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# Application Of Response Surface Methodology And CCD Method For The Biosorption Of Nickel From An Aqueous Solution On To IP Leaves Powder.

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**ABSTRACT**

Improved modern movement after the mechanical transformation has prompted the release of chemicals, which causes natural and general medical issues. The nearness of overwhelming metals in the earth is of significant concern due to their extraordinary poisonous quality and inclination for bioaccumulation in the natural pecking order even in moderately low focuses. The present work explores the growing of Ipomea Palmate leaves powder on biosorption of nickel metal present in a watery liquid course of action. The effects of various parameters (Time, pH, Dosage, Size, Concentration and Temperature) on biosorption of nickel are considered. Expulsion of nickel achieved a balance most extreme of 30 minutes. The trial data gave strong match with Langmuir isotherm taken after by Freundlich and Temkin isotherms. The Central Composite Design (CCD) programming was utilized to outline the analyses and to decide the optimum conditions.

**Key words**: Biosorption; Ipomea palmate; Isotherms.

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## INTRODUCTION

Metal pollution has been a great stress as far back as couple of decades. It is assumed that the wide usage of man-made chemicals, anthropogenic lifestyle, and fast industrialization is the huge wellspring of metal harmful quality[1]. Nickel is outstanding as a considerable metal poison, show in effluents of electroplating endeavors, cleaning, and composite gathering, mining, and refining industries [2]. Nickel has been caught as an embryotoxin and teratogen[3].The higher gathering of Nickel causes dermatitis, squeamishness, hurling, behavioral, and respiratory issues despite cyanosis, gastrointestinal inconvenience, and shortcoming [4]. All these natural issue results alert the need of nickel expulsion from the earth and to raise its levels beneath as far as possible from its sources. The established physicochemical strategies are generally utilized for the expulsion of nickel from the mechanical effluents, to be specific, evaporative recuperation, filtration, particle trade, and layer advances. in spite of the fact that they are promising to some degree, however these procedures have high reagent or vitality prerequisites and create lethal slime that requires watchful disposal[5].Biosorption is a procedure that utilizations economical biomaterials to sequester metals from fluid arrangements and the biomaterials utilized as a part of this procedure are named as biosorbents. The side-effects from agribusiness, nourishment and pharmaceutical businesses give monetarily suitable wellsprings of biosorbent; this makes biosorption a cheap option treatment strategy. Late research on biosorption has demonstrated that biomaterials containing acidic gatherings, for example, hydroxyls and carboxyls were compelling in restricting metal cations[6].

## MATERIALS & METHODS

### Preparation of the bisorbent

*Ipomea palmate* leaves were collected from a nearby Tenali canal place in Guntur District, Andhra Pradesh, India. The collected biosorbent was washed with distilled water several times until the dirt particles are removed. After through washing with distilled water, biosorbent was sun dried for ten days until they became crispy, cut into small pieces, powdered and sieved.

### Preparation of Nickel stock solution:

$Nicl_2.6H_2O$ is used as the source of nickel stock solution. All the required solutions are prepared with double distilled water.1000mg/L of nickel stock solution was prepared by dissolving appropriate amount of $Nicl_2.6H_2O$.Later the mother was solution was diluted to get the test solution like 20,40,80,120&160mg/L.

## RESULTS AND DISCUSSION

### Effect of agitation time

From the Fig.1The maximum percentage of biosorption is attained at 30 min of agitation. The percentage removal of Nickel becomes constant after 30 min indicating the attainment of the equilibrium. Therefore all other experiments are conducted at this agitation time[7,8,9,10].

### Effect of biosorbent size:

It was observed from fig. 2 that the percentage biosorption of nickel as a function of biosorbent size is decreases with increasing particle size due lack of lack of specific surface area[11].

### Effect of pH of the aqueous solution:

The effect of pH on the percentage of the Congo red is shown in figure 5 under various other fixed operating conditions. The initial pH of adsorption medium is one of the most important parameters affecting the adsorption process. It can be seen (fig.3) here that the percentage removal was increased from pH from 2 to 5[12].

**Effect of initial concentration of nickel in the aqueous solution:**

It was noted from the graph 4 that the % removal decreases with increase in metal concentration due to the adsorbent cannot accommodate much more adsorbate available in the solution[13].

**Effect of biosorbent dosage:**

The percentage removal of nickel is drawn against biosorbent dosage for biosorbent size 53µ and shown in figure.5. It is evident from the plots that the percentage removal of nickel metal from the aqueous phase increases with increase in the biosorbent dosage due to availability of surface area.[14]

**Effect of Temperature:**

The effect of changes in the temperature on the nickel uptake is shown in fig. 6. It was noted that the % Biosorption increase with increase in temperature up to 310K thereafter there is mild increase in removal of Nickel.

