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Application of photoactivated periodate to the decolorization of reactive dye: reaction parameters and mechanism

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Abstract

The decolorization of the reactive dye, reactive black 5 (RB5), by the photoactivated periodate (UV/IO_4^-) was investigated under ultraviolet (UV) irradiation at 254 nm, and its possible mechanism based on periodate photochemistry was discussed, in order to elucidate the main oxidants involved in the decolorization of RB5 as the reaction parameters, such as the light intensity, periodate concentration, dissolved oxygen, and solution pH, were varied, with the intermittent employment of an $^\bullet OH$ scavenger. The decolorization rate of RB5 increased linearly with increasing light intensity. The decolorization rate was found to increase at low periodate concentrations (0–5 mM), but slightly decrease at higher concentrations (>5 mM). Dissolved oxygen did not have any measurable effect on the decolorization rate of the reactive dye. No pH dependence of the decolorization rate was observed over the pH range of 1.5–10, regardless of the periodate speciation. Based on these results, the main oxidants responsible for the RB5 decolorization with UV 254 were found to be $^\bullet OH$ and $IO_3^\bullet$ ($I^{VI\bullet}$), after considering the six intermediate species which are reported to be produced as a result of periodate photolysis, namely the radical species ($^\bullet OH$, $IO_3^\bullet$, and $IO_4^\bullet$) and the non-radical species (O_3, IO_4^-, and IO_3^-).
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1. Introduction

The effective decolorization of colored effluents has become an important problem in the treatment of wastewater originating from the textile industry. Conventionally, physical (adsorption, filtration, and flotation), chemical (coagulation, oxidation, reduction, and electrolysis), and biological methods have been employed to treat wastewater containing organic dyes and pigments. However, it is difficult to find a convenient method which can effectively treat all types of dyestuff wastewater. In particular, conventional biological treatment alone is not sufficient to treat modern dyes, due to the large degree of aromaticity and stability of the molecules involved [1,2]. Accordingly, advanced oxidation processes (AOPs) are under investigation as an alternative means of overcoming the limitations of the conventional techniques [3–5].

AOPs which generate $^\bullet OH$ as a non-selective oxidant, are emerging technologies in the effective degradation of the recalcitrant organic pollutants present in wastewater. The UV/O_3, UV/H_2O_2, UV/TiO_2, and $h\nu$/Fe(III)/H_2O_2 pro-

cesses (photo Fenton process), to mention only a few of the AOPs currently being studied employ ultraviolet irradiation of an aqueous solution, in order to produce $^\bullet OH$ or enhance the $^\bullet OH$ production rate.

Recently, the photoactivated periodate (UV/IO_4^-) system was proposed as a novel advanced oxidation technology, and which was shown to remove aqueous organic compounds in several studies [6–11]. Although, the periodate ion (IO_4^-) itself is a well-known oxidant, whose reduction potential was reported to be +1.60 V, oxidation reactions initiated by this ion are known to be selective and significantly slow compared with those involving $^\bullet OH$ [12]. Periodate is effectively applicable to the oxidation of the compounds in which the carbonyl group or hydroxyl group is adjacent to a second carbonyl or hydroxyl group, such as α-diols, α-ketols, α-diketones, and α-ketonic aldehydes. Despite the selective reactivities of periodate to substrates with specific functional groups, UV light can greatly enhance the reactivity and reduce the selectivity of the periodate oxidation reaction. This is because periodate produces $^\bullet OH$ and other reactive radical and non-radical oxidants, through the photodecomposition which occurs when it absorbs UV light at wavelengths of less than 300 nm. Although the photochemistry of the periodate ion in the aqueous phase has been extensively investigated,
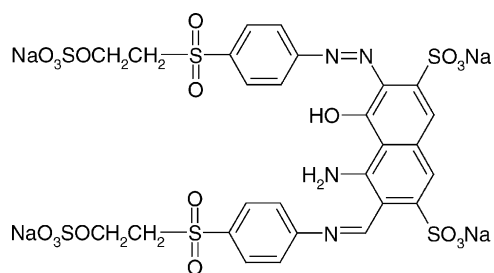
Fig. 1. Chemical structure of reactive black 5.

