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# Anaerobic Treatment of Textile Dye Wastewater using Mixed Culture in Batch Reactor

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### ARTICLE DETAILS

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### ABSTRACT

Anaerobic treatment of textile dye wastewater was carried in a batch reactor using mixed culture obtained from cow dung. The textile dye wastewater was collected from a small scale industry. Statistical design (Response surface methodology, RSM) was employed to study the effect of variables like pH, temperature, textile dye wastewater dilution ratio and MLVSS on dye decolourization and COD removal efficiency. The optimum condition was found for the maximum decolourization and COD removal efficiency. They are: pH – 7.3, temperature –  $30.6\,^{\circ}$ C, wastewater dilution ratio 1:2, and MLVSS – 7000 mg/L. The maximum decolourization and COD reduction was found to be 60.8% and 66.7% at the RSM optimized conditions.

### 1. Introduction

Dyes are widely used in various industries like textile, paper, printing, cosmetics, pharmaceuticals, petroleum etc. Of these industries, textile dyeing industries releases large amount of wastewater. During dyeing process, about 10-15% of the dyes are lost in the wastewater [1] and causes detrimental effects to the human being and environment. Along with color, it also comprises pollutants like degradable organics, nutrients, pH altering agent, salts, sulfur, toxicants and refractory organics [2, 3].

Treatment of dye wastewater is done by physical, chemical and biological methods. Physical and chemical methods are mostly ineffective, expensive and produce side reactions, high sludge and by products formation and not suited to degrade all dyes [4, 5]. Biological treatment is found to be the best alternative because of low operational cost [6, 7]. Many works have been reported for decolorization of dye using aerobic microbes [8, 9]. Few works are carried out on anaerobic digestion of textile dye wastewater [10-12]. Hence this study is focused on anaerobic digestion of textile dye wastewater using mixed culture obtained from cow dung.

Response Surface Methodology (RSM) is widely used to study the effects of the variables towards their response. This method is suitable for fitting a quadratic surface and it helps to optimize the effective parameters with a minimum number of experiments, as well as to analyze the interaction between the parameters [13]. It is successfully employed in biotechnology and environmental biotechnology [14, 15].

### 2. Experimental Methods

### 2.1 Materials

Textile dye wastewater was collected from a small scale industry located at Tiruppatur, Tamilnadu, India. The collected wastewater was characterized by analyzing pH, colour, COD, BOD, TS etc. The analysis was performed according to the procedure given in APHA (1999) [16] and given in Table 1. The wastewater was stored at 4±1 °C in air tight plastic containers. Mixed culture was obtained using cow dung. It is maintained at 4 °C in a freezer.

In this study, Box-Behnken design was employed for the optimization of process variables. In order to determine the existence of a relationship

between the factors and the response variables, the data collected is analyzed in a statistical manner, using regression. A regression design is normally employed to model a response as a mathematical function of a few continuous factors and good model parameter estimates are desired.

The coded values of the process variables are determined by the following equation.

$$x_{i} = \frac{X_{i} - X_{o}}{\Delta X} \tag{1}$$

where  $x_i$  – coded value of the ith variable,  $X_i$  – uncoded value of the i<sup>th</sup> test variable and  $X_0$  – uncoded value of the i<sup>th</sup> test variable at center point.

The regression analysis was performed to estimate the response function as a second order polynomial.

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i=1,i,j}^{k-1} \sum_{i=2}^{k} \beta_{ij} X_i X_j$$
 (2)

where Y is the predicted response,  $b_i$ ,  $b_j$ ,  $b_{ij}$  are coefficients estimated from regression. They represent the linear, quadratic and cross products of  $x_1$ ,  $x_2$ ,  $x_3$  on response.

The regression and graphical analysis with statistical significance are carried out using Design-Expert software (version 7.1.5, Stat-Ease, Inc., Minneapolis, USA). To find the relationship between the experimental variables and responses, the response surface and contour plots are generated from the models. The optimum values of the process variables are obtained from the response surface.

