



APPLICATION OF RESPONSE SURFACE METHODOLOGY FOR OPTIMIZATION OF LEAD (II) REMOVAL FROM AQUEOUS SOLUTION BY *LEMNA MAJOR* BIOMASS

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ABSTRACT

The main aim of this study was to investigate the individual and combined effect of four process parameters, *i.e.* initial lead (II) concentration, adsorbent dose, initial solution pH, and contact time on lead (II) adsorption from aqueous solution using *Lemna major* biomass. In the present study, response surface methodology (RSM) approach using Box-Behnken design (BBD) was applied to develop mathematical model and to optimize process parameters by performing thirty batch experiments and the adsorption efficiency was modelled using second-order regression polynomial equation. Analysis of variance (ANOVA) showed the relative significance of process parameters in removal process. The R^2 , adjusted R^2 and predicted R^2 values were found to be 0.9875, 0.9758 and 0.9643, respectively, which indicates that the actual and the predicted values are in good agreement with each other. Based on the removal efficiency and economic use of adsorbent, the independent variables were optimized by two procedures. The desirability of first and second optimization procedures were found to be 1.00 and 0.864, respectively, which shows that the estimated function may well explain the experimental model. The optimized result obtained from RAMP plots suggested that the *Lemna major* biomass was supposed to be an effective and economically feasible biosorbent for the removal of lead (II) from aqueous solution.

Keywords: Response surface methodology, *Lemna major*, adsorption, biomass, desirability.

1. INTRODUCTION

The removal of heavy metal pollutants from aqueous solutions is one of the most important environmental concerns due to their high toxicity and impact on human health. Lead exposure in human cause severe damage to brain, kidney, stomach and even can cause miscarriage in pregnant women [1]. It is also regarded as a general metabolic poison and enzyme inhibitor [2]. Lead is widely used in painting, petrochemical, newsprint, smelting, metal electroplating, mining, plumbing and battery industries producing large quantities of effluents containing the toxic metal. According to the US Environmental Protection Agency (EPA) the permissible level for lead in drinking water is 0.05 mg/L and according to Bureau of Indian Standards (BIS) limit is 0.1 mg/L [3]. But the industrial effluents contain much higher concentration of lead compared to the permissible limit. Therefore, the concentration of such a toxic heavy metal must be reduced to levels in terms of protection of public health and environment. Traditional techniques for removing heavy metal ions from water and wastewater include chemical precipitation, ion-exchange,

electrochemical deposition, solvent extraction, membrane filtration, reverse osmosis and adsorption. Among these, adsorption is effective and economical for waste minimization [4]. Many heavy metal adsorption studies have focused on the application of activated carbons [5-6] but it is quite expensive. Nowadays, researchers are focusing more on the development of low cost and efficient adsorbents to remove heavy metals from aqueous solution. A variety of substances have been tried as adsorbents to remove lead from aqueous solutions and a number of studies have been reported using adsorbents like sawdust [7], rice husk [8], walnut shell [9], maize leaf [10], Clay [11], banana stem [12], peat [13], tree fern [14], red mud [15], lateritic minerals [16], alluvial soil [17], etc.

However in these studies optimization of process parameters was carried out by conventional method, *i.e.* investigating a process by varying one factor at a time whilst maintaining all other factors involved at constant levels. This classical approach is time consuming, requires lots of experiments and of low efficiency in optimizing a given process. Furthermore, the

conventional optimization process could not depict the interactive effects of process variable. In this study response surface methodology (RSM) approach have been used for optimization of process parameters. RSM is a combination of statistical and mathematical techniques used to determine the optimum operational conditions of the process and analysis of problems in which a response of interest is influenced by several variables.

In the present study *Lemna major* biomass was used as a potential and low cost adsorbent for the removal of lead from aqueous solution. In literature there have been no reports regarding the optimization of process parameters for lead (II) adsorption onto *Lemna major* biomass. The main objective of this study is to examine the individual and combined effect of four operating parameters like initial lead (II) concentration, adsorbent dose, pH and contact time on the removal of copper (II) onto *Lemna major* biomass. The RSM approach using Box-Behnken design (BBD) was adopted to develop the mathematical model and study the interactive effect of process parameters.

2. MATERIAL AND METHODS

2.1. Preparation of the synthetic metal solution

A stock solution of lead (II) was prepared (100 mg/L) by dissolving 0.15984 g of analytical grade lead nitrate ($\text{Pb}(\text{NO}_3)_2$) obtained from E. Merck Ltd., India in double distilled water. The working solutions of metal were prepared by diluting the stock solution to the desired concentrations. Before mixing the adsorbent, the pH of each lead solution was adjusted to the required value by 0.1 M NaOH or 0.1 M HCl solution.

2.2. Preparation of biosorbent

Lemna major, a floating macrophyte was collected from the surrounding area of University of Burdwan, West Bengal, India. The macrophyte was thoroughly washed with distilled water to remove mud and dirt and then it was initially sun dried for 7 days followed by drying in hot air oven at 253 K for 10 h and cooled at room temperature in desiccator. The dried material was crushed and sieved to give a fraction of 150 mesh screen with standard testing sieve, stored in sterile, closed glass bottle and used as biosorbent without any pretreatment for the removal of lead.

2.3. Analysis

Adsorbent characterization was performed by means of spectroscopic and quantitative analysis. The physico-chemical characteristics of *L. major* biomass were

determined using standard procedures [18]. Sodium, potassium and phosphate concentrations were determined by Flame Photometer (Model No. SYSTRONICS 126). Residual lead (II) concentration was determined by atomic absorption spectrophotometer (Model No. GBC HG3000). Magnetic stirrer (TARSONS, Spinot digital model MC02, CAT No. 6040, S. No. 173) is used for stirring purpose.

