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Optimization of batch process parameters using response surface methodology for dye removal by a novel adsorbent

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Abstract

Adsorption of Astrazone Blue FRR (Basic Blue 69) and Teflon Blue ANL (Acid Blue 125) was investigated using a hybrid adsorbent that was prepared by pyrolysing a mixture of carbon and fly ash in 1:1 ratio. A 2^4 full factorial central composite design was successfully employed for experimental design and analysis of the results. The combined effect of pH, temperature, particle size and time on the dye adsorption was studied and optimized using response surface methodology. The optimum pH, temperature, particle size and time were found to be 12.8, 27.75 °C, 0.0555 mm, 230 min, respectively for basic blue and those for acid blue 25 were 1.5, 27.5 °C, 0.0565 mm and 245 min, respectively. Complete removal (100%) was observed for both the dyes using the hybrid adsorbent.
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Keywords: Adsorption; Hybrid adsorbent; Dye removal; Response surface methodology; Statistical analysis

1. Introduction

The presence of dyes in effluents is a major concern due to their adverse effect to many forms of life. Coloured waters are also objectionable on aesthetic grounds for drinking and other municipal and agricultural purposes [1]. Industries such as textile, leather, paper, plastics, etc., are some of the sources for dye effluents. The treatment of aqueous water containing soluble dyes thus requires complete removal followed by secure disposal [2]. The most commonly used techniques for colour removal include chemical precipitation, ion exchange, reverse osmosis, ozonation and solvent extraction, etc. However, these techniques have certain disadvantages such as high capital cost and operational costs or secondary sludge disposal problem [3]. The adsorption technique has been proved to be an excellent way to treat effluents, offering significant advantages over conventional process especially from an energetic and environmental point of view [2]. However, increasing costs of carbon result in economic difficulties for developing countries like India. Hence, alternate adsorbents with equivalent potential of activated carbon are current thrust area of research.

The adsorption of dyes on various types of materials has been studied in detail. These include: activated carbon [4]; Peat [5]; Chitin [6]; Silica [7]; the hardwood sawdust [8]; Hardwood [9]; Bagasse pith [10]; Flyash [11,12]; mixture of flyash and coal [13]; Chitosan fiber [14]; Paddy straw [15]; Rice husk [16]; Slag [17]; Chitosan [18]; Acid treated spent bleaching earth [19]; Palm fruit bunch [20]; and Bone char [21].

Conventional and classical methods of studying a process by maintaining other factors involved at an unspecified constant level does not depict the combined effect of all the factors involved. This method is also time consuming and requires a number of experiments to determine optimum levels, which are unreliable. These limitations of a classical method can be eliminated by optimizing all the affecting parameters collectively by statistical experimental design such as response surface methodology (RSM) [22]. RSM is a collection of mathematical and statistical techniques useful for developing, improving and optimizing processes and can be

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Nomenclature

C_0	initial concentration of dye solution (mg/l)
C_t	concentration of dye solution at the desired time, t (mg/l)
Exp.	experimental value
Pred.	predicted value
x_i	dimensionless coded value of the variable, X_i
X_0	value of the X_i at the center point
X_1	time (min)
X_2	pH
X_3	temperature (°C)
X_4	particle size (mm)
δX	step change
Y	predicted response

Greek letters

β_0	offset term
β_i	linear effect
β_{ii}	squared effect
β_{ij}	interaction effect
η	removal efficiency (%)

used to evaluate the relative significance of several affecting factors even in the presence of complex interactions. The main objective of RSM is to determine the optimum operational conditions for the system or to determine a region that satisfies the operating specifications [23]. The application of statistical experimental design techniques in adsorption process development can result in improved product yields, reduced process variability, closer confirmation of the output response to nominal and target requirements and reduced development time and overall costs [24].

