Statistical analysis of *Litchi chinensis*'s adsorption behavior toward Cr(VI)

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Abstract

The adsorption results of Cr(VI) removal from aqueous solutions on *Litchi chinensis* have been optimized by the Box–Behnken design of response surface methodology. Three experimental parameters (dose, temperature, and pH) were chosen as independent variables. The maximum Cr(VI) adsorption was obtained at the initial pH of 2. Analysis of variance (ANOVA) of the results was successfully used to check the significance of the independent variables and their interactions. The three-dimensional (3D) response surface plots were used to study the interactive effects of the independent variables on % Cr(VI) removal. These figures successfully interpret the effect of interaction between pH (0.1–1.0), adsorbent dose (0.1–1.0 g.) and temperature (0–50 °C). The second-order polynomial equation was generated for the response. A statistical hypothesis test was conducted to critically analyze the experimental data by applying *t* test, paired *t* test, and Chi-square test. The comparison of *t*-calculated and *t*-tabulated values showed that the results were in favour of the conducted experiment.

Keywords *Litchi chinensis* · Box–Behnken design · Statistical analysis · Hypothesis test

Introduction

From many times, the presence of heavy metals in the water sources is a serious environmental problem and is dangerous to the human health worldwide. These inorganic pollutants are highly toxic because they are not biodegradable; thus, researches are going on for decades to find some methods to adsorb metal ions from aqueous solution. Among them, chromium has become a serious health concern because it causes many severe diseases. Strong exposure to Cr(VI) causes cancer in the digestive tract and may cause epigastric pain, nausea, vomiting, severe diarrhoea, and haemorrhage (Mohanty et al. 2005). Chromium is one of the contaminants, which exist in hexavalent [Cr(VI)] and also in the trivalent form [Cr(III)] (Rao et al. 2015). Chromium and its compounds are widely used in many industries such as metal finishing, dyeing, pigments, inks, glass, ceramics, tanning, textile, wood preserving, electroplating, steel fabrication, and canning industries (Rao et al. 2015). Chromium (VI) is more toxic to human physiology because of its mutagenic

and carcinogenic properties, which may even lead to death (Rao et al. 2015). The chromate (CrO_4^{2-}) and dichromate ($\text{Cr}_2\text{O}_7^{2-}$) forms of Cr(VI) are extremely hazardous (Elidoro et al. 2017), and the maximum acceptable limit by various standards has been set (Uddin 2017). Permissible limits for Cr(VI) in drinking water (mg/L) according to various standards are very low: 0.050 (IS 10500) (FAD 25: Drinking Water 2012), 0.050 (WHO) (Uddin 2017), 0.100 (EPA) (Uddin 2017), 0.050 (EU standard) (Wikipedia).

Many processes like precipitation (Esalah and Husein 2008), chemical oxidation (Kaur and Crimi 2014), reverse osmosis (Çimen 2015), electrochemical treatment (Ruotolo et al. 2006), emulsion (Nosrati et al. 2011), ultrafiltration (Muthumareeswaran et al. 2017), photo-catalysis (Machado et al. 2014), ion exchange (Kononova et al. 2009), pre-concentration (Rao and Kashifuddin 2012a), evaporation (Sachitanand et al. 2013), sedimentation (Vukić et al. 2008), adsorption (Khatoon et al. 2018; Naushad et al. 2015; Alqadmi et al. 2016; Uddin and Bushra 2017) have been developed to remove this toxic Cr(VI) from aqueous solution. Out of these methods, adsorption is a widely used and most efficient method to eliminate heavy metals from contaminated water (Rao and Kashifuddin 2012a, b). This technique is superior because of its low cost, ease of operation, efficiency in treatment, good applicability, high capacity,

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reliability, less energy consumption, and simplicity (Rao and Kashifuddin 2016; Khan et al. 2014). Various effective and low-cost adsorbents have been used for the removal of Cr(VI) recently (Eliodorio et al. 2017; Suriga 2017; Ali et al. 2016; Eldin et al. 2017; Lee et al. 2017; Mullick et al. 2018; Fan et al. 2017; Qi et al. 2016; Panda et al. 2017; Ali et al. 2016; Gorzin and Abadi 2018).

