

Determination of the optimum conditions in the removal of Bomaplex Red CR-L dye from the textile wastewater using O_3 , H_2O_2 , HCO_3^- and PAC

Ensar Oguz^{a,*}, Bülent Keskinler^b, Cafer Çelik^c, Zeynep Çelik^d

^a Atatürk University Environmental Problems Research Center, 25240 Erzurum, Turkey

^b Gebze Institute of Technology, Environmental Engineering Department, 41400 Çayirova, Kocaeli, Turkey

^c Atatürk University, Industrial Engineering Department, 25240 Erzurum, Turkey

^d Atatürk University, Environmental Engineering Department, 25240 Erzurum, Turkey

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Abstract

Bomaplex Red CR-L textile dye was used in the experimental studies. Taguchi method was applied to determine optimum conditions in the removal of dye from synthetic textile wastewater. After the parameters were determined to remove Bomaplex Red CR-L dye from synthetic textile wastewater, the experimental studies were realized. The chosen experimental parameters and their ranges: HCO_3^- (mM), 0–39; temperature ($^{\circ}C$), 18–70; ozone–air flow rate ($l\ min^{-1}$), 5–15; the dye concentration (ppm), 200–600; particulate activated carbon (PAC) (g), 0–1.5; H_2O_2 (mM), 0–0.056; pH, 3–12; and treatment time (min), 10–30, respectively. An orthogonal array L_{18} ($2^1 \times 3^7$) for experimental plan and the smaller the better performance statistics formula were selected to define optimum conditions. The optimum conditions were found to be as follows: HCO_3^- (mM), 0; temperature ($^{\circ}C$), 70; ozone–air flow rate ($l\ min^{-1}$), 10; the dye concentration (ppm), 200; particulate activated carbon (PAC) (g), 1; H_2O_2 (mM), 0.056; pH, 12; and time (min), 20. Under these optimum conditions, it was determined that the Bomaplex Red CR-L removal efficiency from textile wastewater was 99%.

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1. Introduction

Ozone reacts with organic compounds dissolved in water through either direct ozone attack or indirect free radical attack. The hydroxyl radicals are generated by ozone decomposition in aqueous solutions [1]. The predominance of the oxidation reactions by molecular ozone at a lower pH has already been established, while beyond some critical pH level, hydroxyl radicals might become the predominant oxidizing species. The critical pH is expected to differ for different chemical composition in the aqueous phase [2]. During the ozonation process, dyes lose their color by the oxidative cleavage of the chromophores. The cleavage of carbon–carbon double bonds and other functional groups, which have high electron densities, will shift the

absorption spectra of the molecule out of the visible region [3].

Different types of dyes are used in many industries such as textile, paint, ink, plastics and cosmetics. A certain amount of them are lost in the process of their manufacturing and utilization and often cause environmental problems. Considerable amount of high quality water is needed for textile industry for dyeing and finishing operations. Color and recalcitrant compounds are among the vital environmental concerns in effluent treatment. Wastewater from these industries represents a variety of pollutants, high strength load and high in color depending on the stage of the process and nature of discharge. The high pollution load is generated from spent dyeing baths, first and second rinses carried out after dyeing processes. These effluents consist of unbound colorants or reaction products, which remain in the substrate and are washed out in subsequent rinses, dye impurities, auxiliaries and surfactants. These compounds exhibit slow degradation kinetics for conventional biological processes and

* Corresponding author. Tel.: +90 442 231 4601.
E-mail address: eoguz@atauni.edu.tr (E. Oguz).

resulting effluent is high in color and violates the discharge limits imposed by legislation [4–8].

One of the main problems in the treatment of textile dyeing wastewater and dye manufacture wastewater is the removal of dye color. The color of wastewater from today's new dyes is much more difficult to treat by physical techniques such as adsorption and chemical coagulation to achieve complete decolorization, especially for highly soluble dyes. Apart from the physical methods of decolorization, chemical oxidation using oxidants such as ozone, chlorine or hypochlorite, hydrogen peroxide, potassium permanganate can be used to destroy the dye to a colorless solution.

