Int. J. Bio-Inorg. Hybr. Nanomater., 6(1): 5-12, Spring 2017

National Conference on
Nanostructure, Nanoscience
And Nanoengineering
February 15, 2017
NCNNN2017

Optimization of photo-degradation of direct blue 258 using nano Titanium Oxide with response surface method

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Received: 18 January 2017; Accepted: 21 February 2017

ABSTRACT: The photodegradation of Direct Blue 258, a member of the group of azo dyes which are commonly used in the various branches of the industry, was studied. The photostability of this dye was not previously surveyed. Photocatalytic degradation method was evaluated. The both light source include solar simulated (UV 400 W lamp) and sun light (E= 400 W/m^2) and titanium dioxide nanoparticles were used as irradiation sources and photocatalysts, respectively. Several factors such as reaction time, pH and nano-TiO_2 concentration and UV source were investigated. Using a 24 factorial matrix, the pH and the nano-TiO_2 concentration are the main parameters influencing the degradation rate of DB258. Subsequently, a central composite design methodology has been investigated to determine the optimal experimental parameters for DB258 degradation. After only 30 min of treatment time, high removal of DB258 was achieved by the photo catalyst process under day light (96 %) compared to the UV 400 W lamp. The day light and UV 400 W lamp applied under optimal operating conditions (at 30 min, 0.5 g/L nano-TiO_2 and under pH 7) is capable to degrade around 96 % and 96.4 % of DB258, respectively. Since, UV source is not only hazardous but also expensive because of large input of electric power to generate UV irradiation. According to the results, sunlight assisted nano-TiO_2 could be effectively used for photocatalytic degradation of pollutants in wastewater.

Keywords: *Day Light; Discoloration; Ecofriendly; Nano TiO_2; Optimization; Photodegradation*

INTRODUCTION

Textile industry wastewater is heavily charged with unconsumed dyes, surfactants and sometimes traces of metals (Hathaisamit, *et al.*, 2012). These effluents cause a lot of damages to the environment. In most countries, researchers are looking for appropriate treatments in order to remove these pollutants and to obtain the decolorization and/or full degradation of dye house effluents (Khataee, *et al.*, 2010, Torrades & Garcia-Montano, 2014). The obtained results show that solar photo catalysis is very efficient to discolor the textile effluent of the dyes investigated (Kumar, *et al.*, 2014). Dyes can be degraded in the presence of photo catalyst upon irradiation with visible light because of their absorption in the visible region (Ajmal,

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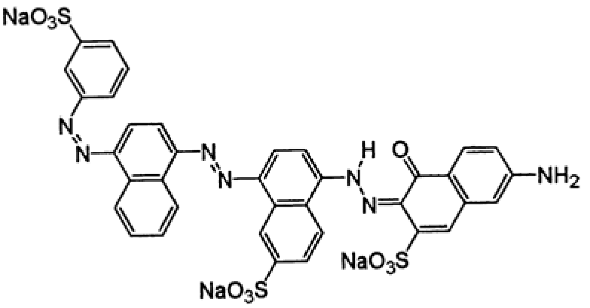
(*) Corresponding Author-e-mail:*Sayedmir@yahoo.com,**nasirizadeh@iauyazd.ac.ir *et al.*, 2014, Pereira, *et al.*, 2014). It has been proven that the presence of catalysts enhances the rate of photodecomposition. Among the various types of photo catalyst, TiO_2 is the most commonly applied semiconductor photo catalyst for destroying organic dye pollutants (Munusamy, *et al.*, 2013, Tao, *et al.*, 2015). This choice is mainly due to the availability, low cost, chemical stability and non-toxicity of TiO_2 (Mital & Manoj, 2011). TiO_2 is the most studied semiconductor mainly due to its high photochemical stability in a broad pH range, its low cost, and its non-toxicity. Commercial titania P25 of Degussa has been widely used as a catalyst in the photo degradation of dyes (Nabizadeh Chianeh & Basiri Parsa, 2014, Colpini, *et al.*, 2008). Studies have been carried out on photo degradation of the dyes pre adsorbed on the surface of TiO_2 particles with visible light (Kansal, *et al.*, 2007). The well-known new technology of wastewater treatment in last decade is the photo catalysis process by using TiO_2 (Asl, *et al.*, 2012, Martinez, *et al.*, 2011). It was shown that the photo degradation rate of azo dyes under UV irradiation considerably depends on the chemical structure in the presence of TiO_2. UV irradiation and TiO_2 catalysis were used for the decomposition of Acid Blue 40 (Forgacs, *et al.*, 2004). Response surface methodology is a collection of statistical and mathematical techniques useful for developing, improving and optimizing processes. The application of statistical experimental design techniques in the development of the photocatalytic process can result in reduced process variability combined with the requirement of less resources (time, reagents and experimental work) (Etemadifar, *et al.*, 2014, Nazari, *et al.*, 2015, Nasirizadeh, *et al.*, 2012, Kasiri, *et al.*, 2013). In this work, the central composite design (CCD) has been applied to the optimization of photocatalytic treatment of Direct Blue 258 (DB258). The factors (variables) investigated were the reaction time, nano-TiO_2 concentration, pH and type of irradiation source.