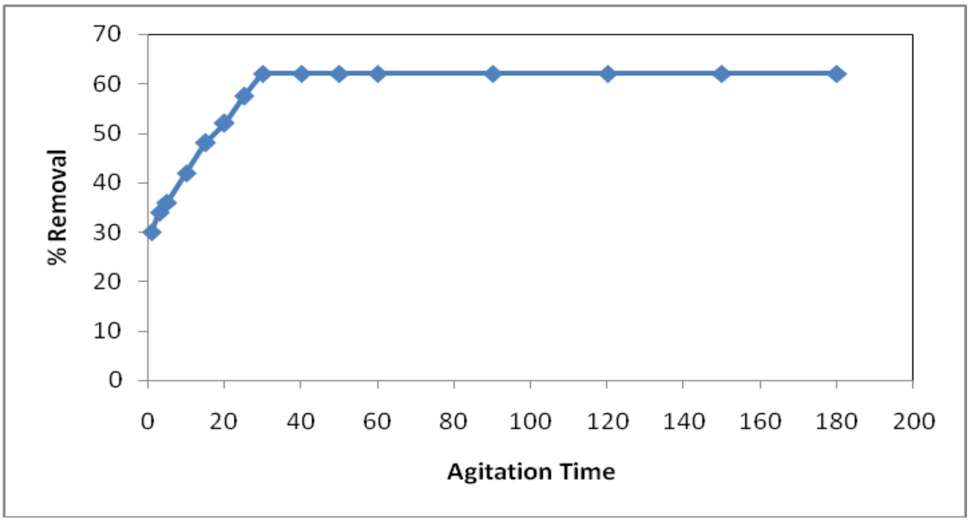


**Fig. 1 Effect of agitation time on % biosorption of nickel using Ipomea palmate leaves**

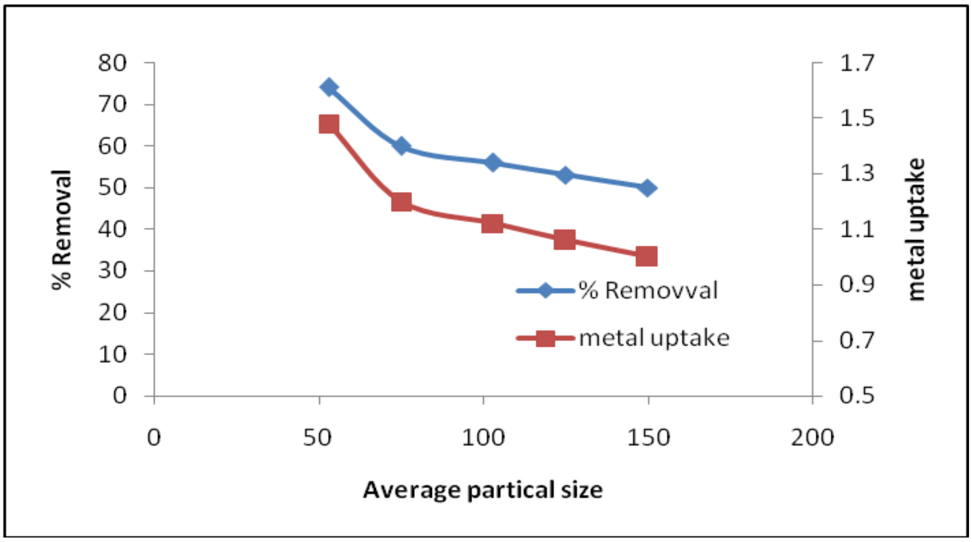


**Fig. 2 Measurement of % removal of nickel for biosorbent size**

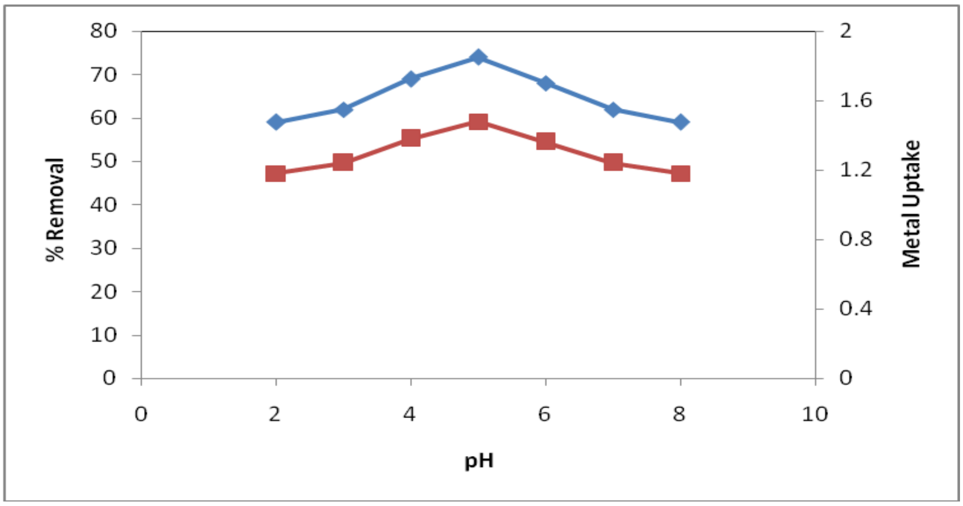


Fig.3 Dependence of % biosorption on pH of aqueous solution using Ipomea palmate

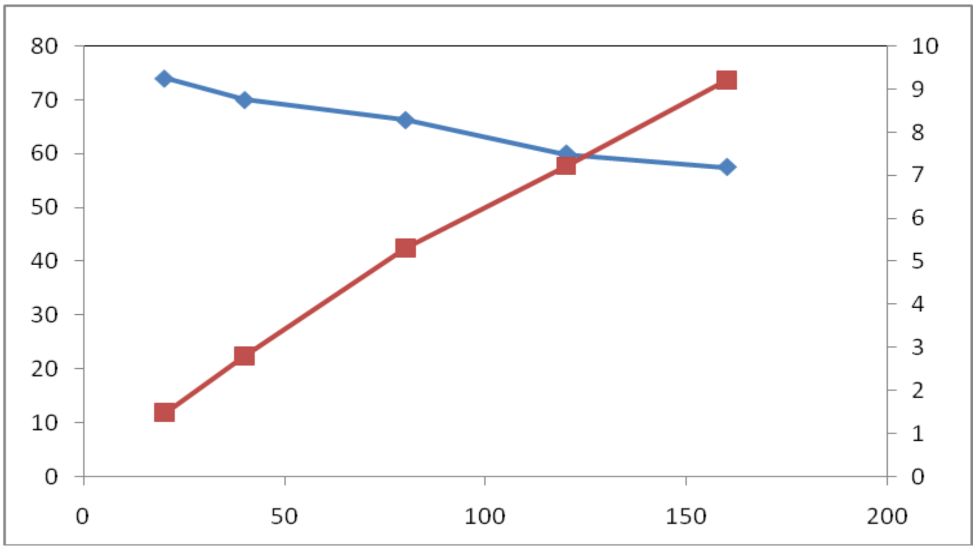


Fig. 4. % biosorption as a function of initial concentration of nickel

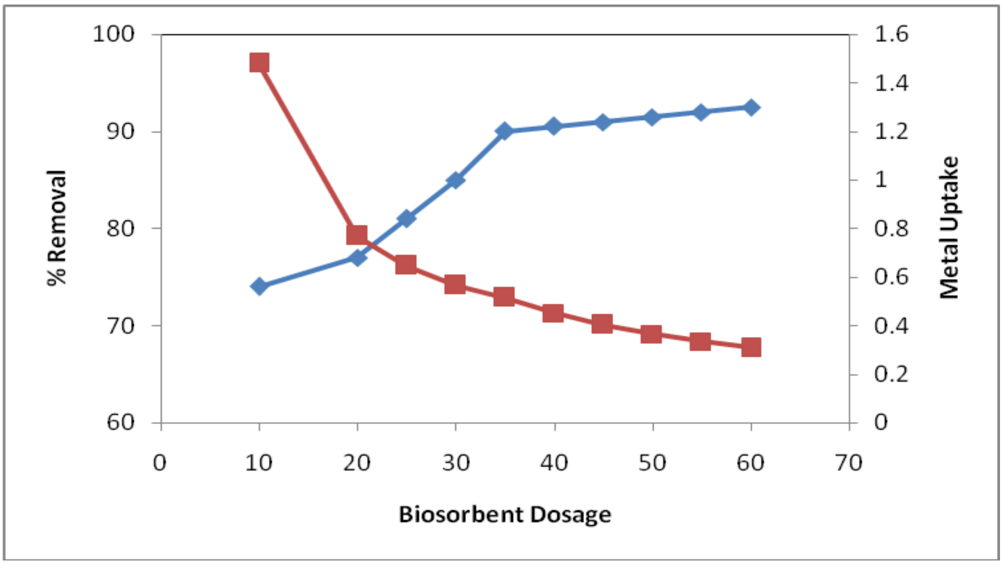


Fig.5. Dependence of % biosorption of nickle on biosorbent dosage

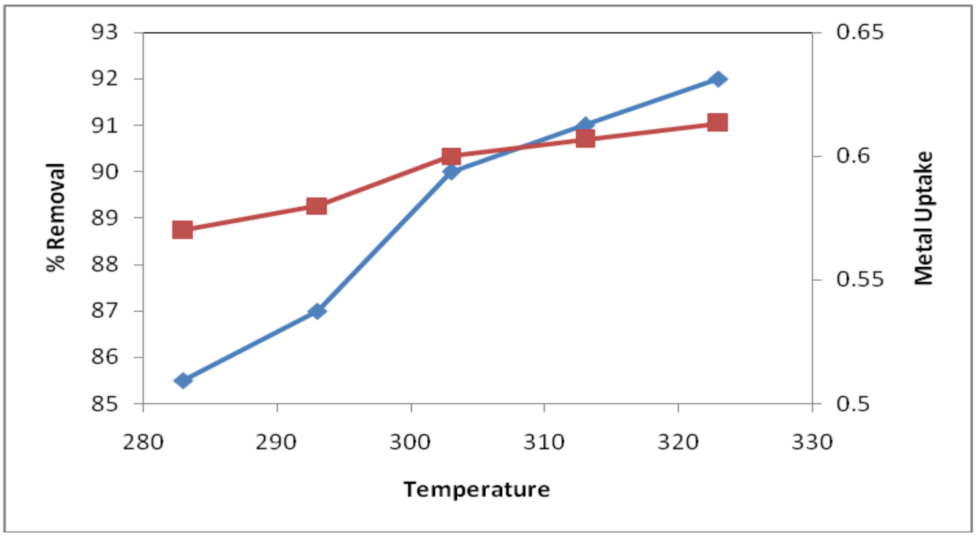


**Fig. 6. Variations of % biosorption of nickel on temperature**

**Isotherms:**

From the Table 1 it was observed that the Freundlich Isotherm gave good agreement for the removal and followed the Langmuir and Temkin isotherms.

**Kinetics of biosorption:**

From the Table 2 it was observed that second order was well fitted for sorption than the second order.

**Thermodynamics of biosorption:**

Enthalpy is the most commonly used thermodynamic function due to its practical significance. The negative value of ΔH will indicate the endothermic nature of biosorption and the physical/chemical in nature of sorption. It can be easily reversed by supplying the heat equal to calculated ΔH[15].

