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Research Article

Optimization studies for removal of Remazol Brilliant Blue dye from aqueous solution using acid treated red mud

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Abstract

The present study focuses on the utilization of the acid treated red mud as an adsorbent for removal of Remazol Brilliant Blue dye, a reactive dye from dye synthetic water. Red mud is a waste product in production of alumina and it poses serious pollution hazard. Adsorption of Remazol Brilliant Blue, from synthetic solution was studied by adsorption on powdered dilute sulphuric acid treated red mud. The adsorption of the dye on red mud was studied to determine the effect of initial dye concentration, initial pH, and adsorbent dosage. Langmuir isotherm model has been found to represent the equilibrium data for adsorption system better than Freundlich model. The adsorption capacity of red mud was found to be 26.7 mg dye per gram of adsorbent at 30^oC. A 2^4 factorial design analysis was performed to screen the parameters affecting Remazol Brilliant Blue dye removal efficiency. The results of the study showed that percentage adsorption was found to be 99% with optimal conditions of initial dye concentration 100 mg/l, red mud dosage 4.8 gm/l, initial pH 1 and temperature 33^oC respectively. It was found that small change in dosage and pH will drastically affect the percentage removal of dye.

Keywords: *Remazol Brilliant Blue; Red mud; Isotherm*

1. Introduction

At present synthetic dye compounds can be exist in the effluents of wastewater from different industries, i.e. textiles, paper, leather, plastics industries, etc. (R. Gong, et al 2005). Discharge of colored wastewater from the industries into near water streams and rivers poses severe environmental problems. Even minute quantities of dyes can color large water bodies, which not only affects aesthetic conditions but also reduces light penetration and photosynthesis. In addition, most of dyes are either toxic or carcinogenic (R. Gong, M 2007; S. Wang et al 2007). Hence the waste water containing dyes must be treated prior to its discharge from the industry. The colored wastewater is treated by physical, chemical and biological methods. Adsorption is one of the processes, which being widely used method for removal of dye and has wide applicability in wastewater treatment. Reactive dyes are typically azo-based chromophores combined with different types of reactive functional groups. These are presently used for coloring cotton fibers. They differ from all other classes of dyes in that they bind to the textile fibers such as cotton to form covalent bonds (Aksu et al. 2000). Some of the conventional adsorbents which are generally used for dye wastewater treatment are alumina, silica gel, zeolite and activated carbons. Studies have shown that these adsorbents are more efficient adsorbents for the removal of different types of dyes, but due to their high cost their use is restricted (Walker et al. 1999).This has initiated many researchers to find low cost adsorbents which may replace the above adsorbents. A number of non-conventional adsorbents such as Sawdust (Khattri et al. 1999), Clay minerals (Lopezgalindo et al. 2007), Cellulose based waste (Annadurai et al. 2002), Wheat straw (Robinson et al. 2001), AC Rice husk (Mohamed et al. 2004), and Rice husk (McKay et al. 1999) have been extensively used as adsorbents for removal of dye.

The present work focuses on the possibility of utilization of the dilute sulphuric acid treated red mud as an adsorbent for removal of Remazol Brilliant Blue dye. Response Process Optimization was carried out using the results of experiments designed as per central composite design.

2. Materials and methods

2.1. Red mud and Dye

Red Mud (Composition: Fe_2O_3- 42%, Al_2O_3-20%, TiO_2-9%, SiO_2-10-12%, Na_2O-4-5%) was obtained from Hindalco Aluminium Industry, Belgaum, India. Remazol Brilliant Blue dye was obtained from Campbell Knitwear

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Ltd, Belgaum, India. Dye contains NH and SO_3 functional groups. The dye concentration was measured at 608 nm using a UV–VIS bio-spectrophotometer (Elico BL-198).

