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Biosorption of reactive dye using acid-treated rice husk: Factorial design analysis

V. Ponnusami *, V. Krithika, R. Madhuram, S.N. Srivastava

School of Chemical & Biotechnology, SASTRA Deemed University, Thanjavur, India

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Abstract

A factorial experimental design technique was used to investigate the biosorption of reactive red RGB (λ_{max} = 521 nm) from water solution on rice husk treated with nitric acid. Biosorption is favored because of abundance of biomass, low cost, reduced sludge compared to conventional treatment techniques and better decontamination efficiency from highly diluted solutions. Factorial design of experiments is employed to study the effect of four factors pH (2 and 7), temperature (20 and 40), adsorbent dosage (5 and 50 mg/L) and initial concentration of the dye (50 and 250 mg/L) at two levels low and high. The efficiency of color removal was determined after 60 min of treatment. Main effects and interaction effects of the four factors were analyzed using statistical techniques. A regression model was suggested and it was found to fit the experimental data very well. The results were analyzed statistically using the Student's *t*-test, analysis of variance, *F*-test and lack of fit to define most important process variables affecting the percentage dye removal. The most significant variable was thus found to be pH.

Keywords: Rice husk; Biosorption; Reactive dye; Red RGB; Color removal; Factorial design of experiments

1. Introduction

Synthetic dyes are widely used in industries such as textiles, leather, paper, plastics, etc. to color their final products [1]. Reactive dyes are the most common dyes used due to their advantages, such as bright colors, excellent colorfastness and ease of application [2,3]. Colored dye effluents are highly toxic to aquatic life. It reduces the photosynthetic activity and primary production. These dyes are mostly resistant to biodegradation and therefore are not removed by conventional treatment techniques. In general, there are seven main methods used for the treatment of dye-containing effluent: adsorption, oxidation, ozonation, biological treatment, coagulation, flocculation and membrane processes [4,5]. Adsorption technique has been proved to be an excellent way to treat dye effluents, offering advantages over conventional process [6,7]. Adsorption is one of the most efficient methods of removing pollutants from wastewater. Also, the adsorption provides an attractive alternative treatment, especially if the adsorbent is inexpensive and readily available [8]. Many studies have been made on use of different adsorbents like activated carbon [9,10], peat [11], coir pith [8], chitin [1,12], silica [13], fly ash [14], and many others like hardwood sawdust, bagasse pith, rice husk, rice hull, paddy straw, slag, fenugreek mucilage and various blends of these [15–25,29,30]. However search for cost-effective, efficient adsorbent is continuing.

Carbon is widely used as an adsorbent for many species because of its high efficiency. However because of the cost involved search for alternative adsorbent that could provide an economical solution is very important in developing countries like India. In agricultural countries where abundant source of biomass is available biosorption is becoming more and more attractive.

Rice husk can prove to be better alternative for sorption process because it is freely available in countries like India where agriculture is among the major businesses. It is cheap and shows good sorption capacities when properly treated.

In a multivariate experiment, all of the important variables are changed during each run of trials. The need for this arises because the variables often interact with each other. For example, if pH conditions are optimized at one temperature, this work may have to be repeated if it is subsequently found that a different temperature works better. In a multivariate approach, variation

* Corresponding author. Tel.: +91 98943 46540.
E-mail address: vponnusami@hotmail.com (V. Ponnusami).

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of temperature is included in the first round of experiments. This will show the best direction to move within the multidimensional space defined by the major variables. There are several important problems with the conventional approach of changing only one or two variables in a run. It may be take many rounds of experiments to find the optimum point. In cases where variables must be changed in large steps the optimum may not be found at all. The factorial design would reveal the effect of the interaction of process variables and improve optimization process. The relative importance of all the factors can be evaluated simultaneously with less number of experiments [26–28].

Temperature, pH, initial concentration of dye and adsorbent particle dosage are important parameters in adsorption. Thus in the present study capacity of rice husk to remove color was investigated by varying these factors at two levels. Interaction between these factors were studied and optimization done.

2. Materials and methods

2.1. Preparation of adsorbent

Rice husk obtained from a nearby rice mill was screened and then washed with distilled water till the supernatant solution becomes clear. It was then soaked in 2.0 mol/L nitric acid for an hour and then rinsed with distilled water till the supernatant solution reached 7 pH. The washed husk was dried at 105° in oven. The dried husk was screened and BSS −30/+36 mesh size particles were collected and used for the study.

