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OPTIMIZATION OF OPERATIONAL PARAMETERS FOR DECOLORIZATION AND DEGRADATION OF C. I. REACTIVE BLUE 29 BY OZONE

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ABSTRACT

Because conventional wastewater treatment of effluent containing anthraquinone dye causes notable environmental problems, it is important to find effective alternative methods for dye removal. This study evaluated the efficacy of ozonation for dye removal and Chemical Oxygen Demand reduction and identified optimal operational conditions for parameters such as pH, contact time and concentration of C.I. Reactive Blue 29 dye in a semi-batch reactor. Values of pH between 3 and 11 and contact times between 15 and 120 minutes were investigated. Dye concentrations were based on the American Dye Manufacture Institute standards and ranged from 1000 to 5000. Although results showed that Chemical Oxygen Demand removal by ozone alone was not very efficient (58%), ozonation proved to be an efficient method for decolorizing Reactive Blue 29 (96%). pH was found to significantly influence the effectiveness of Chemical Oxygen Demand removal, and optimal pH conditions (95% confidence interval) were between 9 and 11. For decolorization, pH adjustment was not necessary. Degradation and decolorization of dye were found to be strongly influenced by the contact time, optimal conditions (95% confidence interval) for degradation and decolorization were 60 and 30 minutes, respectively. The optimal dye concentration was 1000 American Dye Manufacture Institute.

Key words: Decolorization; C.I .Reactive Blue 29; Ozonation; Operational parameters

INTRODUCTION

Textile wastewater generates common environmental problems (Hunger, 2003; Peternal *et al*., 2007; Behnajady *et al*., 2009) because it contains dyes, typically organic compounds with complex structures that are highly soluble in water and easily hydrolyzed. Approximately 20% - 40% of these dyes remain in the effluent (Song *et al*., 2008) and some are known to be toxic and carcinogenic (Epolito *et al*., 2005; Lung-chyuan chen *et al*, 2009; Novotny *et al*., 2006; Mohamadian Fazli *et al*., 2010).

Conventional wastewater treatment methods are not well suited for oxidizing textile wastewater because the ratio of BOD to COD is in the range of 0.1-0.25, which indicates a large amount of non-biodegradable organic matter (Kdasi *et al*., 2004; Georgiou *et al*., 2004; Deng *et al*., 2008; Mehrali *et al*., 2010). Physical processes such as adsorption onto carbon and reverse osmosis are non-destructive and merely transfer the pollutants

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to other media. Chemical methods also have significant restrictions (Robinson *et al.,* 2001; Zou and Zhu, 2007).

Advanced Oxidation Processes (AOPs) have shown the greatest promise for mitigating these problems and treating textile wastewater (Forgacs *et al*., 2004; Zou and Zhu, 2007; Arsalan, 2007). The aim of any AOP system is the generation of OH^- radicals, which have the greatest electrochemical oxidation potential (EOP) among oxidizing agents (Kdasi *et al*., 2004; Movahedian *et al*., 2009).

Ozone is one of the most important oxidizing agents because it is highly reactive with organic compounds (Chu and Wai, 2000; Zou and Zhu, 2007; Wu *et al*., 2007). Ozone can effectively decolorize dye wastewater by two reaction pathways: a direct path corresponding to molecular activity and an indirect path corresponding to the activity of the free radical, in acidic pH, the ozone is available as molecular and in alkaline pH , ozone decomposes to OH^- radicals (Masten and Davies, 1994; Kdasi *et al*., 2004; Shu and Chang, 2005; Lee *et al*., 2007).

According to Jiangning Wu, ozonation has been very effective in decolorizing textile effluents (Wu *et al*., 2007). Evidence from Turkey suggested that the decolorization of dyestuff wastewater by ozone treatment was excellent (Turhan and Turgut, 2009); Srinvasan *et al*. documented upto 97% dye color removal by ozonation (Srinivasan *et al*., 2009); Amat reported that ozone was very effective for decolorizing textile effluents (Amat *et al*., 2007). However, it has been reported that conventional ozonation process has not been not very efficient for COD removal by ozone alone (Szpyrkowicz *et al*., 2001).

