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Statistical Optimization of Biosorption of Hexavalent Chromium Using Water Hyacinth

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ABSTRACT: Several industrial operations release Hexavalent Chromium as a leading toxin. In this study aquatic weed Water hyacinth in crude and completely pyrolysed forms were used for the reduction of hexavalent chromium concentration from synthetic waste water. The biosorption experiments were performed in batch operation. Response Surface Methodology is used to optimize process parameters. A quadratic model was developed to correlate the variables to the response based on central composite design. Statistical tools were used to identify the most significant factor for all the adsorbents. Through Analysis of Variance (ANOVA) it was found that pH and biomass loading are significant for water hyacinth powder and concentration is significant for pyrolysed adsorbent. Between both the adsorbents, pyrolysed water hyacinth adsorbed maximum chromium of 99.24% at optimized conditions pH 1.924, temperature of 400C, initial concentration 133.607 mg/l and biomass loading of 8.041g/ 100ml.

KEYWORDS: Biosorption, Water Hyacinth, Hexavalent chromium, Pyrolysis

I. INTRODUCTION

Waste water from industries containing heavy metals causes danger to human beings and other forms of fauna. Conventional methods used for removal of heavy metals from waste water are often too expensive and have poor efficiencies at low metal concentrations. In the recent years the biosorption of metal ions has received considerable attention for the development of an efficient, clean, cheap and sustainable technology for waste water treatment at low metal concentrations. Chromium in aqueous system occurs in two forms, trivalent chromium (Cr (III)) and hexavalent chromium (Cr (VI)). Chemical, biological and environmental characters exhibited by both the forms of chromium are diverse and distinct [1]. Cr (III) is essential trace metal nutrient required for microorganisms in minute quantities [2], whereas Cr (VI) is all types of fauna and flora [3-5]. Huge quantities of chromium is discharged into the water bodies from various industries such as electroplating, leather tanning, mining, paints and pigments [6-8]. Due to threat caused by chromium to environment and mankind, permissible limits of chromium have been closely monitored and regulated by most industrial countries. Industrial effluents generally contain chromium concentration in the range 0.5 to 270 mg/l [7]. The maximum value for chromium into inland surface water and potable water is 0.1 and 0.05 mg/l respectively [9, 10].

Various technologies are employed to reduce the level of chromium in effluents such as reduction followed by chemical precipitation [11], ion exchange process [12], electrochemical precipitation [13], reduction [14], adsorption [15], solvent extraction [16], membrane separation [17], concentration [18], evaporation and reverse osmosis [19, 20]. It is reported adsorption is easily adaptable and efficient method for removal of heavy metals. Adsorption solves the difficulty of sludge disposal and renders the system more economically feasible, especially if low cost adsorbents are used [21]. Recently many researchers have worked in identifying economical and efficient adsorbent for chromium removal from waste water. In the current work aquatic weed, water hyacinth is explored as biosorbent for removal chromium as it is freely and abundantly available. Response Surface Methodology (RSM) is statistical tool often used for multivariable studies in many processes [22-24]. RSM is used for design of experiments, model building, evaluation



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of the effects of parameters and optimizing the parameters for maximum removal of chromium. The research work aims to quantify the biosorption of chromium by water hyacinth and use RSM to optimize the variables for maximum uptake of chromium.

II. MATERIALS AND METHODS

A. Preparation of Biosorbent

1) Water Hyacinth Collection

Natural agro waste biosorbent Eichhornia crassipes collected from Kengeri Lake situated in Bengaluru district, Karnataka were used for removal of chromium. Natural biosorbent along with pyrolysis process were employed for comparative metal removal efficiency.

2) Crude Water Hyacinth (WH)

Water hyacinth was washed, sun dried then dried in oven at 60° C, powdered, sieved using 90/120 mesh BSS Standard sieve to get uniform sized particles. The fraction that was retained on 120 meshes were collected, washed gently with distilled water and dried in the hot oven for 2 hours at 80 °C.