and has been relatively well elucidated in many previous studies [13–17], there are still few reports on the application of photoactivated periodate to water treatment. In particular, no research has been done to investigate the characteristics of the dyestuff decolorization process induced by the photoactivated periodate. Hence, in this study, we attempted to investigate the decolorization of wastewater containing RB5 in the photoactivated periodate system, as the reaction parameters affecting the decolorization of RB5, such as the light intensity, periodate concentration, dissolved oxygen, and solution pH, were varied, with the intermittent employment of an $^\bullet$OH scavenger. The objectives of this study were two-fold: (1) to investigate the effects of the reaction parameters on the reactive black 5 (RB5, Fig. 1) decolorization process and (2) to elucidate the main oxidants responsible for the decolorization of RB5 in the photoactivated periodate system.

2. Photochemistry of periodate in aqueous solution

The photolysis of periodate and periodic acid in aqueous solution have been extensively studied, and the reactions involved in periodate photolysis have been discussed in a number of studies [13–17]. In spite of the complicated nature of periodate photolysis, Wagner and Strehlow [15] suggested the simple reaction scheme as shown in Table 1. According to this scheme, periodate can be photodecomposed by two photo-initiated pathways. As shown in Table 1, periodate can be photodecomposed to $IO_3^\bullet$ (I^{VI}) and $O^{\bullet-}$ by one

electron transfer (reaction (1) in pathway I) or to IO_3^- (I^{VI}) and $O(^3P)$ by two electron transfer (reaction (3) in pathway II). The $O^{\bullet-}$ generated by reaction (1) is present as its conjugate acid, $^\bullet$OH, in the pH range of <11.9 (pK_a of $^\bullet$OH; [18]). On the other hand, the atomic oxygen, $O(^3P)$ generated by reaction (3) rapidly reacts with dissolved oxygen to produce O_3 by reaction (4) [19]. The primary products of periodate photolysis, such as $^\bullet$OH, $IO_3^\bullet$, and O_3, undergo self-reaction or reactions with other products, as shown in reactions (5)–(10). The reaction mechanism indicated in Table 1 shows that the potential oxidants in the UV/IO_4^- system can be classified into radical species ($^\bullet$OH, $IO_3^\bullet$, and $IO_4^\bullet$) and non-radical species (O_3, IO_4^-, and IO_3^-).

3. Experimental

3.1. Reagents

All reagents used were of reagent grade and used without further purification. Sodium periodate ($NaIO_4$), sodium iodate ($NaIO_3$), RB5, perchloric acid ($HClO_4$), sodium hydroxide (NaOH), and *tert*-butanol (*t*-BuOH) were purchased from Aldrich (USA). All stock solutions were prepared in distilled and deionized water (Barnstead NANO Pure, USA). RB5 has a molecular weight of 991.82 and its chemical structure is depicted in Fig. 1. However, the RB5 reagent used in this study has an ash content of 55% and this was not taken into consideration when expressing the concentration of the RB5. The $100\,mg\,l^{-1}$ aqueous stock solution of RB5 was prepared by adding the dye to distilled deionized water, and fully dissolving it by continuous mixing for at least 5 h. The RB5 solution showed a maximum visible absorption at 597 nm, which remained invariant in the pH range of 1.5–10. The dye stock solution was stored under dark conditions and its variance was spectrophotometrically checked prior to use. The 50 mM stock solutions of IO_4^-, and IO_3^- were prepared by dissolving $NaIO_4$ and $NaIO_3$ in distilled deionized water, respectively, and were stored at room temperature.

Table 1
Reaction mechanism for the periodate photolysis [16]

Photo-initiation	Pathway I		Pathway II	
	$IO_4^-(I^{VII}) + h\nu \Rightarrow IO_3^\bullet(I^{VI}) + O^{\bullet-}$	(1)	$IO_4^-(I^{VII}) + h\nu \Rightarrow IO_3^-(I^V) + O(^3P)$	(3)
	$O^{\bullet-} + H^+ \Leftrightarrow {}^\bullet OH$	(2)	$O_2 + O(^3P) \Rightarrow O_3$	(4)
Subreactions	$^\bullet OH + IO_4^- \Rightarrow OH^- + IO_4^\bullet$	(5)		
	$O_3 + IO_3^\bullet \Rightarrow IO_4^\bullet + O_2$	(6)		
	$2IO_4^\bullet \Leftrightarrow I_2O_8$	(7)		
	$I_2O_8 + H_2O \Rightarrow IO_3^- + IO_4^- + 2H^+ + O_2$	(8)		
	$2IO_3^\bullet \Leftrightarrow I_2O_6$	(9)		
	$I_2O_6 + H_2O \Rightarrow IO_4^- + IO_3^- + 2H^+$	(10)		