The aim of this study was to investigate the application of ozone for the decolorization and degradation of anthraquinone dye C.I. Reactive Blue 29. Because ozone performance is influenced by parameters such as pH, treatment time and pollutant concentration, it is necessary to identify optimal process parameters through laboratory tests. Thus, a series of experiments were conducted in order to establish optimal values of these parameters.

MATERIALS AND METHODS

Reagents

C.I. Reactive Blue 29 was purchased from Dyestar Hoechst Corporation (Frankfurt, Germany). The chemical structure of the dye is shown in Fig.1. Other chemicals were sulfuric acid (H_2SO_4, 96%), ammonium Ferro sulfate, potassium dichromate, potassium iodide and sodium hydroxide, all of which were purchased from Merck, Germany.

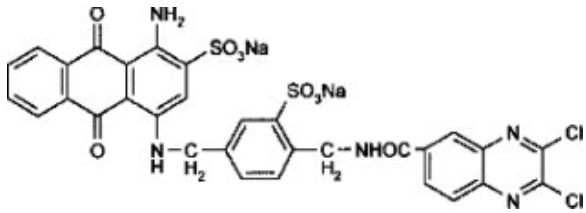

Fig.1: Chemical structure of C.I. Reactive Blue 29

Experimental procedure

As shown in Fig. 2, ozone was generated by a system including an oxygen concentrator followed by an ozone generation (ARDA, model 6-5-11015). The ozone flow rate was adjusted to 160 mg per hour. The unused ozone was taken out of the reactor and collected into a 10% potassium iodide solution, where it was measured by the indigo method (APHA, 2005). Ozonation was performed in a 1 L aluminum column reactor (5 cm diameter and 55 cm high).

All experiments were performed using initial dye concentrations based on the American Dye Manufacture Institute (ADMI) standards (1000, 2000, 3000, 4000 and 5000 ADMI). The initial pH values of model solutions were adjusted between 3 and 11 with the addition of 0.1 M H_2SO_4 or NaOH. The experiments were conducted consecutively for contact times of 15, 30, 45, 60, 75 and 120 minutes. All solutions were prepared with deionized water with conductivity of less than 5 µs/cm.

To determine the frequency of each test and the level of each factor in the context of previous researches, the Standard Deviation was estimated to be 10% (Peternal *et al*., 2007; Zou and Zhu, 2007) and Φ was evaluated based on the equation (1). Then, using graphs for the choice of sample size, the frequency of each test was selected with

a confidence interval of 95% and a power of test of 80%. A mean difference of about 15 or higher between two levels of each factor in the test indicated that the test was statistically meaningful. In this way, the best level for each factor was determined. As the result of the calculations, the frequency of every test was determined to be 4 (Neter and Wasserman, 1974).

$$\Phi = \frac{1}{\sigma}\sqrt{\frac{\sum T_j^2}{r}} = \frac{1}{10}\times\sqrt{\frac{5\times 15^2}{5}} = 1.5 \qquad (1)$$

In which:
σ = Standard Deviation
T_j^2= Mean difference between the two groups
r = Number of groups

Analytical method

pH was measured by pH-Meter, E 520, and color was monitored by the ADMI weighted ordinate method with a DR-5000 spectrophotometer (Hach Co., USA) and measured by Color-ADMI-10048.fm (APHA, 2005). The extent of dye mineralization was assessed using COD, which was measured by standard methods (APHA, 2005). The study was performed according to 'Design and Analysis of Experiments' (Montgomery, 2001) and all calculations were done with SPSS 11.1.