3) Preparation of Activated Carbon by Pyrolysis (WHP)

The activated carbon was prepared by complete pyrolysis using crude Water hyacinth in a muffle furnace. The complete pyrolysis Water hyacinth (WHP) adsorbent is obtained by just keeping the crucible in the muffle furnace.

B. Preparation of Chromium Stock Solution

Preparation of Synthetic chromium solution includes dissolving 2.828g of potassium dichromate ($K_2Cr_2O_7$) in 1000ml double distilled water. 1000 ppm of stock chromium solution was prepared. The stock solution was diluted to prepare other required concentrations. Thenadjustment of pH of the solution was done to the required value.

C. Preparation of Diphenylcarbazide (DPC) Solution

Preparation of Diphenylcarbazide (DPC) solution includes dissolving of 250mg of DPC in 50ml of acetone.

D. Analysis of Chromium

0.25ml of phosphoric acid was added to 1ml of standard sample containing known concentration of chromium, pH was adjusted to 1.0 ± 0.3 using 0.2N sulphuric acid. The solution was mixed well and then diluted to 100ml in a volumetric flask using double distilled water. Further 2ml of DPC solution was added and mixed well. After full colour development for 10min, 4ml of this solution was used in an absorption cell and the concentrations were measured spectrometrically at 540nm in UV-double beam spectrophotometer. By measuring the absorbance, the calibration curve was drawnfor different known concentrations of chromium solutions and plotting a graph between concentrations versus absorbance. A straight line is obtained with R² of 0.9965.

E. Initial Experiments

The initial experiments were conducted to fix limits of the parameters to be varied. Maximum chromium removal is observed with the range of initial metal ion concentrations of 10 - 250 ppm, pH 1-7, temperature $30-50^{\circ}$ C and biosorbent dosage of 1 - 10 g/100 ml. These ranges of variables were employed for further study.

F. Design of Experiments using Central Composite Design (CCD)

The parameters such as pH, temperature, initial metal ion concentration and biosorbent dosage were chosen as independent variables and chromium removal efficiency was considered as the output response. A 2^4 full factorial experimental design, with seven replicates at the centre point and using the statistical software, MINITAB 16, a group of 31 trails were engaged in this study. Every independent variable had 5 levels which were -2, -1, 0, +1 and +2. Therefore, a total of 31 different arrangements were chosen in casual order according to a CCD. Table I shows the levels and ranges of independent variables used in the experiment for the chromium removal. The analysis aimed on



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how the removal efficiency is influenced by independent variables, pH (X_1), temperature (X_2), Initial metal ion concentration (X_3) and biosorbent dosage (X_4). The maximum removal efficiency (Z) is the dependent output variable [25].

G. Response Surface Methodology (RSM)

Response surface methodology is a statistical toolwhich is used for multiparameter regression analysis by using quantitative information obtained from well-designed experiments to solve multivariate equations at the same time. The independent variables pH, metal ion concentration, temperature and adsorbent dosage were considered for the removal of chromium in CCD.

	Levels and Ranges						
Independent Variable	-2	-1	0	1	2		
pH (X1)	1	2.5	4.0	5.5	7		
Temperature (°C) (X ₂)	30	35	40	45	50		
Initial Chromium ion concentration (ppm) (X ₃)	50	87.5	125	162.5	200		
Biomass loading (g/100 ml) (X ₄)	1	3.25	5.5	7.75	10		

TABLE I CENTRAL COMPOSITE DESIGN FOR BIOSORPTION OF CHROMIUM

A second order polynomial equation is shown inequation 1. The regression analysis of the polynomial equation was performed to estimate the response function.

$$\begin{split} Z = & \delta_0 + \delta_1 X_1 + \delta_2 X_2 + \delta_3 X_3 + \delta_4 X_4 + \delta_{11} X_{12} + \delta_{22} X_{22} + \delta_{33} X_{32} + \delta_{44} X_{42} + \delta_{12} X_1 X_2 + \delta_{13} X_1 X_3 + \delta_{14} X_1 X_4 + \delta_{23} X_2 X_3 + \delta_{24} X_2 X_4 \\ + & \delta_{34} X_3 X_4 \end{split}$$