### 2.2 Experimental Procedure

Experiments were carried out in a 500 mL Erlenmeyer flask, according to the Box-Behnken design given in Table 1. The process parameters chosen for this study were pH, temperature, wastewater dilution ratio and inoculum concentration. The pH of the wastewater was adjusted to 6.5, 7.5 and 8.5 by adding acid or base as required. Experiments were carried out by varying the temperature to 28 °C, 32 °C and 36 °C respectively. Wastewater dilution was varied as raw, 1:1 and 1:2 with normal water. Mixed liquor volatile suspended Solids (MLVSS) was varied between 4500 to 7000 mg/L. Dye concentration was measured in bio-spectrophotometer (Model: BL-200, ELICO, India) at a wavelength of 390nm. COD of the sample was analysed using the procedure given in APHA.

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**Table 1** BBD based experimental conditions and results for anaerobic digestion of textile dye wastewater using mixed culture obtained from cow dung

Run	Α	В	С	D	%Decolorization		COD removal efficiency		
no.					Experimental Predicte		Experimental	erimental Predicted	
1	1	-1	0	0	40.6	39.95	48.9	46.72	
2	0	1	-1	0	38.6	37.56	40.6	39.92	
3	1	0	-1	0	37.6	36.75	42.9	41.53	
4	1	0	0	1	43.2	45.71	50.4	52.93	
5	0	1	0	1	46.8	45.80	52.3	50.07	
6	0	-1	1	0	51.6	52.86	56.7	58.58	
7	-1	-1	0	0	43.4	39.66	49.6	44.72	
8	0	0	1	-1	53.9	53.13	60.9	58.83	
9	0	0	1	1	59.8	57.45	64.5	63.20	
10	-1	0	0	-1	42.6	40.31	46.8	45.47	
11	0	0	0	0	49.3	48.83	52.6	53.28	
12	1	0	0	-1	43.6	41.80	49.2	48.82	
13	-1	1	0	0	37.8	36.40	39.8	39.38	
14	0	0	-1	1	46.7	45.41	50.4	49.87	
15	-1	0	0	1	43.8	45.83	49.6	51.18	
16	1	1	0	0	35.8	37.50	40.2	42.48	
17	1	0	1	0	49.9	49.03	55.9	55.02	
18	0	-1	-1	0	37.9	38.08	40.2	41.80	
19	0	0	0	0	48.6	48.83	53.4	53.27	
20	0	1	0	-1	39.4	41.13	44.5	45.95	
21	0	1	1	0	47.6	47.64	51.3	50.90	
22	0	-1	0	1	48.6	48.71	55.7	55.65	
23	0	0	-1	-1	40.0	40.30	45.7	44.40	
24	0	-1	0	-1	41.1	43.95	46.3	49.93	
25	-1	0	-1	0	33.2	35.91	36.3	38.58	
26	0	0	0	0	48.6	48.83	53.8	53.27	
27	-1	0	1	0	45.8	48.50	50.1	53.27	

### 3. Results and Discussion

The effect of pH, temperature, waste water dilution ratio and inoculum concentration on decolourization and COD removal efficiency were studied. The experimental and predicted values of percentage decolourization and COD removal efficiency were given in Table 1. A second order polynomial coefficients for each term of the equation were determined using the Design Expert 7.1.5 and it was given in Table 2.

Table 2 ANOVA for the decolourization and COD reduction of textile dye wastewater

Source	%Decolorizat	ion				
	Coefficient	F Value	P value	Coefficient	F - value	p-value
Model	48.83	9.69	0.0002	53.27	9.51	0.0002
Α	0.34	0.2176	0.6492	1.27	2.4364	0.1445
В	-1.43	3.8304	0.0740	-2.39	8.5732	0.0127
С	6.22	72.056	< 0.0001	6.94	72.222	< 0.0001
D	2.36	10.369	0.0074	2.46	9.0578	0.0109
AB	0.20	0.0248	0.8773	0.28	0.0378	0.8491
AC	-0.075	0.0035	0.9538	-0.20	0.0199	0.8899
AD	-0.40	0.0994	0.7579	-0.40	0.0799	0.7822
BC	-1.17	0.8580	0.3725	-1.45	1.0504	0.3256
BD	-0.025	0.0004	0.9846	-0.40	0.0799	0.7822
CD	-0.20	0.0249	0.8773	-0.27	0.0378	0.8491
A^2	-5.98	29.583	0.0002	-5.37	19.215	0.0009
B^2	-4.49	16.687	0.0015	-4.57	13.917	0.0029
C^2	-0.31	0.0809	0.7809	-0.90	0.5346	0.4787
D^2	0.55	0.2507	0.6257	1.70	1.9346	0.1895
Lack of		47.086	0.0910		25.535	0.0833
Fit						