2.2. Pretreatment of red mud

Red mud was washed thoroughly with distilled water, dried at 110^{o}C for 24 hours. 10 grams of washed red mud was soaked in 200 ml of 1N H_2SO_4 for 24 hours, washed with water several times and dried at 110^{o}C overnight**.** The acid treated red mud sample thus prepared was sieved and the sample of average size 120 microns was used for the studies. Scanning Electronic Microscopy (SEM) was carried out for surface morphology to compare the relative performance of raw and acid treated red mud. The surface areas of original red mud and acid treated red mud were determined by BET analysis using ASAP 2020 V3.04 H, Micromeritics, USA surface area analyzer.

2.3 Batch adsorption experiments

Batch adsorption experiments were carried out in 100 ml conical flask containing the dye solution of the desired concentration and the known amount of acid treated red mud. The solution was agitated till equilibrium conditions are reached. The dye solution was then separated from the adsorbent by centrifugation and the remaining dye concentration of supernatant was determined by using UV spectrophotometer. Batch experiments were performed at different adsorbent dosages, initial dye concentration, temperature and pH.

2.4. Design of experiments for optimization

To help in the optimization of parameters such as initial pH, initial dye concentration, red mud dosage and temperature for efficient removal of the reactive dye, batch experiments were designed as per CCD with four factors at five levels. The range of levels in coded and un coded form for different parameters are presented in Table.1. The design matrix of experimental conditions with 31 sets of experiments in un coded form of factors is shown in Table 2.The response variable in this study is the percentage adsorption of dye at equilibrium. Batch adsorption experiments were carried out as conditions shown in Table 2.

2.5. Analysis and optimization

The results obtained from the batch experiments based on central composite design were analyzed using response surface methodology (RSM). The effect of four factors as individuals and in combination as interacting effect on the % adsorption of the dye was evaluated using RSM. Multiple regression analysis on the experimental input-output data was performed using MINITAB 14 software. The results were fitted into the regression equation and the effects of four factors on the percentage adsorption were analyzed by RSM. Thus, the equation giving percentage dye adsorptions on red mud, as a function of the factors are presented using a second-order polynomial model with 15 coefficients. The optimum values of initial pH, red mud dosage, initial dye concentration and temperaturewere obtained by using response optimizer of MINITAB 14. Response surface plots were generated using these input-output data.

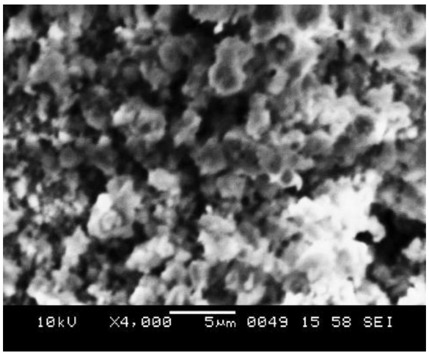
Table.1. Factors and levels used in the central composite design study

Factors	**-2**	**-1**	**0**	**+1**	**+2**
pH	1	2	3	4	5
Initial concentration, mg/l	10	32.5	55	77.5	100
Red Mud Dosage, g/l	0.3	1.8	3	4.2	5.4
Temperature, ^{o}C	20	25	30	35	40

3. Results and discussion

3.1. Red mud characterization

Red mud is found to be a complex mixture of phases mainly comprising of Hematite (Fe_2O_3), Goethite FeO(OH) and Quartz (SiO_2). The surface of the red mud activated by acid pretreatment was compared with that of raw red mud using the results of scanning electron microscopy. The SEM-EDAX micrograph of raw red mud and acid treated red mud samples are shown in Fig 1 and 2. EDAX analysis shows that the intensity for metals like Al, Si, Ti and Fe in raw red mud are high. Acid treatment of red mud has resulted in drastically decreased intensities for Fe, Ti, Si and Al. Similar results were obtained by Agatzini et al.(2008) and Uzun and Mustafa (2007).The raw red mud and acid treated red mud have specific surface area of 20.2 and 27.3 m^2/g respectively. The pore size of raw red and acid treated red mud was 20.98 nm and 20.09 nm respectively. The surface area and pore volume of acid treated red mud is higher than the raw red mud. The acid treatment leads to removal of sodium ions and compounds (Shing–Jen 1997; Pratt et al. 1982; SnigdhaSushil et al. 2012), further it causes dispersion of dissolved metal oxides as hydroxides which leads to formation of pores thereby increasing the surface area.