Where, X₁, X₂, X₃, X₄are linear effects,

 $\mathbf{X}_{12}, \mathbf{X}_{22}, \mathbf{X}_{32}, \mathbf{X}_{42}$ are squared effects,

 X_1X_2 , X_1X_3 , X_1X_4 , X_2X_3 , X_2X_4 , and X_3X_4 are interaction effects and Z is the predicted response.

 δ_0 is constant coefficient, δ_1 , δ_2 , δ_3 , δ_4 are linear coefficients, δ_{11} , δ_{22} , δ_{33} , δ_{44} are squared coefficients, and δ_{12} , δ_{13} , δ_{14} , δ_{23} , δ_{24} , δ_{34} are interactive coefficients, respectively.

Estimation of the coefficients of the regression equation and obtaining regression analysis was performed using a statistical software package Minitab 16. The statistical test called Analysis of Variance (ANOVA) was used to validate the equations. In each case estimating the goodness of fit was the importance of each term in the equation. Response surface was used to understand the linear, square and interactive effects of test variables on percentage removal of chromium.

H. Batch Experiments

Batch adsorption studies were performed by shaking 100 ml of different solutions in 250 ml conical flasks with cork lid in constant temperature shaker at the conditioned mentioned by central composite design to obtain the equilibrium data. The experiments were conducted in triplicate and the values obtained were averaged. After specified time interval, the samples were analysed by the spectrophotometric method.

I. Optimization of Parameters

The second degree polynomial equation is solved and for the process parameters the optimum values are obtained using response optimizer in Minitab 16.



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III. RESULTS AND DISCUSSION

A. Central Composite Design Analysis[CCD]

According to the CCD analysis, the results of experiments executed are tabulated in Table II. Table II gives the experimental results along with the expected values for two adsorbents WH and WHP. It may be observed from Table 2 that the percentage removal is higher at lower pH. In acidic pH, the biosorbent surface may be protonated and hence the positively charged biosorbent removes higher amounts of Chromium in the anionic form HCrO⁴⁻. Further, the effect of temperature is significant on pyrolysed type of adsorbent suggests that the biosorption between chromium and pyrolysed water hyacinth is because of chemical interaction and physical adsorption. With the temperature increment, more surface area obtained for diffusion and adsorption by enlargement of the pores in the adsorbent [26].

B. Response Surface Methodology (RSM)

Analysis of regression was done to fit the response function as per the equation 1 and the results are reported as eq.2 and eq. 3 for WH, WHP respectively.

$\begin{array}{l} Y=\!83.294\!-\!6.746X_1\!-\!0.745X_2\!-\!1.464X_3\!+\!8.275X_4\!-\!3.455X_{12}\!-\!4.083X_{22}\!+\!3.988X_{32}\!-\!2.134X_{42}\!-\!3.104X_1X_2\!+\!1.712X_1X_3\!-\!1.567X_1X_4\!+\!5.227X_2X_3\!+\!0.455X_2X_4\!+\!0.819X_3X_4\!-\!.....(2) \end{array}$

These above equations explain the effect of individual variable (linear and squared) and interactive effects on Chromium adsorption onto different adsorbent. The chromium removal was predicted using eq.2 & eq.3 at experimental parameters and the obtained values are also reported in Table II.