**Optimization using Response Surface Methodology (RSM):**

**Optimization using CCD**

The number of tests required for the CCD includes the standard 2n factorial with its origin at the center,2n points fixed axially. For four variables,the recommended number of tests at the center is six.Hence the total number (N) of tests required for the four independent variable is

$$N=2^n+2n+6=2^4+2\times4+6=30 \qquad \text{(i)}$$

The regression equation for % biosorption of Nickel ($Y$) is function of pH ($X_1$), $C_o$ ($X_2$), w ($X_3$) and T ($X_4$). The variations in the corresponding coded values of four parameters and response are presented in table 3.

$$\mathbf{Y = -715.744 + 29.334 X_1 + 1.165 X_2 + 2.340 X_3 + 4.415 X_4 - 3.108 X_1^2 - 0.043 X_2^2 - 0.026 X_3^2 - 0.007 X_4^2 + 0.111 X_1X_2 - 0.011 X_1X_3 - 0.0 X_1X_4 - 0.005 X_2X_3 - 0.0 X_2X_4 - 0.001 X_3X_4} \qquad \textbf{(ii)}$$

The following equation represents multiple regression analysis of the experimental data for the biosorption of Nickel:

$$\mathbf{Y = -715.744 + 29.334 X_1 + 1.165 X_2 + 2.340 X_3 + 4.415 X_4 - 3.108 X_1^2 - 0.043 X_2^2 - 0.026 X_3^2 - 0.007 X_4^2 + 0.111 X_1X_2 - 0.011 X_1X_3 - 0.005 X_2X_3 - 0.001 X_3X_4} \qquad \textbf{(iii)}$$

Table-4 represents the results obtained in CCD.

Experimental conditions [Coded Values] and observed response values of central composite design with $2^4$ factorial runs, 6- central points and 8- axial points. Agitation time fixed at 25 min and biosorbent size at 53 µm Response obtained from regression in eq.(iii) in the form of ANOVA is presented.