3.2 Effect of pH, initial dye concentration and red mud dosage.

The batch adsorption experiments were conducted at different initial pH conditions ranging from 2 to12. The

Table 2. Experimental design matrix and results for the dye removal percentage

S.N	pH	Initial Concentration, mg/l	Dosage, g/l	Temperature, ^oC	% Removal	
					Experimental	Predicted
1	2	32.5	4.2	25	83.148	80.70
2	3	55	3.0	30	50.134	50.13
3	1	55	3.0	30	94.429	103.10
4	3	55	3.0	20	43.050	44.50
5	4	32.5	4.2	35	45.631	46.07
6	4	32.5	1.8	35	47.407	45.53
7	2	77.5	1.8	25	47.530	44.61
8	3	55	3.0	30	50.134	50.13
9	4	77.5	4.2	25	46.087	49.52
10	3	55	3.0	30	50.134	50.13
11	3	55	3.0	30	50.134	50.13
12	3	55	3.0	30	50.134	50.13
13	3	55	3.0	30	50.134	50.13
14	2	77.5	4.2	25	64.531	63.82
15	2	77.5	1.8	35	58.067	55.396
16	4	77.5	1.8	25	40.467	37.023
17	2	77.5	4.2	35	79.758	75.246
18	4	32.5	1.8	25	43.148	44.73
19	3	55	3.0	30	50.134	50.13
20	3	100	3.0	30	41.456	44.03
21	3	10	3.0	30	72.785	75.70
22	4	77.5	1.8	35	31.230	30.74
23	2	32.5	1.8	35	97.852	91.49
24	3	55	0.6	30	30.355	37.74
25	2	32.5	4.2	35	98.333	99.021
26	4	32.5	4.2	25	45.000	45.09
27	3	55	5.4	30	59.839	57.95
28	4	77.5	4.2	35	42.339	43.43
29	2	32.5	1.8	25	76.852	73.2
30	5	55	3.0	30	46.019	42.48
31	3	55	3.0	40	52.767	52.76

Table 3.Analysis of Variance for % Adsorption

Source	DF	Seq SS	Adj SS	Adj Ms	F	P
Regression	10	9786.7	9786.70	978.670	47.73	0.000
Linear	4	7788.3	3256.30	814.076	39.70	0.00
Square	3	1090.2	1090.24	363.412	17.72	0.00
Interaction	3	908.1	908.14	302.712	14.76	0.000
Residual Error	20	410.1	410.08	20.504		
Lack of Fit	14	410.1	410.08	29.291		
Pure Error	30	10196.8				
Total	30	10196.8				

effect of pH on percentage adsorption at equilibrium was studied with initial dye concentration of 30 mg/l and adsorbent dosage of 1.2g/l. The percentage removal of dye by adsorption has decreased with the increase in pH from 2 to 8. At pH above 6, a decrease in adsorption takes place. The decrease in adsorption with increase in pH may be explained on the basis of acid-base dissociation at solid/liquid interface (Namasivayam et al. 2001). The effect of initial dye concentration on percentage adsorption of the dye was studied. The adsorption of the dye at equilibrium has decreased from 94% to 71 % with increase in dye concentration from 10 mg/l to 70 mg/l for a fixed red mud dosage of 3.2 g/l. This may be attributed to surplus dye molecules being present in the solution, with respect to the available active adsorption sites owing to fixed surface area of red mud (G M Ratnamala et al 2012). The variation of removal of the dye with varying amount of red mud dosage was studied for different initial dye concentrations (10-50 mg/l). The uptake increases with increasing adsorbent dosage. The percentage adsorption of the dye increased by 10% when the quantity of adsorbent used was doubled, i.e., from 0.6 to 1.2 g/l. Further increase in dosage did not show much increase in uptake of dye. The increase in dye removal with adsorbent dosage is due to greater availability of adsorbent surface area (Namasivayam et al. 2001) and more active sites.

