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Evaluation and Optimization of Process Parameters towards Decolourization of Methyl Red by Oxidative Process

U. Arulmozhi[1], G. Preethi[1], S. Gurukarthikeyan[1], Helan Chandra[2] and S. Sridhar[3]

[1]Department of Biotechnology, Jeppiaar Engineering College, Chennai, Tamil Nadu, India.
[2]Professor, Department of Biotechnology, Jeppiaar Engineering College, Chennai, Tamil Nadu, India.
[3]Assistant Professor, Department of Biotechnology, Jeppiaar Engineering College, Chennai, Tamil Nadu, India.

Abstract

Azo dyes comprise largest class of synthetic organics, cementing it with wide commercial applications in various processing and manufacturing industries. Metabolic breakdown products of azo dyes, generates amines, which are found to be carcinogenic and mutagenic by nature, so these components have to detoxified, before its release into water bodies. Existing physic-chemical methods are not cost-effective and eco-friendly; with each methodology have their own limitations. Methyl Red (2-(N, N-Dimethyl-4 aminophenyl) azobenzene carboxylic acid) is our choice of dye under investigation. In our study, strong oxidizing agent such as sodium hypochlorite was used in our study to investigate the decolorizability of azo dyes. Preliminary studies were carried out using five process variables such as pH, temperature (), incubation time (minutes), dye concentration (mM) and sodium hypochlorite concentration (mM), to predict the operating levels and ranges for optimization. A Box-Benkhen Design matrix with seventeen runs was used to study the interaction among three variables such as pH, temperature (°C) and incubation time (minutes). The optimal predicted experimental conditions were found to be pH 3.0, temperature of 50˚C and incubation time of 20 minutes, yielding a maximal decolourization of 87.17% which was found to be in good agreement with that of the predicted conditions (88.92%). As our study is at bench scale, further validation has to be performed using analytical tools, assisting us to predict the insights of interaction among the reactant and the oxidizing agent and model mechanism.

Keywords: Sodium hypochlorite, Azo dye, Degradation, Response Surface Methodology, One-Factor Variable at a Time (OFAT) approach.

1. Introduction

The extent industrial water pollution and industrialization have a direct impact on rural and urban populations. Textile industries consumes gallons of water for processing and manufacturing textile garments, which in turn releases quantum of spent water in the form of effluent into surrounding water bodies. This discharge totally affects the physical, chemical and biological properties of aquatic environment. The main components of effluents are breakdown dye products, unused dyes, and volatile organic and inorganic compounds. The presence of dyes even in minute amount either in surface and subsurface water makes them aesthetically objectionable, causing many water borne diseases. So, color removal plays an important role in water environment remediation (Wu 2008). Reactive Azo dyes are one of most extensively utilized dyes in textile industry as a coloring agent. The presence of azo bond in association with one or more aromatic structures generates toxic amines during degradation process, making azo dyes as most persistent and recalcitrant synthetic organics worldwide. Water pollution control requires monitoring, action and abatement of toxic pollutants to zero levels at all levels of hierarchical framework (Jamison et al 2006). Literatures have reported an array of chemical, physical and biological methodology, but each and every process has their own limitations. Chemical treatment techniques such as oxidative process involving a strong oxidizing agent like sodium hypochlorite found a wide application in treating effluent containing textile dyes at preliminary level, due to its ability to completely mineralize the prototypical synthetic organic compound. Statistical methodology like RSM is an empirical modeling system for improvising and optimizing complex processes, evaluation the linear and multiple relationship between the responses and independent variables, thereby explaining the influence of the parameters, alone or in combination towards the response (Chen et al 2002). In our study, we optimize the effect of process influencing variables towards decolorization/degradation of azo dye by a strong oxidizing agent such as sodium hypochlorite, in order to predict the optimal operational conditions using statistical tools such as response surface methodology (RSM).

2. Methods

2.1 Methodology

Stock dye solution of 1% (w/v) was prepared, centrifuged and stored at room temperature in brown amber bottles.

The reaction mixture consists of 9.5mL of dye solution of standard dye concentration and 0.5 mL of sodium hypochlorite (mM), were mixed and incubated at appropriate temperature without shaking at room temperature. The decrease in absorbance due to dye decolourization was measured using a double beam UV-Visible spectrophotometer at dyes absorption maximum (420nm). Five process parameters such as effect of pH (2, 3, 4, 5, 6), Temperature (40^0C, 50^0C, 60^0C, 70^0C), dye concentration (0.37, 55, 74, 92, 111 mM), NaOCl concentration (13.43, 26.86, 40.30, 53.73 mM), incubation time (10, 20, 30, 40, 50, 60 minutes) was studied using One Factor at a Time (OFAT) approach.