3.2. Experimental procedure and apparatus

In the control experiments without UV irradiation, the direct oxidation of the reactive dyes by IO_4^- and IO_3^- (product of periodate oxidation) at various pH conditions was examined in a 50 ml vial, which was protected from all sources of light by covering it with aluminum foil. Neither IO_4^- nor IO_3^- alone in the absence of UV irradiation significantly affected the oxidation of RB5 over the pH range of 1.5–10. UV alone did not affect the oxidation of RB5 either.

All of the experiments using UV light were performed in a 150 ml Pyrex reactor with a quartz window equipped with low-pressure mercury vapor lamps (99% emission at 253.7 nm, Philips, USA). The reactor was sealed with a rubber septum, and gas purging was performed with a needle-type diffuser. Hundred milliliter of reaction solution was used and the solution temperature was controlled by means of a thermostatically controlled water bath. The lamps were preheated for about 30 min in order to stabilize them prior to the experiment. The light intensity was controlled by adjusting the distance between the lamp and the reactor. The incident photon flow (Einstein $l^{-1} s^{-1}$) was measured using ferrioxalate actinometry [20]. The photolytic production of Fe(II) was limited to <10% of the initial amounts of Fe(III) to insure complete light absorption by the ferrioxalate actinometer. An overall quantum yield for the ferrioxalate photolysis of 1.2 was used in the calculation [21]. The initial pH was adjusted by adding an appropriate amount of 0.1N $HClO_4$, and NaOH solutions. The pH was not controlled during the reaction time. The pH of the reaction solution slightly dropped during the reaction, but no significant variance was observed. The reaction temperature was maintained at $20 \pm 0.2\,^{\circ}C$ during the reaction, and mixing was performed by means of a vigorous magnetic stirrer.

The general experimental procedure was as follows. The 50 mg l^{-1} dye solutions for the experiments were prepared by adjusting the solution pH. The periodate concentration of the solution was adjusted by adding concentrated IO_4^- stock solution. The prepared reaction solution was put into a thermostatically controlled container, in order to adjust the solution temperature to 20 °C. Finally, the reaction was started by applying irradiation. Samples were withdrawn at predetermined intervals and quickly analyzed using a spectrophotometer.
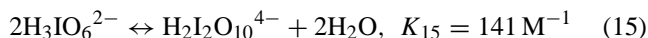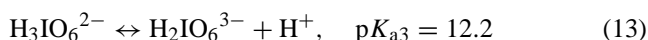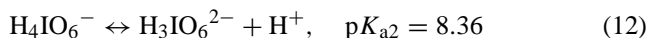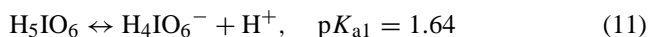
3.3. Analysis

The Fe(II) produced in the ferrioxalate actinometry measurement was analyzed using the 1,10-phenanthroline method ($\varepsilon_{510} = 11\,050\,M^{-1}\,cm^{-1}$ for Fe(II)–phenanthroline complex; [22]). The decolorization of the reactive dye was analyzed by measuring the visible light absorbance at the maximum absorption wavelength (λ_{max}) with a Hewlett-Packard 8452 diode array spectrophotometer (USA). The solution pH was measured by means of an Orion 710A pH meter (USA).

4. Results and discussion

4.1. Periodate speciation

The various I^{VII} species (H_5IO_6, $H_4IO_6^-$, $H_3IO_6^{2-}$, IO_4^-, $H_2I_2O_{10}^{4-}$, and $H_2IO_6^{3-}$) coexist in an aqueous solution of $NaIO_4$. The equilibria among the species are presented in Eqs. (11)–(15) [6].