2.2. Preparation of dye solution

Reactive red RGB dye was collected from a textile unit of Thirupur (India). It is a monoazo based, vinylsulphone/monochlorotriazine bifunctional dye. Stock solutions of the dye were prepared in deionized water and were diluted to working concentrations. pH was adjusted by adding either H_2SO_4 or Na_2CO_3 as required.

Table 1
High and low levels of factors

Factor	Low level (−1)	High level (+1)
pH	2	7
Initial concentration (mg/L)	50	250
Dosage (g/L)	5	50
Temperature (°C)	20	40

2.3. Batch adsorption

Stock solution of 1000 mg/L of the dye was prepared and suitably diluted to required initial concentrations (50 and 250 mg/L). The four factors initial concentration of dye, pH, temperature and adsorbent dosage are varied at two levels as shown in the Table 1. For each run, 50 ml of dye solution taken in conical flasks were agitated in incubated shaker at 150 rpm for 1 h. After one hour of contact time samples were withdrawn and filtered. Absorbance was determined using UV–vis double beam Spectrophotometer (Systronics 2201) at a maximum wavelength of 521 nm. Sixteen experiments with all possible combinations of variables were conducted in duplicate, and a matrix was established according to their high and low levels, represented by +1 and −1, respectively.

3. Results and discussion

Results are shown in the Table 2. Removal efficiency was defined as

$$\eta = \left(\frac{C_i - C_{\mathrm{f}}}{C_i}\right) \times 100 \tag{1}$$

where C_i is initial concentration and C_{f} is the final concentration of the dye in the solution. The results were analyzed using MINITAB 14 for windows. The main effects and interaction between factors were determined. The effect of a factor is the change in response, here, percentage color removal produced by

Table 2
Experimental data

Run	pH	D (g/L)	C (mg/L)	T (°C)	Removal efficiency (%)		Removal efficiency (%)
					Trial 1	Trial 2	Average
1	−1	−1	−1	−1	89.36	95.78	92.57
2	1	−1	−1	−1	53.67	52.02	52.84
3	−1	1	−1	−1	86.97	93.76	90.37
4	1	1	−1	−1	72.39	80.55	76.47
5	−1	−1	1	−1	68.46	64.99	66.73
6	1	−1	1	−1	32.44	28.44	30.44
7	−1	1	1	−1	93.19	93.69	93.44
8	1	1	1	−1	88.17	91.41	89.79
9	−1	−1	−1	1	97.25	95.41	96.33
10	1	−1	−1	1	76.42	56.51	66.47
11	−1	1	−1	1	76.24	90.83	83.53
12	1	1	−1	1	79.54	73.21	76.38
13	−1	−1	1	1	84.31	82.84	83.58
14	1	−1	1	1	53.32	44.96	49.14
15	−1	1	1	1	94.77	96.53	95.65
16	1	1	1	1	89.32	90.75	90.04

Table 3
Statistical parameters for 2^4 design

Term	Effects	Coefficients	Standard error	T	P
Constant		77.11	0.9506	81.11	0.000
pH	−21.33	−10.66	0.9506	−11.22	0.000
D	19.70	9.85	0.9506	10.36	0.000
C	−4.52	−2.26	0.9506	−2.38	0.030
T	6.06	3.03	0.9506	3.19	0.006
pH × D	13.75	6.87	0.9506	7.23	0.000
pH × C	1.33	0.67	0.9506	0.70	0.494
pH × T	2.06	1.03	0.9506	1.08	0.294
$D \times C$	15.06	7.53	0.9506	7.92	0.000
$D \times T$	−7.18	−3.59	0.9506	−3.77	0.002
$C \times T$	3.44	1.72	0.9506	1.81	0.089
pH × $D \times C$	1.62	0.81	0.9506	0.85	0.408
pH × $D \times T$	−0.87	−0.43	0.9506	−0.46	0.655
pH × $C \times T$	−2.09	−1.05	0.9506	−1.10	0.288
$D \times C \times T$	−1.10	−0.55	0.9506	−0.58	0.572
pH × $D \times C \times T$	−0.09	−0.04	0.9506	−0.05	0.964

a change in the level of a factor, pH, temperature, initial concentration of dye or adsorbent dosage from lower to higher level. The codified model employed for 2^4 factorial designs was