There is a wide range of applications of Taguchi method, from chemistry to engineering and from microbiology to agriculture [9–14]. The advantage of the Taguchi method on the conventional experimental design methods, in addition to keeping the experimental cost at a minimum level, is that it minimizes the variation in product response while keeping the mean response on target. Its other advantage is that the optimum working conditions determined from the laboratory work can also be reproduced in the real production environment [15–17].

This investigation was aimed to determine optimum process conditions for the removal of Bomaplex Red CR-L dye from synthetic textile wastewater using Taguchi fractional design method. In this study, eight controllable factors were identified for the removal of dye. The parameters used in the optimization are HCO_3^- , temperature, ozone–air flow rate, dye concentrations, PAC, H_2O_2 , pH and treatment time, respectively. The eight parameters were set (one parameter was set at two levels and seven parameters were set at three levels) to observe the main effects. At the result of an oxidation time of 20 min, the dye removal efficiency by the combined processes was 99%.

2. Materials and method

2.1. Experimental system

The experimental set-up includes an air dryer, compressor, ozone generator and semi-batch reactor having a capacity of 1 l (see Fig. 1).

The air dryer consisted of a column which was filled with a high adsorptive anhydrous CaCl_2 . Ozone was generated using an ozonizer Model OG-24; water was used as the cooling medium. The ozone–oxygen mixture was then fed into the contact place through a porous plate gas sparger located at the semi-batch reactor's base. All experiments were carried out in a 1000 ml

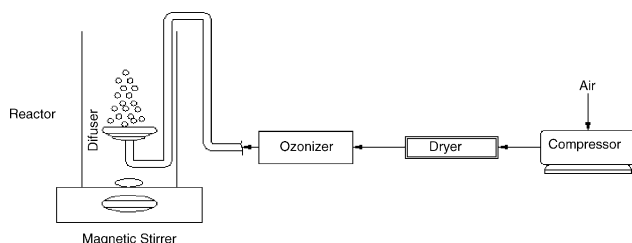


Fig. 1. Diagram of ozonation system.

cylindrical semi-batch reactor. The reactor had a glass column of 7 cm diameter, 40 cm height and a water-cooling jacket to keep the reactor contents at constant temperature. The dye solution of 250 ml was used during each batch ozonation. A magnetic stirrer was used with the gas diffuser to achieve sufficient recirculation of the dye solution.

Bomaplex Red CR-L dye was ensured from a textile mill located in Türkiye. Solutions of this dye were prepared with distilled water in concentrations of 1000 ppm. The dye concentration time data during dye removal were detected using spectrophotometry. The dye used in the present work was water-soluble. It was defined that the Bomaplex Red CR-L dye gave a peak at 505 nm using spectrophotometry (UV Spectrophotometer, Shimadzu 160A) as a function of color. The dye removal capacity was determined by absorbance measurements at the maximum visible absorbance wavelength of 505 nm. All the samples to measure dye concentrations was analyzed at 505 nm. The chemical structure of the water-soluble dye was given in Fig. 2.

Ozone was generated from air, and was supplied into the system through an Opal OG-24 model ozonizer at rates of 5, 10 and 15 l min^{-1} . The combined processes were performed in a cylindrical semi-batch glass reactor (volume: 1 l). The ozone–air mixture percentages (0.7, 1.1 and 1.4% O_3) were continuously sparged through a diffuser.

2.2. Taguchi method

Taguchi method is a systematic application of design and analysis of experiments for the purpose of designing and improving product quality. There are some differences of this method from other statistical experimental design methods. The main difference is parameters affecting an experiment can be investigated as controlling and not controlling (noise factor). The use of the parameter design in the Taguchi method to optimize a process with multiple performance characteristics includes the following steps [18–20]: (a) to identify the performance characteristics and select process parameters to be evaluated; (b)