Red- or pink-red-colored smooth fruit peel of litchi tree (*Litchi chinensis*) covered with small sharp protuberances was successfully utilized as a low cost, efficient, waste adsorbent for the removal of Cr(VI) from wastewater (Rao et al. 2012; Yi et al. 2017). The results of the research studies investigated by Rao et al. 2012 and Yi et al. 2017 concluded that Litchi peel exhibited remarkable adsorption capacity toward Cr(VI) ions, as investigated by the effect of various parameters such as pH, contact time, temperature, adsorbent amount, and initial Cr(VI) concentration. Different isotherms, thermodynamics, and kinetics parameters also showed the effectiveness of Litchi peel to treat hexavalent chromium containing wastewater (Rao et al. 2012).

To confirm the reliability of experimental data, a statistical optimization process that is known as response surface methodology (RSM) was used (Mondal et al. 2017). RSM is a multivariate, computational statistical technique in which the experimental adsorption data were fitted in a second-order polynomial equation, and finally, it was analyzed by performing tests of variance and lack of fit (Mondal et al. 2017). The Box–Behnken design (BBD) is one of the available designs of response surface methodology that is used to optimize the adsorption process (Simsek et al. 2015). It was used in many successful recent studies to validate the experimental results (Mondal et al. 2017; Loqman et al. 2016; Okwadha and Nyingi 2016; Siva Kiran et al. 2017; Igberase et al. 2017; Kavitha and Thambavani 2016; Ma et al. 2016; Wei et al. 2016; Perez et al. 2017). The objective of the present work is to optimize the efficiency of litchi fruit peel in Cr(VI) removal from electroplating wastewater (Rao et al. 2012). The variables like pH, solution temperature, and adsorbent dose were optimized to evaluate the combined and interactive effects of the variables in the process for the removal of chromium ions from aqueous solution.

Materials and experimental methods

The dried peel of Litchi fruit was used in the form of powder to remove the Cr(VI) using the batch experimental procedure, the same as investigated before by Rao et al. 2012. Cr(VI) adsorption experiments were then conducted by defined concentration and volume of the metal ion with different doses of adsorbent (0.1, 0.5, and 1.0 g) at different pH values (2.0, 6.0, and 10.0) and temperatures (30, 40,

and 50 °C). Double-distilled water (DDW) was used in all adsorption experiments.

Percent adsorption of hexavalent chromium ion was calculated as follows:

$$\% \text{ adsorption} = \frac{C_0 - C_e}{C_0} \times 100$$

where C_0 and C_e are initial and equilibrium concentrations of Cr(VI), respectively. The adsorption capacity of Cr(VI) was calculated as follows:

$$q_e = \left(\frac{C_0 - C_e}{m} \right) \times V$$

where m is mass of adsorbent and V is the volume of solution.

Statistical analysis of adsorption data

In order to test the effect of different variables in terms of statistical design for adsorption process, a three-level, three-factorial Box–Behnken methodology (Box and Behnken 1960a, b) was employed to analyze the optimal combination of independent variables resulting in maximum Cr(VI) adsorption capacity. Box–Behnken model may be represented as

$$Y = \beta_0 + \sum_{i=1}^n \beta_i X_i + \sum_{i=1}^n \beta_{ii} X_i^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^n \beta_{ij} X_i X_j + \varepsilon$$