$$H_5IO_6 \leftrightarrow H_4IO_6^- + H^+, \quad pK_{a1} = 1.64 \tag{11}$$

$$H_4IO_6^- \leftrightarrow H_3IO_6^{2-} + H^+, \quad pK_{a2} = 8.36 \tag{12}$$

$$H_3IO_6^{2-} \leftrightarrow H_2IO_6^{3-} + H^+, \quad pK_{a3} = 12.2 \tag{13}$$

$$H_4IO_6^- \leftrightarrow IO_4^- + 2H_2O, \quad K_{14} = 40 \tag{14}$$

$$2H_3IO_6^{2-} \leftrightarrow H_2I_2O_{10}^{4-} + 2H_2O, \quad K_{15} = 141\,M^{-1} \tag{15}$$

The speciation of the IO_4^- solution was calculated using the Mathematica 4.0 program (Wolfram Research, USA) on the basis of the above equations and constants. Fig. 2 shows the concentration–pH profile of the I^{VII} species for an $NaIO_4$ concentration of 0.5 mM. IO_4^- species dominated at pH <8, whereas the dimerized form, $H_2I_2O_{10}^{4-}$, was the dominant species at a pH higher than 8.

4.2. Effects of the reaction parameters

Fig. 3 shows the RB5 decomposition produced by the UV/IO_4^- system while varying the incident photon flow
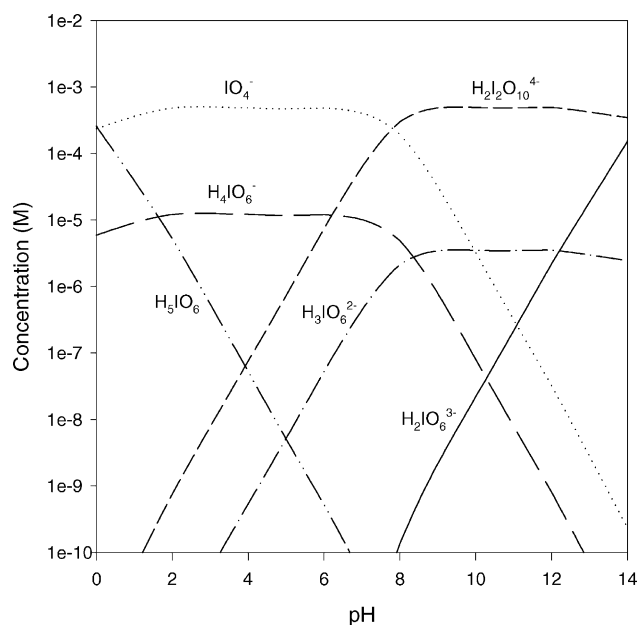

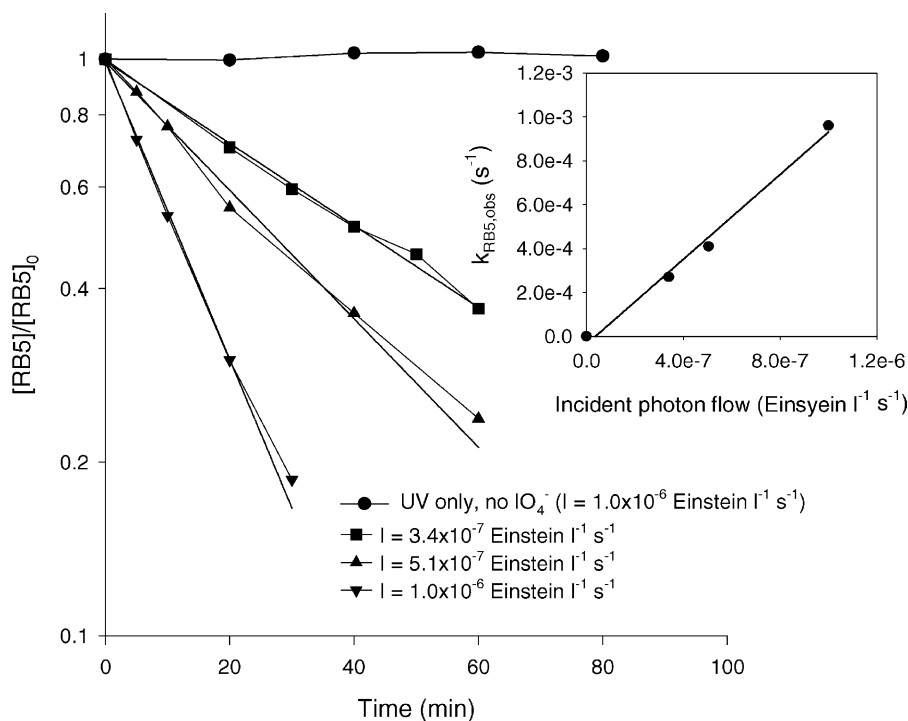Fig. 2. Speciation of 0.5 mM periodate.