For any batch adsorption process, the main parameters to be considered are pH, temperature, particle size and time [25]. Hence, it is necessary to investigate extensively on the relationship between adsorption efficiency and the parameters affecting it. Owing to high cost of activated carbon, an adsorbent that is cheap and easily available would be a better alternative. In the present study, a novel adsorbent consisting of 1:1 mixture of carbon and flyash was investigated for its efficiency to remove two classes of dyes namely Astrazone Blue FRR (BB 69) and Teflon Blue ANL (AB 25) from aqueous solution and their chemical structure is shown in Figs. 1 and 2, respectively. The interaction between the parameters was studied and optimized using response surface methodology.

2. Materials and methods

2.1. Preparation of hybrid adsorbent

Fly ash was obtained from Ennore Thermal Power Plant, Chennai, Tamilnadu. The fly ash was washed with distilled water, dried under sunlight and subsequently in hot air oven at 60 °C. Hybrid adsorbent was prepared by mixing carbon (supplied by SD Fine Chemicals) with flyash in 1:1 ratio by pyrolysing in an isothermal reactor powered by an electric furnace. High purity nitrogen was used as the purging gas. The isothermal reactor was heated to the desired temperature of 650 °C at a heating rate of 15 °C/min, and a holding time of 3 h. After pyrolysis, the product was activated at the same temperature for 3 h using CO_2 as oxidizing agent and subsequently used as adsorbent. The scanning electron micrograph image (SEM) (Fig. 3) shows irregular and porous structure of the hybrid adsorbent, owing to their exposure to a combustion environment, which indicates very high surface area. Chemical analysis of the hybrid adsorbent showed that carbon was the major constituent along with silica, lime and alumina. The origin of carbon constituents could be reasoned by analyzing the process and material used for carbon manufacture. Silica

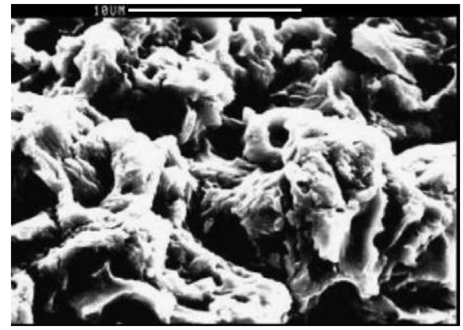

Fig. 1. SEM image of the hybrid adsorbent.

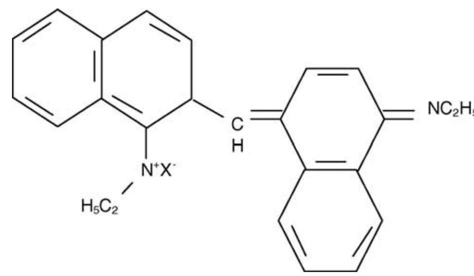

Fig. 2. Structure of Astrazone Blue FRR (BB 69).

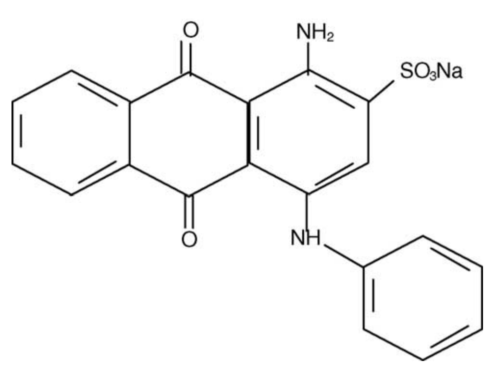

Fig. 3. Structure of Teflon Blue ANL (AB 25).

and alumina content were due to the constituents present in the flyash.

2.2. Methods

2.2.1. Batch adsorption

Stock solutions of dyes (AB 25 and BB 69) were prepared in deionized water and were diluted according to the working concentration. The required pH was adjusted by 0.1 N HCl or 0.1 N NaOH. Dye concentration was measured using UV–vis Spectrophotometer (Shimadzu UV 1600, Japan) at a wavelength corresponding to the maximum absorbance for each dye, 600 nm (λ_{max} maximum absorbance) for AB 25 dye and 585 nm (λ_{max} maximum absorbance) for BB 69. The dye solution (100 ml) at desired pH value, taken in 250 ml Erlenmeyer flasks was contacted with 10 g/l of hybrid adsorbent. The flasks were kept under agitation in a rotatable orbital shaker at 150 rpm for desired time. Experiments were performed according to the central composite design (CCD) matrix given in Table 3. The response was expressed as % dye removal calculated as $\frac{(C_0-C_t)}{C_0} \times 100$.

