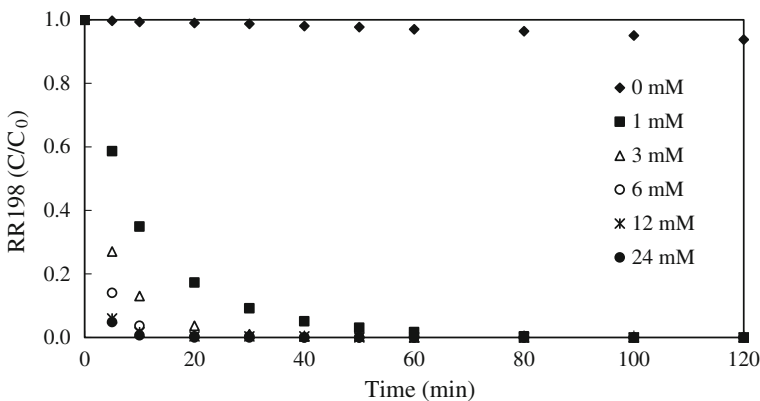


Fig. 2 Effects of $\text{Na}_2\text{S}_2\text{O}_8$ dosage under 254 nm irradiation ($[\text{RR198}] = 20$ mg/L and pH = 7)

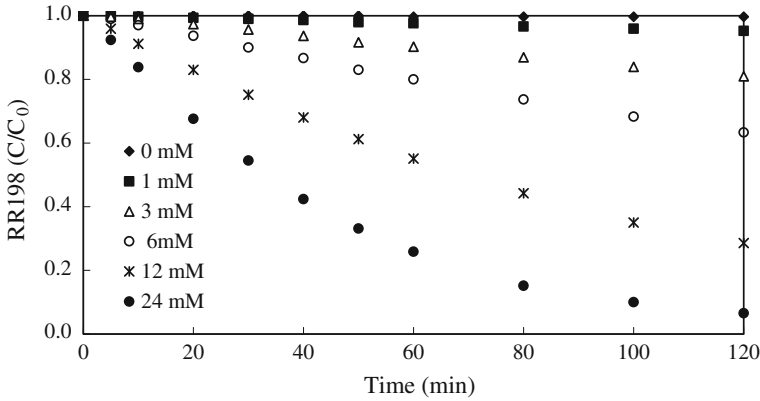
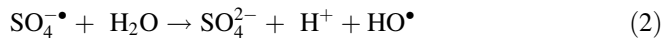
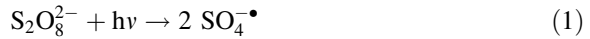
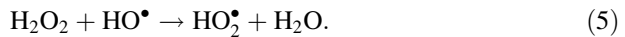


Fig. 3 Effects of $\text{Na}_2\text{S}_2\text{O}_8$ dosage under 365 nm irradiation ($[\text{RR198}] = 20 \text{ mg/L}$ and $\text{pH} = 7$)



In a UV/ H_2O_2 system, the decolorization efficiency increases with the H_2O_2 concentration from 1 to 6 mM; however, at concentrations >6 mM, no further improvement occurred (Table 1). Eq. 4 describes the reaction of the UV/ H_2O_2 system. Moreover, H_2O_2 may act as a HO^{\bullet} scavenger to form hydroperoxyl radicals, which detrimentally affect photolysis (Eq. 5). Numerous works have indicated that the rate of degradation of organic compounds increases with H_2O_2 concentration up to a threshold; as the H_2O_2 concentration increases further, the degradation efficiency declines as H_2O_2 scavenges HO^{\bullet} , when H_2O_2 is present at a high concentration [13, 19, 20], generating hydroperoxyl radicals, which have lower oxidation capacity than hydroxyl radicals. Accordingly, the dose of H_2O_2 in the UV/ H_2O_2 system must be carefully controlled.



Under 254 nm irradiation, the k values obtained by adding 1, 3, 6, 12, and 24 mM NaBrO_3 to UV/ NaBrO_3 were 2.38, 7.34, 13.37, 24.71, and 35.68 h^{-1} (Table 1). Zuo and Katsumura [21] investigated the mechanisms of UV/ NaBrO_3 , as in Eqs. 6–11. However, the scavenging of HO^{\bullet} by BrO_3^- was not identified herein (Eq. 11). Hence, the k values increased with the dose of NaBrO_3 (Table 1).