The results were analyzed by analysis of variance (ANOVA). The ANOVA of the quadratic regression model indicates the model is significant. In this work, the model F-value 9.69 and 9.507 for decolourization and COD removal efficiency implies that the models were significant. The smaller the magnitude of the P, more significant is the corresponding coefficient. P value less than 0.05 indicate the model terms are significant. From the P values it is found that, the variables, C, D, A², B² were significant model terms for decolourization and B, C, D, A², B² were significant model terms for COD removal. From the ANOVA table it was found that the linear effect of dye wastewater concentration and MLVSS are more significant for textile dye wastewater treatment.

The fit of the model is also expressed by the coefficient of regression  $R^2$ , which is found to be 0.9188 for decolourization and 0.9173 for COD reduction, indicating that more than 90% of the variability in the response could be explained by the model. This implies that the prediction of

experimental data is quite satisfactory. Adeq precision measures the signal to noise ratio. A ratio greater than 4 is desirable. In this work, the ratio was found to be 11.388 and 11.672 for decolourization and COD reduction, which indicates an adequate signal.

To investigate the interactive effect of two factors on the decolourization and degradation of dye wastewater, response surface method-ology was used and contour plots were drawn. Response surface plots as a function of two factors at a time, maintaining all other factors at fixed levels are more helpful in understanding both the main and the interactive effects of two factors. The response surface curves for the decolourization and degradation of textile dye wastewater were shown in Figs. 1-12. The nature of the response surface curves shows the interaction between the variables. The elliptical shape of the curve indicates good interaction between the two variables and circular shape indicates no interaction between the variables. From the figures it is observed that the elliptical nature of the contour in graphs depicts the mutual interaction of all the variables. There is a relative significant interaction between every two variables, and there is a maximum predicted yield as indicated by the surface confined in the smallest ellipse in the contour diagrams.

Fig.1 shows the interactive effect of pH and temperature on textile dye decolourization. From the figure, it was inferred that increase in pH (up to 7.3) increases the dye decolourization efficiency. After that the decolourization efficiency decreases. Similar trend was observed in Figs. 2 and 3. The pH has a major effect on the efficiency of dye decolorization, and the optimal pH for color removal is often between 6.0 and 7 for most of the dyes. It is clear from Fig.1 that percentage decolourization of dye increased with an increase in temperature from 28 to 30.6 °C. The percentage decolourization of dye decreased with further increase in temperature. Decolourizing activity was significantly suppressed at higher temperatures. This may be due to the loss of cell viability. From Fig. 3, it was observed that, the percentage of decolourization increases with increase in dilution ratio. Similar trend is observed in Figs. 4 and 6. The increase in MLVSS upto 7000 mg/L increases the decolourization efficiency. This is clearly depicted in Figs. 3, 5 and 6.

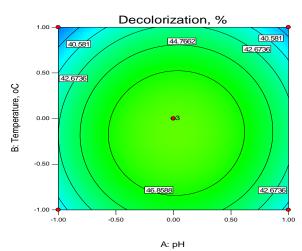


Fig. 1 Interactive effect of pH and temperature on decolorization of textile dye wastewater.

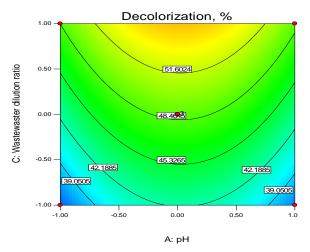


Fig. 2 Interactive effect of pH and wastewater dilution ratio on decolorization of textile dye wastewater

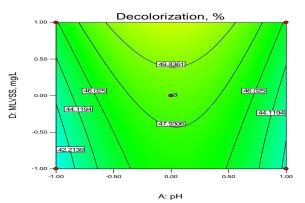


Fig. 3 Interactive effect of pH and MLVSS on decolorization of textile dye wastewater

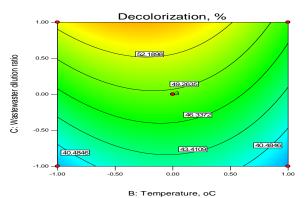
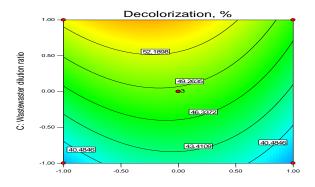