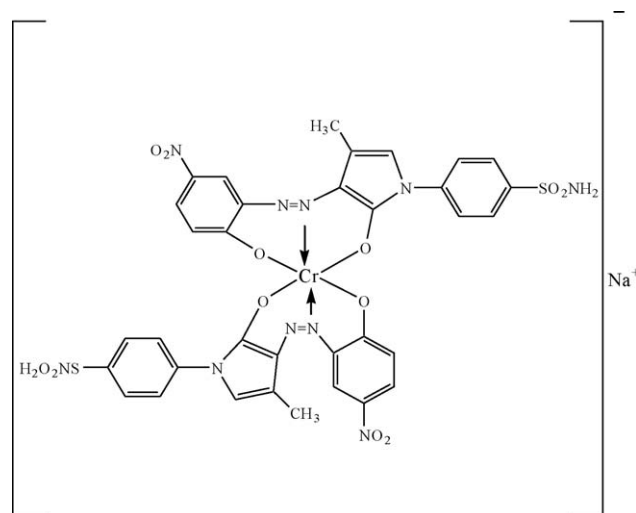


Fig. 2. The general chemical structure of the Bomaplex Red CR-L group dyes.

Table 1
Experimental parameters and their levels

Parameters		Parameters levels		
		1	2	3
A	HCO ₃ ⁻ (mM)	0	39	–
B	Temperature (°C)	18	40	70
C	Ozone–air flow rate (l min ⁻¹)	5	10	15
D	Dye concentration (ppm)	200	400	600
E	PAC dosage (g)	0	1	1.5
F	H ₂ O ₂ (mM)	0	0.028	0.056
G	pH	3	9.3	12
H	Treatment time (min)	10	20	30

to determine the number of parameter levels for the process and possible interaction between the process parameters; (c) to select the appropriate orthogonal array (OA) and assignment of process parameters to the orthogonal array; (d) to conduct the experiments based on the arrangement of the orthogonal array; (e) calculate the performance statistics; (f) to analyze the experimental result using the performance statistics and ANOVA; (g) to select the optimal levels of process parameters; and (h) to verify the optimal process parameters through the confirmation experiment.

This investigation was aimed to determine optimum process conditions for the removal of Bomaplex Red CR-L dye from synthetic textile wastewater. The experimental parameters effect on the removal of dye and their levels determined in the light of preliminary tests are given in Table 1.

The orthogonal array experimental design method was chosen to determine experimental plan, L₁₈ (2¹ × 3⁷) as seen Table 2, because it is the most suitable for the conditions being investigated; one parameter with two levels and seven parameters with three levels [20].

Table 2
Chosen L₁₈ (2¹ × 3⁷) experimental plan table and the results of experiment

Experiment no.	Parameters and their levels								The results of experiment Yi			SN _s = -10 log $\left(\frac{1}{n} \sum_{i=1}^n Y_i^2 \right)$
	A	B	C	D	E	F	G	H	1	2	3	
1	1	1	1	1	1	1	1	1	0	0	0.2	18.327
2	1	1	2	2	2	2	2	2	0	0	0	3070.000
3	1	1	3	3	3	3	3	3	0	0	0	3070.000
4	1	2	3	1	2	2	3	3	0	0	0	3070.000
5	1	2	1	2	3	3	1	1	0	0	0	3070.000
6	1	2	2	3	1	1	2	2	31	28	36	-29.960
7	1	3	3	2	1	3	2	3	0	0	0	3070.000
8	1	3	1	3	2	1	3	1	0	0	0	3070.000
9	1	3	2	1	3	2	1	2	0	0	0	3070.000
10	2	1	1	3	3	2	2	1	13	7	0	-18.501
11	2	1	2	1	1	3	3	2	0	0	0	3070.000
12	2	1	3	2	2	1	1	3	0	0	0	3070.000
13	2	2	2	2	3	1	3	2	0	0	0	3070.000
14	2	2	3	3	1	2	1	3	8	5	6	-15.860
15	2	2	1	1	2	3	2	1	0	0	0	3070.000
16	2	3	2	3	2	3	1	2	0	0	0	3070.000
17	2	3	3	1	3	1	2	3	0	0	0	3070.000
18	2	3	1	2	1	2	3	1	4	3	3	-10.820

The interactive effects of parameters other than that between HCO₃⁻ and temperature were not taken into account in the theoretical analysis because some preliminary tests show that they could be neglected. The validity of this assumption was checked by confirmation experiments conducted at the optimum conditions.