2.3. Factorial experimental design and optimization of parameters

Temperature, pH, particle size and time were chosen as independent variables and the efficiency of colour removal as dependent output response variable. Independent variables, experimental range and levels for AB 25 and BB 69 removal are given in Tables 1 and 2, respectively. A 2^4 full-factorial experimental design [23], with nine replicates at the center point and thus a total of 31 experiments were employed in this study. The center point replicates were chosen to verify any change in the estimation procedure, as a measure of precision property. Experimental plan showing the coded value of the variables together with dye removal efficiency for AB 25 and BB 69 are given in Table 3. For statistical calculations, the variables X_i were coded as x_i according to the following relationship:

$$x_i = \frac{(X_i - X_0)}{\delta X} \quad (1)$$

The behaviour of the system was explained by the following quadratic equation

$$Y = \beta_0 + \Sigma\beta_i x_i + \Sigma\beta_{ii} {x_i}^2 + \Sigma\beta_{ij} x_i x_j \quad (2)$$

The results of the experimental design were studied and interpreted by MINITAB 14 (PA, USA) statistical software to estimate the response of the dependent variable.

3. Results and discussion

The most important parameters, which affect the efficiency of an adsorbent are time, pH and temperature of the solution and particle size of the adsorbent. In order to study the combined effect of these factors, experiments were performed at different combinations of the physical parameters using statistically designed experiments. The pH range studied was between 1 and 6 for AB 25 and 8–14 for BB 69.The temperature was between 27 and 45 °C, particle size was small (0.0585 mm), large (0.618 mm) and the time varied between 5 and 255 min for AB 25 and 5–240 min for BB 69.

The main effects of each parameter on dye removal are given in Figs. 4 and 5 for AB 25 and BB 69, respectively. From the figures, it was observed that the maximum removal was found to be at 240 min and 255 min for AB 25 and BB 69, respectively. This indicates that the higher the contact time between dye and adsorbent, the higher the removal efficiency until equilibrium time is reached. The same trend was found to be correct by McKay 1983 [6] for dye removal on chitin. Maximum adsorption of AB 25 and BB 69 occurred at pH 1 and 12, respectively. This is due to the fact that dyes adsorb poorly when they are ionized [26].

Table 1
Experimental range and levels of independent process variables for AB 25 removal

Independent variable	Range and level				
	$-\alpha$	-1	0	1	α
Time (X_1, min)	5	15	135	255	375
pH	0.5	1	3.5	6	8.5
Temperature (X_3, °C)	18	27	36	45	54
Particle size (X_4, mm)	0.0343	0.0585	0.33825	0.618	0.89775

Table 2
Experimental range and levels of independent process variables for BB 69 removal

Independent variable	Range and level				
	$-\alpha$	-1	0	1	α
Time (X_1, min)	5	15	127.5	240	352.5
pH	7.5	8	11	12	14
Temperature (X_3, °C)	18	27	36	45	54
Particle size (X_4, mm)	0.0343	0.0585	0.33825	0.618	0.89775

Table 3
Full factorial central composite design matrix for AB 25 and BB 69 removal