2.4. Batch mode biosorption experiments

In this investigation, 30 batch adsorption experiments, designed by BBD model were conducted to study the effect of initial lead concentrations (25-100 mg/lit), biomass dosage (0.1-0.5 g/50 ml), pH (2.0-8.0) and contact time (5-30 min) on the removal of lead from aqueous solution using 50 mL of lead (II) test solution in 250mL Erlenmeyer flask. All the experiments were conducted at a constant temperature 303 K. Solution pH was adjusted with 0.1 M HCl or 0.1 M NaOH, respectively. At the end of each experiment, samples were collected from the flasks, filtered at predetermined time intervals for analyzing the residual lead (II) concentration in the solutions. The amount of lead (II) ions adsorbed in milligram per gram was determined using the following mass balance equation:

$$q_e = \frac{(C_i - C_e)V}{m} \quad (1)$$

Where q_e is the metal uptake (mg/g), C_i and C_e are the initial and equilibrium concentrations of lead (II) (mg/L). V is the volume of solution in liter and m is the mass of biosorbent (g). The percentage of removal of lead (II) ions was calculated from the following equation:

$$\text{Removal (\%)} = \frac{(C_i - C_e)}{C_i} \times 100 \quad (2)$$

All the experiments were performed in duplicate and the average values were recorded.

2.5. Experimental design and optimization

To investigate the optimum condition for lead (II) removal, RSM based BBD model with three level and four independent variables like initial lead (II) concentration (x_1), adsorbent dose (x_2), pH (x_3) and contact time (x_4) were studied with the help of design-expert software (Stat-Wase, Inc., version 8.0.7.1, Minneapolis, USA). The total number of experiments (N) needed for BBD model can be calculated by using Eq. (3):

$$N = 2K(K - 1) + C_p \quad (3)$$

Where k is the total number of variables and C_p is the number of centre point replicates.

The coded values of process variables were determined by the following equation:

$$\text{Coded value} = x_i = \frac{x_i - X_0}{\Delta X} \quad (i=1,2,3,\dots,K) \quad (4)$$

Where x_i is the dimensionless value of a process variable, X_i is the real value of the i^{th} factor of an independent variable, X_0 is the value of X_i at the centre point and ΔX is the value of step change. In order to get true functional relationship between independent process variables and the response and to identify the relevant model terms, a second order polynomial regression equation (Eq. 5) was used to describe the effect of variables in terms of linear, quadratic and cross product terms.

$$Y = \beta_0 + \sum_{i=1}^n \beta_i x_i + \sum_{i=1}^n \beta_{ii} x_i^2 + \sum_{i,j=1(i \neq j)}^n \beta_{ij} x_i x_j + \varepsilon \quad (5)$$

where, Y is the predicted response variable (percentage removal of lead (II)), β_0 is the offset term, β_i is the coefficient of linear effect, β_{ii} is the coefficient of square effect, β_{ij} is the coefficient of interaction effect between the input factor x_i and x_j and ε is the error. Analysis of variance (ANOVA) was carried out to assess the precision and reliability of developed model and to describe the coefficients of the quadratic equation [19]. The significance and adequacy of the model were verified from Fisher variation ratio (F), probability value (prob>F) and adequate precision value [20]. Furthermore, each variable is investigated for individual and interactive effect on removal process. Equation (5) can be written for four independent variables with Y as ultimate response in their coded values in the following equation:

$$Y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_4 x_4 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{14} x_1 x_4 + \beta_{23} x_2 x_3 + \beta_{24} x_2 x_4 + \beta_{34} x_3 x_4 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{33} x_3^2 + \beta_{44} x_4^2 \quad (6)$$

3. RESULTS AND DISCUSSIONS

3.1. Characterization of biosorbent

The physicochemical properties of *L. major* biomass are summarized in Table 1.

Table 1: Physicochemical characteristics of adsorbent

Analysis	Value
Specific gravity	0.139
Moisture content (%)	0.04
Bulk density (g/cm ³)	0.141
Particle density (g/cm ³)	0.165
Porosity (%)	14.54
Conductivity (µS/cm)	1.07
Na ⁺ (mg/L)	7.0
K ⁺ (mg/L)	20.0
PO ₄ ³⁻ (mg/L)	1.92

3.2. Design of Experiments

Biosorption of lead (II) on *L. major* biomass was optimized by applying response surface methodology (RSM) through Box–Behnken Design (BBD). In order to understand the effect of various independent process parameters such as initial lead concentration (x_1), pH (x_2), biosorbent dose (x_3) and contact time (x_4) on % removal of lead (II), 30 batch experiments were conducted. The coded values of the independent process variables were determined by equation 4. The range and levels of independent process variables were summarized in Table 2. The behavior of the percentage removal of lead (II) was explained by the second order polynomial equation (Equation 6).

Table 2: Experimental factor levels used in factorial design

Independent variable	Factor	Coded levels		
		-1	0	+1
Initial concentration (mg/L)	x_1	25	62.5	100
pH	x_2	2	5	8
Adsorbent dose (g)	x_3	0.1	0.4	0.7
Contact time (min)	x_4	5	17.5	30

3.3. Evaluation of model

In order to justify the fitment of the different models such as linear, two-factor interaction (2FI), quadratic and cubic, evaluation was done on the basis of scores obtained from the sequential model sum of squares (Table 3), and it shows that the quadratic model has a high score. The larger magnitude of F (106.57) and smaller value of p (< 0.0001) indicates that the quadratic model model is highly significant, and was found to be good.

3.4. Quadratic model for lead (II) adsorption process

To examine the combined effect of four different independent process parameters on percentage removal of lead (II) 30 batch experiments were performed. The experimental design is given in Table 4, along with experimental data and predicted responses. Regression analysis was performed to fit the response functions, i.e. percentage removal of lead (II). The second order polynomial equation developed represent responses as functions of initial metal concentration (x_1), adsorbent dose (x_2), pH (x_3), and contact time (x_4). An empirical relationship between the response and the input test variables in coded units can be expressed by the following equation:

$$\%R_{\text{Lead (II)}} = 67.74 - 6.11 x_1 + 5.55 x_2 + 13.60 x_3 - 0.014 x_4 + 6.15 x_1x_2 - 7.15 x_1x_3 - 3.76 x_1x_4 + 2.97 x_2x_3 + 0.43 x_2x_4 + 0.22 x_3x_4 - 24.33 x_1^2 - 16.49 x_2^2 - 7.20 x_3^2 + 34.85 x_4^2 \quad (7)$$

The above equation demonstrated how lead (II) adsorption onto biomass was affected by the individual variables (linear and quadratic) or doubles interaction.

