

Application of Response Surface methodology (RSM) for the Removal of Nickel Using Rice Husk ASH as Biosorbent

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ABSTRACT

The Rice Husk Ash is a cheap and readily available biosorbent for the removal of nickel ions from aqueous solutions. This investigation comprises of the equilibrium and kinetics study for biosorption of nickel ions from aqueous solutions using Rice Husk Ash powder as a biosorbent in a batch process. The Rice Husk Ash is obtained from a hotel near Andhra University campus, is dried in sunlight for 15 days. The biosorption process is carried out in batch process by varying four parameters: pH, Dosage, Concentration and Temperature of the solution of the process.

The results indicate that biosorption of nickel is increased with an increase in biosorbent dosage. A significant increase in percentage biosorption of nickel is observed as pH is increased from 2 to 5 and the percentage biosorption decreased beyond pH 5. Increased initial concentration of nickel in the aqueous solution resulted in lower percentage of biosorption. Langmuir, Freundlich and Temkin isotherm models describe the present data very well indicating favorable biosorption. The biosorption followed pseudo-second-order kinetics.

The present study involves the use of statistical design to optimize process conditions for maximal biosorption of nickel from aqueous solution using BBD involving RSM

Hence the Rice Husk Ash is highly effective as biosorbent for biosorption of nickel ions from aqueous solutions and can be appreciably considered as most versatile, economical, feasible and efficient biosorbent for biosorption of nickel ions from aqueous solutions.

Keywords: Rice Husk Ash, Biosorption, Nickel, Response Surface Methodology, Box-Behnken Design, Optimization, Equilibrium isotherms, Low cost biosorbent.

INTRODUCTION:

A vast number of raw materials for industrial processes originate from agricultural activities, which result in the production of chemical and solid wastes. The chemical wastes arise from the use of pesticides, dyes and fertilizers while the solid wastes include bagasse, sawdust, rice husk, peanut shell and coffee husk, among others. Interestingly, the agricultural solid wastes can be converted to adsorption media and used to treat the chemical wastes; a concept of using waste to treat waste. By this concept, the cost of adsorption material for wastewater treatment, which is a major constraint in wastewater management, is generally reduced. Due to environmental concerns and the demand for high-quality water, there has been an increase in regulations controlling the discharge of heavy metals and non-biodegradable toxic compounds into water bodies. This has resulted in developing toxic waste removal techniques such that only minute quantities remain in the wastewater discharged into water bodies.[1]

Traditional methods for removal of lead ions from solution include chemical precipitation, ion exchange, electro dialysis and membrane separations. All these methods have various disadvantages, specifically, high capital investment and operating cost, incomplete removal, low selectivity and high energy consumption. Therefore, there is a need for a cost effective treatment method that is capable of removing low concentrations of lead from solution. For the last decades, biosorption or sorption of contaminants by sorbents of natural origin has gained important credibility due to the good performance and low cost of these complexing materials.[2]

Biosorption, which is defined as the accumulation and concentration of pollutants from aqueous solutions by the use of biological materials, appears to offer a technically feasible and economically attractive approach. The biosorption mechanism of heavy metals is theorized to be a combination of active and passive transport starting with diffusion of the metal ions to the surface of the microbial cell. The coordination of metal ions to different functional groups, such as amino, thioether, carboxyl, hydroxyl, carbonyl, phosphate, phenolic, etc., groups, in or on the algal cell biomass makes it a good biosorbent for removal of heavy metals from aqueous solutions.[3]

In recent years, biosorption has been suggested as being cheaper and more effective than chemical (precipitation) or physical (ion exchange and membrane) technologies. Biosorption involves the use of biological materials that form complexes with metal ions using their ligands or functional groups. Most metal sorption reported in literature is based on bacterial, algal and fungal biomass, which needs to be cultured, collected from their natural habitats and pre-processed, with the result of additional costs. The use of biosorbents from numerous lignocellulosic agro wastes is a very constructive approach and has received much attention in sorption of heavy metals, because they are inexpensive and have high adsorption properties resulted from their ion exchange capabilities. [4]