Fig. 3. Effect of light intensity (I) on RB5 degradation: $[IO_4^-]_0 = 0.5\,mM$, $[RB5]_0 = 50\,mg\,l^{-1}$, $pH_0 = 7.0$. Light source = low-pressure mercury lamp (99% emission at 253.7 nm).

(Einstein $l^{-1}\,s^{-1}$). In the absence of periodate, UV irradiation alone had no measurable effect on the decolorization of RB5. The half-life of RB5 was approximately 10 min under the incident photon irradiance of 1.0×10^{-6} Einstein $l^{-1}\,s^{-1}$, and decreased with decreasing incident photon flow. As shown in Fig. 3, the RB5 decomposition rate obeyed pseudo-first-order kinetics and the observed rate constant of RB5 decomposition, $k_{obs,RB5}$ (s^{-1}), can be readily determined by Eq. (16).

$$-\frac{d\ln[RB5]}{dt} = k_{obs,RB5} \tag{16}$$

The inset of Fig. 3 shows the linear increase of $k_{obs,RB5}$ with respect to the increase of incident photon flow. This linearity results from the direct relationship between the formation of the main oxidants responsible for the RB5 decolorization and the photolysis of IO_4^-.

Fig. 4 shows the effect of initial periodate concentration on the RB5 decolorization process over the range of periodate concentration of 0.25–15 mM. The RB5 decolorization rate followed pseudo-first-order kinetics, regardless of the initial concentration of periodate. As shown in Fig. 4, the $k_{RB5,obs}$ value increased with increasing initial periodate concentration up to 5 mM, but somewhat decreased at higher IO_4^- concentration. This result can be explained by the fact that at low periodate concentration, the increased photolysis of periodate produces more $^{\bullet}OH$, which reacts with RB5, resulting in an increase of $k_{RB5,obs}$. On the other hand, at high periodate concentration, $k_{RB5,obs}$ decreases with increasing

periodate concentration, because of the competitive reaction involving periodate itself, since periodate can act as an $^{\bullet}OH$ scavenger by reaction (5) (Table 1) whose rate constant was reported as being $4.8 \times 10^8\,M^{-1}\,s^{-1}$ [23].

The effects of gas purging and the presence of an $^{\bullet}OH$ scavenger (t-BuOH) were investigated and the results are
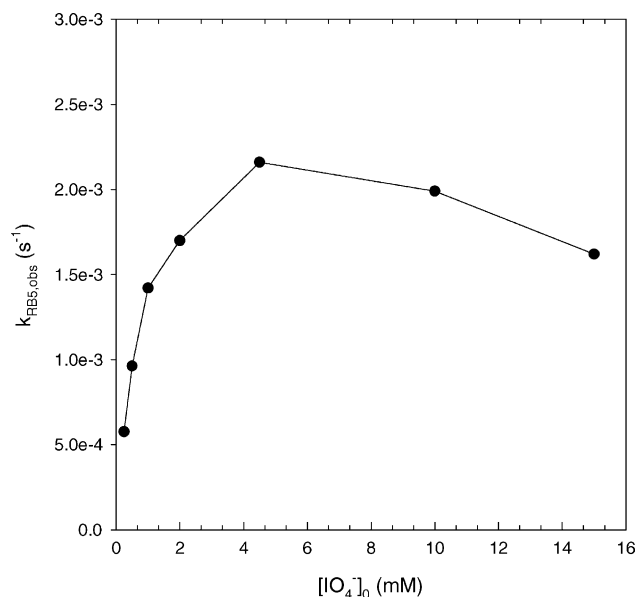