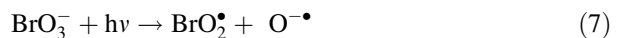
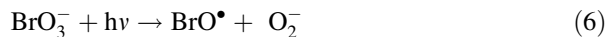
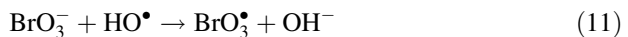


Table 1 Effects of oxidant dosage on the pseudo-first order decolorization rate constants (k , h^{-1}) in UV/oxidant systems ($[\text{RR198}] = 20 \text{ mg/L}$, $\text{pH} = 7$ and $25 \text{ }^\circ\text{C}$)

	1 mM	3 mM	6 mM	12 mM	24 mM
254 nm					
Na ₂ S ₂ O ₈					
k	5.05 (0.08)*	9.73	17.93	26.41	31.19
r^2	0.978 (0.996)*	0.961	0.977	0.947	0.981
H ₂ O ₂					
k	4.82 (0.05)*	8.71	10.24	9.81	7.00
r^2	0.992 (0.973)*	0.994	0.995	0.994	0.996
NaBrO ₃					
k	2.38 (1.03)*	7.34	13.37	24.71	35.68
r^2	0.997 (0.994)*	0.991	0.960	0.978	0.985
NaIO ₄					
k	14.08 (1.94)*	15.47	11.90	10.34	7.37
r^2	0.999 (0.938)*	0.990	0.988	0.983	0.991
365 nm					
Na ₂ S ₂ O ₈					
k	0.02	0.10	0.23	0.62	1.36
r^2	0.992	0.996	0.998	0.998	0.997
NaIO ₄					
k	0.52	1.00	1.51	2.17	3.34
r^2	0.999	0.998	0.999	0.998	0.992

* 1,200 mg/L C₂H₅OH addition

Weavers et al. [5] proposed Eq. 10 and Eqs. 12–16 as UV/NaIO₄ mechanisms. The high activity of UV/IO₄⁻ is attributable to the formation of numerous highly reactive radicals (O^{-•}, HO[•], IO₃[•], and IO₄[•]) and non-radical (O₃, IO₄⁻, and IO₃⁻) intermediates. Increasing the concentration of IO₄⁻ increases the number of radicals formed. Under 254 nm irradiation, the k value increases with NaIO₄ dose up to a threshold (3 mM); as the NaIO₄ dose increases beyond this threshold, the degradation efficiency declines as NaIO₄ scavenges HO[•] (Eq. 16) when NaIO₄ is present at a high concentration (Table 1). However, the scavenging of HO[•] by IO₄⁻ was not observed under 365 nm irradiation. Hence, the k values increased with the NaIO₄ dose. Lee and Yoon [22] found that the k values of RB5 increased with low IO₄⁻ concentrations (0–5 mM), but slightly fell as the concentration of IO₄⁻ increased to high values (>5 mM) in a UV/NaIO₄ system. The excess IO₄⁻ ions in a

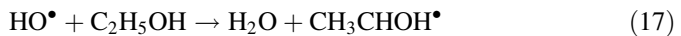
solution may scavenge HO^\bullet , which may attack dye; therefore, the decolorization rate declined when a high concentration of IO_4^- was added [22].



Effects of UV wavelength and ethanol addition

The wavelength of UVA and UVC was 320–400 and 200–290 nm, respectively. This study selected 254 and 365 nm UV light to clarify the effects of UVA and UVC in UV/ TiO_2 and UV/oxidant systems. All oxidants that were adopted herein can be photolyzed under 254 nm irradiation; however, only $\text{Na}_2\text{S}_2\text{O}_8$ and NaIO_4 can be photolyzed by 365 nm irradiation (Table 1). The k value of 254 nm exceeded that of 365 nm. Lo et al. [3] and Lin et al. [4] both indicated that the photodegradation efficiency of 4-chlorophenol increased with a decrease in light wavelength. Additionally, the k values of C.I. reactive red 2 decolorization by 254 nm irradiation exceeded those by 365 nm in the photo-fenton and photo-Fenton-like systems [23]. The dosage of H_2O_2 and NaIO_4 has a threshold value, suggesting that the HO^\bullet was scavenged by excess oxidant under 254 nm irradiation; however, no such scavenging occurred under 365 nm irradiation. This work suggests that the UV wavelength affected not only the decolorization efficiency but also the optimal dosage of oxidant.

Ethanol is known to act as an HO^\bullet scavenger [11]. Buxton et al. [24] suggested the mechanism on the HO^\bullet scavenger was as Eq. 17. In the experiments of ethanol addition, the concentration of ethanol and oxidant was 26 and 1 mM. On a mole basis, the amount of added ethanol must exceed the amount of oxidant in order to suppress the generated reactive free radicals; therefore, a high ethanol dose (1,200 mg/L) was used. Notably, adding ethanol reduced the decolorization rate in UV/oxidant systems (Fig. 4). At 1,200 mg/L ethanol addition, the k values for UV/ H_2O_2 and UV/ $\text{Na}_2\text{S}_2\text{O}_8$ systems (Table 1) were approximately 100 times less than those in the absence of ethanol, revealing that the decolorization proceeded mainly via HO^\bullet . However, adding 1,200 mg/L ethanol to UV/ NaBrO_3 and UV/ NaIO_4 systems does not completely prevent decolorization. This experimental finding suggests that another reactive species, which does not react with ethanol, participates in decolorization. This species is most likely one of the BrO^\bullet , BrO_2^\bullet , and BrO_3^\bullet that are formed in UV/ NaBrO_3 and the $\text{O}^{-\bullet}$, IO_3^\bullet , IO_4^\bullet , and O_3 that are produced in UV/ NaIO_4 .