$$\eta = A_0 + A_1X_1 + A_2X_2 + A_3X_3 + A_4X_4 + A_5X_1X_2 + A_6X_1X_3 + A_7X_1X_4 + A_8X_2X_3 + A_9X_2X_4 + A_{10}X_3X_4 + A_{11}X_1X_2X_3 + A_{12}X_1X_2X_4 + A_{13}X_1X_3X_4 + A_{14}X_2X_3X_4 + A_{15}X_1X_2X_3X_4 \quad (2)$$

where A_0 represents the global mean and A_i represents the regression coefficient corresponding to the main factor effects and interactions. The effects, regression coefficients, standard errors, T and P are shown in Table 3. The main effects represent deviations of the average between high and low levels for each one of them. When the effect of a factor is positive, removal efficiency increase as the factor is changed from low to high levels. In contrast, if the effects are negative, a reduction in removal efficiency occurs for high level of the same factor. Fig. 1 shows

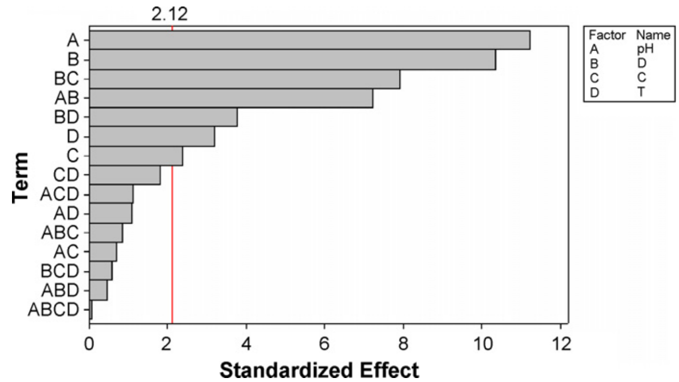

Fig. 2. Pareto chart of standardized effects on the removal efficiency for reactive red RGB—full model.

the main effects of the four factors on percentage color removal. Substituting the regression coefficients in Eq. (2) we get model equation relating the level of parameters and color removal efficiency:

$$\eta = 77.11 - 10.66X_1 + 9.85X_2 - 2.26X_3 + 3.03X_4 + 6.87X_1X_2 - 0.67X_1X_3 + 1.03X_1X_4 + 7.53X_2X_3 - 3.59X_2X_4 + 1.72X_3X_4 + 0.81X_1X_2X_3 - 0.43X_1X_2X_4 - 1.05X_1X_3X_4 + 0.55X_2X_3X_4 - 0.04X_1X_2X_3X_4 \quad (3)$$

3.1. Student's t-test

The Pareto chart (Fig. 2) gives the relative importance of the individual and interaction effects. Student's t-test was performed to determine whether the calculated effects were significantly different from zero and these values for each effect are shown in Pareto chart by horizontal columns. For a 95% confidence level and sixteen degrees of freedom t-value is equal to 2.12. The vertical line in the chart indicates the minimum statistically significant effect magnitude for 95% confidence level.

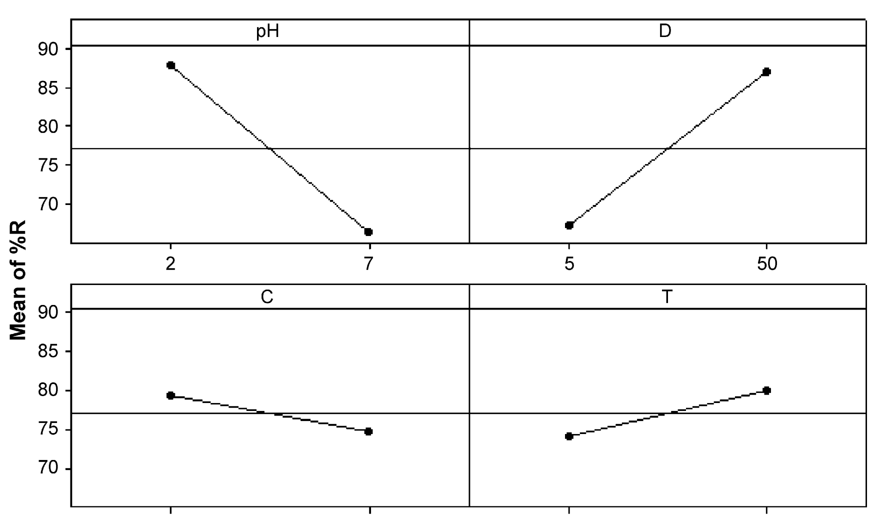

Fig. 1. Main effects plot for percentage dye removal (%R).