where Y is the objective to optimize the response, β_0 is the constant coefficient, β_i is the linear coefficient, β_{ii} is the quadratic coefficient, β_{ij} is the interaction coefficient, ε is random error, while X_i and X_j are the coded values of the independent process variables. The variables were dose (0.1, 0.5, and 1.0 g), pH value (2.0, 6.0, and 10.0), and temperature (30, 40, and 50 °C). These parameters of adsorption test were considered as independent variables, while the response was percentage Cr(VI) removal. Experimental results were analyzed by estimated determination and regression coefficients using Minitab 17 (statistical software). Analysis of variance (ANOVA) was conducted to test the statistical significance of the designed model. The predicted values were then plotted in surface response and 3D graphs for the final optimization procedure. To verify the prediction model, three experimental units under optimal conditions were performed. Experimental range and various levels of independent variables are listed in Table 1. As shown in Table 1, the three factors (dose, temperature, and pH) chosen for this study were designated as X_1 , X_2 , and X_3 (coded) and prescribed into three levels as +1, 0, −1 for high, intermediate, and low values, respectively.

Table 1 Levels and codes of selected variables for Box–Behnken design

Variables	Symbol	Coded levels		
		Coded	− 1	0
Dose	X_1	0.10	0.55	1.00
Temperature	X_2	30	40	50
pH	X_3	2	6	10

The applicability of the adsorption process can also be monitored, qualitatively and quantitatively, by applying statistical hypothesis testing (Kaushal and Singh 2016). In this type of statistical analysis, the effect of different factors like pH value, time, and concentration on % adsorption of Cr(VI) was examined. In each case, the null hypothesis (H_0 : $\mu_1 = \mu_2$) was assumed that the factors did not effect on the % adsorption, while the alternative hypothesis (H_a : $\mu_1 > \mu_2$) was assumed that the experimental parameters were effective and caused an increase in % adsorption. The analysis was tested using t test, paired t test, and Chi-square test within 5% level of confidence.

Results and discussion

Response surface methodology and statistical analysis

Box–Behnken design

The Box–Behnken experimental design is a very powerful tool to determine the optimal level of process parameters

with less number of experiments when compared with other designs of experimental models. In this paper, the selected variables (temperature, dose, and pH) were taken for optimization. The design matrix of experimental results by putting all three independent variables, in their reaction conditions, and their observed response values are (given in Table 2). A comparison of observed and predicted Cr(VI) percentage removal is also given in the same Table 2. The difference between the values is less, indicating that the experimental values are quite accurate and have reliability with the predicted values.

The empirical model equation for Cr(VI) adsorption performance of *Litchi chinensis* was then generated and can be described by the following equation:

$$\text{response}(Y) = 9.7 + 89.20X_1 + 2.69X_2 - 4.55X_3 - 69.8X_1^2 - 0.363X_2^2 + 0.039X_3^2 + 0.722X_1X_2 + 0.83X_1X_3 - 0.0125X_2X_3$$

The linear term of coefficients X_1 and X_2 showed positive, favorable, and a significant effect on the response, while X_3 showed a negative effect, which is opposite, if compared, to quadratic terms in which X_1^2 and X_2^2 showed negative, while X_3^2 showed a positive effect on the response (Y). Interaction terms (X_1X_2 , X_1X_3) of the same parameter indicated positive and favorable effect, while X_2X_3 showed negative and unfavorable effect on percentage removal of Cr(VI).

To determine the interaction effects of independent variables on Cr(VI) adsorption onto *L. chinensis*, an ANOVA (analysis of variance) test was also performed as shown in Table 3. The results of ANOVA demonstrated that the effect of various factors on Cr(VI) adsorption was highly significant in describing the observed experimental data as per model F value of 44.45 and low p value (0.000). Table 4 also

Table 2 Box–Behnken design matrix (BBD) in terms of coded values and the comparison of experimental and predicted % Cr(VI) removal