Fig. 4 Interactive effect of temperature and wastewater dilution ratio on decolorization of textile dye wastewater



B: Temperature, oC Fig. 5 Interactive effect of temperature and MLVSS on decolorization of textile dye wastewater

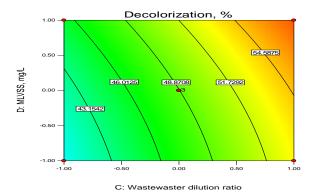


Fig. 6 Interactive effect of wastewater dilution ratio and MLVSS on decolorization of textile dye wastewater

pH is one of the important factor in the treatment of textile dye wastewater by microorganism. The interactive effect of pH and temperature on biodegradation of textile wastewater was shown in Fig. 7. From the figure it was observed that increase in pH upto 7.3 increases the COD reduction. Further increase in pH leads to decrease in COD reduction. The interactive effects of other parameters were shown in Figs. 8-12. The trend observed for other parameters was similar to the decolourization profile. The optimum conditions were also same.

The second order polynomial equation obtained from RSM was used to find the optimum conditions. Equation was solved in MATLAB 7.0. The optimum condition for the maximum decolourization was found to be: pH – 7.3, temperature – 30.6 °C, wastewater dilution ratio 1:2, and MLVSS – 7000 mg/L. The optimal conditions predicted using RSM has been validated using experiments. At the optimized condition, the maximum colour removal and COD reduction were found to be 60.8% and 66.7% respectively.

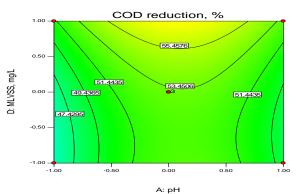
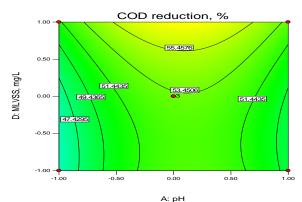
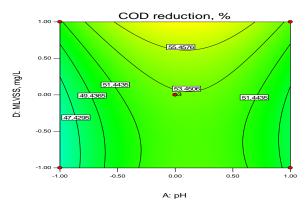


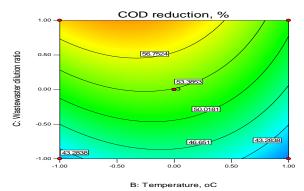
Fig. 7 Interactive effect of pH and temperature on COD removal efficiency of textile dye wastewater



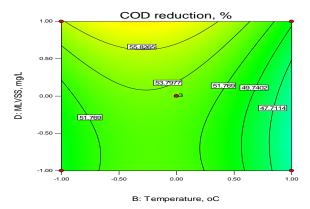
 $\textbf{Fig. 8} \ \ \textbf{Interactive effect of pH and was tewater dilution ratio on COD removal efficiency of textile dye was tewater \\$ 



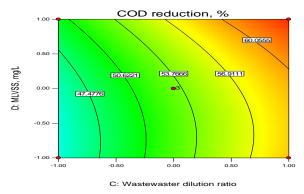
 $\textbf{Fig. 9} \ Interactive \ effect \ of \ pH \ and \ MLVSS \ on \ COD \ removal \ efficiency \ of \ textile \ dye was tewater$ 



 $\textbf{Fig. 10} \ \ \textbf{Interactive effect of temperature and was tewater dilution ratio on COD removal efficiency of textile dye was tewater}$ 



 $\textbf{Fig. 11} \ \, \textbf{Interactive effect of temperature and MLVSS on COD removal efficiency of textile dye was tewater}$ 



 $\textbf{Fig. 12} \ \, \textbf{Interactive effect of was tewater dilution ratio and MLVSS on decolorization of textile dye was tewater}$ 

### 4. Conclusion

In this study anaerobic digestion of textile dye wastewater was carried out using mixed culture obtained from cow dung. RSM was applied to optimize the process parameters. From the results it was found that a maximum of 60.8% colour removal and 66.7% COD reduction occurs at the optimized condition. Hence it was concluded that mixed culture can be utilized for the effective treatment of textile dye wastewater.

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