In order to observe the effects of noise sources on the dye removal, each experiment was repeated three times under the same conditions at different times. The performance statistics were chosen as the optimization criteria. There are three categories of performance statistics, the larger-the-better, the-smaller-the-better and the nominal-the-better. In this study, the performance statistics of the-smaller-the-better was used to define the optimum conditions [21,22]. The smaller-the-better performance statistics was given by Eq. (1).

$$SN_s = -10 \log \left(\frac{1}{n} \sum_{i=1}^n Y_i^2 \right) \quad (1)$$

where SN_s is performance statistics, *n* the number of repetition done for an experimental combination, and *Y_i* the performance value of *i*th experiment. In Taguchi method, the experiment corresponding to optimum working conditions might not have been done during the whole period of the experimental stage. In such cases, the performance value corresponding to optimum working conditions can be predicted by utilizing the balanced characteristic of OA. For this, the additive model may be used Eq. (2) [23]:

$$Y_i = \mu + X_i + e_i \quad (2)$$

where μ is the overall mean of performance value; *X_i* the fixed effect of the parameter level combination used in *i*th experiment and *e_i* the random error in *i*th experiment. Because Eq. (2) is a point estimation, which is calculated by using experimental

data in order to determine whether results of the confirmation experiments are meaningful or not, the confidence interval must be evaluated. The confidence interval at chosen error level may be calculated by Eq. (3) [15]:

$$Y_i \mp \sqrt{F_{\alpha;1,DF_{MSe}} MS_e \left(\frac{1+m}{N} + \frac{1}{n_i} \right)} \quad (3)$$

where F is the value of F -table, α the error level, DF_{MSe} the degrees of freedom of mean square error, m the degrees of freedom used in the prediction of Y_i , N the number of total experiments, and n_i the number of repetitions in the confirmation experiment.

The order of the experiments was obtained by inserting parameters into columns of OA, $L_{18} (2^1 \times 3^7)$, chosen as the experimental plan given in Table 2. The order of experiments was made random in order to avoid noise sources which had not been considered initially and could occur during an experiment and affect results in a negative way.

A confirmation experiment is a powerful tool for detecting the presence of interactions among the control parameters. If the predicted response under the optimum conditions does not match the observed response, then it implies that the interactions are important. If the predicted response matches the observed response, then it implies that the interactions are probably not important and the additive model is a good approximation [20].

3. Results and discussion

The collected data were analyzed by PC for evaluation of the effect of each parameter on the optimization criteria. In order to see effective parameters and their confidence levels on the dye removal process, an analysis of variance was performed. A statistical analysis of variance (ANOVA) was performed to see which process parameters are statistically significant. F -test is a tool to see which process parameters have a significant effect on the dye removal value. The F -value for each process parameter is simply a ratio of mean of the squared deviations to the mean of the squared error.

The removal of dye process from the synthetic textile wastewater was investigated in different experimental conditions. To determine the optimum conditions for the removal of dye in the dye solutions, the effects of HCO_3^- ions, temperature, ozone–air flow rate, dye concentrations, (PAC), H_2O_2 , pH and treatment time were investigated, respectively. The reaction conditions in which the effect of parameters investigated and the experimental results are given in Table 2. The degrees of the influences of parameters on the performance statistics are given at the graphs in Figs. 3–11.

The numerical value of the maximum point in each graph marks the best value of particular parameter. From Fig. 3, the HCO_3^- ions which have scavenger effect on the OH^\bullet radicals negatively affected the dye removal efficiency. The performance statistics value is proportion to the dye removal efficiency. With the decreasing the HCO_3^- ions level, the performance statistics value belonging to the dye removal efficiency increased as seen from Fig. 3. From Fig. 3, the fact that the HCO_3^- ions are not