EXPERIMENTAL

Materials

The dye, Direct Blue 258 (DB258), was obtained from Imperial Chemical Industries, U.K, (Molecular formula = $C_{36}H_{22}N_7Na_3O_{10}S_3$, color index number = 34138, max = 492 nm, Mw = 877.7 g/mol). The investigated photo catalyst was nano-TiO_2 P25 (NTO) (anatase > 99% (DEGUSSA, Germany). According to the manufacturer's specifications, the crystallites mean size was 21 nm and the specific surface area was >300 m^2/g. The buffer solutions were made from H_3PO_4 +NaOH (for pH= 2.0-11.0). The pHs were adjusted to 2.0 M of H_3PO_4 with 2.0 M NaOH.



Scheme 1. Chemical structure of Direct Blue 258

Instruments

A UV-Vis spectrophotometer, Varian Cary 100 model (Australian) was used to determine the initial and final concentration DB258. Artificial irradiation device (320-400 hm UV-A) was included HPA 400 S lamp (400 W, Philips, the Belgium) with peak intensity at 254 nm. The lamps were turned on at the beginning of each experiment. The pH measurement was performed with a Metrohm model 691 pH/mV meters.

Experimental design

In the present study central composite design, which is a widely used form of RSM, was employed for optimization of photocatalytic decolorization process of DB258 solution. In order to evaluate the influence of operating parameters on the photocatalytic decolorization efficiency of DB258, four main factors were cho-

Table 1. Experimental ranges and levels of the independent variables

Variables	Lower	Upper
Nano TiO_2 (NTO) (g/L)	0.01	0.50
pH	3	10
Time (min)	10	30
Irradiation source	Day Light	UV 400 nm

sen: UV light source, reaction time (min), pH solution and NTO concentration (% W.V). The experimental ranges and the levels of the independent variables for DB258 removal are given in Table 1.

A total of 33 experiments were employed in this work. Experimental data were analyzed using Design of Expert 8.0.1. Details of pretreatment design with NTO concentration, time, pH and light irradiation source are presented in Table 2 (Run 1-33). As usual, the experiments were performed in random order to avoid systematic error. The color removal efficiency (DR %) was taken as the response of the design experiments.

Generally, 2.5 mL of DB258 (0.5 g L^{-1}), 5.0 mL buffer solution (pH= 7.0) were added to 50 mL volumetric flask and diluted to the volume with double-distilled water. The 0.05 g TiO_2 was added to this solution. The resulting solutions were moved under light irradiation for different reaction times obtained with experimental design. The percent dye removal (DR

Table 2. The 4 factors central composite design matrix and the value of response function