**Interaction effects of biosorption variables:**

The three-dimensional view of response surface contour plots [Fig.10 (a) to (f)] show % biosorption as a function of four various combinations of independent variables. The % biosorption of Nickel is strongly influenced as evident from figs. 10 (a) & (f).

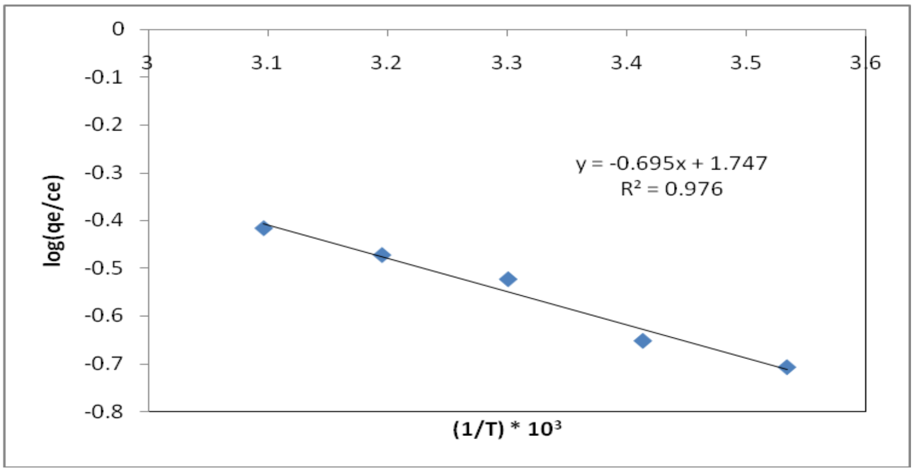


**Fig.7 Vant Hoff's plot for biosorption**

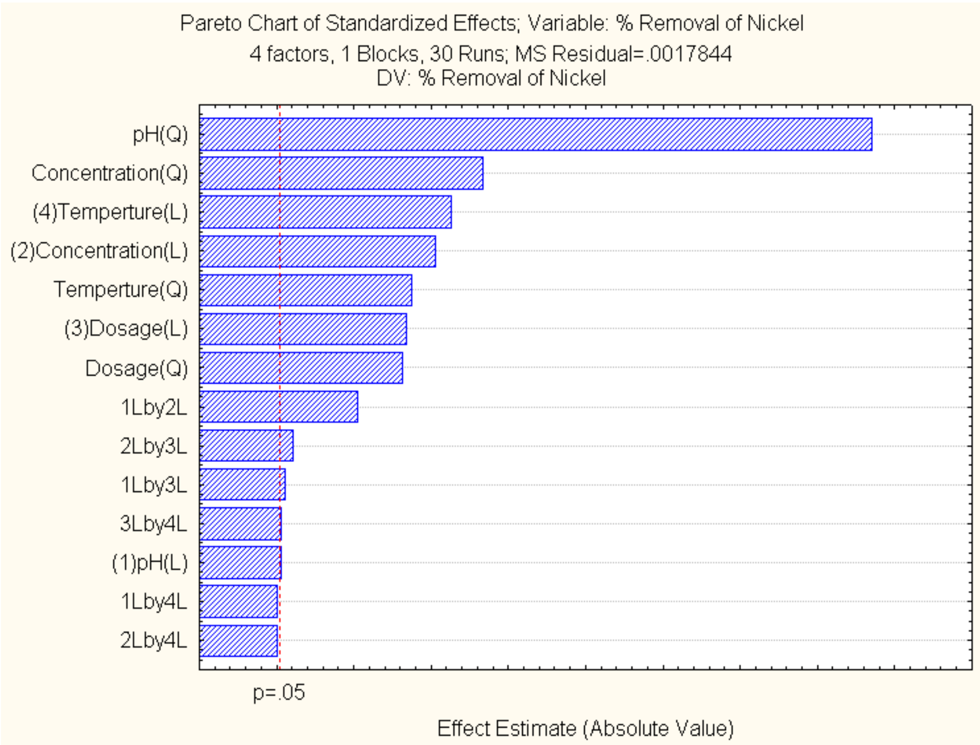