Observations	Time (X_1, min)	pH	Temperature (X_3, °C)	Particle size (X_4, mm)	Removal efficiency (η, %)			
					AB 25		BB 69	
					Exp.	Pred.	Exp.	Pred.
1	−1	−1	−1	−1	08.75	03.76	27.50	27.50
2	1	−1	−1	−1	99.62	99.63	92.90	91.67
3	−1	1	−1	−1	09.75	09.73	42.75	33.78
4	1	1	−1	−1	90.92	90.82	99.35	99.35
5	−1	−1	1	−1	10.50	10.52	18.25	18.46
6	1	−1	1	−1	82.00	82.01	71.75	77.11
7	−1	1	1	−1	03.75	03.75	31.85	31.73
8	1	1	1	−1	60.45	60.46	92.85	91.77
9	−1	−1	−1	1	09.85	09.66	20.50	20.50
10	1	−1	−1	1	87.35	87.33	85.50	85.31
11	−1	1	−1	1	02.85	02.86	36.85	25.64
12	1	1	−1	1	65.75	65.74	91.85	91.84
13	−1	−1	1	1	02.25	03.99	11.50	11.03
14	1	−1	1	1	61.50	57.28	62.85	70.32
15	−1	1	1	1	.0001	15.56	22.85	23.16
16	1	1	1	1	08.50	22.95	83.85	83.84
17	−α	0	0	0	00.10	01.51	22.85	23.16
18	α	0	0	0	62.50	72.10	83.85	91.96
19	0	−α	0	0	39.50	39.48	13.50	10.30
20	0	α	0	0	01.50	08.76	21.35	30.10
21	0	0	−α	0	46.50	52.95	23.75	31.24
22	0	0	α	0	18.25	16.91	16.50	14.20
23	0	0	0	−α	27.85	45.36	38.75	40.08
24	0	0	0	α	27.50	27.53	27.65	33.40
25	0	0	0	0	35.50	35.50	34.85	34.85
26	0	0	0	0	35.50	35.50	34.85	34.85
27	0	0	0	0	35.50	35.50	34.85	34.85
28	0	0	0	0	35.50	35.50	34.85	34.85
29	0	0	0	0	35.50	35.50	34.85	34.85
30	0	0	0	0	35.50	35.50	34.85	34.85
31	0	0	0	0	35.50	35.50	34.85	34.85

This is also because of the fact that, when the pH is such that dyes are in ionized form, adjacent molecules of the dyes on the hybrid adsorbent surface will repel each other to a significant degree, because of their same electrical charge. Thus, both the dyes AB 25 and BB 69 that are acidic and basic in nature respectively could not get packed very densely on the hybrid adsorbent surface and at pH values other than 1 and 12, respectively, the equilibrium amount of the adsorbed solute was only modest. This explains the common observation that the non-ionized form of acidic and basic compounds adsorb much better than their ionized counterparts.

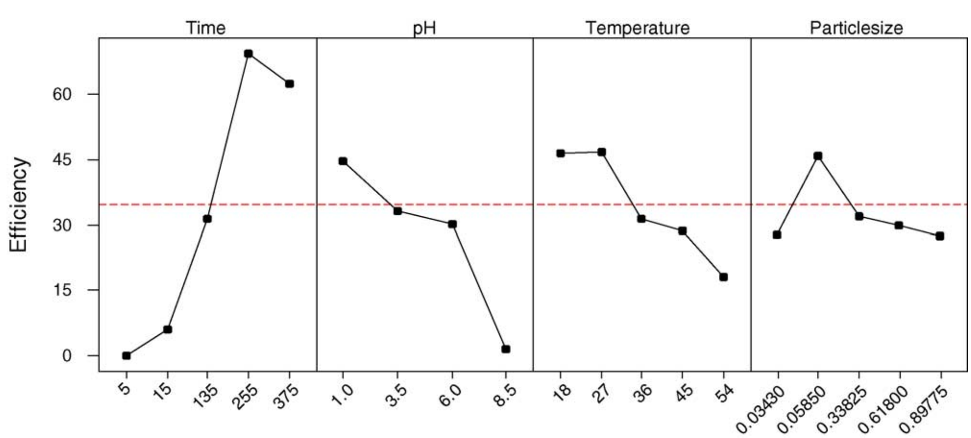

Fig. 4. Main effects plot of parameters for AB 25 removal.