Table 3: Sequential model sum of squares

Source	Sum of squares	df	Mean square	F-value	p-value, prob> F	
Mean vs Total	1.083E+005	1	1.083E+005			
Linear vs Mean	3044.63	4	761.16	3.53	0.0205	
2Fl vs Linear	2283.64	6	380.61	2.33	0.0747	
Quadratic vs 2Fl	3004.14	4	751.03	106.57	< 0.0001	Suggested
Cubic vs Quadratic	104.87	10	10.49	62.92	0.0001	Aliased
Residual	0.83	5	0.17			
Total	1.167+005	30	3890			

Table 4: BBD matrix for the experimental design and predicted responses for lead (II) removal

Run order	Coded Value				Lead (II) removal (%)	
	x ₁	x ₂	x ₃	x ₄	Obse-rved	Predi-cted
1	25.00	8.00	0.10	5.00	32	32.42
2	62.50	5.00	0.30	25.00	80	80.28
3	25.00	8.00	0.10	30.00	40	40.32
4	37.50	5.00	0.30	17.50	55	61.01
5	25.00	8.00	0.50	30.00	86	88.22
6	25.00	2.00	0.50	30.00	82	82.60
7	25.00	2.00	0.10	30.00	46	46.60
8	62.50	5.00	0.70	17.50	67.3	66.16
9	50.00	2.00	0.10	5.00	65	61.13
10	62.50	8.00	0.30	17.50	60	56.80
11	62.50	5.00	0.30	17.50	68	67.74
12	100.00	2.00	0.10	30.00	29	28.86
13	100.00	2.00	0.50	30.00	37	36.26
14	25.00	6.00	0.30	30.00	90	86.24
15	100.00	8.00	0.50	30.00	66	66.46
16	100.00	2.00	0.50	5.00	43	44.22
17	100.00	8.00	0.10	30.00	47	47.16
18	62.50	5.00	0.10	17.50	46	46.94
19	12.50	5.00	0.30	17.50	36	32.65
20	25.00	2.00	0.10	5.00	38	38.00
21	62.50	5.00	0.30	17.50	67	67.74
22	25.00	8.00	0.50	5.00	79	79.43
23	100.00	8.00	0.50	5.00	73	72.71
24	62.50	5.00	0.30	20.00	68	69.14
25	62.50	5.00	0.30	17.50	68	67.74
26	62.50	5.00	0.30	17.50	68	67.2
27	25.00	2.00	0.50	5.00	76	75.51
28	62.50	5.00	0.30	17.50	68	67.2
29	62.50	5.00	0.30	17.50	68	67.74
30	100.00	8.00	0.10	5.00	54	54.31

Negative coefficient values indicate that individual or double interactions factors negatively affect lead (II) adsorption while positive coefficient values represents that factors increase lead (II) removal efficiency. For instance, among all linear factors initial concentration and contact time had a negative effect but adsorbent dose and pH had a positive effect on lead (II) removal.

With the increase of initial lead (II) ions concentration percentage removal decreased. This can be explained on the basis of the fact that all adsorbents have a limited number of active binding sites and at a certain concentration the active sites become saturated. However a sharp increase in the lead ion removal is recorded when the pH value of the solutions changed from 2.0 to 6.0. From pH 6 onwards a steady decrease of adsorption of lead is noticed. The percentage of lead ion removal increased with increase in adsorbent dose. Such a trend is mostly attributed to an increase in the sorptive surface area and the availability of more active binding sites on the surface of the adsorbent¹⁷. Furthermore, rate of adsorption increased with contact time.

The adequacy and significance of the quadratic model was justified by the analysis of variance (ANOVA). The ANOVA summary is given in Table 5. The significance of each term is determined by the corresponding F value, p value and sum of square (SS). Larger the value of F, the corresponding term is highly significant. Furthermore, null hypothesis is rejected and the variable is said to be significant when p value is small. With the increase of the value of SS, the significance of that particular variable also increases [21]. It is evident from Table 5 that model F value is 84.46 with the corresponding p value <0.0001 and high SS (8332.41). The parameters having an F-statistics probability value

less than 0.05 are said to be significant [22]. In this present study the probability of model F statistics value is <0.0001 implies that the model suggested by the software is highly significant. In this case x_1 , x_2 , x_3 , x_1x_2 , x_1x_3 , x_1x_4 , x_2x_3 , x_1^2 , x_2^2 , x_3^2 and x_4^2 are statistically significant ($p < 0.05$) model terms at the 95% confidence level. ANOVA analysis suggested that among the four process parameters biomass dose ($p < 0.0001$, $SS = 3140.80$, $F = 445.69$) has the most influential effect on lead removal followed by initial lead concentration ($p < 0.0001$, $SS = 519$, $F = 73.73$) and pH ($p < 0.0001$, $SS = 480.47$, $F = 68.18$). Contact time ($p = 0.9842$) has no significant effect on the response. The lack of fit test is employed to verify whether the selected quadratic model is adequate to describe the observed data or whether a more complicated model should be used. The non significant value of lack of fit ($p = 0.2135$) confirmed the validity of developed model [23]. Furthermore, The R^2 , adjusted R^2 and predicted R^2

values were found to be 0.9875, 0.9758 and 0.9643, respectively, which indicates that there was a good reasonable agreement between the actual and the predicted values. The signal-to-noise ratio is measured by adequate precision ratio and a ratio > 4 is desirable. In this study the ratio of 31.625 obtained for lead adsorption indicates an appropriated signal to noise ratio. The quadratic model can be used to navigate the design space and to find the optimal conditions for biosorption process. The plot of actual vs predicted values (Fig. 3) showed that the actual values are distributed relatively near to the straight line, indicating good fitness of the model. The normal residual plot (Fig. 4) between probability and internally studentized residuals showed that the residuals were distributed normally with minimum deviations. This was an indication of better fitment of the model with the experimental data.