**Fig. 8 Pareto Chart**

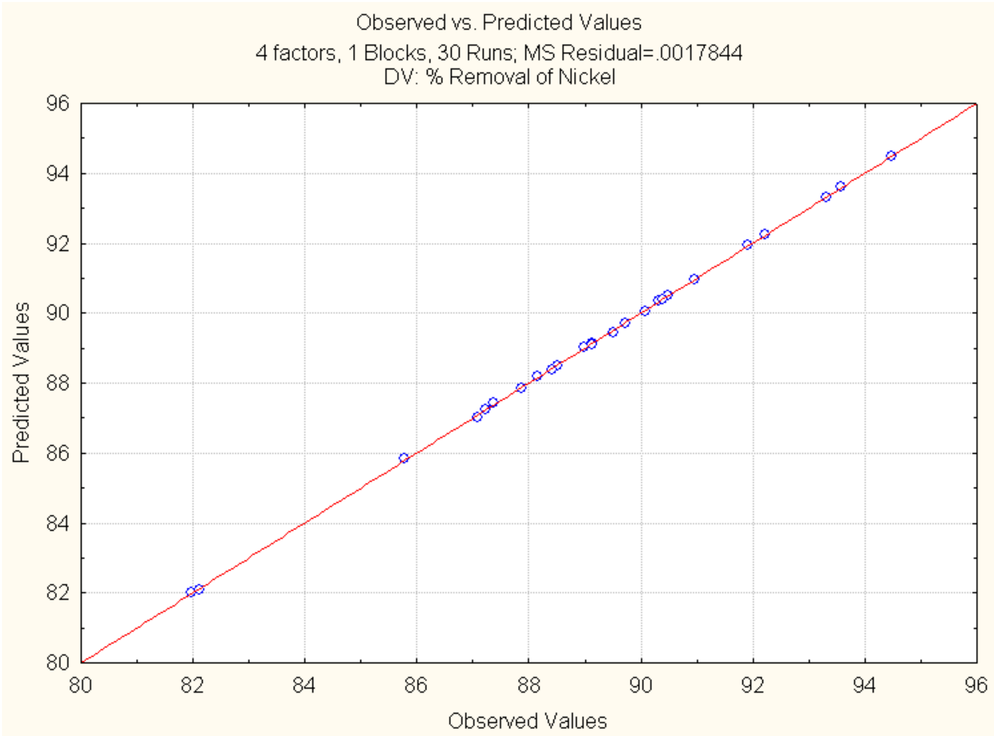


**Fig. 9 Normal probability plot for % biosorption of Nickel**

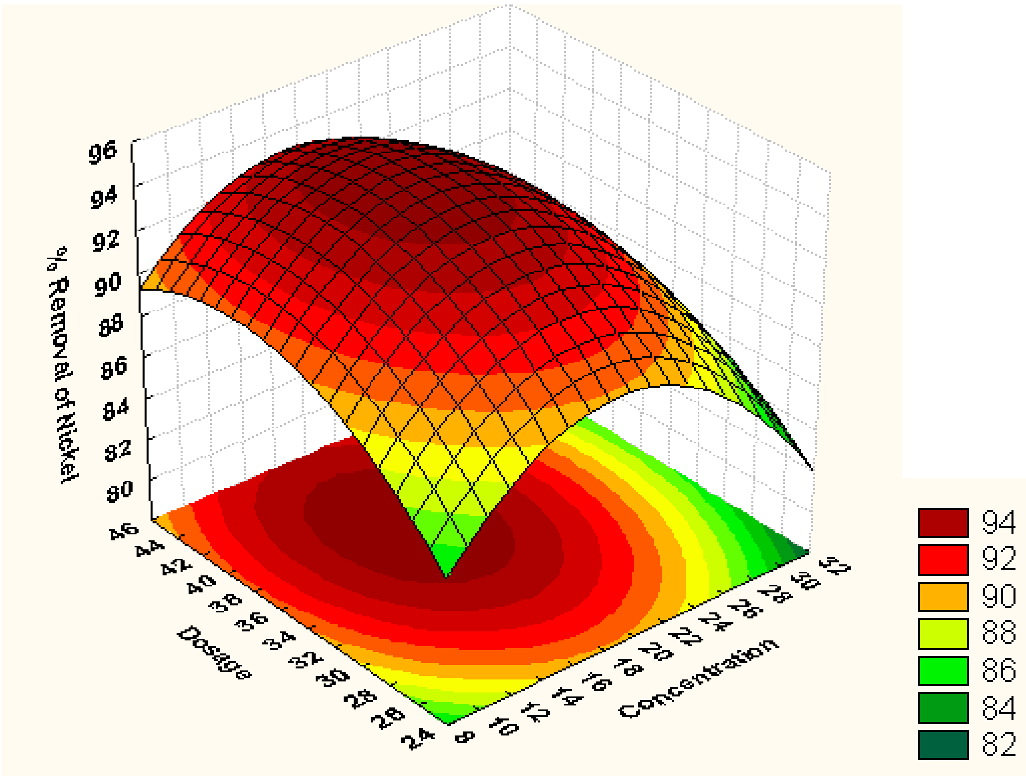


**Fig. 10 (a) Surface contour plot for the effects of dosage and initial concentration of nickel on % biosorption**

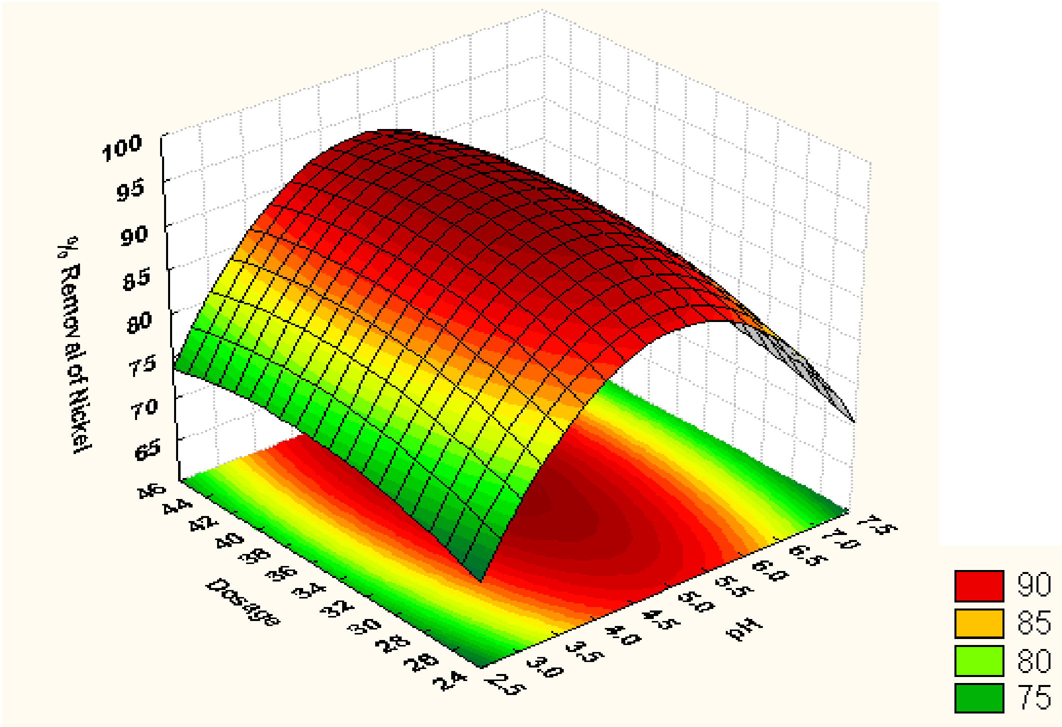