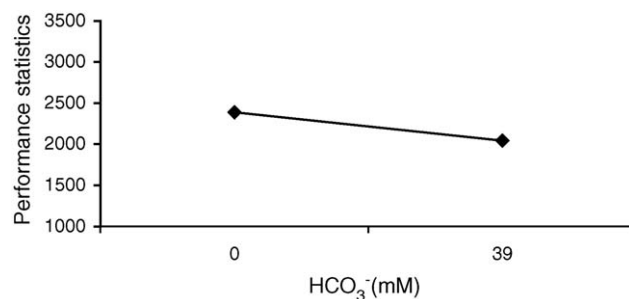


Fig. 3. The effect of HCO_3^- ions on the performance statistics for the dye removal.

present in the dye removal processes is most important because of their scavenger effect. Thus, it was thought that HCO_3^- not finding in the dye removal process is useful for treatment process.

HCO_3^- ions in the O_3/HCO_3^- process were used to scavenge occurring OH^\bullet radicals during ozonation. It is likely that bicarbonate is the principal consumer of the hydroxyl radicals, particularly as relatively high concentrations of bicarbonate are present in water. The scavenging effect of bicarbonate also lies in the fact that it reacts with hydroxyl radicals to generate bicarbonate radicals ($HCO_3^{\bullet-}$). These act as a very selective additional oxidation species and which have a much lower reaction rate constant than hydroxyl radicals for the oxidation of organic micropollutants [25]. It has been reported that bicarbonate ions scavenge hydroxyl radicals to produce intermediates which do not release a radical-type chain carrier, thereby quenching the radical type chain reaction [24].

Fig. 4 shows that removal of the dye has increased with increasing temperature ranges. Ozone solubility in solution decrease with increasing the solution temperature, while the dye removal performance statistics should have decreased with increase of temperature levels because of PAC used together with ozone in the same process and the increase of the rate of adsorption process with temperature, the dye removal performance increased with increase of the temperature at $70^\circ C$ and the degree was defined as the most effective level for the dye removal process. As seen in Fig. 4, the performance statistics values increase with increase of the temperature levels.

As seen in Fig. 5, the dye removal performance statistics value increased with increase of ozone–air flow rate from 5 to 101 min^{-1} but decreased at 151 min^{-1} . It was thought that the

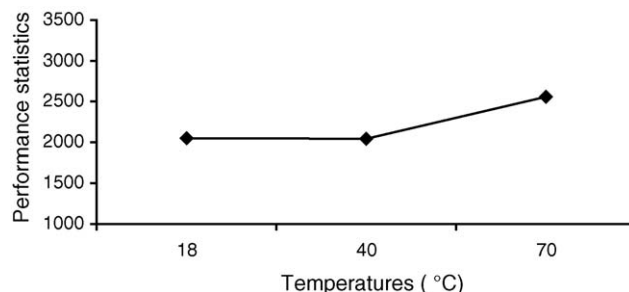


Fig. 4. The effect of temperature on the performance statistics for the dye removal.

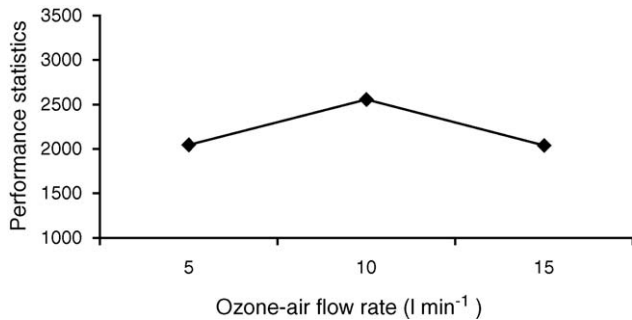


Fig. 5. The effect of ozone-air flow rate on the performance statistics for the dye removal.

decrease in the performance statistics at 15 l min⁻¹ is that the ozone-air flow rate at 15 l min⁻¹ was too much and some of the mixture of ozone-air left the solution of 250 ml without dissolution in the solution or there was stronger competition of ozone molecules on the dye molecules. From Fig. 5, the most effective level of ozone-air flow rate in the dye removal was defined as 10 l min⁻¹.