Table 4
Analysis of variance—full model fitting

Source	Degrees of freedom	Sum of squares (SS)	Mean square (MS)	F	P-value
pH	1	3639.32	3639.32	125.85	0.000
D	1	3103.37	3103.37	107.31	0.000
C	1	163.40	163.40	5.65	0.030
T	1	293.63	293.63	10.15	0.006
pH × D	1	1512.19	1512.19	52.29	0.000
pH × C	1	14.18	14.18	0.49	0.494
pH × T	1	33.97	33.97	1.17	0.294
D × C	1	1815.10	1815.10	62.77	0.000
D × T	1	411.88	411.88	14.24	0.002
C × T	1	94.87	94.87	3.28	0.089
pH × D × C	1	20.89	20.89	0.72	0.408
pH × D × T	1	6.00	6.00	0.21	0.655
pH × C × T	1	34.96	34.96	1.21	0.288
D × C × T	1	9.64	9.64	0.33	0.572
pH × D × C × T	1	0.06	0.06	0.00	0.964
Error	16	462.69	28.92		
Total	31	11616.16			

$S = 5.37758$; $R^2 = 96.02\%$; $R^2_{(adj)} = 92.28\%$; $F = \text{adj. MS}_{factor}/\text{adj. MS}_{Error}$.

3.2. Analysis of variance (ANOVA)

In Table 4 the sum of squares used to estimate the factors' effects and F-ratios are shown. Since $F_{0.05,1,16} = 4.49$, all the effects with F value higher than 4.49 are significant. The effects are statistically significant when P-value, defined as the smallest level of significance leading to rejection of null hypothesis, is less than 0.05.

Based on the student's t-test and F-test, few interaction effects which seem insignificant compared to other effects, were neglected and the effects, regression coefficients, standard error, t and p-value were recalculated with remaining variables. Resultant values are shown in Table 5. Fig. 3 shows the student's t-test results. Reduced model equation with resultant coefficients was

$$\eta = 77.11 - 10.66X_1 + 9.85X_2 - 2.26X_3 + 3.03X_4 + 6.87X_1X_2 + 7.53X_2X_3 - 3.59X_2X_4 \quad (4)$$

For the reduced model ANOVA and F-test and were performed and results of ANOVA are given in Table 6. Interaction plot of effects is shown in Fig. 4. Lack of fit associated elimination of few factors was $F = 0.93$. This was very much lower compared to tabulated value $F = 2.59$. Therefore, these factors did not have statistical significance. The residues should also be examined for normal distribution. Fig. 5 shows normal probability plot of residual values. It could be seen that the experimental points were reasonably aligned suggesting normal distribution.

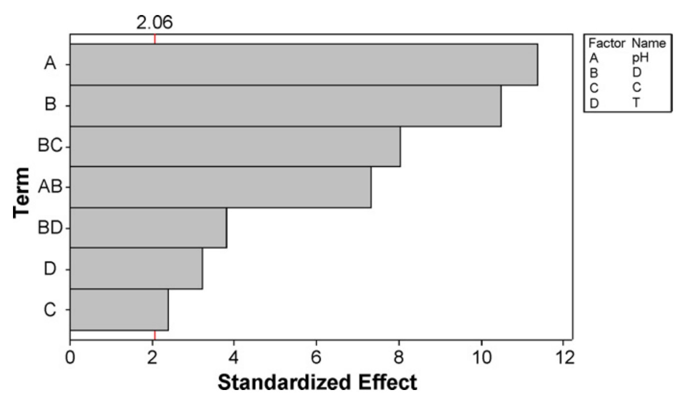

Fig. 3. Pareto chart of standardized effects on the removal efficiency for reactive red RGB—reduced model.

Table 5
Statistical parameters for 2^4 design—reduced model

Term	Effect	Coefficient	Standard error	T	P
Constant		77.11	0.9391	82.11	0.000
pH	−21.33	−10.66	0.9391	−11.36	0.000
D	19.70	9.85	0.9391	10.49	0.000
C	−4.52	−2.26	0.9391	−2.41	0.024
T	6.06	3.03	0.9391	3.23	0.004
pH × D	13.75	6.87	0.9391	7.32	0.000
D × C	15.06	7.53	0.9391	8.02	0.000
D × T	−7.18	−3.59	0.9391	−3.82	0.001