RUN	Factor 1 A:NTO%	Factor 2 B:Time (min)	Factor 3 C:UV type	Factor 4 D:pH	% DR
1	0.38	20	Day Light	7	91.83
2	0.50	30	Day Light	10	90.8
3	0.01	10	400W	10	85.6
4	0.01	30	Day Light	10	70.25
5	0.13	20	Day Light	10	31.17
6	0.38	20	400W	3	95.56
7	0.26	10	400W	3	76.52
8	0.01	30	400W	7	87.87
9	0.50	30	Day Light	10	90.44
10	0.50	30	400W	3	96.19
11	0.01	10	400W	3	57.23
12	0.50	30	Day Light	7	93.28
13	0.26	30	400W	10	55.61
14	0.50	30	400W	3	96.19
15	0.01	20	Day Light	7	85.37
16	0.26	10	Day Light	10	51.71
17	0.50	10	400W	10	96.93
18	0.01	20	400W	3	57.47
19	0.50	20	400W	7	93.54
20	0.26	15	Day Light	3	72.73
21	0.50	10	Day Light	7	96.84
22	0.01	30	Day Light	10	67.53
23	0.26	30	Day Light	3	67.89
24	0.01	20	Day Light	7	85.38
25	0.26	30	Day Light	3	67.03
26	0.26	15	400W	10	42.03
27	0.50	10	Day Light	7	96.84
28	0.26	25	400W	7	91.83
29	0.50	10	Day Light	3	95.05
30	0.13	15	400W	7	88.9
31	0.01	30	400W	7	86.75
32	0.01	10	Day Light	3	53.43
33	0.05	10	400W	10	96.93

%) was expressed as the percentage ratio of decolorized dye concentration to that of the initial one. The initial and final absorbance of the dye solution were determined at the maximum wave length λ_{max} = 492 nm using a UV-VIS spectrophotometer. These analyses were carried out in duplicate. The percentage removal of dye from solution was calculated as follows:

$$\% \text{ Dye Removal} = \left(A_0 - A\right) / A_0 \times 100 \quad (1)$$

where A_0 and A is the initial and final absorbance of solution, respectively and the removal was taken as a response (Y) of the experimental design.

RESULTS AND DISCUSSION

Experimental design and statistical analysis

To find the optimum conditions for dye removal of the direct blue 258 (DB258), the experimental design as a function of the selected main factors has to be determined. The 4-factor CCD matrix and experimental results obtained in the photocatalytic decolorization runs are presented in Table 2. Results obtained for decolorization efficiencies are given in Table 2. Based on these results, an empirical model was generated in terms of actual factors (standardized equation) and expressed by the following second-order polynomial equation (Eqs. (2) for day light and (3) for UV 400 W):

$$\%\text{Dye Removal} = 100.77 - 99.08 \times \left(\text{NTO}\right) - \left(1.50 \times \text{Time}\right) - 0.27 \times \left(\text{NTO} \times \text{Time}\right) + \left(230.40 \times \text{NTO}^2\right) + 0.04 \times \text{Time}^2 \quad (2)$$

$$\%\text{Dye Removal} = 133.73 - 101.51 \times \left(\text{NTO}\right) - \left(2.75 \times \text{Time}\right) - 0.27 \times \left(\text{NTO} \times \text{Time}\right) + \left(230.40 \times \text{NTO}^2\right) + 0.04 \times \text{Time}^2 \quad (3)$$

Response surfaces are drawn via achieved statistical models Eqs. (2) and (3) and the relation between independent variables and accordingly the decolorization of DB258 is found.

Table 3. Analysis of variance (ANOVA) for fit of decolorization efficiency from central composite design

Source	Sum of Squares	df	Mean Square	F Value	P-value Prob > F
Model	8872.91	16	554.56	4.61	0.0026*
A-NTO	2374.52	1	2374.52	19.73	0.004
B-Time	4.12	1	4.12	0.034	0.8556
C- UV type	174.74	1	174.74	1.45	0.2458
D-PH	1936.84	2	968.42	8.04	0.0038
AB	5.52	1	5.52	0.046	0.8331
AC	2.81	1	2.81	0.023	0.8804
AD	1163.58	2	581.79	4.83	0.0228
BC	663.23	1	663.23	5.51	0.0321
BD	65.04	2	35.52	0.27	0.7667
CD	20.62	2	10.31	0.086	0.9184
A^2	896.13	1	896.13	7.44	0.0149
B^2	67.84	1	67.84	0.56	0.4637
Residual	1926.06	16	120.38	-	-
Lack of Fit	1920.78	8	240.10	363.61	0.00111
Pure Error	5.28	8	0.66	-	-
Cor total	10798.97	32	-	-	-

* Significant

R-squared: 0.8216, adjusted R-squared: 0.6433

Analysis of variance (ANOVA)