Fig. 6, respectively, shows the change of the initial dye concentrations (from 200 to 600 ppm) according to performance statistics values. The performance statistics value was high at the level of 200 ppm, but it decreased with increasing of the dye concentration. It was thought that the reason of decreasing of the performance statistics values at 400 and 600 ppm is that increase of the dye concentration in the solution and decrease of the collision frequency of ozone molecules with the dye molecules (the rate of ozone molecules to the dye molecule in the solution decreases with increase of the dye concentration).

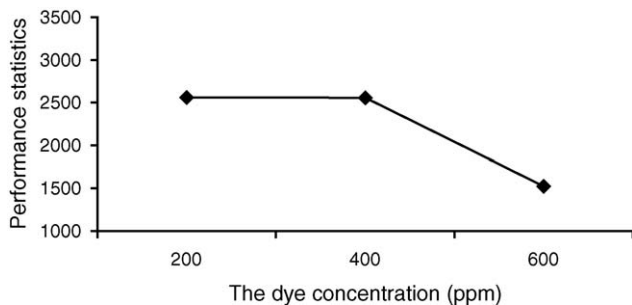


Fig. 6. The effect of dye concentration on the performance statistics for the dye removal.

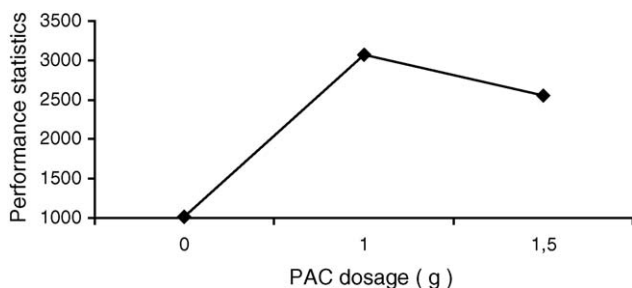
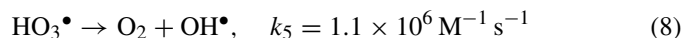
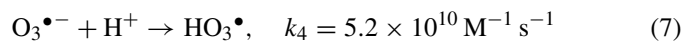
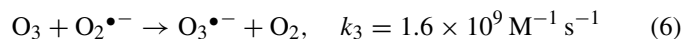
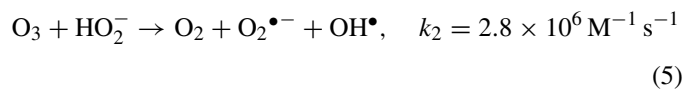
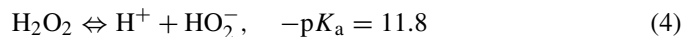


Fig. 7. The effect of PAC dosage on the performance statistics for the dye removal.

As seen in Fig. 7, the dye removal performance statistics value increased with increase of PAC dosage level from 0 to 1 g, but decreased from 1 to 1.5 g. In the present combined process, it was thought that PAC played double roles as both adsorbent and catalyst. The PAC used to remove dye from synthetic textile wastewater has quite a positive effect on the treatment of dye wastewater. For the PAC, the performance statistics value was max at the level of 1 g which was defined as optimum level. As known, the ozone dissolved in the solution is utilized by PAC dosage. Two mechanisms are involved in the oxidation of carbon black by ozone: (i) direct oxidation of elemental carbon to CO₂; and (ii) oxidation of elemental carbon to intermediate that are soluble in alkaline solutions and are subsequently oxidized to CO₂. Because of these mechanisms, it was thought that the PAC particles at level of 1.5 g which were too much in the solution partly prevented ozone molecules to react with the dye molecules. The PAC particles reduced dissolved ozone concentration, formation of more polar groups (which are able to be adsorbed easily on the surface of the PAC particles because of polarization) and synergic effect among the dye, ozone and PAC in the solution. Because of these reasons, the PAC of level of 1 g was optimum level as seen from Fig. 7.

Hydrogen peroxide may react directly or after it is first ionized or dissociated into free radicals. In spite of the powerful oxidizing ability of hydrogen peroxide, it acts as a reductant when reacting with stronger oxidizing agents such as chlorine, potassium permanganate, and potassium dichromate. The reaction mechanism is very complex and may be affected by the reaction conditions and the type of the catalyst. The decomposition of ozone catalyzed by hydroperoxide (HO₂⁻) generating hydroxyl radicals [25] are given in details as follows:



Since the oxidation potential of hydroxyl radicals is much higher than that of the ozone molecule, direct oxidation by O₃ is slower than radical oxidation (OH[•]) according to the Eqs. (4)–(8).