Rice husk are an agricultural waste produced as by-product of the rice milling industry to be about more than 100 million tones, 96% of which is generated in developing countries. Rice husk is mostly used as a fuel in the boiler furnaces of various industries to produce steam. The ash generated after burning the rice husk in the boiler is called rice husk ash. The R.H was collected from the particulate collection equipment attached up stream to the stack of rice-fired boilers. The ash generated got a server disposal problem. The objective of this study was to explore the possibility using R.H and R.H.A. for removing Pb(II) from aqueous solution.[5]

Nickel(II) ion is one such heavy metal frequently encountered in raw wastewater streams from industries such as non-ferrous metal, mineral processing, paint formulation, electroplating, porcelain enameling, copper sulphate manufacture and steam-electric power plants. [6]

2. Materials and methods

2.1 Preparation of the adsorbent

Nickel (II) Nitrate, $Ni(NO_3)_2 \cdot 6H_2O$ is used as the source for Nickel stock solution. All the required solutions are prepared with analytical reagents and double-distilled water. 5.102g of 98% $Ni(NO_3)_2 \cdot 6H_2O$ is dissolved in distilled water of 1.0 L volumetric flask up to the mark to obtain 1000 ppm (mg/L) of Nickel stock solution. Synthetic samples of different concentrations of Nickel are prepared from this stock solution by appropriate dilutions. 100 mg/L Nickel stock solution is prepared by diluting 10 mL of 1000 ppm Nickel stock solution with distilled water in 100 mL volumetric flask up to the mark. Similarly solutions with different metal concentrations such as (20 to 200ppm) are prepared.

2.2 Batch mode adsorption studies

Batch mode adsorption studies for individual metal compounds were carried out to investigate the effect of different parameters such as, agitation time, pH, adsorbate concentration, adsorbent dosage, and temperature. Solution containing adsorbate and adsorbent was taken in 250 ml capacity conical flasks and agitated at 180 rpm in a mechanical shaker at predetermined time intervals. The adsorbate was decanted and separated from the adsorbent using filter paper (Whatman No-1). The filtrates are analyzed in Atomic Absorption Spectrophotometer.

Table 1. Range of different parameters investigated in the present study

Parameter	Values Investigated
Agitation time, t, min	2, 5, 10, 20, 30, 50, 70, 90, 120, 150, and 180.
pH of aqueous solution	2, 3, 4, 5, and 6

Initial Nickel concentration, C_0 , ppm	20, 40, 80, 120, 160, 180, and 200
adsorbent dosage, w, g	0.5, 0.75, 1, 1.25, 1.5, 1.75, 2, 2.5, and 3
Temperature, K	283, 293, 303, 313 and 323 K

3. RESULTS AND DISCUSSION

3.1 Effect of agitation time on biosorption of nickel:

Duration of equilibrium biosorption is defined as the time required for heavy metal concentration to reach a constant value during biosorption. The equilibrium agitation time is determined by plotting the % biosorption of nickel against agitation time as shown fig. 3.1 for the interaction time intervals between 1 to 180 min. For 74 μm size of 0.5, 1, and 1.5 gram biosorbent dosage, 47.48% (0.28949mg/g) of nickel is biosorbed in the first 5 min. The % biosorption is increased briskly up to 90 min reaching 61.56% (0.375343mg/g). Beyond 90 min, the % biosorption is constant indicating the attainment of equilibrium for all dosage of biosorbent.