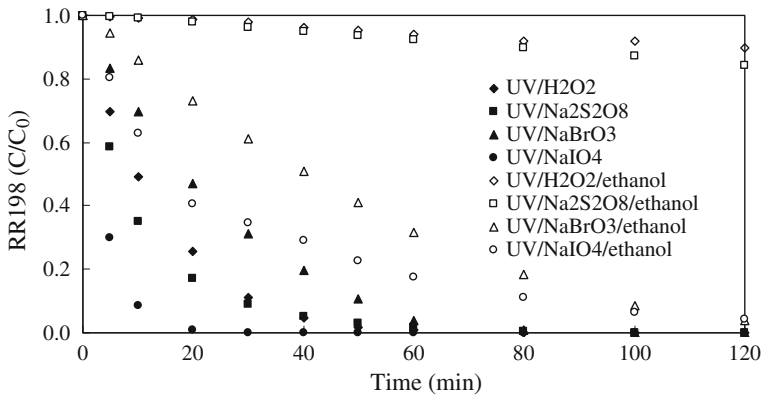


Fig. 4 Effects of ethanol addition in UV/oxidant systems ($[\text{RR198}] = 20 \text{ mg/L}$, $[\text{ethanol}] = 1,200 \text{ mg/L}$, $[\text{oxidant}] = 1 \text{ mM}$, $\text{pH} = 7$ and $\text{UV} = 254 \text{ nm}$)

Effects of added NaIO_4 in UV/TiO_2 system

Under both of 254 and 365 nm irradiation, decolorization rates of RR198 followed the order $\text{UV}/\text{TiO}_2/\text{NaIO}_4 > \text{UV}/\text{NaIO}_4 > \text{UV}/\text{TiO}_2$ (Table 2). Adding inorganic oxidants increased the rate of decolorization by various means, such as, (i) prevention of electron-hole recombination via capturing conduction band electrons; (ii) increased formation of hydroxyl radicals formed, and (iii) production of other species that oxidize intermediate compounds [7]. The net effect is to increase the rate of formation of hydroxyl radicals and, thus, the effective rate of degradation of RR198, according to Eq. 18 [5, 6, 25]. Numerous studies also found that the rate of photodegradation using $\text{UV}/\text{TiO}_2/\text{IO}_4^-$ exceeded that using UV/TiO_2 [6, 9, 25, 26].

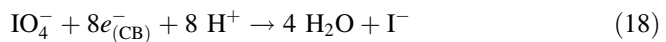


Table 2 Comparisons of the pseudo-first order decolorization rate constants (k , h^{-1}) in UV/TiO_2 , UV/NaIO_4 and $\text{UV}/\text{TiO}_2/\text{NaIO}_4$ systems ($[\text{RR198}] = 20 \text{ mg/L}$, $[\text{TiO}_2] = 1 \text{ g/L}$, $[\text{NaIO}_4] = 1 \text{ mM}$, $\text{pH} = 7$ and $25 \text{ }^\circ\text{C}$)

	UV/TiO_2	UV/NaIO_4	$\text{UV}/\text{TiO}_2/\text{NaIO}_4$
254 nm			
k	1.39	14.08 (7.88)*	Too fast (16.75)*
r^2	0.985	0.999 (0.991)*	-(0.955)*
365 nm			
k	0.50	0.52	11.67
r^2	0.995	0.999	0.985

* 0.5 mM NaIO_4 addition

Conclusion

The decolorization efficiency of RR198 in UV/H₂O₂, UV/Na₂S₂O₈, UV/NaBrO₃, and UV/NaIO₄ systems was determined. All oxidants can be photolyzed by 254 nm irradiation; however, only Na₂S₂O₈ and NaIO₄ can be photolyzed by 365 nm irradiation. At 1 mM oxidant addition, the *k* values followed the order UV/NaIO₄ > UV/Na₂S₂O₈ > UV/H₂O₂ > UV/NaBrO₃. Experimental results indicate that the *k* values increased with the dose of Na₂S₂O₈ and NaBrO₃ under 254 nm irradiation. This study suggests that HO• dominate decolorization in UV/H₂O₂ and UV/Na₂S₂O₈ systems. Additionally, BrO•, BrO₂•, and BrO₃•, which are produced in UV/NaBrO₃, and O⁻•, IO₃•, IO₄• and O₃, which are produced in UV/NaIO₄, are important reactive species in UV/NaBrO₃ and UV/NaIO₄ systems. Addition of NaIO₄ to UV/TiO₂ resulted in quenching of conduction band electrons and generation of reactive radical intermediates, enhancing decolorization of RR198.

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