The experimental results were analyzed by ANOVA test and CCD to obtain an empirical model for the best response of dye removal. In the ANOVA test, the F-ratio value obtained for the percentage of dye removal is higher than the Fisher's F-value ($F_{9,7} = 4.61$), and so one can conclude that the model fitted well. The quality of the polynomial model was expressed by the coefficient of determination, namely, R2 and Adj-R2. It was found that the predicted values matched the experimental values reasonably well with $R^2 = 0.8216$. This implies that 82.16% of the variations for percent color removal are explained by the independent variables. Adjusted R2 (Adj-R^2) is also a measure of goodness of a fit, but it is more suitable for comparing models with different numbers of independent variables. It corrects R2-value for the sample size and the number of terms in the model by using the degrees of freedom on its computations. If there are many terms in a model and not very large sample size, Adj-R^2 may be visibly smaller than R2 (Khataee, *et al.*, 2010, Santos, and Boaventura, 2008, Box and Behnken, 1960). Here, Adj-R^2 value (0.6433) was close to the corresponding R2 value. Next, the suitability of the model was verified. The next point was the identification of significant variables and/or interactions, according to the F value. It can be seen in Table 3 the obtained results. From these results (Table 3), we can affirm that A (NTO) and D (pH) concentrations have a higher effect on the response (P values smaller than 0.05 for 95% confidence level). However, B (time), NTO with time UV type, UV type with pH was negligible, in accordance with the fact that this variable is only important in the first stages of decolorization. NTO has a stronger effect than pH.

Effect of variables on dye removal of BD258

Fig. 1 (a and b) represents the response surfaces of model for dye removal of DB258 under (a) day light and (b) UV 400 W lamp at pH 7.0. As it is obvious from Fig. 1 a and b, dye removal efficiency under day light increased with increasing reaction time, but removal efficiency under UV 400 w lamp at 10 min was higher than longer reaction time. Also, dye removal efficiency under day light was decreased by increasing of the catalyst values up to 0.26 g/L, and next increased up to 100% dye removal at 0.5 g/L nano TiO_2. However, removal efficiency under UV 400 W lamp was increased by increasing of the nano-catalyst values. The results indicate that dye removal of DB258 occur at a faster rate with UV 400 W in comparison to day light. In case of UV 400 W, 96 % dye removal efficiency was observed in 10 min irradiation time under, whereas in the presence of day light irradiation for the same duration, 96.8 % removal efficiency was recorded. Afterward, removal efficiency was increased to 99 % for 30 min.

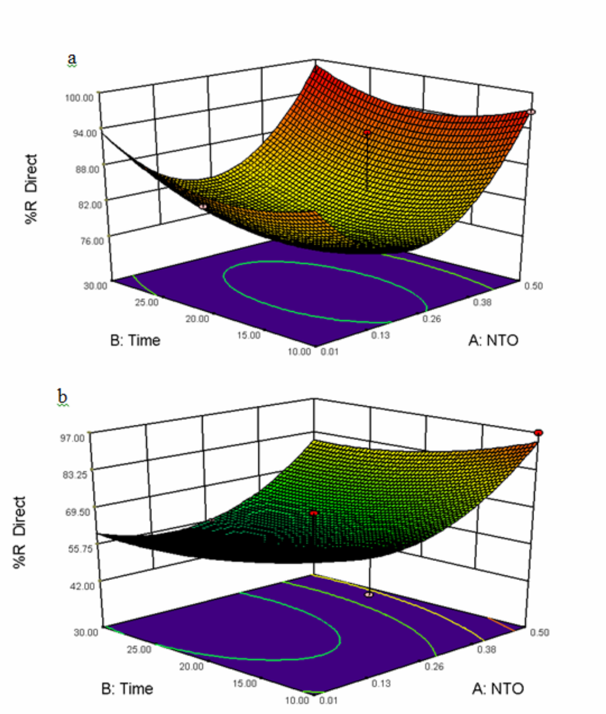


Fig. 1. The response surface of photocatalytic decolorization efficiency (%) as the function of reaction time (min) and NTO concentration (g/L) at pH= 7 (a) under Day light and (b) UV 400 W