Run	Dose	Temp.	pH	X_1	X_2	X_3	% Cr(VI) removal (Y)	
							Experimental	Predicted
1	0.1	30	6	–1	0	–1	40	40.50
2	1	30	6	0	–1	–1	80	75.75
3	0.1	50	6	–1	1	0	32	36.20
4	1	50	6	0	0	0	85	84.50
5	0.1	40	2	0	–1	1	62	60.62
6	1	40	2	–1	–1	0	96	99.37
7	0.1	40	10	1	0	1	28	24.62
8	1	40	10	0	0	0	68	69.37
9	0.55	30	2	0	0	0	88	88.87
10	0.55	50	2	–1	0	1	95	92.12
11	0.55	30	10	1	–1	0	54	56.87
12	0.55	50	10	1	1	0	59	58.12
13	0.55	40	6	0	1	1	77	77.00
14	0.55	40	6	0	1	–1	77	77.00
15	0.55	40	6	1	0	–1	77	77.00

Table 3 Analysis of variance in the regression model for the optimization of Cr(VI) ions

Source of variations	Degree of freedom	Sum of square	Mean square	value	P value
Regression	9	6500.48	722.28	44.45	0.000
Linear	3	5674.25	1891.42	116.39	0.000
Dose (X_1)	1	3486.13	3486.13	214.53	0.000
Temp (X_2)	1	10.13	10.13	0.62	0.466
pH (X_3)	1	2178.00	2178.00	134.03	0.000
Square	3	773.98	257.99	15.88	0.055
(X_1) ²	1	736.67	736.67	45.33	0.001
(X_2) ²	1	48.52	48.52	2.99	0.145
(X_3) ²	1	1.44	1.44	0.09	0.778
Two-way interaction	3	52.25	17.42	1.07	0.329
X_1X_2	1	42.25	42.25	2.60	0.168
X_2X_3	1	9.00	9.00	0.55	0.490
	1	1.00	1.00	0.06	0.814
Error	5	81.25	16.25		
Lack of fit	3	81.25	27.08		
Pure error	2	0.00	0.00		
Total	14	6581.73			

Table 4 Estimated value of coefficient regression for the fitted quadratic polynomial model of Cr(VI) ion

Term	Effect	Coeff.	Standard error	T value	P value
Constant		77.00	2.33	33.08	0.000
X_1	41.75	20.88	1.43	14.65	0.000
X_2	2.25	1.12	1.43	0.79	0.466
X_3	-33.00	-16.50	1.43	-11.58	0.000
X_1X_1	-28.25	-14.13	2.10	-6.73	0.001
X_2X_2	-7.25	-3.63	2.10	-1.73	0.145
X_3X_3	1.25	0.63	2.10	0.30	0.778
X_1X_2	6.50	3.25	2.02	1.61	0.168
X_1X_3	3.00	1.50	2.02	0.74	0.490
X_2X_3	-1.00	-0.50	2.02	-0.25	0.814
R^2		0.987			
R^2 adj		0.965			

indicates that all of the study factors have significant linear effects as X_1 and X_2 have positive effects. The good quality of the developed model can also be judged and confirmed by the high value of model coefficients of determination coefficient ($R^2 = 0.987$) (Table 4). The normal probability plot of the residuals is one of the most important diagnostic tools to determine the systematic departures from the assumption that errors have a normal distribution (Yetilmezsoy et al. 2009). Figure 1 of the plot indicates that the most of the data points in the analysis are close to the straight-line means the ideal normal distribution of the experimental data and support the well fitting of experimental results. Figure 2 shows three-dimensional (3D) response surface plots as a function