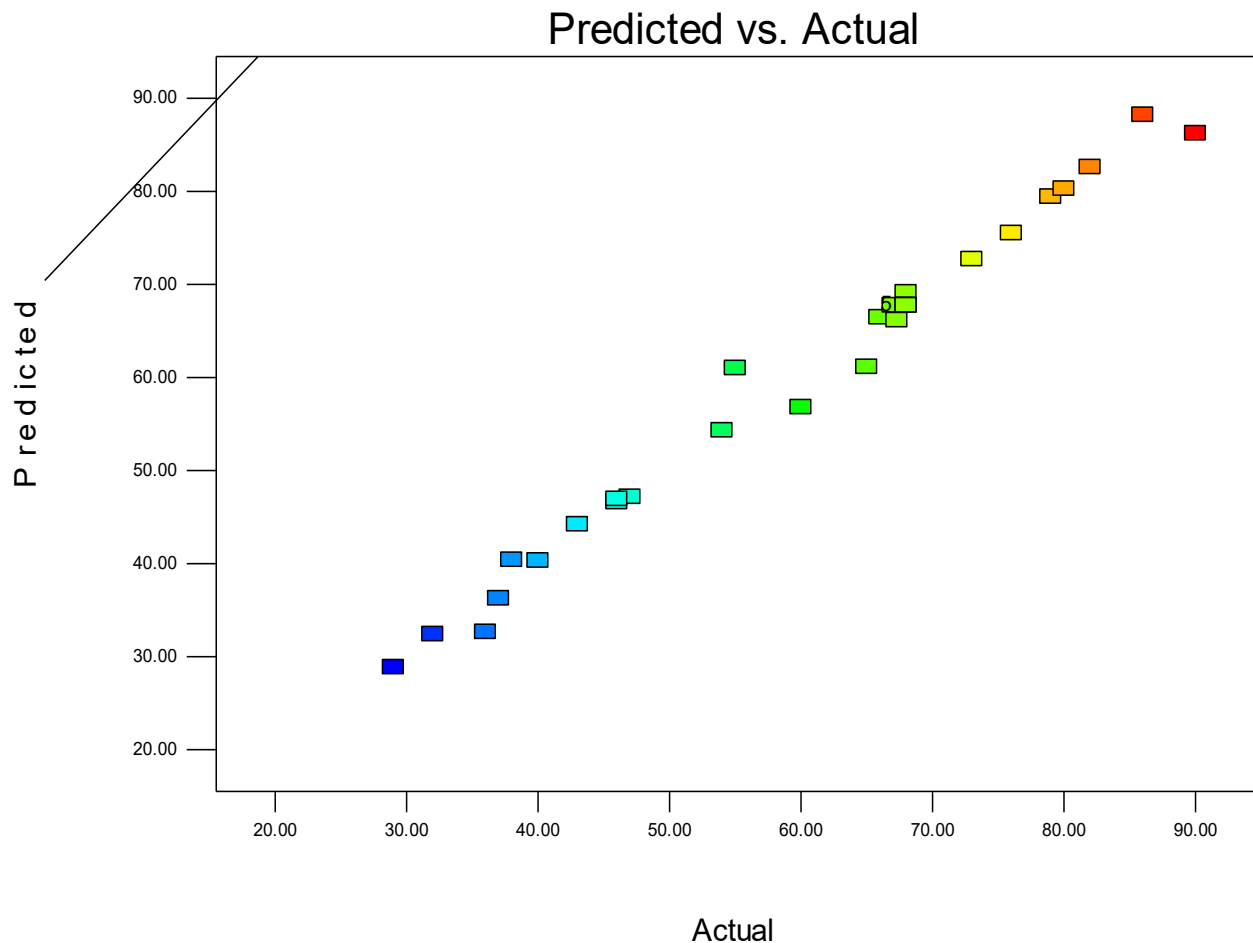


Fig. 3: Comparison between the actual values and the predicted values of RSM model for adsorption of lead (II)