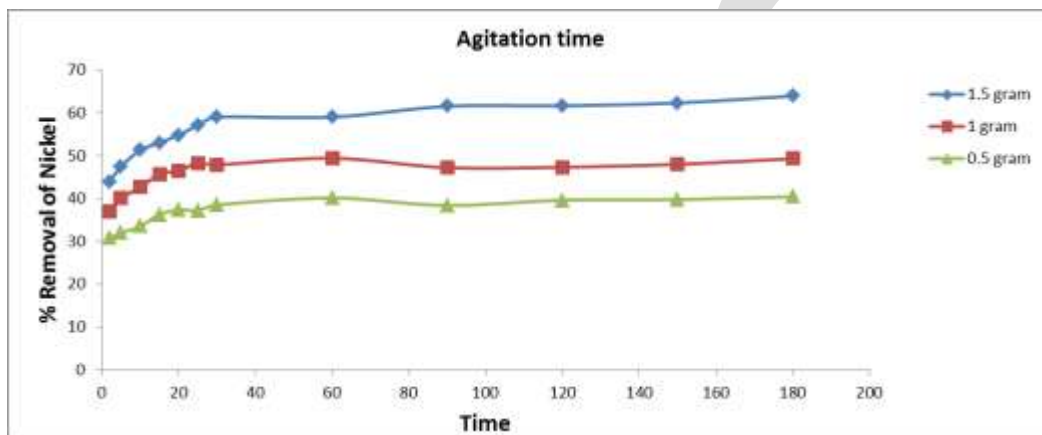


Fig 3.1 Effect of agitation time on biosorption of nickel

3.2 Effect of pH of the aqueous solution:

pH controls biosorption by influencing the surface change of the biosorbent, the degree of ionization and the species of biosorbate. In the present investigation, nickel biosorption data are obtained in the pH range of 2 to 6 of the aqueous solution ($C_0 = 100 \text{ mg/L}$) using 1 gram of 74 μm size biosorbent. The effect of pH of aqueous solution on % biosorption of nickel is shown in fig.3.2. The % biosorption of nickel is increased from 28.19 % (2.2725mg/g) to 52.28 % (4.3935mg/g) as pH is increased from 2 to 5 and decreased beyond the pH value of 5 % biosorption is decreased from pH 5 to 6 reaching 47.00 % (4.2824mg/g) from 52.28 % (4.3935mg/g).