of two variables is the best way to identify the interaction effects of each other. The interactive effect of the combination of independent variables (dose, temperature, and pH) on Cr(VI) percentage adsorption, is shown in Fig. 2a, b. Figure 2a shows the 3D response surfaces representing the influence of adsorbent dose and temperature on Cr(VI) removal, while Fig. 2b represents the influence of adsorbent dose and pH on Cr(VI) removal by *L. chinensis*. It is clear from Fig. 2a, b that adsorbent dosage had a significant effect which led to increase percentage Cr(VI) removal up to maximum along with increasing temperature and decreasing pH. This finding of increase in Cr(VI) adsorption (%) with decrease in solution pH and maximum at pH 2 is common which is supported by loads of previous studies of Cr(VI) adsorption (Rao et al. 2012, 2015; Mullick et al. 2018; Mondal et al. 2017; Mohanty et al. 2005; Lamhamdi et al. 2017; Igberase et al. 2017). It can also be understood by the speciation of Cr(VI) (adsorbate) ion, as solution pH is an important parameter in Cr(VI) adsorption. At pH 2, Cr(VI) ion exists in solution in the form of HCrO_4^- (Doke and Khan 2017; Yang et al. 2009), which could adsorb on protonated surface sites of the adsorbent by electrostatic attraction mechanism, and leads to increase in adsorption. At high pH (more than 6), the existence form of hexavalent chromium is CrO_4^{2-} and surface becomes less protonated that leads to decrease in Cr(VI) adsorption, also due to exchange of Cr(VI) ions with predominant OH^- ions (Rao et al. 2015; Yang et al. 2009). These results confirmed that electrostatic attraction or ion exchange process between the surface and species was the main adsorption mechanism for Cr(VI) uptake. The increase in Cr(VI) removal with the increase in temperature indicated

Fig. 1 Normal probability plot **Fig. 2** **a** 3D surface plot of response (Y) vs adsorbent dosage (X_1) and solution temperature (X_2). **b** 3D surface plot of response (Y) vs adsorbent dosage (X_1) and pH (X_3)

the endothermic process, which means the chemical change with absorption of heat. The same results were experimented using *L. chinensis* by a batch process in previous studies (Rao et al. 2012; Yi et al. 2017).

Hypothesis testing

The hypothesis testing was conducted: (1) to judge the optimum value of pH, concentration and time for maximum Cr(VI) removal from solution, (2) to judge the success of the experiment by checking higher R^2 value, (3) to infer that the higher adsorbent dosage resulted in higher % removal of chromium ions.

In this subsection, the testing hypothesis analysis of the experimental data of pH effect is under consideration. Upon

Table 5 Effect of pH on % removal of Cr(VI) ion

n	pH	% Removal (X_1)
1	2	96
2	4	88
3	6	72
4	8	62
5	10	54

using statistical hypothesis tests, it can confirm that from the result of pH study (Rao et al. 2012), the optimal pH value for maximum removal of Cr(VI) ions from the water solution

was 2. To further analyze this, a two-tailed t test was used for the obtained experimental data as shown in Table 5.

- (a) To describe the hypothesis testing process qualitatively and quantitatively, the following statistical assumptions were made:

null hypothesis (H_0): the optimum pH for Cr(VI) adsorption was 2, and alternate hypothesis (H_a): the optimum pH $\neq 2$. It was determined with significance level $\alpha=0.05$ and $(n-1)$ degree of freedom.

Upon using the two-tailed t -test with a degree of freedom 4 using formula $t = \frac{\bar{X} - \mu}{\frac{s}{\sqrt{n}}}$ (Hogg and Craig

2014), it was found that $t_{\text{calculated}}$ was -2.757 while $t_{\text{tabulated}} = \pm 2.776$, which means that $t_{\text{calculated}} < t_{\text{tabulated}}$. This result of hypothesis testing confirms that null hypothesis H_0 can be accepted (that the optimum pH of Cr(VI) adsorption was 2), which also in accordance with the experimental result. Figure 3 shows probability chart for t distribution for testing.

- (b) Paired t test was used to test the hypothesis of matched pairs of concentration effect (before and after the adsorption experiment), as shown in Table 6. It was assumed that

H_0 : no changes in the final concentration of Cr(VI) ions after adsorption experiment.

H_a : Successful adsorption was achieved after the experiment.

The calculations have been done using the formula: $t = (D^- - \mu) / (S / \sqrt{n})$ (Hogg and Craig 2014), at 6 degree of freedom. It was found that $t_{\text{calculated}}$ (4.056) $> t_{\text{tabulated}}$ (± 2.447), which means that the null hypothesis can be rejected and alternative hypothesis (that the adsorption experiment was successfully achieved) can be accepted. Figure 4 shows the probability chart for t distribution for paired t test.