As seen in Fig. 8, the dye removal performance statistics values increased at levels of 0 and 0.056 mM, but decreased at the level of 0.028 mM. Because the dye removal at the level of 0 mM occurred by ozonide mechanism which increased performance statistics value. But the dye removal at levels of 0.028 and 0.056 mM was realized by ozonide and (OH[•]) radicals. Because of the increase of (OH[•]) radicals at the level of 0.058 mM, performance statistics value increased with increasing (OH[•]) radicals and received the maximum value as seen in Fig. 8.

The performance statistics value at the level of 0.028 mM decreased because of the decreasing of the O₃ molecules which

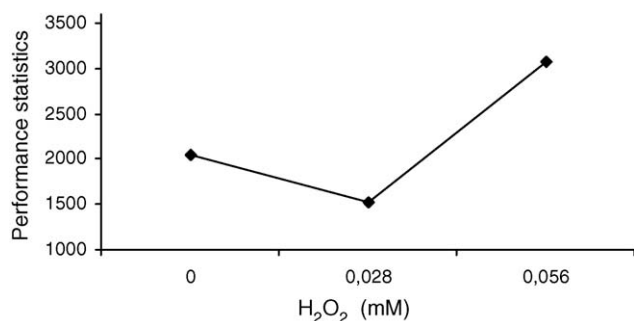


Fig. 8. The effect of H₂O₂ on the performance statistics for the dye removal.

reacted with H₂O₂. As H₂O₂ was not enough concentration in the solution and depleted O₃ molecules in the dye solution, the performance statistics value at the level of 0.028 mM decreased as seen from Fig. 8.

Bomplex Red CR-L dye used in this study is a basic dye. This dye is degraded both at lower pH and high pH values by (H⁺ ions and ozonide) and OH[•] radicals, respectively. The ozonation of the Bomplex Red CR-L dye at various initial pHs (3, 9.3 and 12) was examined, and was shown in Fig. 9. In general, ozone oxidation pathways include direct oxidation by ozone or radical oxidation by OH[•] radical. Direct oxidation is more selective and predominates under acidic conditions, while radical oxidation is less selective and predominates under basic conditions. Since the oxidation potential of hydroxyl radicals is much higher than that of the ozone molecule, direct oxidation is slower than radical oxidation. The increase of solution pH did showed a positive enhancement for the performance statistics because of OH[•] radicals. The decrease of the solution pH showed the decrease of performance statistics value as seen Fig. 9. In this study, it was thought that pH was an important parameter for the performance statistics.

Fig. 10 shows the change of the treatment times respectively (from 10 to 30 min) according to performance statistics values. The performance statistics value was high at the level of 20 min. This time was enough to remove the dye molecules from synthetic textile wastewater as seen in Fig. 10. The performance statistics value in a time of 20 min received a maximum value which was important as economic.

The interactive effects between HCO₃⁻ and temperature on the performance statistics for the dye removal are shown in Fig. 11.

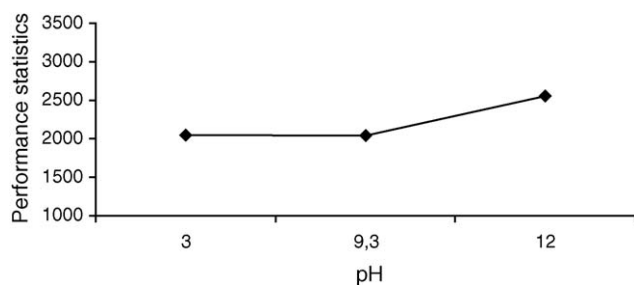


Fig. 9. The effect of pH on the performance statistics for the dye removal.