TABLE II. CENTRAL COMPOSITE DESIGN SPECIFICATIONS ALONG WITH OBSERVED RESPONSE FOR CHROMIUM REMOVAL									
D		T	Initial	Biomass	% Chromium removal				
Order pH	1 emp	Conc'n	Loading	Crude Sample (WH)		Pyrolysed (WHP)			
	()	(ppm)	(g/100ml)	Theoretical	Experimental	Theoretical	Experimental		
1	5.5	45	200	3.25	73.8854	87.31	79.7117	84.40	
2	5.5	35	200	7.75	83.7604	90.46	88.5533	93.72	
3	2.5	45	200	3.25	87.6196	99.54	72.4946	67.84	
4	4.0	40	150	5.50	83.2943	95.20	88.7486	98.36	
5	5.5	45	100	3.25	64.5712	66.57	95.1012	93.88	
6	2.5	45	100	3.25	85.1554	81.75	92.8167	98.72	
7	4.0	50	150	5.50	91.1167	95.12	87.6054	89.60	
8	4.0	30	150	5.50	98.1367	93.80	97.1971	98.27	
9	5.5	35	100	3.25	81.2487	98.36	84.8133	90.00	
10	4.0	40	150	5.50	83.2943	74.28	88.7486	90.51	
11	2.5	45	100	7.75	94.0779	98.34	92.6563	89.72	
12	5.5	35	200	3.25	69.6529	82.43	82.8012	85.60	
13	5.5	35	100	7.75	92.0813	87.20	89.2629	93.78	
14	2.5	35	100	7.75	95.3304	95.20	98.1583	99.54	
15	5.5	45	200	7.75	89.8129	89.88	62.4712	53.14	
16	4.0	40	250	5.50	96.3183	90.72	78.5804	80.00	
17	1.0	40	150	5.50	82.9633	100.00	96.1904	100.00	
18	4.0	40	150	5.50	83.2943	78.56	88.7486	77.25	
19	4.0	40	150	5.50	83.2943	95.20	88.7486	86.84	
20	5.5	45	100	7.75	77.2238	95.10	86.5583	95.17	
21	4.0	40	150	10.00	91.2717	98.88	92.7621	98.54	
22	4.0	40	150	1.00	58.2417	40.30	77.1704	70.46	
23	2.5	35	200	3 25	60 7821	55.20	83 3817	85.84	



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24	4.0	40	150	5.50	83.2943	87.84	88.7486	89.52
25	4.0	40	50	5.50	92.1750	97.44	99.6121	97.26
26	7.0	40	150	5.50	55.9800	28.61	69.5121	54.77
27	2.5	35	200	7.75	90.1596	95.20	97.5162	98.60
28	4.0	40	150	5.50	83.2943	70.46	88.7486	89.24
29	4.0	40	150	5.50	83.2943	81.52	88.7486	89.52
30	2.5	35	100	3.25	88.2279	95.20	80.3262	79.52
31	2.5	45	200	7.75	95.8171	96.00	83.6367	89.52

Multiple regression coefficient R^2 is calculated from the second degree polynomial equation (equation 2 and 3), is R^2 =0.8410 for WH and R^2 =0.8872 for WHP indicates that the predicted values are closer to experimental data as shown in Table II. If R^2 value is closer to 1, then it is a good statistical model. The R^2 value is 0.8410 indicates that 84.10% of experimental data was well-suited with the model and the variations of 15.90% are not described by the model [27].

The experimental results were analysed and"t" test was conducted to find the importance of individual parameters, squared parameters and interactive parameters combination. Table III summarises the regression coefficients, 't' test results and probability values. Lesser the probability value more weightage is the effect. The results show that pH and biomass loading are significant for crude water hyacinth (probability value, p=0.000) whereas concentration and temperature*concentration interactive effect becomes significant for pyrolysed water hyacinth.