Fig. 4. Effect of initial periodate concentration on RB5 degradation. $[RB5]_0 = 50\,mg\,l^{-1}$, $pH_0 = 7.0$. Incident photon flow $= 1.0 \times 10^{-6}$ Einstein $l^{-1}\,s^{-1}$ (99% emission at 253.7 nm).
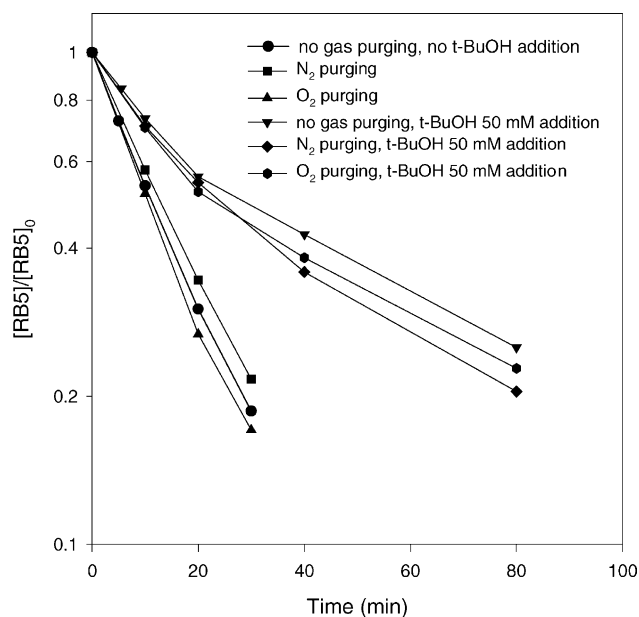
Fig. 5. Effect of gas purging and hydroxyl radical scavenger on RB5 degradation: $[IO_4^-]_0 = 0.5 \, \text{mM}$, $[RB5]_0 = 50 \, \text{mg} \, l^{-1}$, $pH_0 = 7.0$. Light intensity $= 1.0 \times 10^{-6} \, \text{Einstein} \, l^{-1} \, s^{-1}$ (99% emission at 253.7 nm).
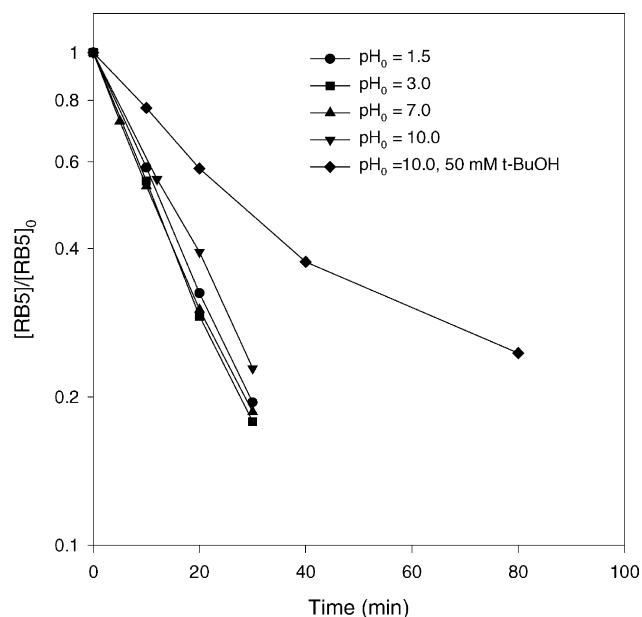

Fig. 6. Effect of initial pH on RB5 degradation: $[IO_4^-]_0 = 0.5 \, \text{mM}$, $[RB5]_0 = 50 \, \text{mg} \, l^{-1}$. Light intensity $= 1.0 \times 10^{-6} \, \text{Einstein} \, l^{-1} \, s^{-1}$ (99% emission at 253.7 nm).

presented in Fig. 5. As shown in Fig. 5, the dissolved oxygen did not have a significant effect on the RB5 decolorization rate. On the other hand, the RB5 decolorization rate was significantly reduced by the addition of excess *t*-BuOH (50 mM), in order to scavenge all the •OHs produced in the system. As shown in Fig. 5, the half-life of RB5 doubled to about 20 min in the presence of excess *t*-BuOH. This indicates that •OH was one of the main oxidants responsible for RB5 decolorization.