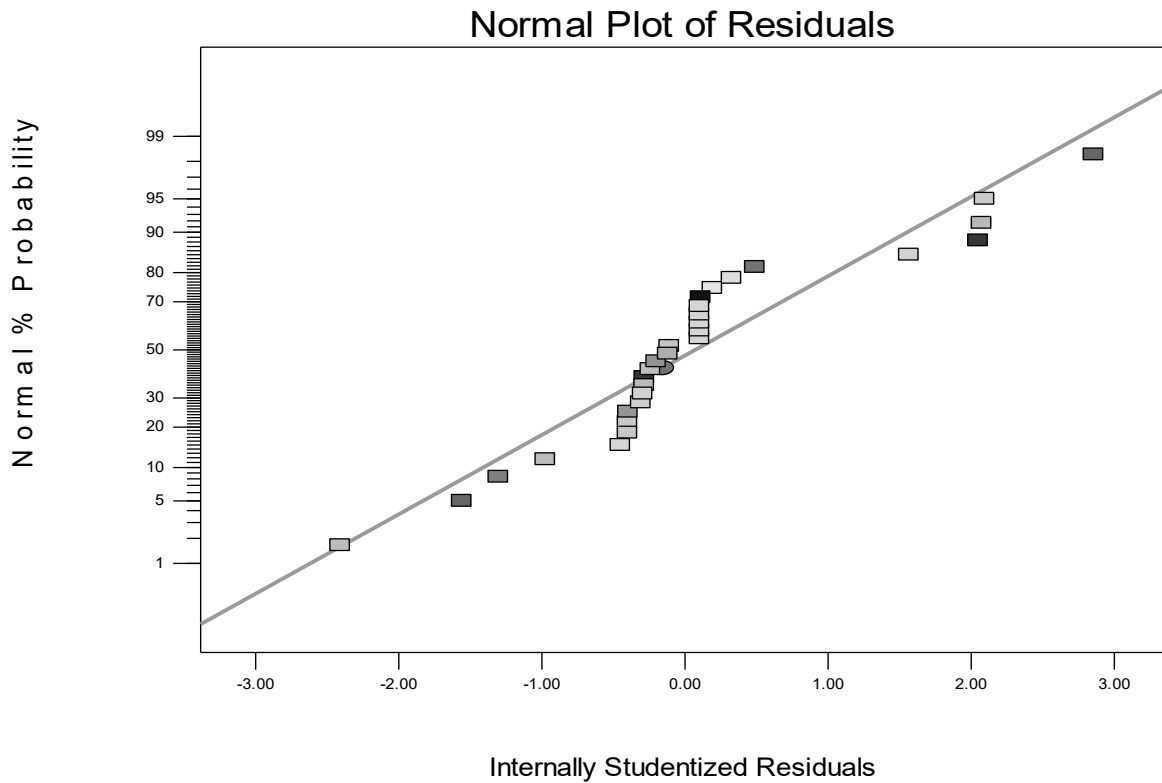


Fig. 4: Plot of Studentized residuals versus experimental run number

Table 5: Analysis of variance (ANOVA) for the quadratic model

Variation source	Coefficient	Sum of squares	DF	Mean square	F	Prob>F
Intercept	67.74		1			
x_1	-6.11	519.59	1	519.59	73.73	<0.0001
x_2	5.55	480.47	1	480.47	68.18	<0.0001
x_3	13.60	3140.80	1	3140.80	445.69	<0.0001
x_4	-0.014	285.2	1	285.2	404.7	0.9842
x_1^2	-24.33	1886.52	1	1886.52	267.70	<0.0001
x_2^2	-16.49	439.41	1	439.41	62.35	<0.0001
x_3^2	-7.20	697.47	1	697.47	98.97	<0.0001
x_4^2	34.85	148.73	1	148.73	211.40	<0.0001
x_1x_2	6.15	512.29	1	512.29	72.70	<0.0001
x_1x_3	-7.15	692.62	1	692.62	98.29	<0.0025
x_1x_4	-3.76	195.82	1	195.82	27.79	<0.0001
x_2x_3	2.97	131.31	1	131.31	18.63	0.0006
x_2x_4	0.43	2.72	1	2.72	0.39	0.5441
x_3x_4	0.22	0.74	1	0.74	0.11	0.7498
Model		8332.41	14	595.17	84.46	<0.0001
Residual		105.71	15	7.05		
Lack of Fit		104.87	10	10.49	8.78	0.2135
Pure Error		0.83	5	0.17		
Cor Total		8438.11	29			
$R^2 = 0.9875$						
$R^2_{Adjusted} = 0.9758$						
$R^2_{Predicted} = 0.9643$						
Adeq Precision =31.625						

Table 6: Diagnostics case statistics

Standard order	Actual value	Predicted value	Residual	Leverage	Internally studentized residuals	Externally studentized residuals	Influence on fitted values DFFITS	Cook's distance
1	38.00	40.40	-2.40	0.518	-1.303	-1.337	-1.386	0.122
2	65.00	61.13	3.87	0.515	2.094	2.405	1.48	0.311
3	32.00	32.42	-0.42	0.691	-0.283	-0.274	-0.411	0.012
4	54.00	54.31	-0.31	0.768	-0.241	-0.233	-0.425	0.013
5	76.00	75.51	0.49	0.683	0.325	0.315	0.463	0.015
6	43.00	44.22	-1.22	0.779	-0.976	-0.974	-1.829	0.224
7	79.00	79.43	-0.43	0.682	-0.284	-0.275	-0.403	0.012
8	73.00	72.71	0.29	0.677	0.192	0.186	0.269	0.005
9	46.00	46.60	-0.60	0.688	-0.401	-0.390	-0.579	0.024
10	29.00	28.86	0.14	0.764	0.110	0.106	0.191	0.003
11	40.00	40.32	-0.32	0.641	-0.201	-0.195	-0.260	0.005
12	47.00	47.16	-0.16	0.675	-0.109	-0.105	-0.152	0.002
13	82.00	82.60	-0.60	0.674	-0.398	-0.387	-0.557	0.022
14	37.00	36.26	0.74	0.675	0.488	0.476	0.686	0.033
15	86.00	88.22	-2.22	0.711	-1.559	-1.645	-1.58	0.400
16	66.00	66.46	-0.46	0.681	-0.308	-0.299	-0.436	0.014
17	36.00	32.65	3.35	0.804	2.855	2.08	1.27	1.23
18	55.00	61.01	-6.01	0.118	-2.409	-2.972	-1.085	0.052
19	90.00	86.24	3.76	0.522	2.049	2.333	1.44	0.306
20	60.00	56.80	3.20	0.663	2.074	2.372	1.33	0.564
21	46.00	46.94	-0.94	0.216	-0.402	-0.390	-0.205	0.003
22	67.30	66.16	1.14	0.925	1.565	1.653	1.80	1.01
23	68.00	69.14	-1.14	0.103	-0.452	-0.439	-0.149	0.002
24	80.00	80.28	-0.28	0.213	-0.120	-0.116	-0.060	0.000
25	68.00	67.74	0.26	0.102	0.102	0.098	0.033	0.000
26	68.00	67.74	0.26	0.102	0.102	0.098	0.033	0.000
27	68.00	67.74	0.26	0.102	0.102	0.098	0.033	0.000
28	67.00	67.74	-0.74	0.102	-0.296	-0.287	-0.097	0.001
29	68.00	67.74	0.26	0.102	0.102	0.098	0.033	0.000
30	68.00	67.74	0.26	0.102	0.102	0.098	0.033	0.000

From the analysis of diagnostic case statistics the value of leverage, internally studentized residuals, externally studentized residuals, DFFITS and Cook's distance of the data can be obtained (Table 6). The proposed model is said to be significantly valid if the leverage value is within 0 to 1, limit of the internally studentized residuals is ± 3 sigma, limit of DFFITS lies in between +2 and -2 and Cook's distance range of ± 1 [24]. From the study of diagnostic case statistics data it is evident that the model fits well to optimize the independent variables (all the values are within the range) for the removal of lead (II) from aqueous solution.