р **Regression Coefficient** t Term WHP WH WHP WH WHP WH Constant 83.294 88.748 14.118 22.449 0.000 0.000 pН -6.746 -4.169 -2.117 -1.953 0.050 0.069 Temperature 0.745 -3.398 0.234 -1.591 0.818 0.131 Concentration -1.464 -5.258 -0.460 -2.463 0.652 0.026 Biomass loading 8.275 3.898 2.592 1.86 0.020 0.087 pH*pH 0.254 -3.455 -2.724 -1.184 -1.393 0.183 Temperature*Temperature 4.083 1.413 1.399 0.722 0.181 0.48 Concentration*Concentration 3.988 0.087 1.366 0.044 0.191 0.965 Biomass loading*Biomass loading -2.134 -0.946 -0.731 -0.483 0.475 0.635 pH*Temperature -3.401 -0.551 -0.872 -0.211 0.396 0.836 pH *Concentration 1.712 -1.267 0.439 -0.484 0.667 0.635 pH*Biomass loading -4.597 -1.567 -0.402 -1.757 0.693 0.098 Temperature* Concentration 5.227 -5.844 1.340 -2.235 0.199 0.040 Temperature*Biomass loading -3.248 0.455 0.117 -1.242 0.909 0.232 Concentration*Biomass loading 0.819 0.326 0.210 0.125 0.836 0.902

TABLE III. STUDENT "T" TEST ANALYSIS FOR BIO SORPTION OF CHROMIUM

C. Analysis of Variance (ANOVA)

Table IV represents the results of multiple linear regressions conducted for the second order response surface model by ANOVA. The Fischer's variance ratio, F value = (Sr^2/Se^2) , is the ratio of mean square of regression to the mean square of the error. The Fischer's variance ratio with higher value and lower the probability P value (< 0.01) demonstrates significance for the regression model. From Table IV it is seen that linear effect was significant in both cases.



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TABLE IV. ANALYSIS OF VARIANCE (ANOVA) FOR THE SELECTED QUADRATIC MODEL FOR THE REMOVAL OF CHROMIUM

Source	DF		Sum of Squares		F		Prob>F	
	WH	WHP	WH	WHP	WH	WHP	WH	WHP
Regression	14	14	5070.47	3126.07	1.49	2.04	0.222	0.086
Linear	4	4	2793.39	1722.50	2.87	3.94	0.005	0.002
Square	4	4	1554.48	318.11	1.59	0.73	0.224	0.586
Interaction	6	6	722.60	1085.46	0.49	1.65	0.803	0.197
Residual error	16	16	3898.72	1750.47				
Total	30	30	8969.19	4876.54				

D. Optimization of Response

The optimization of process variables are done to get maximum % removal using response optimizer in Minitab 16. In this regard second degree polynomial equations (eq.2 and eq.3) were used to get the optimum values for the variables. Table V gives the optimum values for all adsorbents. Maximum chromium removal of 99.24% was achieved with water hyacinth activated carbon at initial metal concentration of 133.607 ppm, pH of 1.924, temperature of 40 °C and lowest biomass loading of 8.041 g/100 ml.

OPTIMIZED PARAMETERS									
Biosorbent	рН	Temperature (°C)	Initial metal ion Concentration (ppm)	Biomass Load (g/100ml)	Experimental % Chromium Removal				
WH	1.978	42.004	150.000	8.947	98.72				
WHP	1.924	40.000	133.607	8.041	99.24				

TABLE V

IV. CONCLUSION

In the present work biosorption of chromium was studied in batch experiments using three types of biosorbent derived from tamarind fruit shell. To study the effect of parameters variation like pH, temperature, initial chromium concentration and adsorbent dosage was studied using Central composite design. For all the adsorbents, it is observed from the student "t" test that for non- pyrolysed type of adsorbents, pH was significant and the temperature becomes significant for pyrolysed samples. ANOVA analysis reveals that for all the adsorbents linear effect was significant and for crude tamarind square effect is also significant. A regression coefficient of 0.8612 from ANOVA shows that the regression model with second order satisfactorily fits with the experimental data. The optimization of process variables indicates that crude tamarind removed maximum chromium of 99.24% with initial metal concentration of 133.607 ppm, pH of 1.924, temperature of 40 °C and lowest biomass loading of 8.041 g/100 ml. The study clearly demonstrating the use of water hyacinth activated carbon may be used to treat a chemical waste containing chromium.

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