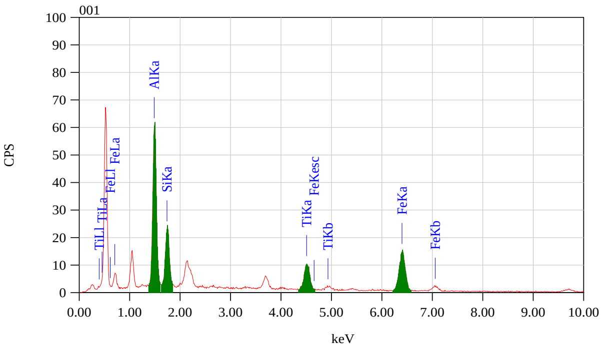
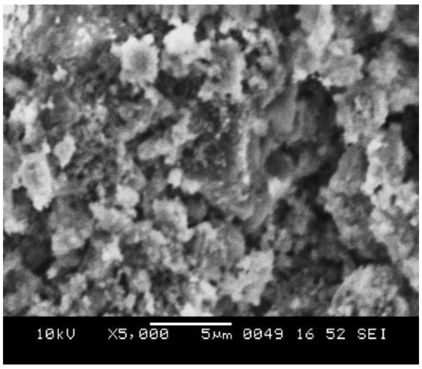


Fig. 1. The SEM –EDAX micrograph of raw red mud



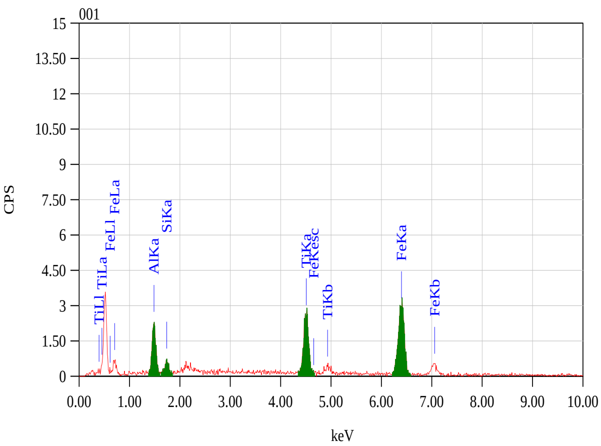


Fig.2. The SEM – EDAX micrograph of red mud treated with H2SO4

3.3. Response surface optimization

The 31 set of Experiments shown in Table 2, designed as per CCD matrix were conducted to study the effect of different factors influencing the adsorption process as an individual and on interaction with each other , as well as with a view to optimize the process using RSM. The matrix of four variables pH, Initial concentration of dye, Adsorbent dosage and Temperature were varied at five levels (-2,-1, 0,+1,+2). The higher level of variable was designed as'+' and the lower level was designed as '−'. Dye removal percentage obtained from the experiments at the end of equilibrium time for each of the experiments is shown in Table 2.

$$\%Adsorption = 90.93 - 31.60X_1 - 1.456X_2 + 4.65X_3 + 3.76X_4 + 5.70{X_1}^2 + 0.0048{X_{12}}^2 - 0.39{X_3}^2 + 0.0052{X_4}^2 + 0.237X_1X_2 - 1.493X_1X_3 - 0.87X_1X_4 + 0.112X_2X_3 - 0.0157X_2X_4 + 0.0076X_3X_4 \quad (1)$$