Table 6
Analysis of variance for percentage color removal—reduced model

Source	Degrees of freedom	Sum of squares (SS)	Mean square (MS)	F	P-value
pH	1	3639.3	3639.3	128.97	0.000
D	1	3103.4	3103.4	109.97	0.000
C	1	163.4	163.4	5.79	0.024
T	1	293.6	293.6	10.41	0.004
pH × D	1	1512.2	1512.2	53.59	0.000
D × C	1	1815.1	1815.1	64.32	0.000
D × T	1	411.9	411.9	14.60	0.001
Residual error	24	677.3	28.22		
Lack of fit	8	214.6	26.82	0.93	0.520
Pure error	16	462.7	28.92		
Total	31	11616.2			

$S = 5.31219$; $R^2 = 94.17\%$; $R^2_{(adj)} = 92.47\%$; $F = \text{MS}_{factor}/\text{MS}_{error}$.

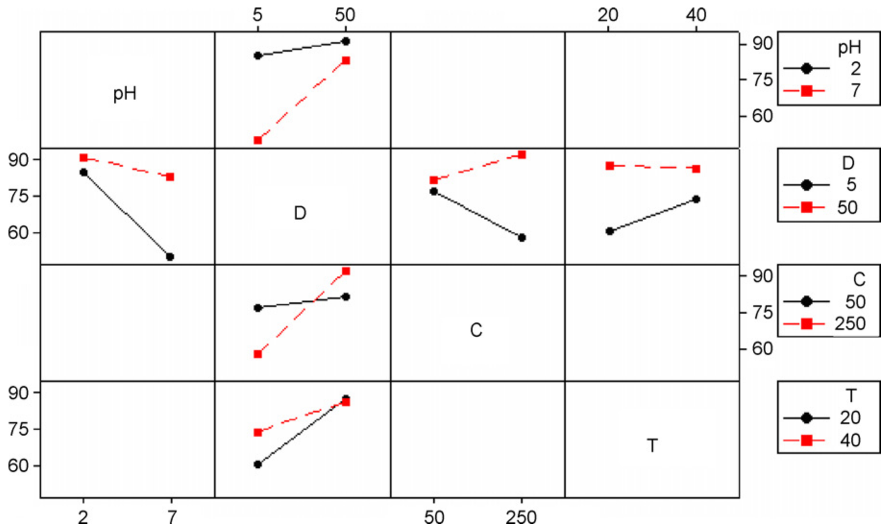

Fig. 4. Interaction effects for percentage dye removal.

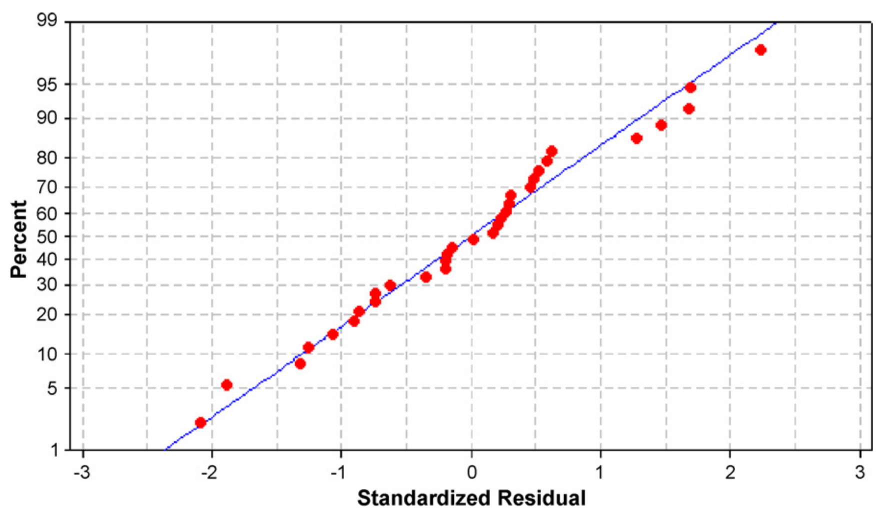

Fig. 5. Normal probability plot of residual values for removal efficiency of reactive red dye vs. their expected values when the distribution is normal.

Plot of percentage dye removal versus residual (Fig. 6) indicated the outliers if any. Except for two points (run number 10) all other points were found to fall in the range of +2 to −2. Points corresponding to run 10 were slightly out of this range.