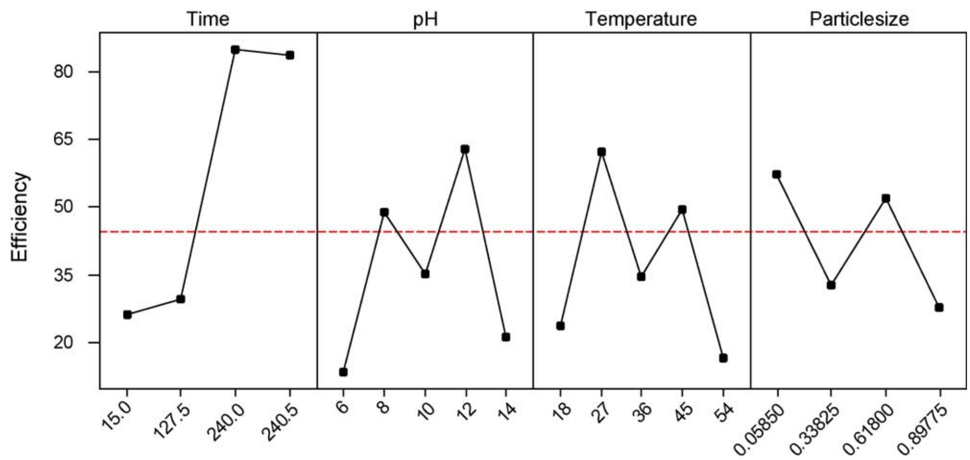

Fig. 5. Main effects plot of parameters for BB 69 removal.

Acid species thus adsorb better at low pH and basic species adsorb much better at higher pH. Hence, the effect of solution pH is extremely important when the adsorbing species is capable of ionizing in response to the prevailing pH. Normal atmospheric temperature (∼27 °C) was found to be better for maximum adsorption of both the dyes and adsorption efficiency decreased with increase in temperature. This indicates the exothermic nature of the process and is due to the enhanced magnitude of the reverse step in the mechanism as the temperature increases.

Similar temperature effects on the adsorption of reactive dye on chitosan beads had also been observed by Chion, 2002 [27]. The present study reveals the fact that in general raising the temperature lead to a decrease in adsorption.

The adsorbed molecules have greater vibrational energies and therefore are more likely to desorb from the surface and hence adsorption will be lower if a system is run at higher temperature. Higher removal efficiency was found in particles of smaller size (0.0585 mm) for both the dyes. The relative increase in adsorption with particles of smaller sizes may be attributed to the fact that they have large surface area. Small particles will have a shorter diffusion path, thus allowing the adsorbate to penetrate deeper into the adsorbent particle more quickly, resulting in a higher rate of adsorption. In 2000, McKay had also observed similar results for the metal ions adsorption on bone char [21]. Using the experimental results, the regression model equation (second order polynomial) relating the removal efficiency and process parameters was developed and is given in Eqs. (3) and (4) for AB 25 and BB 69, respectively. Apart from the linear effect of the parameter for the dye removal, the RSM also gives an insight into the quadratic and interaction effect of the parameters.

These analyses were done by means of Fisher's 'F'-test and Student 't'-test. The student 't'-test was used to determine the significance of the regression coefficients of the parameters. The P-values were used as a tool to check the significance of each of the interactions among the variables, which in turn may indicate the patterns of the interactions among the variables.

The regression equation for AB 25 is

$$\begin{aligned}\eta = {} & (-26.265946) + (0.515665X_1) + (8.986252X_2) \\ & + (0.801103X_3) + (33.553279X_4) + (0.000209{X_1}^2) \\ & + (-0.502472{X_2}^2) + (-0.001769{X_3}^2) \\ & + (25.029406{X_4}^2) + (-0.012317X_1X_2) \\ & + (-0.005643X_1X_3) + (-0.135540X_1X_4) \\ & + (-0.141566X_2X_3) + (-4.568305X_2X_4) \\ & + (-1.234596X_3X_4) \end{aligned} \tag{3}$$

In general, larger the magnitude of t and smaller the value of P, the more significant is the corresponding coefficient term [28]. The regression coefficient, t and P values for all the linear, quadratic and interaction effects of the parameter are given in Tables 4 and 5 for AB 25 and BB 69, respectively.