Table 4. Optimum values of the process parameters for maximum removal of dye

<table>
<tr><td>Parameters</td><td colspan="2">Values</td></tr>
<tr><td>pH</td><td colspan="2">1.00</td></tr>
<tr><td>Initial Concentration,m g/l</td><td>100</td><td rowspan="2">Optimum dye to adsorbent dosage ratio=0.020 g/g</td></tr>
<tr><td>Red mud Dosage, g/l</td><td>4.8</td></tr>
<tr><td>Temperature, °C</td><td colspan="2">33.11</td></tr>
</table>

The results were analyzed statistically using the Student's test, analysis of variance, *F* test and lack of fit. The results of ANOVA on main and interaction effect of parameters are shown in Table 3. It shows that the main and interaction terms are highly significant ($P< 0.05$) and model was applicable very well. Predicted values of percentage adsorption for all the 31 set of experiments are shown in Table 2 and are found to match well with the experimental values. The RSM analysis for adsorption of the dye using red mud indicated that the second-order polynomial model Eq (1) was highly significant and adequate to represent the actual relationship between the response and the variables, with *P* -value of 0.000 and a high value of coefficient of determination ($R^2 = 0.97$).

The surface plots were generated using RSM with MINITAB 14 and are shown in Fig 3 to Fig 8. They show the relative effects of two variables when the remaining variables are kept constant.

The combined effect of initial pH and temperature on adsorption of dye at constant dosage (0.3g/l) and initial concentration (70 mg/l) has been shown in Fig 3. It can be seen that increasing the pH value decreases the percentage adsorption of the dye. Highly acidic pH favors adsorption. Increasing the temperature from 20 to 40°C facilitated the removal of dye. The increase in dye uptake with increasing temperature indicates endothermic nature of adsorption. Effect of temperature on percentage adsorption is very significant at low pH values. High interaction is observed between temperature and pH.

Fig.4 shows the interactive effect of temperature and initial concentration of dye of the solution on percent adsorption of dye onto red mud at constant dosage (0.3 g/l) and pH (2). It shows that as the initial concentration of dye increases, the percentage adsorption decreases. The effect of temperature is found minimum in the range of initial concentrations studied, indicating that the interaction effect of temperature and initial concentration is not very significant.

The three dimensional response surfaces of the combined effect of initial concentration of dye and dosage on adsorption of dye at constant pH (2) and temperature (15°C) is shown in Fig.5. Percentage adsorption decreases with the increase in initial dye concentration and increases with increase in adsorbent dosage. Effect of adsorbent dosage is more significant at higher concentrations. Interaction effect between adsorbent dosage and dye concentration is observed to be highly significant.

The combined effect of pH and adsorbent dose on adsorption of dye at constant temperature (15^{o}C) and initial feed concentration of dye (70 mg/l) is shown in Fig.6.Percentage adsorption decreases with pH up to a certain value of pH and then it increases further with increase in pH, whereas with increase in dosage percentage adsorption increases. Minimum adsorption was found to be at a pH of around 2.5, irrespective of adsorbent dosage. Effect of dosage in entire range of pH seems to be similar. Hence dosage and pH interaction are not very significant.

In Fig.7, It shows the three dimensional response surfaces which were constructed to show the effect of temperature and adsorbent dose on the adsorption of dye at constant pH (2) and initial concentration of dye (70 mg/l). It can be seen from the figure that initially the percentage removal increases very sharply with the increase in adsorbent dosage but beyond a certain value 4 g/l, the percentage removal reaches almost a constant value. Percentage removal increases significantly with the increase in temperature. But the effect of temperature in the entire range of adsorbent dosage is found similar, indicating minimal interaction between these two variables.

Fig.8 shows the three dimensional response surfaces to explain the combined effect of pH and initial concentration on adsorption of dye at constant dosage (0.3 g/l) and temperature (15^{o}C).It is clear from this figure that the minimum adsorption occurs at a certain pH and the value of pH at which minimum adsorption occurs depends on the initial dye concentration. So pH and initial concentration interaction is highly significant. The percentage adsorption is more in lower concentration region and with lower pH values. But the % adsorption decreases with higher concentration and with higher pH values.