3.5. Comparative effect of individual variables

Perturbation plot helps to compare the effect of all the factors at a particular point in the design space. In perturbation plot the response was plotted by changing

only one factor over its range while holding all the other factors constant. Lead (II) removal efficiency was introduced as each variable move from preferred reference with all other factors held constant at the coded zero level. A steep slope or curvature in a factor indicates the sensitivity of the response to that factor however insensitivity of a particular factor is demonstrated by a relatively flat line²⁶. The comparative effects of all the independent variables for lead (II) adsorption are shown in Figure 5. The sharp curvature of all the independent variables showed that the lead (II) removal efficiency was very much sensitive to all the process variables.

3.6. Optimizing the process variables by desirability function

Desirability is an objective function. The ranges of this function lies from zero (outside the limits) to one (at

goal). In numerical optimization process a point was found that maximizes the desirability function by searching in the design space. For optimization of any combination of five goals, namely, initial concentration, adsorbent dose, pH, contact time and lead (II) removal efficiency a multiple response method was adopted. By setting different criteria the optimization was carried

out in two different ways. The desirability for each factor and response individually and collectively was discussed by the histogram plots. Histogram plots for the present study are depicted in Figure 6 a, b respectively. The RAMP plot (Figure 7 a, b) shows the optimum experimental conditions for the removal of lead (II) at maximum desirability.

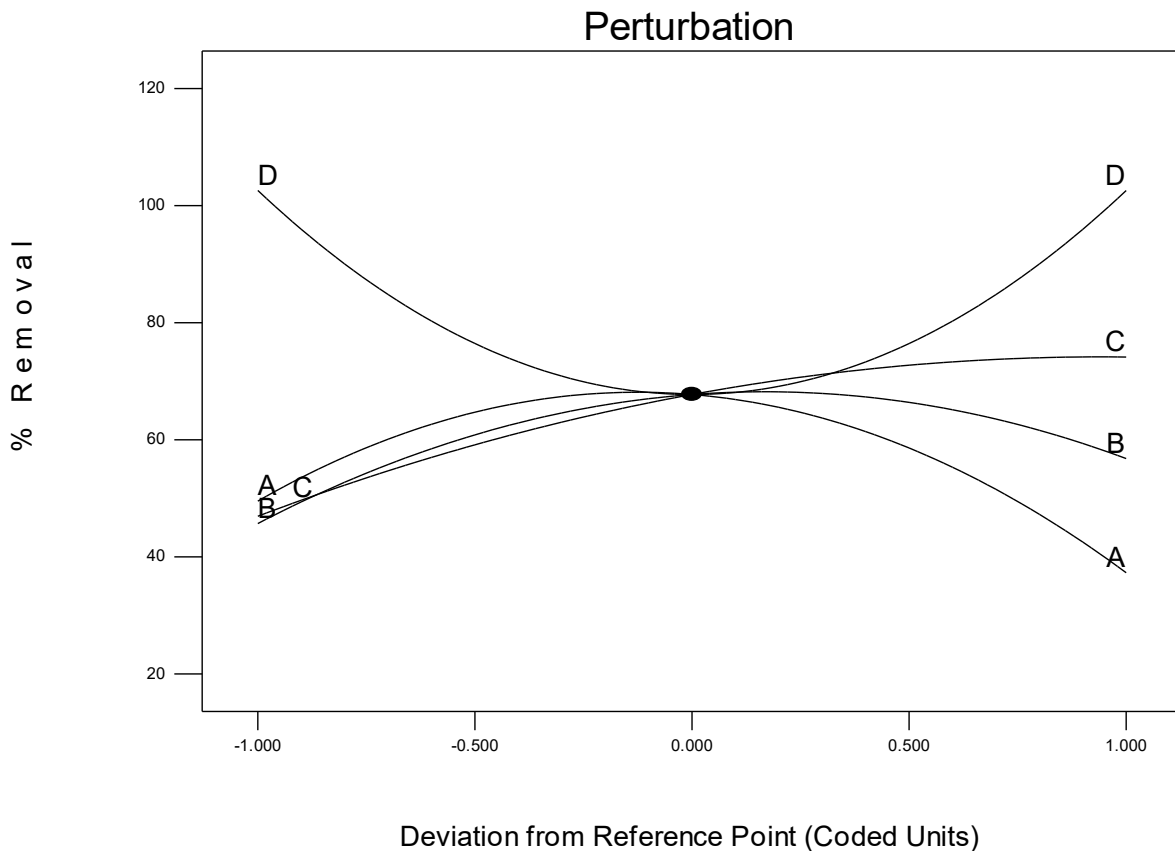


Fig. 5: Perturbation plot (A, B, C and D factors are equivalent to factors x_1 , x_2 , x_3 and x_4).

3.6.1. First optimization procedure

The desirability values of first optimization procedure is shown in Figure 6 a. The criterion of this procedure was set as "in range" for independent variables (initial lead (II) concentration, pH, adsorbent dose and contact time) and "maximum" for dependent variable (removal of lead (II) (R_1 , in percent)). The desirability value was found to be 1 for individual and combination of all these variables. Figure 7 a demonstrates 92.17 % as optimum removal efficiency of lead (II) when the independent variables were 41.11 mg/L (initial lead (II) concentration), 4.55 (pH), 0.57 g/50 mL (*L. Major* biomass dose) and 26.40 minute (contact time) at maximum desirability value.