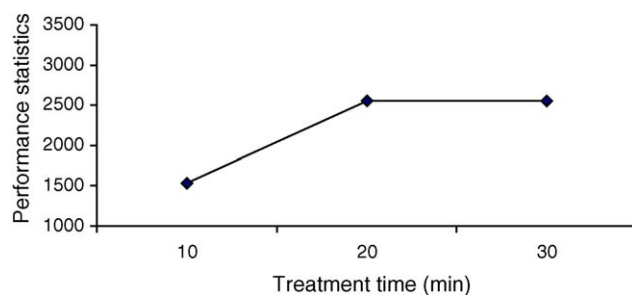


Fig. 10. The effect of treatment time on the performance statistics for the dye removal.

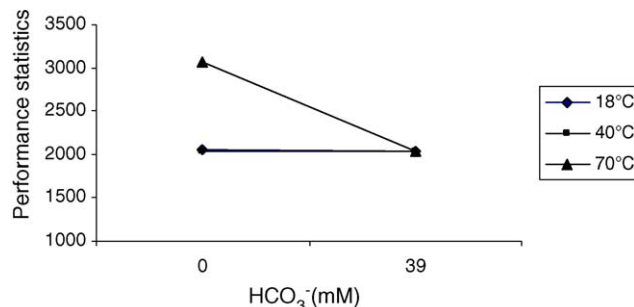


Fig. 11. The interactive effects between HCO₃⁻ and temperature for the dye removal.

The selection of optimum reaction conditions for the removal of the dye from synthetic textile wastewater was done according to maximum amount and minimum cost. The optimum process conditions for the removal of dye are chosen as HCO₃⁻ (mM), 0; temperature (°C), 70; ozone–air flow rate (l min⁻¹), 10; dye concentration (ppm), 200; particulate activated carbon (PAC) (g), 1; H₂O₂ (mM), 0.056; pH, 12; and treatment time (min), 20, as it can be seen in Table 3.

In order to test the predicted results, confirmation experiments were carried out twice at the same working conditions. The fact that the dye removal from confirmation experiments are within the calculated confidence intervals (see Table 3) shows that experimental results are within ±5% in error. This case states that there is a good agreement between the pre-

Table 3
Optimum working conditions, observed and predicted values for the dye removal

Parameters	Optimum working conditions	
	Value	Level
A HCO ₃ ⁻ (mM)	0	1
B Temperature (°C)	70	3
C Ozone-air flow rate (l min ⁻¹)	10	2
D Dye concentration (ppm)	200	1
E PAC dosage (g)	1	2
F H ₂ O ₂ (mM)	0.056	3
G pH	12	3
H Treatment time (min)	20	2
Predicted the dye concentration (ppm)	0	
Predicted confidence interval	0.00–36.91	
Observed the dye concentration (ppm)	0	

dicted values and the experimental values, and interactive effects of parameters other than HCO_3^- and temperature are indeed negligible. It may be concluded that the additive model is adequate for describing the removal of Bomaplex Red CR-L dye from synthetic textile wastewater at the various operational parameters.

4. Conclusions

The major conclusions derived from the present work are: The most important parameter affecting the dye removal from synthetic textile wastewater was PAC. The removal of the dye from synthetic textile wastewater increased with increasing PAC dosage from 0 to 1 g, but decreased with increase of PAC level from 1 g to 1.5 g. Ozone used in the synergic studies cleaves double bonds in the dye molecules and form more polar groups in the solution. Because of polarization, it was thought that PAC particles used in the solution are able to polar groups on their surfaces more effectively. Max performance statistics value of PAC was received at level of 1 g, as seen in Table 3. Parameter levels such as PAC dosage (1 g), ozone–air flow rate (101 min^{-1}), H_2O_2 (0.056 mM), reaction temperature (70°C), HCO_3^- (0 mM), pH (12), the dye concentration (200 ppm) and treatment time (20 min) positively affected on the removal of dye and performance statistics. But the increase of parameter levels such as HCO_3^- and dye concentration negatively affected on the performance statistics. At the result of this study, it was defined that the predicted and observed the values of the dye removal are close to each other (see Table 3), so it was concluded that the Taguchi optimization model applied for the removal of dye was the most successful.

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