RESULTS

Effect of pH on COD removal

The results of the statistical analysis indicated that the effect of pH on COD was significant (F=42.654, Sig.<0.001). Table 1 shows the multiple comparisons for the effects of pH on COD removal and Fig. 3 shows the effect of pH between 3 and 11.

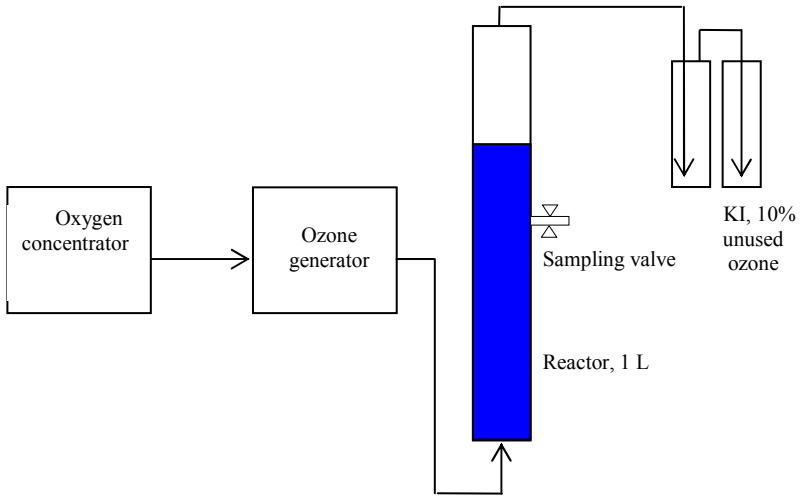

Fig. 2: Schematic diagram of the semi-batch reactor system

Table1: Multiple comparisons for determination of optimal pH for COD removal

(I) pH	(J) pH	Mean Difference (I-J)	S. E.	Sig.	95% Confidence Interval	
					Lower bound	Upper bound
11	3	8.3675(*)	.69165	<0.001	5.9495	10.7855
	5	5.4225(*)	.69165	<0.001	3.0045	7.8405
	7	3.4075(*)	.69165	.004	.9895	5.8255
	9	1.6625	.69165	.268	-.7555	4.0805

The mean difference is significant at the .05 level.
S. E.: Standard Error

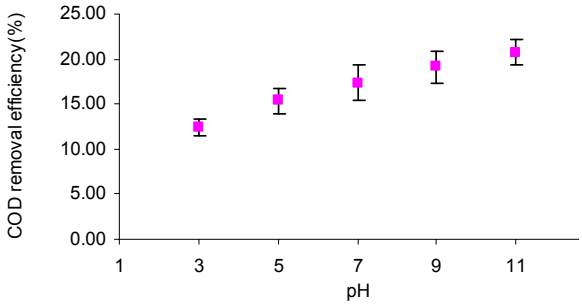

Fig. 3: Effect of pH on COD removal with a 95% confidence interval (T=30 min; ADMI=3000; ozone flow rate: 160mg/h)

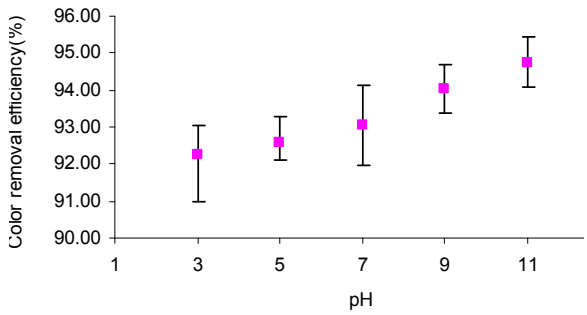

Fig. 4: Effect of pH on color removal with a 95% confidence interval (T=30min; ADMI=3000; ozone flow rate: 160 mg/h)

Effect of pH on color removal

The results of the statistical analysis indicated that the effect of pH on COD was significant (F=6.49, Sig. =0.03). Table 2 shows the multiple comparisons for the effects of pH on color removal and Fig. 4 shows the effects of varying pH between 3 to 11.