In the case of the sample photocatalytic reaction by UV light (sunlight or artificial light), the degradation of direct blue 258 should go through the interaction with the electron hole pair (e^-_{cb}, h^+_{vb}) as usual. The proposed degradation mechanism for DB258 under the irradiated nano-TiO_2 system as follows:

$$TiO_2 + h\upsilon \rightarrow TiO_2\left(e^-_{cb},\, h^+_{vb}\right) \tag{4}$$

$$TiO_2\left(h^+_{vb}\right) + H_2O \rightarrow TiO_2 + H^+ + OH^{\bullet} \tag{5}$$

$$H_2O\left(H^+ + OH^-\right) + h^+_{vb} \rightarrow H^+ + OH^{\bullet} \tag{6}$$

Table 4. Color removal efficiency at optimum values of the process parameters

NTO (g/L)	Time (min)	Irradiation source	pH	%R Estimated	%R Observed
0.50	30	Day Light	7.0	100	96

$$\text{Dye} + \text{OH}^{\bullet} \rightarrow \text{Degradation of Dye} \tag{7}$$

$$\text{Dye} + \text{h}^{+}_{\text{vb}} \rightarrow \text{Oxidation of Dye} \tag{8}$$

The high oxidative potential of the hole ($h^{+}{}_{VB}$) in the nano TiO_2 permits the direct oxidation of the dye to reactive intermediates (Eq. (4)). Another reactive intermediate which is responsible for the degradation is hydroxyl radical (OH•). It is either formed by the decomposition of water (Eq. (6)) or by reaction of the hole with OH^- (Eq. (5)). The hydroxyl radical is an extremely strong, non-selective oxidant (E0 = +3.06 V) which leads to the partial or complete mineralization of several organic chemicals (Hathaisamit, *et al.*, 2012, Pekakis, *et al.*, 2006). The bound radicals can also diffuse away from the surface toward the solution bulk and exist transiently as free OH•. This mechanism suggests that OH• and photogenerated holes ($h^{+}{}_{vb}$) are the primary oxidizing species for dye molecules (Forgacs, *et al.*, 2004). However, sunlight has only 5% of optimum energy for photocatalytic excitation and therewith for degradation of pollutants; it could be safe and cost effective source. UV source is not only hazardous but also expensive because of large input of electric power to generate UV irradiation (Palanisamy and Kavitha, 2011, Domenech and Prieto, 1986). In tropical region like Yazd, intense sunlight is available throughout the year and, hence it could be effectively used for photocatalytic degradation of pollutants in wastewater. Moreover there is no material deterioration in case when sunlight is used as a radiation source (Bansal, *et al.*, 2009). Therefore photocatalytic degradation under sunlight can serve as clean, safe and environmentally friendly method to wastewater treatment.

Determination of optimal conditions for decolorization of DB258

The main objective of the optimization is to identify the optimum values of variables for decolorization of DB258 with photo degradation process from the model obtained using experimental data. By using Design of Expert software the optimum condition of dyeing with acid dyes are evaluated and are shown in Table 4. Then, a specific experiment was performed under these optimum conditions to confirm the agreement of the result achieved from model and experiment. This result also showed the decolorization efficiency for response parameter obtained from the experiment (96 %) and as estimated by model (100%) at day light was satisfactory.

CONCLUSIONS

Comparison of photocatalytic activity of nano TiO_2 under two irradiation light source (day light and UV 400 W lamp) has clearly indicated that direct blue 258 can be successfully degraded under photocatalytic conditions. This degradation depends on several variables include reaction time, pH, nano- TiO_2 concentration. Effect of operational parameters on the decolorization efficiency of DB258 was evaluated by the response surface and 3D plots. In summary, the optimal reaction conditions to degrade of DB258 were: pH = 7.0; time = 30 min, 0.5 g/L nano TiO_2. Analysis of variance showed a high coefficient of determination (R2 = 0.8216 and Adj-R2 = 0.8034), thus ensuring a satisfactory adjustment of the second-order regression model with the experimental data. In addition, we can say that response surface methodology (RSM) was an appropriate technique to optimize the operating conditions and maximize dye wastewater removal. Since, there is no material deterioration and additional reagents not used; therefore photocatalytic degradation under sunlight can serve as clean, safe and environmentally friendly method to wastewater treatment.

ACKNOWLEDGEMENT

This work was financially supported by Yazd Branch, Islamic Azad University and is gratefully for acknowledged for conducting this research "Optimization of the Photocatalytic decomposition of waste containing acid and direct dyes using the nano-titanium dioxide".

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