**Fig. 10 (b) Surface contour plot for the effects of dosage and pH on % biosorption of nickel**

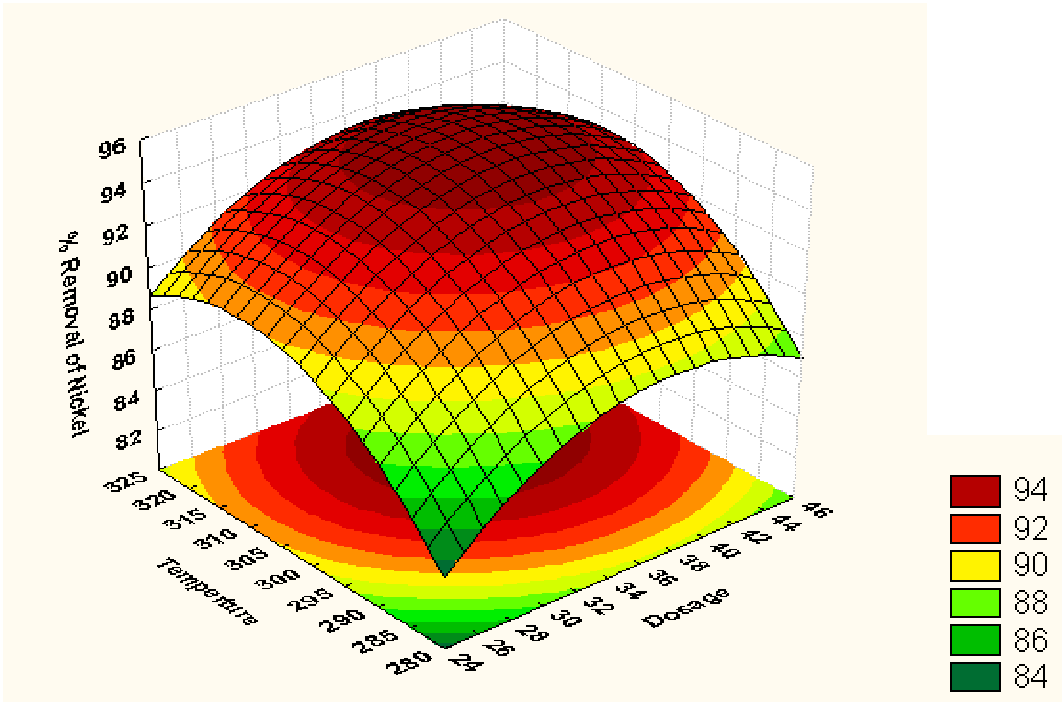


**Fig. 5.10 (c) Surface contour plot for the effects of dosage and Temperature on % biosorption of nickel**

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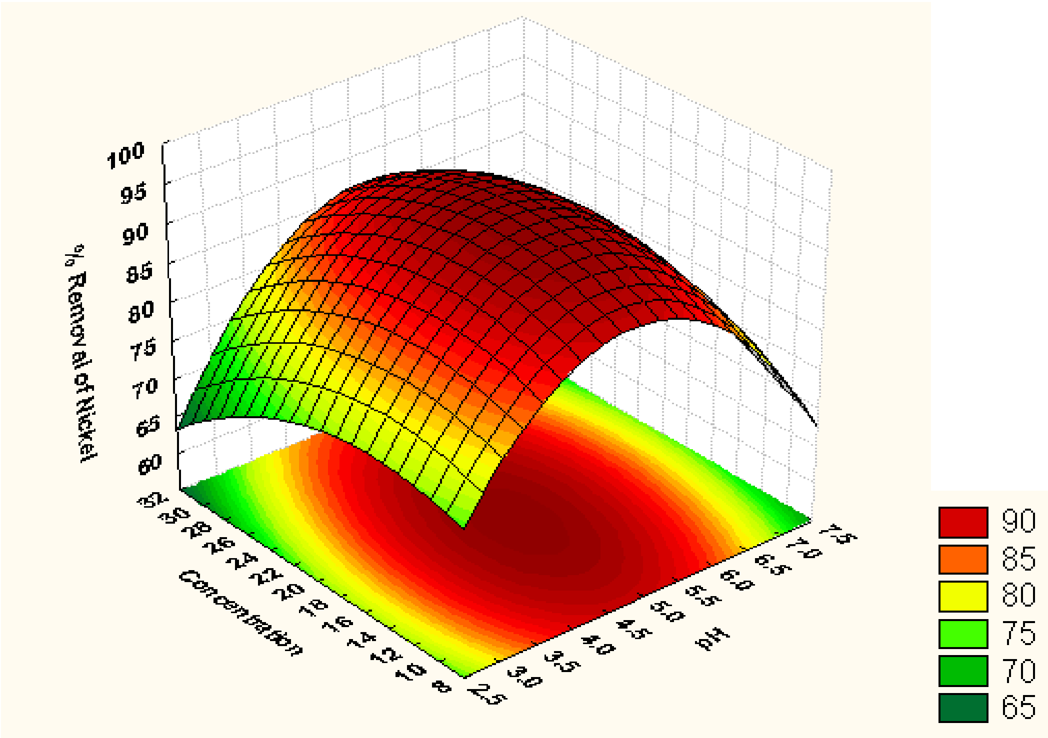


Fig. 10 (d) Surface contour plot for the effects of initial concentration and pH on % biosorption of nickel