Table 4
Estimated regression co-efficient and corresponding t and P value for AB 25

Term	Co-efficient	S.D.	t	P
Constant	−26.265946	24.939	−1.054	0.307
X_1	−0.515665	0.0789	6.812	0.000
X_2	8.986252	3.5487	2.510	0.023
X_3	0.801103	1.1734	0.511	0.616
X_4	33.553279	31.206	1.053	0.308
X_1X_1	0.000209	0.0002	1.857	0.082
X_2X_2	−0.502472	0.2634	−1.849	0.083
X_3X_3	−0.001769	0.0152	0.163	0.872
X_4X_4	25.029406	21.800	1.152	0.260
X_1X_2	−0.012317	0.0055	−3.402	0.004
X_1X_3	−0.005643	0.0015	−4.411	0.000
X_1X_4	−0.135540	0.0492	−3.487	0.003
X_2X_3	0.141566	0.0734	−1.789	0.093
X_2X_4	−4.568305	2.3604	−1.812	0.089
X_3X_4	−1.234596	0.6557	−1.560	0.138

Table 5
Estimated regression co-efficient and corresponding t and P value for BB 69

Term	Co-efficient	S.D.	T	P
Constant	−80.166088	43.8002	−1.831	0.086
X_1	−0.205107	0.0904	−2.568	0.021
X_2	17.267683	5.8932	3.011	0.008
X_3	1.439565	1.2081	1.169	0.260
X_4	−20.36608	34.6306	−0.588	0.565
X_1X_1	0.002017	0.0002	12.289	0.000
X_2X_2	−0.915549	0.2541	−3.505	0.003
X_3X_3	−0.037438	0.0125	−2.841	0.012
X_4X_4	19.213511	18.0655	1.067	0.302
X_1X_2	0.001549	0.0060	0.076	0.940
X_1X_3	−0.001362	0.0013	0.699	0.494
X_1X_4	−0.005105	0.0430	0.192	0.851
X_2X_3	0.097038	0.0752	1.045	0.311
X_2X_4	−0.510258	2.4202	−0.062	0.951
X_3X_4	−0.0422521	0.5378	0.270	0.791

It was observed that the coefficients for the linear effect of time, pH ($P = 0.000$, 0.023, respectively) for AB 25 and pH, time ($P = 0.008$, 0.021, respectively) for BB 69 was highly significant and coefficient for the linear effect of temperature for AB 25 and particle size for BB 69 was the least significant. The coefficient of the quadratic effect of time ($P = 0.082$) for AB25 was slightly significant and for BB 69 ($P = 0.000$) was highly significant. The coefficient of the quadratic terms of temperature ($P = 0.872$) was least significant for BB 69. The coefficients of the interactive effects among the variables did not appear to be very significant in comparison to the linear effects of BB 69. However, the interaction effect between time and temperature ($P = 0.000$), time and particle size ($P = 0.003$), pH and temperature ($P = 0.093$) and pH and particle size ($P = 0.089$) were found to be significant. The significance of these interaction effects between the variables would have been lost if the experiments were carried out by conventional methods.