3.6.2. Second optimization procedure

The desirability values of second optimization procedure is shown in Figure 6 b. The essential condition of this procedure was set as "minimum" for adsorbent dosage, "maximum" for initial lead (II) concentration, "in range" for pH and contact time and the goal was set as "maximum" to analyze economically viable optimal condition. The aim of this process was to find the maximum removal percentage by utilizing less amount of biosorbent. In this procedure, the desirability value ranges from 0.84 to 1 for individual variables and 0.864 for combination of all the variables. Figure 7 b connotes 80.42 % as the optimal response when the independent variables are 88.88 mg/L (initial lead (II)

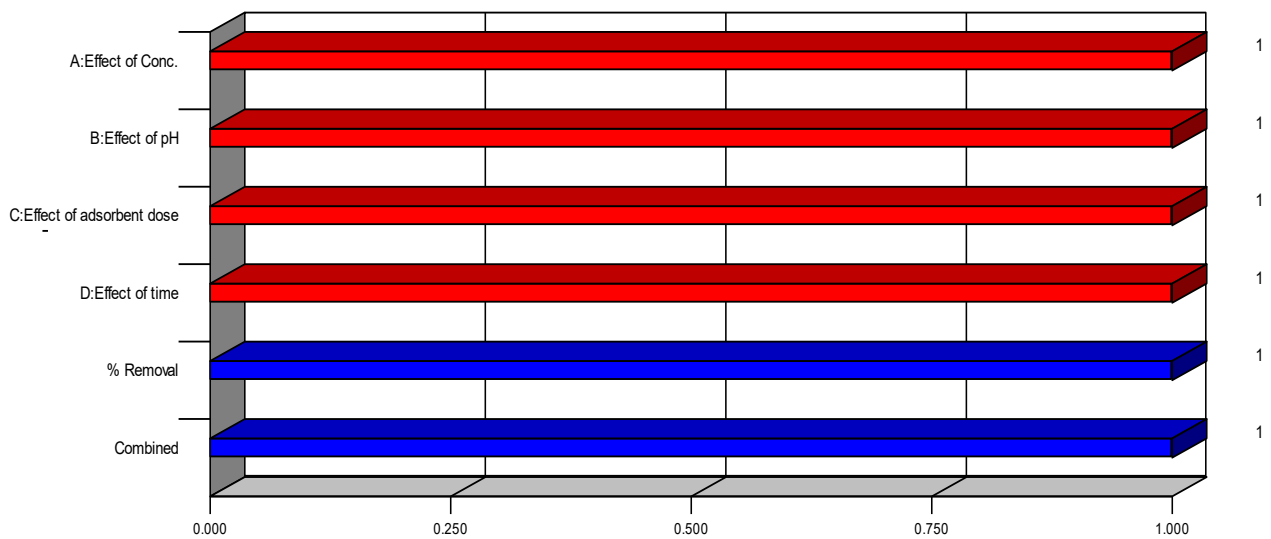
concentration, 5.67 (pH), 0.16 g/50 mL (adsorbent dose) and 5 minute (contact time) at maximum desirability value.

However, second optimization procedure was preferred for the removal of lead (II) by *Lemna major* biomass on the basis of uptake capacity and economical usage of

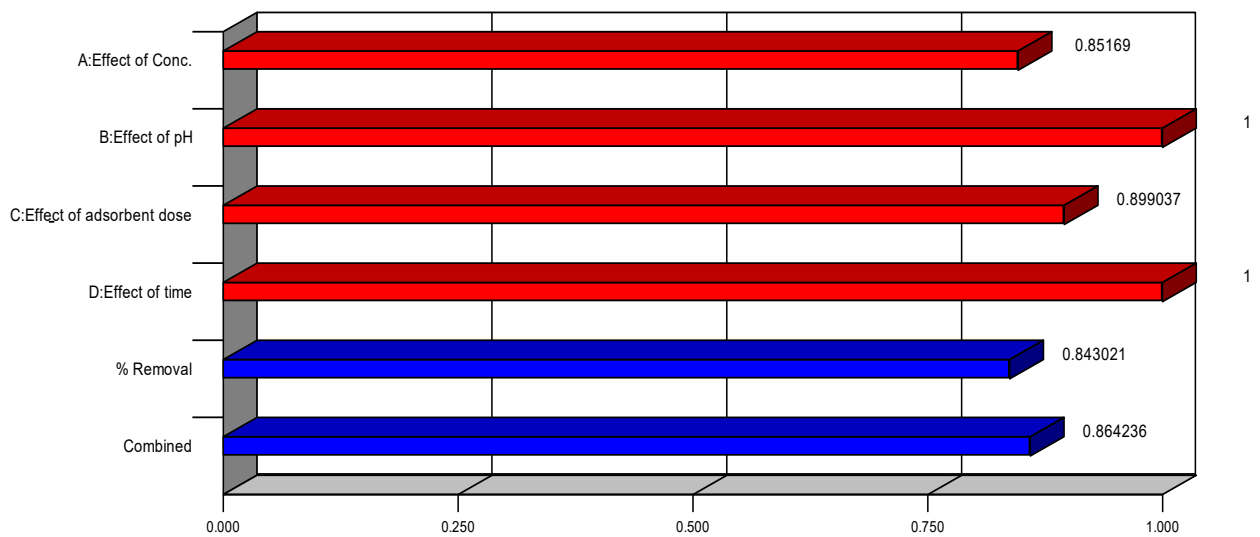
biosorbent. Furthermore, confirmatory experiments were carried out on the two approaches obtained from the software to validate the accuracy of the predicted results. By applying the above said conditions it was observed that predicted percentage removal of lead (II) matches with the experimental values well (Table 7).

Table 7: Optimized results from model and experimental verification for lead (II) removal

Initial concentration (mg/L)	pH	<i>L. major</i> dose (g/50 mL)	Contact time (min)	Removal (%)		Remarks
				Predicted	Experimental	
41.11	4.55	0.57	26.40	92.17	90.25	First procedure
88.88	5.67	0.16	5.0	80.42	77.87	Second procedure