Effect of dye concentration on COD removal by ozone

ANOVA results for the effects of dye concentration on COD removal showed that dye concentration had a very significant effect on COD removal (F=136, Sig.<0.001). Table 3 shows the multiple comparisons for the effects of dye concentration on COD removal and Fig. 5 shows the effects of varying dye concentration.

Effect of dye concentration on color removal by ozone

ANOVA results for the effects of dye concentration on color removal showed that dye concentration had a significant effect on color removal (F=5.79, sig=0.005). Table 4 presents the multiple comparisons for the effects of dye concentration on color removal and Fig. 6 shows the effect of dye concentration evaluated by varying the concentration between 1000 and 5000 ADMI at pH=11.

Effect of ozonation contact time on COD removal

Statistical analysis of the experimental data indicated that contact time had a significant effect on COD removal (F= 184.11, Sig.= <0.001).).

Table 2: Multiple comparisons for determination of optimal pH for color removal

(I) pH	(J) pH	Mean Difference (I-J)	S. E.	Sig.	95% Confidence Interval	
					Lower bound	Upper bound
3	5	-.34	.57	0.984	-2.35	1.65
	7	-.82	.57	0.731	-2.82	1.18
	9	-1.80	.57	.089	-3.81	.19
	11	-2.48(*)	.57	.012	-4.49	-0.47

The mean difference is significant at the .05 level.
S. E.: Standard Error

Table 3: Multiple comparisons for determination of optimal dye concentration for COD removal

(I) Dye ADMI	(J) Dye ADMI	Mean Difference (I-J)	S. E.	Sig.	95% Confidence Interval	
					Lower bound	Upper bound
5000	1000	-25.61 (*)	1.28	<0.001	-30.09	-21.12
	2000	-14.23 (*)	1.28	<0.001	-18.71	-9.74
	3000	-4.74 (*)	1.28	.035	-9.22	-.25
	4000	-2.03	1.28	.651	-6.51	2.45

The mean difference is significant at the .05 level.
S. E.: Standard Error

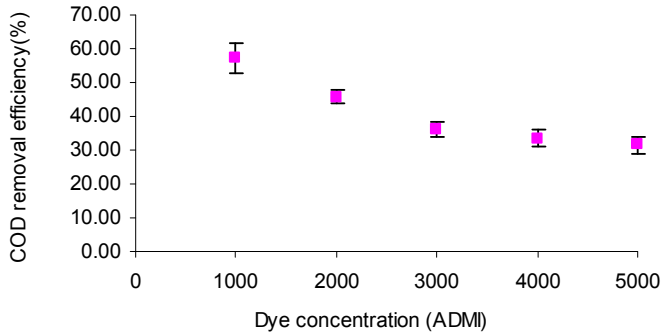

Fig.5: Effect of dye concentration on COD removal with a 95% confidence interval (T=60 min; pH=11; ozone flow rate: 160 mg/h)

Table 4: Multiple comparisons for determination of optimal dye concentration for color removal

(I) Dye (ADMI)	(J) Dye (ADMI)	Mean Difference (I-J)	S. E.	Sig.	95% Confidence Interval	
					Lower bound	Upper bound
5000	1000	-2.30 (*)	.57	.020	-4.30	-.29
	2000	-2.13 (*)	.57	.034	-4.13	-.13
	3000	-1.88	.57	.071	-3.88	.11
	4000	-.88	.57	.672	-2.88	1.11

The mean difference is significant at the .05 level.
S. E.: Standard Error

Table 5 shows the multiple comparisons for the effects of contact time on COD removal by ozone and Fig. 7 shows the effect of contact time evaluated by varying contact time.

Effect of ozonation contact time on color removal

The statistical analysis of the experimental data indicated that contact time had a significant effect on color removal (F=300.81, Sig.= <0.001). Table 6 shows the multiple comparisons for the effects of contact time on color removal by ozone and Fig. 7 shows the effect of contact time was evaluated by varying contact time.