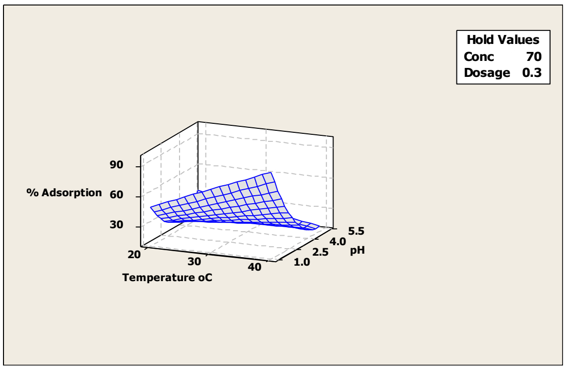


Fig. 3. Surface plot for effect of pH and Temperature on % Adsorption

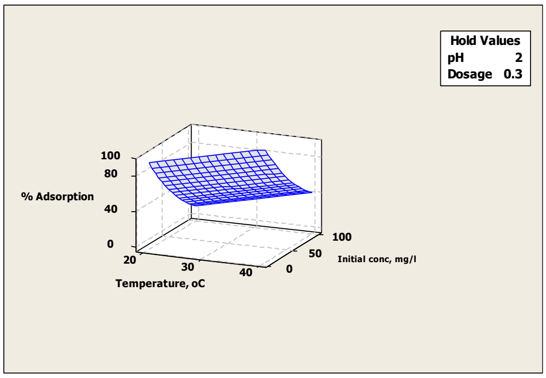


Fig.4. Surface plot for effect of Initial Concentration and Temperature on % Adsorption

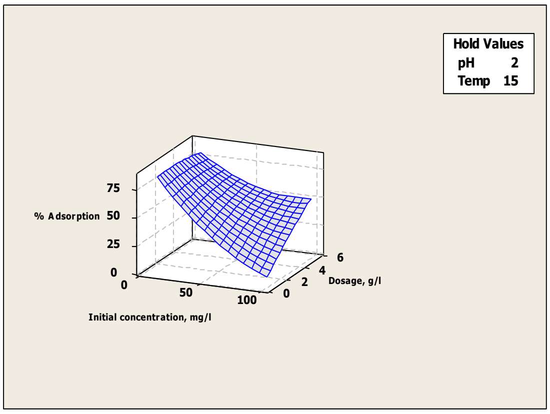


Fig.5. Surface plot for effect of initial concentration and dosage on % Adsorption

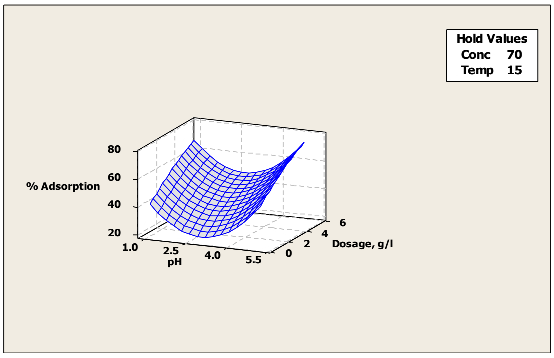


Fig.6. Surface plot for effect of pH and Dosage on % Adsorption

3.4 Adsorption isotherm analysis

The relationship between the amount of dye adsorbed and the equilibrium dye concentration remaining in solution is described by an adsorption isotherm. The linear form of monolayer Langmuir adsorption isotherm given in Eq (2)