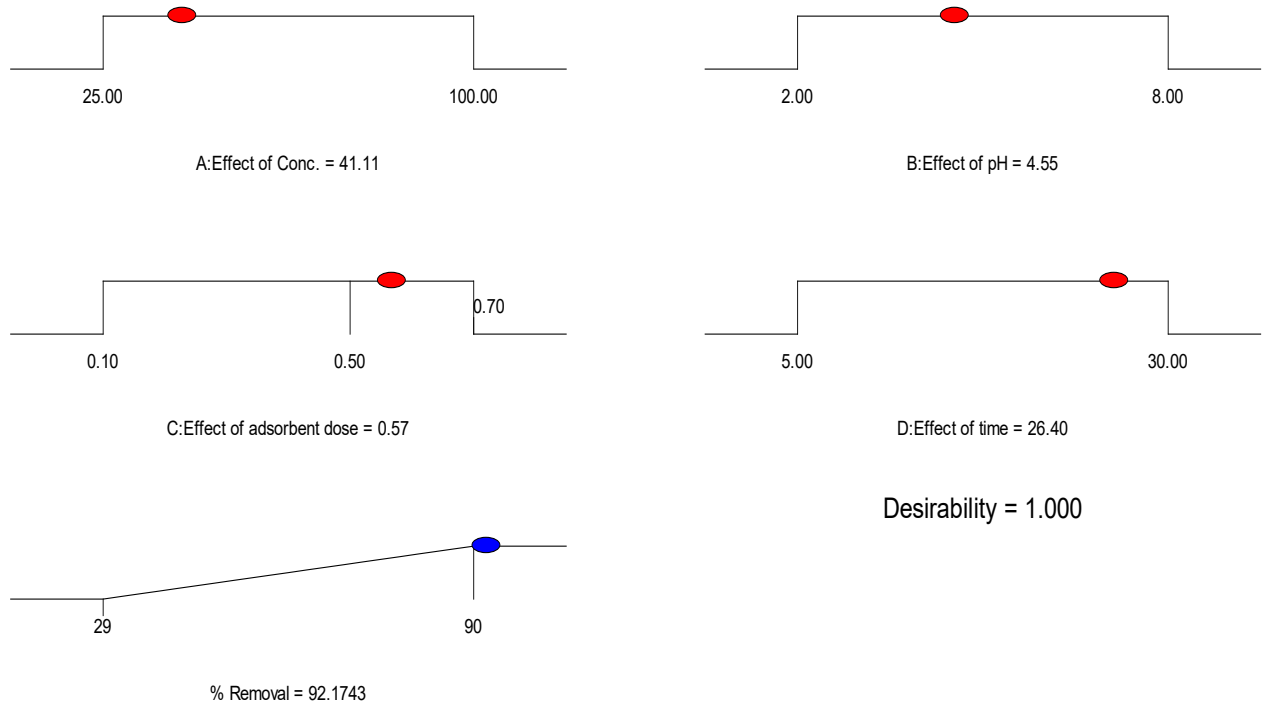


(a)

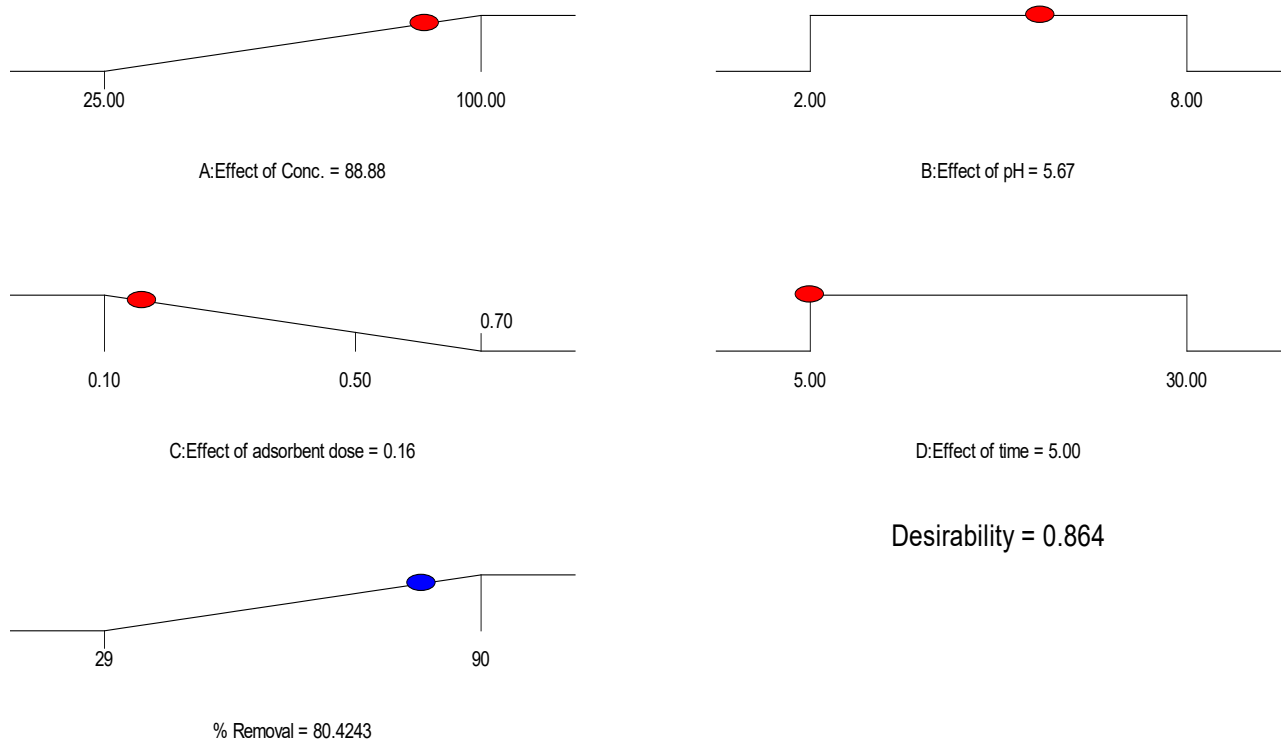


(b)

Fig. 6: Bar graph for (a) first optimization procedure and (b) second optimization procedure



(a)



(b)

Fig. 7: RAMP plots for (a) first optimization procedure and (b) second optimization procedure

4. CONCLUSIONS

This study focused on the adsorption of lead (II) ions onto *Lemna major* biomass from aqueous solution. Response surface methodology based BBD model was used to determine the optimum reaction conditions. According to the ANOVA analysis, all the interaction (except x_4 , x_2x_4 and x_3x_4) terms are statistically significant. The quadratic model represents adequately the response surface space based on the adjusted determination coefficient ($R^2_{Adj} = 0.9758$) and the adequate precision ratio (31.625). The high similarity between the experimental value and the predicted ones suggested that the model was a good fit. The optimized result obtained from RAMP plots revealed that the use of *Lemna major* biomass is an economically viable option for the removal of lead (II) from aqueous solution.

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