Table 5: Multiple comparisons for determination of optimal time for COD removal

(I) Time (min)	(J) Time (min)	Mean Difference (I-J)	S. E	Sig.	95% Confidence Interval	
					Lower bound	Upper bound
60	15	28.10 (*)	1.30	<.001	23.23	32.97
	30	15.40 (*)	1.30	<.001	10.53	20.27
	45	6.39 (*)	1.30	<.006	1.52	11.25
	75	-1.75	1.30	.870	-6.61	3.11
	120	-5.03 (*)	1.30	.040	-9.90	-.16

The mean difference is significant at the .05 level.
S. E.: Standard Error

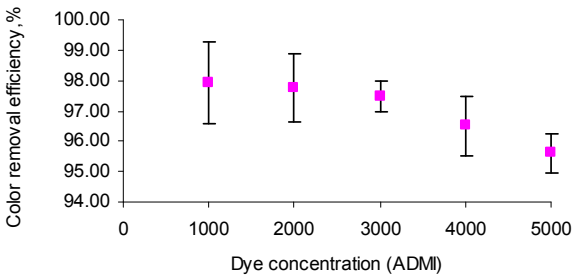

Fig. 6: Effect of dye concentration on color removal with a 95% confidence interval (T=60 min; pH=11; ozone flow rate: 160 mg/h)

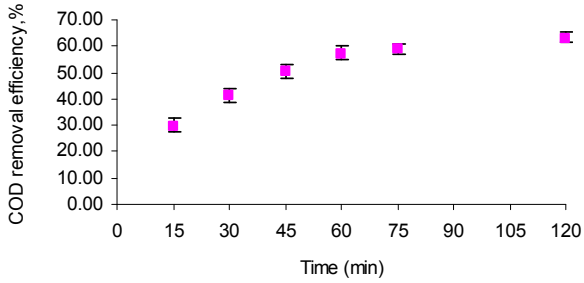

Fig7: Effect of contact time on COD removal with a 95% confidence interval (ADMI=1000; pH=11; ozone flow rate: 160 mg/L)

Table 6: Multiple comparisons for determination of optimal time for color removal

(I) Time (min)	(J) Time (min)	Mean Difference (I-J)	S. E.	Sig.	95% Confidence Interval	
					Lower bound	Upper bound
30	15	4.67 (*)	.61	<0.001	2.37	6.97
	45	-1.50	.61	.356	-3.80	.79
	60	-2.70 (*)	.61	.015	-5.00	-.40
	75	-2.31 (*)	.61	.048	-4.61	-.01
	120	-4.76 (*)	.61	<0.001	-7.06	-2.4613

The mean difference is significant at the .05 level.
S. E.: Standard Error

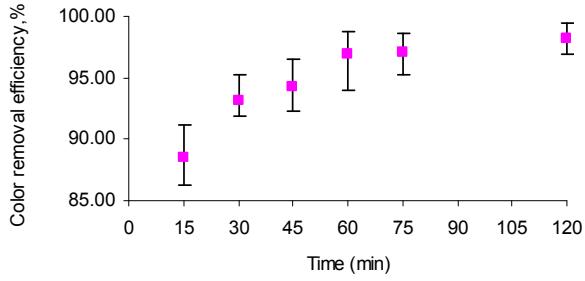

Fig.8: Effect of contact time on color removal with a 95% confidence interval (ADMI=1000; pH=11; ozone flow rate: 160 mg/L)

DISCUSSION

Effect of pH on COD by ozone

Statistical analysis indicated that pH significantly influenced the effectiveness of COD removal (F=42.654, Sig.<0.001). Referring to Table 1 only pairs of mean values were compared, which can frequently be identified by testing the differences between all pairs of treatment means. According to the literature, the optimal pH range for removal of COD with a 95% confidence interval is 9 - 11. Fig. 3 shows the effect of pH levels between 3

and 11, from which, it can be observed that an increase in pH significantly enhanced COD removal, likely because alkaline pH accelerated COD removal by generating OH^- radicals, which have an EOP that is stronger than that of the ozone molecule (2.8/2.08). This finding is in accordance with those of Wu *et al*. (Wu *et al*., 2007), Kdasi (Kdasi *et al.*, 2004), Kusic (Kusaic *et al.,* 2006), Yasar (Yasar *et al*., 2007) and Turhan and Turgut (Turhan and Turgut, 2009).