Table 6 Cr(VI) ion concentrations in the solution before (X_i) and after (Y_i) the experiment

n	X_i	Y_i	$D_i = X_i - Y_i$
1	10	0.86	9.14
2	20	1.80	18.20
3	30	2.79	27.21
4	50	4.54	45.46
5	60	5.38	54.62
6	80	7.31	72.69
7	100	9.28	90.72

- (c) Chi-square test was applied to test the effectiveness of time on final concentration and equilibrium capacity. To describe this statistically, the assumptions were

H_0 : There was no effect of time on initial concentration

H_a : Adsorption was rapid, and equilibrium adsorption capacity (q_e) increased with the increase in concentration

Under significance level $\alpha=0.05$ and using the experimental data (as shown in Table 7), the formula $\chi^2 = \sum_{i=1}^n \frac{(O_{ij} - E_{ij})^2}{E_{ij}}$ (Hogg and Craig 2014) was used

to conduct Chi-square test. It was found that $\chi^2_{\text{calculated}} = 0.8220 < \chi^2_{\text{tabulated}} = 32.3600$. Hence, the null hypotheses did not fall in the accepted region and cannot be accepted, which also means that the alternative hypothesis H_a can be accepted at value level of 0.05. It means that adsorption of Cr(VI) was fast and equilibrium capacity (q_e) increased with the increase in Cr(VI) concentration and reached equilibrium in short time (Fig. 5).

Fig. 3 Probability chart for t distribution for two-tailed test

Fig. 4 Probability chart for t distribution for paired t test

Table 7 Observed frequencies on the effect of time (min) and concentration (mg/L) for the Cr(VI) ion equilibrium adsorption capacity (qe)

Time (min)	10 mg/L qe (mg)	20 mg/L qe (mg)	30 mg/L qe (mg)	50 mg/L qe (mg)	60 mg/L qe (mg)	80 mg/L qe (mg)	100 mg/L qe (mg)
5	0.02	1.31	2.17	3.70	4.52	6.35	8.20
10	0.68	1.42	2.25	3.79	4.62	6.43	8.27
15	0.73	1.64	2.49	3.96	4.7	6.51	8.46
20	0.86	1.82	2.63	4.08	4.97	6.77	8.61
30	0.86	1.80	2.72	4.22	5.08	6.83	8.68
60	0.86	1.80	2.79	4.53	5.36	7.28	9.16
120	0.86	1.80	2.79	4.54	5.38	7.31	9.24
180	0.86	1.80	2.79	4.54	5.38	7.31	9.28
240	0.86	1.80	2.79	4.54	5.38	7.31	9.28
S_D	0.34	0.22	0.23	0.21	0.23	0.21	0.20

 S_D cumulative = 0.049 **Fig. 5** Probability chart for Chi-square distribution

Conclusion

In total, 96% Cr(VI) from polluted water can be removed using the fruit peel of *L. chinensis* by a batch process, which demonstrated its efficient analytical applicability. Critical analysis of the interactive effects of independent variables: initial pH of the solution, dose, and temperature for better understanding of Cr(VI) adsorption onto *L. chinensis* was successfully studied by Box–Behnken design. The results showed that the values of R^2 and adjusted R^2 were quite close to each other, indicated that the model analyzed the experimental data quite well. The linear terms (X_1 , X_2 , X_3), square values (X_1^2 , X_2^2 , and X_3^2), and their two-way interaction (X_1X_2 , X_2X_3) were found to be significant with low P values, suggesting that these variables have important role in Cr(VI) removal. Hypothesis testing was further studied to confirm the fitting of experimental results. Two-tailed t test, paired t test, Chi-square test within 5% level of confidence were tested, and the results showed that the calculated values were inside the

acceptance region in probability chart. Experimental and predictable data of adsorption experiments were close to each other which also confirmed that *L. chinensis* was an excellent adsorbent to bind Cr(VI) ion.

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