2.2 Optimization of process parameter

Three level three factorial Box-Behnken Design was chosen to study the interactions of three variables such as pH, temperature (˚C), incubation time (minutes) (Box and Draper, 1987). The design matrix was generated using Design Expert (Trial version, 8.0); Stat Ease, Inc. Statistical indicators were to investigate model statistics and adequacy to predict optimal operating ranges in order to obtain optimum decolorization.

3. Results and Discussion

Table 1 the experimental levels and ranges and the 17 trial experimental runs of Box-Benhken Design (BBD) matrix design to study the decolourization pattern of Methyl Red by Sodium hypochlorite. The experiments were performed and the experimental responses in terms of % decolourization were recorded. **Table 2** represents the ANOVA analysis of the quadratic equation. pH has the major effect on the efficiency of dye decolourization and the optimal pH was found to be 3. The decolourization percentage was found to be increasing with rise in temperature and the optimal temperature at which the color removal was found to be 60˚ C. The absorbance gets reduced with increase in dye concentration, probably due to steric hindrance or complex structure at the transition state. The optimal dye concentration was found to be 74mM at optimal pH and temperature. On varying the sodium hypochlorite concentration, the decolourization percentage gets varied, while the decolourization increased linearly with incubation time at optimal pH, temperature and dye concentration.

Table 2: Design matrix for Methyl red decolourization using sodium hypochlorite.

Std order	Actual values			Coded values			Decolourization%	
	Low (X1)	Centre (X2)	High (X3)	pH (X1)	Temperature (˚C) (X2)	Incubation time (Minutes)(X3)	Actual Value	Predicted Value
1	-1	-1	0	2	50	40	83.67	82.00
2	1	-1	0	4	50	40	77.54	76.5

3	-1	1	0	2	70	40	81.29	82.00
4	1	1	0	4	70	40	78.7	76.5
5	-1	0	-1	2	60	20	84.15	84.49
6	1	0	-1	4	60	20	81.25	78.98
7	-1	0	1	2	60	60	78.92	79.52
8	1	0	1	4	60	60	68.51	74.01
9	0	-1	-1	3	50	20	87.17	88.92
10	0	1	-1	3	70	20	87.01	88.92
11	0	-1	1	3	50	60	86.69	83.95
12	0	1	1	3	70	60	85.57	83.95
13	0	0	0	3	60	40	85.25	86.44
14	0	0	0	3	60	40	88.46	86.44
15	0	0	0	3	60	40	85.89	86.44
16	0	0	0	3	60	40	85.41	86.44
17	0	0	0	3	60	40	86.53	86.44

Table 1 represents the design matrix with the experimental and predicted value for Methyl red decolorization. Statistical indicators such as model P-value, determination of coefficient and ANOVA parameters supported model adequacy (Myers and Montgomery, 1995). Backward regression elimination analysis was performed in order to predict the optimal decolourization conditions by removing statistically insignificant variables. Polynomial equation for Methyl red decolourization using Sodium Hypochlorite after regression analysis is represented in equation 1

$$\text{Response} = 34.97 + 40.37X_1 - 0.12X_3 - 7.18X_1^2 \qquad (1)$$

Statistical indicators such as F-test and ANOVA were selected to check investigate the regression equation and model adequacy. The model has been found to be highly significant, as represented by model F-value and low p-value (<0.001). Lower value of C.V (2.77), represented higher precision of the model (Box and Hunter, 1978). Determination of coefficient (R^2) assisted us to check the model precision, where model R^2 was found to be 0.826 and adjusted R^2 was found to be 0.789. Joglekar and May suggested that R^2 value greater than 0.80 is recommendable (Joglekar and May 1987). Model correlation was found out with experimental values and predicted values from the optimal conditions. So the model can be reproduced to 98.62%. The error was found to be 1.38, which might be due to experimental errors, handling errors, etc.

4. Conclusion

A Box-Benkhen Design matrix with seventeen runs was used to study the interaction among three variables such as pH, temperature (°C) and incubation time (minutes). The optimal conditions predicted by the decolourization process was be pH 3.0, temperature of 50˚C and incubation time of 20 minutes, yielding a maximal decolourization of (87.17%) which was found to be in good agreement with that of the predicted conditions (88.92%). The mineralization of dye might produce some toxic compounds; this has to be confirmed by further analysis using analytical tools.

Table 2: ANOVA for Methyl red decolourization using sodium hypochlorite.

Source	Sum of Squares	df	Mean Square	F Value	p-value
Model	328.971	3	109.657	20.714	< 0.0001
X_1	60.665	1	60.665	11.459	0.0049
X_3	49.451	1	49.451	9.341	0.0092
X_1^2	218.855	1	218.855	41.341	< 0.0001
Residual	68.819	13	5.293		
Lack of Fit	62.038	9	6.893	4.066	0.095
Pure Error	6.780	4	1.695		
Cor Total	397.791	16			
*significant at 95% interval					

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