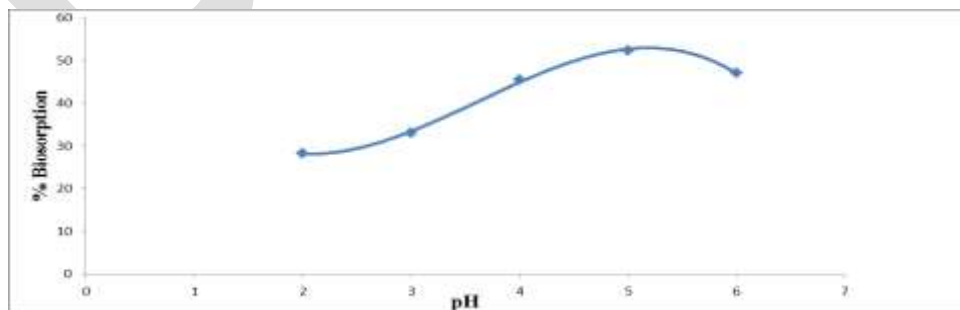


Fig. 3.2 Dependence of % biosorption of nickel on pH

3.3 Effect of initial concentration of Nickel:

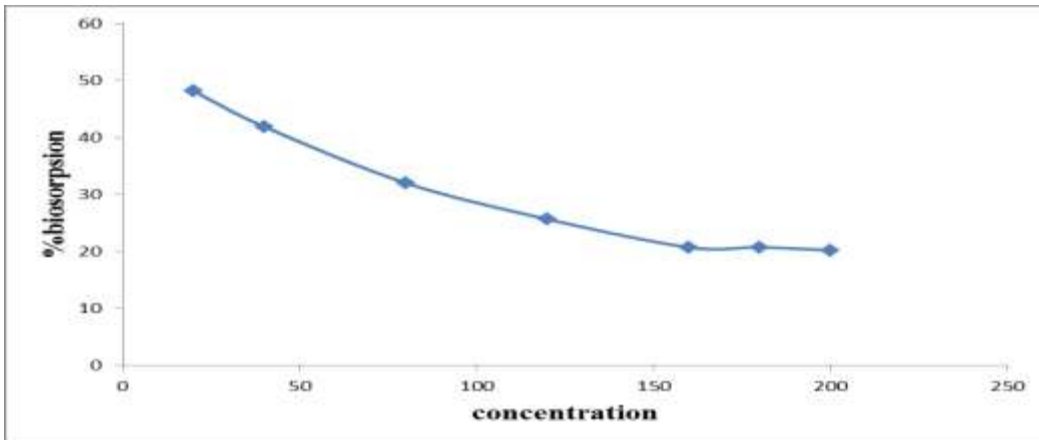


Fig.3.3 % Biosorption as a function of initial concentration of nickel

The effect of initial concentration of nickel in the aqueous solution on the percentage biosorption of nickel is shown in fig.3.3. The percentage biosorption of nickel is decreased from 48.09% (1.6564mg/g) to 20.19 % (4.0602mg/g) with an increase in C_0 from 20 mg/L to 200 mg/L. Such behavior can be attributed to the increase in the amount of biosorbate to the unchanging number of available active sites on the biosorbent (since the amount of biosorbent is kept constant)

3.4 Effect of biosorbent dosage:

The percentage biosorption of nickel is drawn against biosorbent dosage for 74 μm size biosorbent in fig.3.4. The biosorption of nickel increased from 34.23% (0.4122mg/g) to 92.02% (0.1847 mg/g) with an increase in biosorbent dosage from 0.5 to 3 gm. Such behavior is obvious because with an increase in biosorbent dosage, the number of active sites available for nickel biosorption would be more. Hence all other experiments are conducted at 1.25 gram dosage where the % biosorption of nickel is 94.56% (0.45552 mg/g).

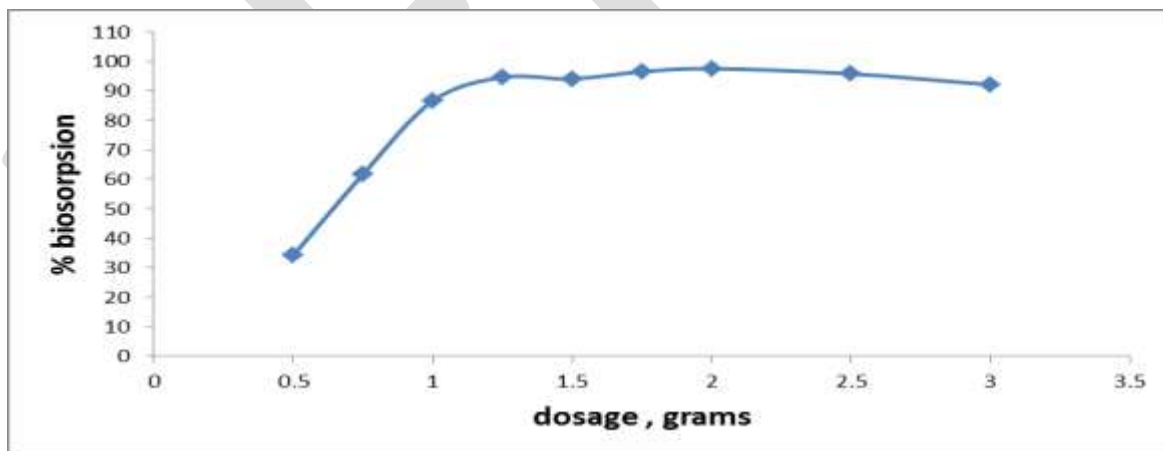


Fig. 3.4 Dependence of %biosorption of nickel on biosorbent dosage

3.5 Effect of Temperature:

The effect of temperature on the equilibrium metal uptake was significant. The effect of changes in the temperature on the nickel uptake is shown in Fig.3.5. When temperature was lower than 303 K, Nickel uptake increased with increasing temperature, but when temperature was over 303 K, the results were on the contrary. This response suggested a different interaction between the ligands on the cell wall and the metal. Below 303 K, chemical biosorption mechanisms played a dominant role in the whole biosorption process, biosorption was expected to increase by increase in the temperature. While at higher temperature and physical biosorption became the main process. Physical biosorption reactions were normally exothermic, thus the extent of biosorption generally is constant with further increasing temperature.