Fig. 6 shows the effect of pH on the RB5 decolorization process. As shown in Fig. 6, RB5 decolorization was not significantly affected by varying the pH. The main species of periodate is IO_4^- between pH 1 and 8, while it is $H_2I_2O_{10}^{4-}$ at pH 10. The observation that the RB5 decolorization rates at pH 10 are similar to those at pH 7, even in the presence of *t*-BuOH, indicates that $H_2I_2O_{10}^{4-}$ has a similar photochemistry to IO_4^-, as reported by Kläning and Sehested, since the dimerized form ($H_2I_2O_{10}^{4-}$) is the dominant species at pH 10 (Fig. 1 and [13]). Kläning and Sehested suggested that $H_2I_2O_{10}^{4-}$ was photodecomposed to •OH and the corresponding $I^{VI}\bullet$ radical, as described in reaction (17). The precise chemical structure for $I^{VI}\bullet$ in Eq. (17) was not reported by Kläning and Sehested, although it may be $IO_3\bullet$.

$$H_2I_2O_{10}^{4-}(I^{VII}) + h\nu \rightarrow \, ^\bullet OH + I^{VI\bullet} \tag{17}$$

4.3. Elucidation of the main oxidants for RB5 decolorization

The radical species (•OH, $IO_3^\bullet$ and $IO_4^\bullet$) and the non-radical species (O_3, IO_4^- and IO_3^-) described in

the reaction mechanism given in Table 1 were considered as potential candidates for the decolorization of RB5 in the UV/IO_4^- system. The influences of each oxidant on the decolorization of RB5 were subsequently described in Figs. 2–5.

As mentioned earlier, the control experiments without UV irradiation showed that neither IO_4^- nor IO_3^- influenced the decolorization of RB5. RB5 was slightly decolorized at pH < 5 by its direct reaction with IO_4^- (data not shown), however, its half-life was about 3 h, which represents a much longer decolorization time, compared with that observed in the case of photoactivated periodate (10 min).

The result shown in Fig. 4 indirectly demonstrated that $IO_4^\bullet$ was not involved in the RB5 decolorization process. This is because if $IO_4^\bullet$ is one of the oxidants responsible for the decolorization of RB5, then $k_{RB5,obs}$ should be proportional to the photon flow absorbed by IO_4^- (I_{a,IO_4^-}) in Eq. (18)), which can be calculated based on Beer's Law.

$$k_{RB5,obs} \propto I_{a,IO_4^-} = I_0(1 - 10^{-\varepsilon_{IO_4^-} L[IO_4^-]}) \tag{18}$$

where, I_0 is the incident photon flow (Einstein $l^{-1} \, s^{-1}$), $\varepsilon_{IO_4^-}$ the molar absorption coefficient of IO_4^- at 254 nm ($M^{-1} \, cm^{-1}$), and L the optical pathlength (cm)

Based on Eq. (18), it would be expected for the value of $k_{RB5,obs}$ to show a logarithmic increase with increasing periodate concentration. However, the maximum value of $k_{RB5,obs}$ was observed at $[IO_4^-] = 5 \, \text{mM}$. This observation can be interpreted by that IO_4^- itself reacts as a •OH scavenger, as described in reaction (5), and $IO_4^\bullet$ produced

by reaction (5) was not involved in the decolorization of RB5. Therefore, increasing IO_4^- concentration does not only enhance $^\bullet OH$ production by Eq. (18) but also linearly increase scavenging rate of the produced $^\bullet OH$. However, the exact quantitative interpretation requires a more detailed information about the $^\bullet OH$ production and scavenging in this system such as the quantum yield for the IO_4^- photolysis, and the rate constant of the reaction between $^\bullet OH$ and RB5.

The fact that dissolved oxygen had no effect on the decolorization of RB5 provides indirect evidence that the O_3 produced via reaction (4) does not significantly influence the RB5 decolorization process (Fig. 5). According to reaction mechanism II, $O(^3P)$ is produced as a result of the photolysis of periodate, and this reacts with oxygen to produce ozone (reaction (4)) which is certainly effective in bringing about the decolorization of RB5. If no oxygen is present, $O(^3P)$ can react with water to produce the $^\bullet OH$ (reaction (19), [24]) which is also highly effective in instigating the decolorization of RB5.

$$O(^3P) + H_2O \rightarrow 2^\bullet OH \tag{19}$$

Therefore, as shown in Fig. 5, no enhancement of the RB5 decolorization process in the presence of dissolved oxygen, is possible if the rate of the $^\bullet OH$ induced decolorization via reaction (19) is similar to that of the decolorization produced by O_3 resulting from reaction (4).