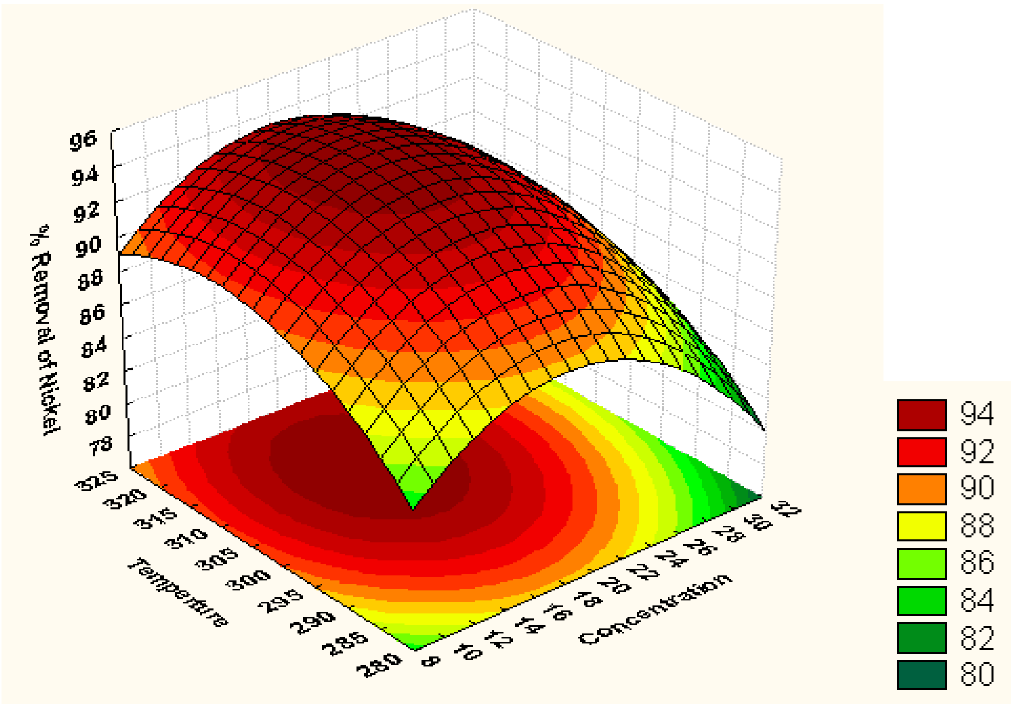


Fig. 10 (e) Surface contour plot for the effects of initial concentration and Temperature on % biosorption of nickel

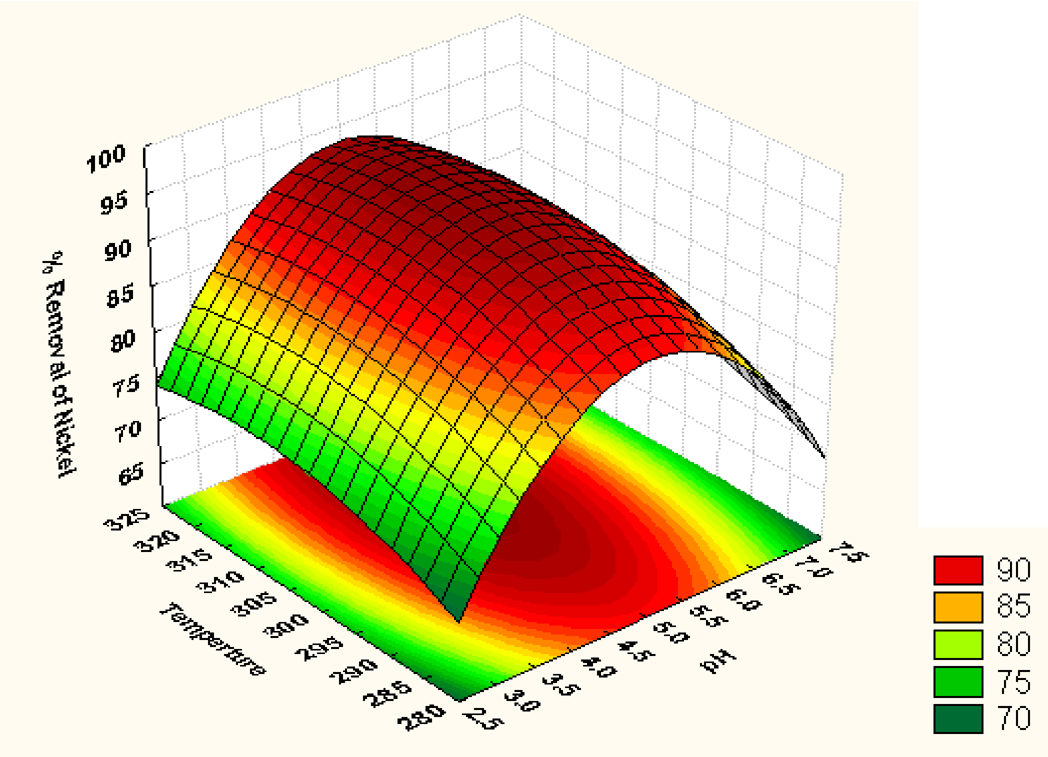


**Fig. 10 (f) Surface contour plot for the effects of pH and Temperature on % biosorption of nickel**

The optimal sets of conditions obtained with CCD and experimental are shown in table-7 along with experimental values [16,17,18].

**Table–1 Isotherm constants**

| Langmuir | Freundlich | Temkin |
|---|---|---|
| $q_m$ = 16.39344 | $k_f$ = 0.45113 | $A_T$ = 0.260375 |
| b = 0.017873 | n = 0.600508 | $b_T$ = 852.7901 |
| $R^2$ =0.981 | $R^2$ =0.996 | $R^2$ =0.963 |

**Table–2 Equations and rate constants**

| Order | Equation | Rate constants | $R^2$ |
|---|---|---|---|
| Lagergren first order | $\log(q_e\text{-}q_t)$=–0.032 t–0.120 | 0.073696 $min^{-1}$ | 0.953 |
| Pseudo second order | $t/q_t$=0.833 t+2.243 | 0.309358 g/(mg-min) | 0.980 |

**Table-3 Levels of different process variables in coded and un-coded form for % biosorption of Nickel using *ipomea palmate* leaves powder**

| Variable | Name | Range and levels | | | | |
|---|---|---|---|---|---|---|
| | | -2 | -1 | 0 | 1 | 2 |
| $X_1$ | pH of aqueous solution | 3 | 4 | 5 | 6 | 7 |
| $X_2$ | Initial Ni concentration, $C_o$, mg/L | 10 | 15 | 20 | 25 | 30 |
| $X_3$ | Biosorbent dosage, w, g/L | 25 | 30 | 35 | 40 | 45 |
| $X_4$ | Temperature, T, K | 283 | 293 | 303 | 313 | 323 |