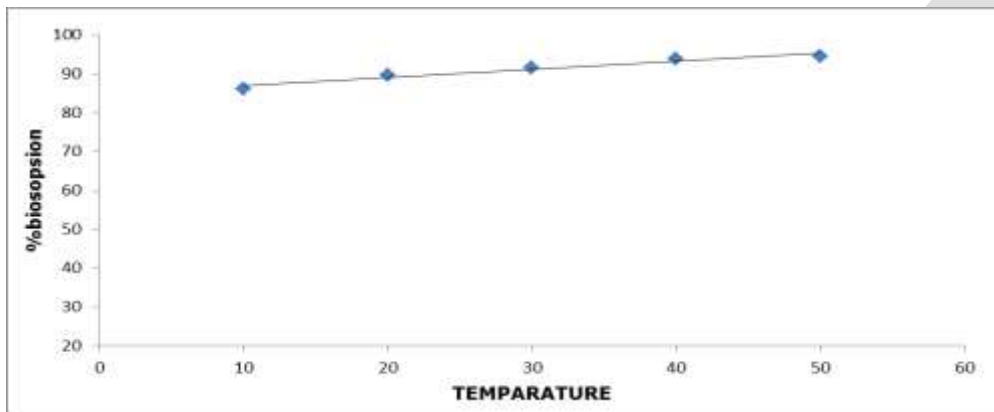


Fig 3.5 Dependence of %biosorption of nickel on biosorbent temperature

3.6 Freundlich isotherm for adsorption of Nickel:

Freundlich isotherm is drawn between $\ln C_e$ and $\ln q_e$ as shown in the figure for the present data. The resulting equation has a correlation coefficient of 0.9945. The following equation are obtained from the plot drawn in the fig 3.6. $\ln q_e = 0.4024 \ln C_e - 0.2718$

The slopes (n) of the above equation are varied between the 'n' value in the above equations satisfies the condition of $0 < n < 1$ indicating favorable biosorption.

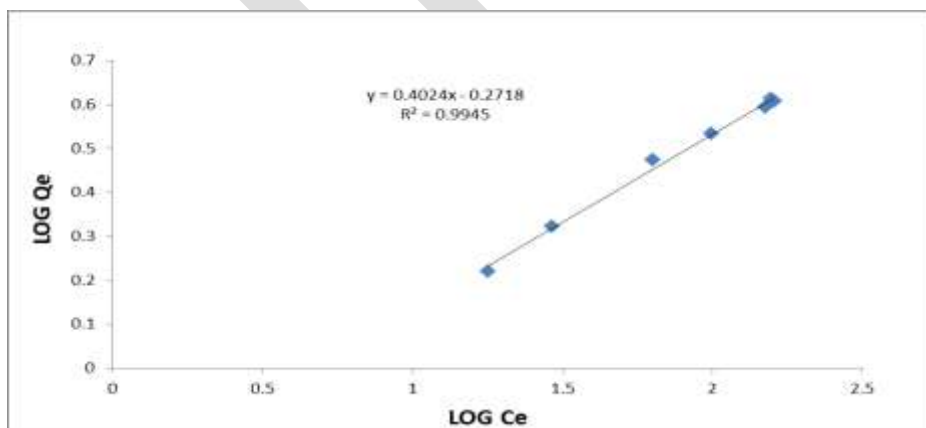


Fig.3.6 Freundlich isotherm for biosorption of Nickel

3.7 Langmuir isotherm for adsorption of Nickel:

Langmuir isotherm is drawn for the present data and shown in Fig.3.7 have good linearity (correlation coefficient, $R \sim 0.9958$) indicating strong binding of Nickel ions to the surface of rice husk ash. The separation factor (R_L), obtained is 0.6976 Shows favorable adsorption.

The following equation obtained from 3.6

$$C_e/q_e = 0.1977 C_e + 8.1535$$

The isotherm constants for nickel- rice husk ash interactions at 303°K , $t=90$ min, $C_o=20\text{mg/L}$, $d_p=74\mu\text{m}$ and $w = 1.25\text{g}$ are shown below.