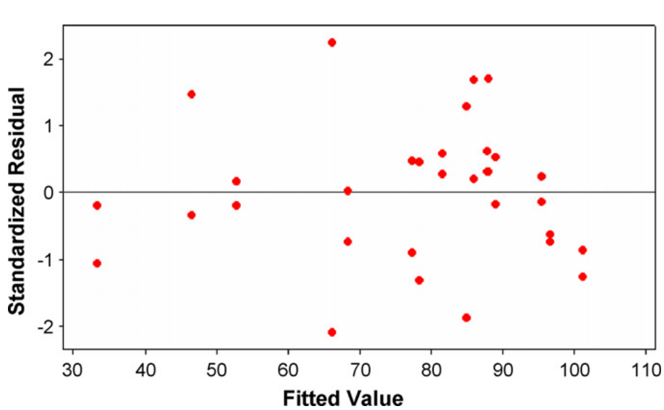

Fig. 6. Percentage dye removal (predicted) vs. residual.

However, elimination of these points did not reduce but increase the lack of fit suggesting that they were not really outliers.

3.3. *Effect of pH*

pH plays a major role in removal of red dye. $P < 0.05$, as indicated in Table 3, shows that pH has a significant effect. Increase in pH from 2 to 7 decreases the adsorption efficiency by about 21.33%. Low pH favors adsorption of reactive red dye on rice husk treated with nitric acid. Similar trends biosorption were reported for three similar reactive dyes by Gong et al. [35] in terms of dye adsorption ratio.

3.4. *Effect of temperature*

Adsorption is favored by increase in temperature. This in an unexpected behavior, but it has been reported by some authors when studying the adsorption of different type of dyes and other organic compounds over several adsorbents McKay et al. [31, Moreira et al. [32–34], Ravikumar et al. [27], Asfour et al. [19]. McKay et al. [31] suggested that this behavior is due to the possibility of an increase in the porosity and total pore volume of the adsorbent with the increase of the temperature. Ravikumar et al. [27] suggested that increase in temperature produce swelling of the internal structure of the adsorbent causing increased penetration of the larger dye molecules. The increase of the color removal efficiency with the increase of the temperature is still not well understood and it is a phenomenon worthy of further investigation. The observations of strong influence of the effects pH, and increase in percentage color removal with temperature suggest ion-exchange mechanism [36].

3.5. *Effect of adsorbent dosage and initial concentration of dye*

Adsorption rate increase, as expected, with increase in adsorbent dosage. Increase of adsorbent dosage from 5 to 50 g/L increases adsorption rate by 19.7%. This is due to the fact that, increase in adsorbent dosage increase area available for adsorption. Also as expected increase of initial concentration decrease adsorption rate. Increase from 50 to 250 mg/L result in decrease of decolorisation efficiency by 4.52%. At higher concentration adsorption efficiency decrease due to saturation of all adsorption sites.

Apart from these linear effects of the parameters used, the interaction between these parameters gives better explanation of the process. As mentioned above *F*-test and student's *t*-test were used to study the interaction effects. Interaction plot (Fig. 4) clearly indicates strong interaction between adsorbent dosage and initial concentration of dye, pH and adsorbent dosage and dose and temperature. When pH is decreased from 7 to 2, decolorisation efficiency increases from 49.72 to 84.80% at 5 g/L dose and 83.17 to 90.75% at 50 g/L. That is the effect of pH is high when the dosage is low, but at higher dosage pH effects are not that high. Similarly at low concentration color removal efficiency was only 77.05 and 81.69% at 5 and 50 g/L dosage level, respectively. But at higher initial concentration, 250 mg/L, change of adsorbent dosage from 5 to 50 g/L increases color removal efficiency by 34.76%, i.e. from 57.47 to 92.23%. Similarly change adsorbent dosage produced 26.87 and 12.52% decrease in percentage color removal at lower and higher temperatures respectively. In conventional design of experiments these interaction will not be revealed, and so missed.

4. Conclusion

The results of present study clearly show that nitric acid treated rice husk is effective in removal of reactive dye and can provide an economical solution for removal of such dyes from aqueous solutions. Up to 96.33 and 95.65% color removal could be achieved in 1 h contact from aqueous solutions with initial concentrations 50 and 250 mg/L, respectively. For 50 mg/L solution maximum color removal 96.33% achieved with 2 pH, 5 g/L dose, and 40 C. Like wise for 250 mg/L solution maximum color removal was achieved with 2 pH, 50 g/L dose and 40 C. The most significant effect was found to be pH. Then adsorbent dosage, adsorbent dosage and initial concentration of dye interaction and pH–initial concentration of dye interaction were also found highly significant.

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