**Table-4 Results from CCD for Nickel biosorption by *ipomea palmate* leaves powder**

| Run No. | $X_1$, pH | $X_2$, $C_o$ | $X_3$, W | $X_4$, T | % biosorption of nickel | |
|---|---|---|---|---|---|---|
| | | | | | Experimental | Predicted |
| 1 | 4 | 15 | 30 | 293 | 88.52 | 88.48 |
| 2 | 4 | 15 | 30 | 313 | 90.48 | 90.50 |
| 3 | 4 | 15 | 40 | 293 | 90.32 | 90.34 |
| 4 | 4 | 15 | 40 | 313 | 92.22 | 92.23 |
| 5 | 4 | 25 | 30 | 293 | 85.78 | 85.84 |
| 6 | 4 | 25 | 30 | 313 | 87.88 | 87.86 |
| 7 | 4 | 25 | 40 | 293 | 87.22 | 87.23 |
| 8 | 4 | 25 | 40 | 313 | 89.12 | 89.12 |
| 9 | 6 | 15 | 30 | 293 | 87.38 | 87.44 |
| 10 | 6 | 15 | 30 | 313 | 89.52 | 89.45 |
| 11 | 6 | 15 | 40 | 293 | 89.12 | 89.09 |
| 12 | 6 | 15 | 40 | 313 | 90.96 | 90.96 |
| 13 | 6 | 25 | 30 | 293 | 87.08 | 87.01 |
| 14 | 6 | 25 | 30 | 313 | 88.98 | 89.02 |
| 15 | 6 | 25 | 40 | 293 | 88.16 | 88.20 |
| 16 | 6 | 25 | 40 | 313 | 90.08 | 90.07 |
| 17 | 3 | 20 | 35 | 303 | 82.12 | 82.09 |
| 18 | 7 | 20 | 35 | 303 | 81.98 | 82.00 |
| 19 | 5 | 10 | 35 | 303 | 91.92 | 91.94 |
| 20 | 5 | 30 | 35 | 303 | 88.42 | 88.40 |
| 21 | 5 | 20 | 25 | 303 | 90.38 | 90.39 |
| 22 | 5 | 20 | 45 | 303 | 93.32 | 93.30 |
| 23 | 5 | 20 | 35 | 283 | 89.72 | 89.70 |
| 24 | 5 | 20 | 35 | 323 | 93.58 | 93.60 |
| 25 | 5 | 20 | 35 | 303 | 94.48 | 94.48 |
| 26 | 5 | 20 | 35 | 303 | 94.48 | 94.48 |
| 27 | 5 | 20 | 35 | 303 | 94.48 | 94.48 |
| 28 | 5 | 20 | 35 | 303 | 94.48 | 94.48 |
| 29 | 5 | 20 | 35 | 303 | 94.48 | 94.48 |
| 30 | 5 | 20 | 35 | 303 | 94.48 | 94.48 |

**Table-5 ANOVA of Nickel biosorption for entire quadratic model**

| Source of variation | SS | df | Mean square(MS) | F-value | *P* > F |
|---|---|---|---|---|---|
| Model | 337.0183 | 14 | 24.0727 | 13473.5261 | 0.00000 |
| Error | 0.0268 | 15 | 0.001786 | | |
| Total | 337.0451 | 29 | | | |

df- degree of freedom; SS- sum of squares; F- factor F; *P*- probability $R^2$=0.99996; $R^2$ (adj):0.99988:

**Table-6 Regression coefficients for the Nickel biosorption onto *ipomea palmate* leaves powder**

| Terms | Regression coefficient | Standard error of the coefficient | t-value | *P*-value |
|---|---|---|---|---|
| **Mean / Intercept** | -715.744 | 8.126507 | -88.075 | 0.000000 |
| **Dosage, w, g/L (L)** | 29.334 | 0.340913 | 86.044 | 0.000000 |
| **Dosage, w, g/L (Q)** | -3.108 | 0.008066 | -385.344 | 0.000000 |
| **Conc, Co, mg/L (L)** | 1.165 | 0.067789 | 17.182 | 0.000000 |
| **Conc, Co, mg/L (Q)** | -0.043 | 0.000323 | -133.665 | 0.000000 |
| **pH (L)** | 2.340 | 0.069222 | 33.798 | 0.000000 |
| **pH (Q)** | -0.026 | 0.000323 | -81.594 | 0.000000 |
| **Temperature, T, K (L)** | 4.415 | 0.049903 | 88.465 | 0.000000 |
| **Temperature, T, K (Q)** | -0.007 | 0.000081 | -87.793 | 0.000000 |
| **1L by 2L** | 0.111 | 0.002112 | 52.435 | 0.000000 |
| **1L by 3L** | -0.011 | 0.002112 | -5.090 | 0.000133 |
| **1L by 4L** | -0.000 | 0.001056 | -0.355 | **0.727465** |
| **2L by 3L** | -0.005 | 0.000422 | -11.008 | 0.000000 |
| **2L by 4L** | -0.000 | 0.000211 | -0.118 | **0.907350** |
| **3L by 4L** | -0.001 | 0.000211 | -3.196 | 0.006015 |

[a]insignificant ($P \geq 0.05$)

**Table – 7 Comparison between optimum values from CCD and experimentation**

| Variable | CCD | Experimental |
|---|---|---|
| pH of aqueous solution | **4.95** | **5** |
| Initial nickel concentration, mg/L | **17.72** | **20** |
| Biosorbent dosage, w, g/L | **37.88** | **35** |
| Temperature, K | **309.74** | **303** |
| % biosorption | **95.22** | **92.0** |

## CONCLUSIONS

The experimental results were analytically discussed and the following conclusions could be drawn from the study of biosorption of Nickel from aqueous solution using biosorption technique.The equilibrium agitation time for biosorption of nickel is 30 min.

1. The optimum dosage is 35 g/L
2. % biosorption is increased upto pH = 5.
3. The RSM optimized values are: w = 37.88 g/L, pH = 4.95, $C_o$ = 17.72 mg/L, T = 309.74 K and extent of biosorption = 95.22 %.
4. The experimental data were well fitted with freundlich isotherm.
5. The kinetic studies show that the biosorption of nickel is described by both second order.The thermodynamic investigation reveals the spontaneity ($\Delta G$ = –9890), irreversibility ($\Delta S$ =32.68) and endothermic ($\Delta H$ = 13.307) nature of biosorption.

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