However, the RB5 decolorization rate was not significantly affected by the presence of dissolved oxygen, even in the presence of 50 mM t-BuOH (Fig. 5). This observation contrasts with the previous explanation based on reaction mechanism I, indicating that $O(^3P)$ is not measurably produced in the system.

On the other hand, the significant reduction of the RB5 decolorization rate in the presence of t-BuOH supported the hypothesis that $^\bullet OH$ is one of the main oxidants responsible for the decolorization of the dye. Another remaining oxidant out of the six possible species is $IO_3^\bullet$ which can be produced as a result of the photolysis of periodate, according to reaction mechanisms I and II, since all other species responsible for the decolorization of RB5 other than $IO_3^\bullet$ were considered or eliminated. $IO_3^\bullet$ can be reduced to IO_3^- as part of the process of decolorizing the dye molecule selectively, as described in reaction (20).

$$IO_3^\bullet(I^{VI}) + \text{dye molecule}$$
$$\rightarrow IO_3^-(I^V) + \text{oxidized product} \tag{20}$$
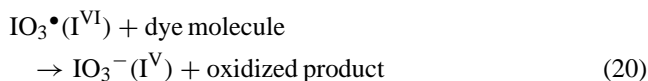
Therefore, the results of this study show that the decolorization of RB5 was achieved only by means of reaction mechanism I in Table 1. This explanation was also supported by the result shown in Fig. 6, in which the RB5 decolorization rates at pH 10 were similar to those at pH 7, in both the absence and presence of t-BuOH, since $H_2I_2O_{10}^{4-}$ was photodecomposed by a mechanism similar to reaction pathway I in the case of IO_4^- photolysis.

4.4. Implications of UV/IO_4^- system to wastewater treatment

Since the UV/IO_4^- system is a kind of novel AOP, in which it is possible to produce not only $^\bullet OH$, but also $IO_3^\bullet$, the UV/IO_4^- system can be applied not only to the decolorization of aqueous pollutants, but also to the removal of chemical oxygen demand (COD) or total organic carbon (TOC) in contaminated water [6,9]. Considering its high molar absorption coefficient (max. value $\gg 8000 \, M^{-1} \, cm^{-1}$ at 222 nm; $1500 \, M^{-1} \, cm^{-1}$ at 254 nm) and the wide range of pH for which it can be used, the UV/IO_4^- system can be more effective in bringing about homogeneous photocatalytic oxidation, as compared with the UV/H_2O_2, UV/TiO_2 and (photo) Fenton systems, etc.

However, the UV/IO_4^- system has several limitations which needs to be overcome. First, a method of regenerating IO_4^- should be put in place since, otherwise, IO_4^- (I^{VII}) is eventually reduced to IO_3^- (I^V), which then does not produce the oxidative species any more (Table 1). The regeneration of IO_3^- to IO_4^- is therefore essential for sustaining the wastewater treatment. Another important problem is the removal of the remaining reagents (IO_4^- and IO_3^-) after treatment. Although no discharge requirements for iodine species have yet been established, toxicity data for these species need to be evaluated. The electrochemical regeneration of IO_4^- or the use of the immobilized UV/IO_4^- system constitute potential solutions to this problem.

5. Conclusion

This study demonstrated that the UV/IO_4^- system constituted an effective process for the treatment of the colored wastewater containing the reactive dye, RB5. The principle results obtained by varying the reaction parameters were as follows. Increasing the light intensity linearly increased the RB5 decolorization rate. The RB5 decolorization rate linearly increased with increasing periodate concentration, until IO_4^- itself became a significant $^\bullet OH$ scavenger. The RB5 decolorization rate was not dependent on pH or dissolved oxygen. From the subsequent discussions about the effects of each reactive species (radical species: $^\bullet OH$, $IO_3^\bullet$, $IO_4^\bullet$, non-radical species: O_3, IO_4^-, and IO_3^-) on the RB5 decolorization process, $^\bullet OH$ and $IO_3^\bullet$ were suggested to be the main oxidants responsible for the decolorization of RB5, which is consistent with reaction mechanism I. Consequently, the compound-selective oxidant ($I^{VI\bullet}$) and the non-selective oxidant, $^\bullet OH$, effectively decolorized RB5 over the entire pH range in an aqueous phase.

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