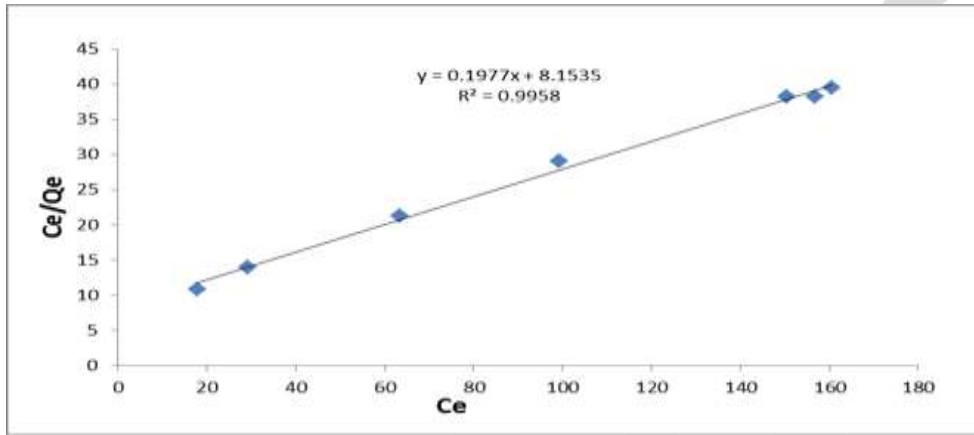


Fig. 3.7 Langmuir isotherm for biosorption of nickel

3.8 Temkin isotherm for adsorption of Nickel:

The present data are analyzed according to the linear form of Temkin isotherm and the linear plot is shown in Fig.3.8. The equation obtained for nickel biosorption is: $q_e = 61.019 \ln C_e - 97.183$ with a correlation coefficient 0.9563. The best fit model is determined based on the linear regression correlation coefficient (R). From the Figs 3.6, 3.7 & 3.8, it is found that biosorption data are well represented by Langmuir isotherm with higher correlation coefficient of 0.9985, followed Freundlich and Temkin isotherms with correlation coefficients of 0.9945 and 0.9692 respectively.

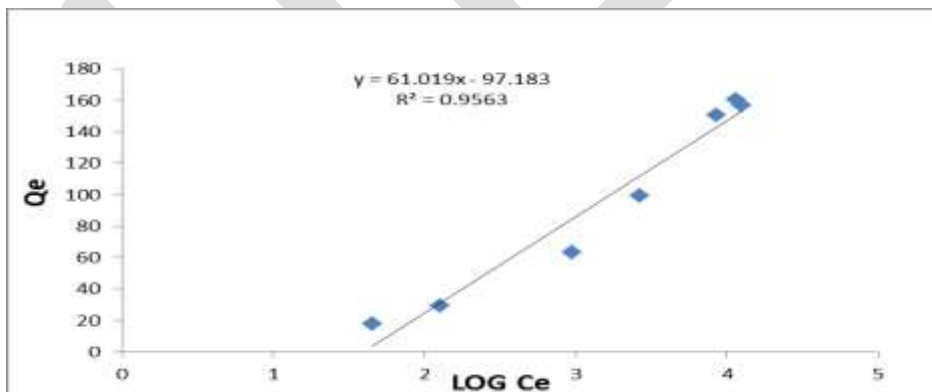


Fig. 3.8 Temkin isotherm for biosorption of Nickel

Table – 2 Isotherms constants

Isotherm	Parameters	At temp 303° K
Langmuir	q_m , mg/g	5.05816
	b , L/g	0.024247
	R^2	0.9985
Freundlich	N	0.4024
	K_f , mg/g	0.762
	R^2	0.9945
Temkin	AT , L/mg	0.09499
	bT	41.2845
	R^2	0.9692

3.9 Kinetics of biosorption

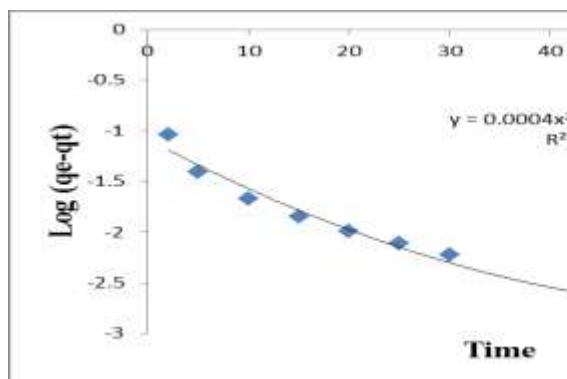


Fig.3.9 First order kinetics for biosorption of Nickel

Plot of $\log (q_e - q_t)$ versus t' gives a curve for first order kinetics, facilitating the computation of adsorption rate constant (K_{ad}).