The regression equation for BB 69

$$\begin{aligned}\eta = {} & (-80.166088) + (-0.205107X_1) + (17.267683X_2) \\ & + (1.439565X_3) + (-20.36608X_4) \\ & + (0.002017({X_1}^2)) + (-0.915549{X_2}^2) \\ & + (-0.037438{X_3}^2) + (19.213511{X_4}^2) \\ & + (0.001549X_1X_2) + (-0.001362X_1X_3) \\ & + (-0.005105X_1X_4) + (0.097038X_2X_3) \\ & + ((-0.510258X_2X_4) + (-0.0422521X_3X_4) \qquad (4)\end{aligned}$$

The model equations (3) and (4) were optimized using a multistage Monte–Carlo Optimization technique [29]. The optimal values of the process parameters were first obtained in coded units and then converted to uncoded units by using Eq. (1). The optimum values of the process variables for the maximum removal efficiency are shown in Table 6. These results closely agree with those obtained from the response surface analysis, confirming that the RSM could be effectively used to optimize the process parameters in complex processes using the statistical design of experiments. Although few studies on the effects of parameters on adsorption have been reported in the literature, no attempt has been made to optimize them using statistical optimization methods. The predicted values (using the model equations) were compared with experimental result for both dyes and the data are shown in Table 3.

Table 6
Optimum values of the process parameter for maximum efficiency

Parameter	Optimum value	
	AB 25	BB 69
η (Efficiency)	100%	100%
X_1 (time)	245 min	230 min
X_2 (pH)	1.5	12.80
X_3 (temperature)	27.5 °C	27.75 °C
X_4 (particle size)	0.0565 mm	0.0555 mm

The statistical significance of the ratio of mean square due to regression and mean square due residual error was tested using analysis of variance (ANOVA). ANOVA is a statistical technique that subdivides the total variation in a set of data into component parts associated with specific sources of variation for the purpose of testing hypotheses on the parameters of the model [30]. According to the ANOVA (Tables 7 and 8), the $F_{\text{Statistics}}$ values for all regressions were higher. The large value of F indicates that most of the variation in the response can be explained by the regression model equation. The associated P-value is used to judge whether $F_{\text{Statistics}}$ is large enough to indicate statistical significance. A P-value lower than 0.01 indicates that the model is considered to be statistically significant [31].

The P-values for all of the regressions were lower than 0.01. This means that at least one of the terms in the regression equation has a significant correlation with the response variable. The ANOVA table also shows a term for residual error, which measures the amount of variation in the response data left unexplained by the model. The form of the model chosen to explain the relationship between the factors and the response is correct.

The $F_{\text{Statistics}}$ values of 41.05 for AB 25 and 55.51 for BB 69 are greater than tabulated $F_{14,16}$, indicate that the second-order polynomial model (Eqs. (3) and (4)) was highly significant and adequate to represent the actual relationship between the response (percent removal efficiency) and the variables, with very small P-value (0.00001 for AB 25 and 0.00001 for BB 69) and a high value of coefficient of determination ($R^2 = 0.973$ for AB 25 and $R^2 = 0.98$ for BB 69).

This implies that 98% and 97.3% of the sample variation for BB 69 and AB 25 are explained by the independent variables and this also means that the model did not explain only about 2% and 2.7% of sample variation for BB 69 and AB 25, respectively. The response surface contour plots to estimate the removal efficiency surface over independent variables pH

Table 7
ANOVA of removal efficiency for AB 25: effect of temperature, pH, time and particle size

Source	d.f.	Sum of squares (SS)	Mean square (MS)	$F_{\text{Statistics}}$	P
Model	14	25056.2	1789.731	41.05	0.00001
Linear	4	22416.1	618.076	14.18	0.0001
Square	4	368.5	92.133	2.11	0.000
Interaction	16	2271.6	378.601	8.68	0.000
Residual error	16	697.6	43.601		
Lack of fit					
Pure error					
Total	30	25753.9			

$R = 0.9864$; $R^2 = 0.97$.

Table 8
ANOVA of removal efficiency for BB 69: effect of temperature, pH, time and particle size

Source	d.f.	Sum of squares (SS)	Mean square (MS)	$F_{\text{Statistics}}$	P
Model	14	22800.0	1628.57	55.51	0.00001
Linear	4	17215.7	125.72	4.29	0.015
Square	4	5534.30	1383.58	47.16	0.0000
Interaction	6	49.9	8.32	0.28	0.936
Residual error	16	469.4	29.34		
Lack of fit					
Pure error					
Total	30	23269.4			

$R = 0.9899$; $R^2 = 0.98$.