$$\frac{C_e}{q_e} = \frac{1}{Q_o b} + \frac{1}{C_e Q_o} \qquad (2)$$

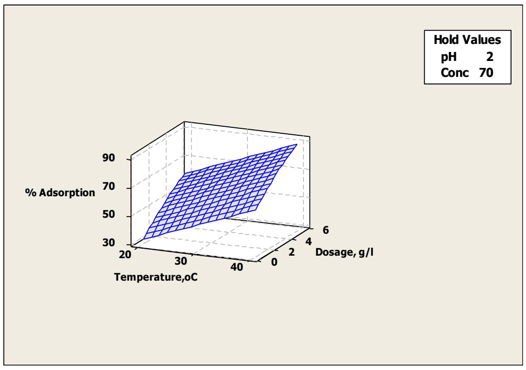


Fig.7. Surface plot for effect of dosage and Temperature on % Adsorption

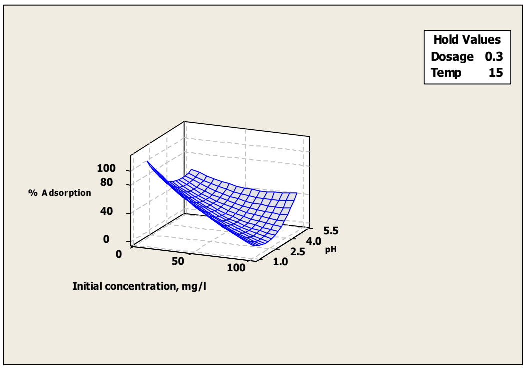


Fig.8. Surface plot for effect of pH and concentration on % Adsorption

Where C_e is the bulk solution concentration of Remazol brilliant blue dye (mg/l) at equilibrium, q_e is the amount of adsorbed dye per unit mass of adsorbent (mg/g), Q_o the monolayer capacity of the adsorbent (mg/g) and b is the Langmuir adsorption constant indicating the adsorption energy (l/mg). The Freundlich isotherm is derived to model the multi layer adsorption and for the adsorption on heterogeneous surfaces. The linear form of equation given in Eq (3)

$$ln q_e = ln k + \frac{ln C_e}{n} \quad (3)$$

Where,k is the sorption capacity (mg/g) and *n* is an empirical parameter which is an indicator of adsorption intensity (Senthilkumar et al. 2009) or the bonding energy between the adsorbent and the dye molecule. The equilibrium data obtained from the experiments were fitted into these two types of isotherms to test the validity of these for dye red mud adsorption system. The values of parameters of the isotherms at three different temperatures of 30°C, along with the corresponding R^2 values representing the goodness of fit are presented in Table 5.The R^2 values indicate that the equilibrium for dye red mud adsorption system can be represented by both Langmuir and Freundlich isotherms under the conditions of the study, but Langmuir model has been found to fit better owing to higher R^2 values. With Freundlich isotherm, the values of n obtained are greater than one. It indicates that adsorption is much more favorable (Rozada et al. 2002).

Table.5. Adsorption Isotherm parameters at 30°C

Temperature °C	Langmuir model	Freundlich Model
30	Q_o=26.17 mg/gram	n =2.17,
	b =0.44 l/mg	k=6.29 mg/g
	R^2=0.98	R^2=0.97

4. Conclusion

The adsorption of Remazol Brilliant Blue dye was studied with red mud treated with dilute Sulphuric acid. The acid treatment was found to increase the surface area of red mud by 7 m^2/g. Langmuir isotherm model has been found to represent the equilibrium data for Remazol Brilliant Blue-Acid Treated Red Mud adsorption system better than Freundlich model for red mud sample. The maximum percentage adsorption of 94% was obtained at 2 pH with initial concentration 10 mg/l and with red mud dosage of 3.2 g/l. Optimization studies were conducted using CCD to analyze the effect of each parameters and interaction of parameters using RSM method for removal of dye. The optimum conditions for red treated with concentrated acid were pH=1, Initial concentration=100 mg/l, red mud dosage=4.8 g/l, and temperature=33°C.

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