Effect of pH on color removal by ozone

Statistical analysis indicated that pH did not significantly influence the effectiveness of color removal (F=6.49, Sig. =0.03). In Table 2 on the basis of the multiple comparisons test, there is a significant statistical difference between pH values of 3 and 11; other pH changes were not significant by this method. Therefore, the adjustment of pH is not necessary for color removal. This unexpected finding can be extrapolated from Fig. 1 of Srinivasan *et al*. (Srinivasan *et al.*, 2009). Based on the results shown in Fig. 4, the effects of varying pH from 3 to 11, color removal rate increased by 3% over this range.

Effect of dye concentration on COD removal by ozone

ANOVA results for the effects of dye concentration on COD removal showed that dye concentration had a very significant effect on COD removal (F=136, Sig.<0.001). Referring to Fig. 5, it can be observed that COD removal decreased with increasing dye concentration, either due to a higher initial dye concentration caused more ozone consumption or due to more intermediates, which consume more ozone, were generated when the initial dye concentration was high. This finding is in accordance with those of Wu and *et al*. (Wu *et al*., 2007) and Kdasi (Kdasi *et al.*, 2004). According to the multiple comparisons test shown on Table 3, the optimal dye concentration was 1000 ADMI.

Effect of dye concentration on color removal by ozone

Referring to Fig. 6, it can be observed that color removal decreased with increasing dye concentration. The maximum decolorization results were 98% and 95.5% at 1000 and 5000 ADMI, respectively. On the basis of the multiple comparisons test (Table 4), the difference between 1000 and 5000 ADMI is significant with a 95% confidence interval. This finding is consistent with previous work by Srinivasan *et al*. (Srinivasan *et al.*, 2009), Wu and *et al*. (Wu *et al*., 2007) and Turhan and Turgut (Turhan and Turgut, 2009).

Effect of ozonation contact time on COD removal by ozone

Statistical analysis of the experimental data indicated that contact time had a significant effect on COD removal (F= 184.11, Sig.= <0.001). On the basis of the multiple comparisons test (Table 5), the difference between contact times of 60 to 75 minutes was not significant (95% confidence interval); therefore, the optimal ozonation contact time in this research was 60 minutes. Referring to Fig. 7, the maximum observed removal of COD was 58% for an initial concentration of 1000 ADMI. Therefore, some of the organic compounds were not completely degraded, which is likely due to the fact that some dyes can produce carboxylic acids when directly attacked by ozone, and this organic product cannot be oxidized by ozone alone. This finding is in accordance with those of Zou and Zhu (Zou and Zhu, 2007) and Constapel *et al*, 2009).

Effect of ozonation contact time on color removal

Statistical analysis of the experimental data indicated that contact time had a significant effect on color removal (F=300.81, Sig.= <0.001). On the basis of the multiple comparisons test (Table 6), it was found that the difference between results for 30 and 45 minutes is not significant for a 95% confidence interval; therefore, the optimal time for ozonation was 30 minutes. Referring to Fig. 8, the maximum color removal was 94% for a concentration of 1000 ADMI. This finding was consistent with research by Turhan and Turgut (Turhan and Turgut, 2009). In addition, this finding can be extrapolated from Srinivasan *et al*. (Srinivasan *et al*, 2009), who achieved more than 90% decolorization in less than 30 minutes for all pH values and Song (Song *et al* , 2008).

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