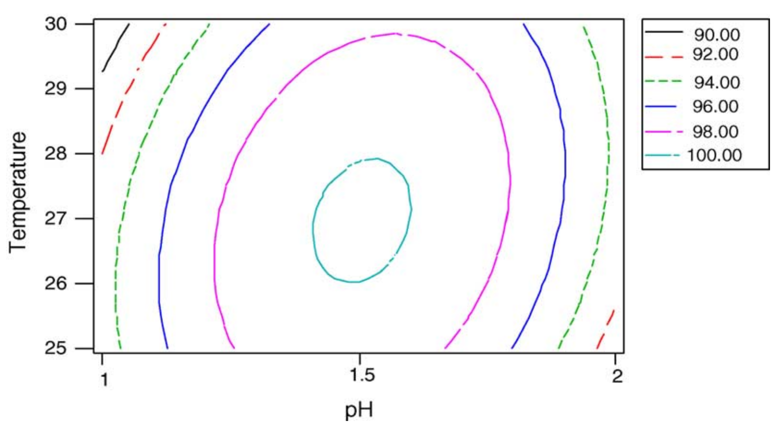

Fig. 6. Response surface contour plot of AB 25 dye removal (%) showing interactive effect of temperature and pH.

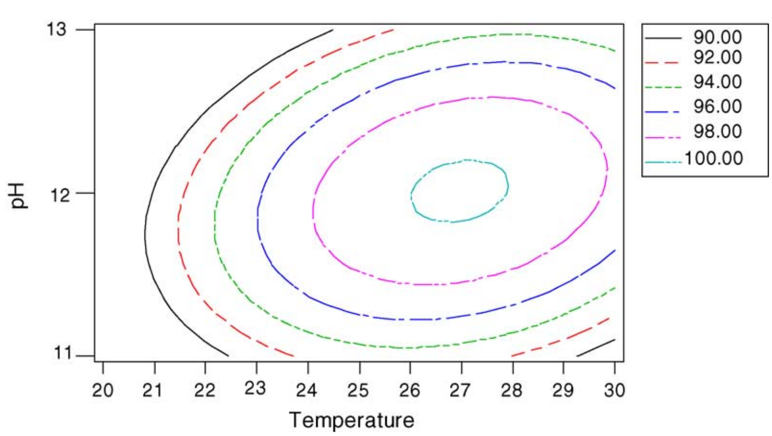

Fig. 7. Response surface contour plot of BB 69 dye removal (%) showing interactive effect of temperature and pH.

and temperature for both the dyes are shown in Figs. 6 and 7. The contour plots given in figures show the relative effects of any two variables when concentration of the remaining variables is kept constant.

The response surfaces of mutual interactions between the variables were found to be elliptical. A similar trend was observed for heavy metal removal using biosorbent by Gopal et al., 2002 [32]. The stationary point or central point is the point at which the slope of the contour is zero in all directions. The coordinates of the central point within the highest contour levels in each of these figures will correspond to the optimum values of the respective constituents. The maximum predicted yield is indicated by the surface confined in the smallest curve of the contour diagram [32]. The optimum values drawn from these figures are in close agreement with those obtained by optimizing the regression model Eqs. (3) and (4).

4. Conclusion

The present study clearly demonstrated the applicability of a hybrid adsorbent containing carbon and fly ash in equal proportion for dye removal. Under optimal values of process parameters, complete removal (100%) was found for both the dyes using the hybrid adsorbent. This study clearly showed that response surface methodology was one of the suitable methods to optimize the best operating conditions to maximize the dye removal. Graphical response surface and contour plots were used to locate the optimum point. A 2^4 full factorial central composite design was successfully employed for experimental design and analysis of results. Satisfactory prediction equations were derived for both the dyes using RSM to optimize the parameters.

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