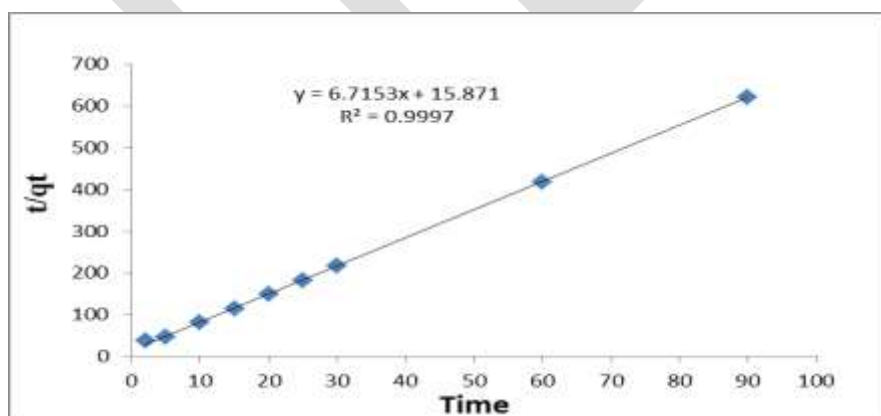


Fig.3.10 Second order Kinetics for biosorption of nickel

In the present study, the kinetics are investigated with 20 mL of aqueous solution ($C_0 = 20$ mg/L) at 303 °K with the interaction time intervals of 1 min to 180 min. Lagrager plots of $\log (q_e - q_t)$ versus agitation time (t) for biosorption of Nickel the biosorbent

size (74 μm) of *rice husk ash* in the interaction time intervals of 1 to 180 min are drawn in figs.3.9 & 3.10. It is second order kinetics as the line is best fit the t/q_t vs. t graph with R^2 value of 0.9984.

Table3 Equations and Rate constants

Order	Equation	Rate constant	R ²
Lagergren first order	$\log (q_e - q_t) = -0.0276 t - 1.31$	0.06356 Min ⁻¹	0.9753
Pseudo second order	$t/q_t = 6.5322t + 21.606$	1.97 g/(mg-min)	0.9984

3.11 Optimization using Box-Behnken Design:

The experiments conducted with different pH values ranging from 4-6. Different nickel concentrations of 15-25mg/L, different biosorbent dosage of 1 to 1.5 gram and different temperatures coupled to each other and varied simultaneously to cover the combination of parameters in BBD. The levels and ranges are chosen independent parameters are given in the Table-3., Table-4. is employed for the optimization of the parameters.

The regression equation for the optimization of medium constitutes: % biosorption of nickel (Y) is a function of pH (X₁), initial nickel concentration C_o (X₂), biosorbent dosage (X₃) and temperature T (X₄). Multiple regression analysis of the experimental data has resulted in the following equation for the biosorption of nickel:

$$Y = -332.126 + 66.68X_1 + 8.958 X_2 + 206.067X_3 + 2.923X_4 - 6.674 X_1^2 - 0.225X_2^2 - 82.487X_3^2 - 0.048X_4^2 \text{-----} \rightarrow (1)$$

The result of above regression model in the form of analysis of variance (ANOVA) for Eq. (1) is given in Table-5.6. The optimal set if condition for maximum percentage biosorption of nickel is pH =4.99, biosorption dosage (w) = 1.249 gram and initial nickel concentration of Co = 19.87428 mg/L calculated at these optimum condition is 96.46 %. Fig. 3.11 shows the comparison between the % biosorption obtained through experiments and that predicted. The experimental values are in good agreement with predicted values.

The correlation coefficient (R²) provides a measure of the models variability in the observed response values. The closer the R² value to 1, the stronger the model is and it predicts the response better. In the present study the value of the regression coefficient (R² =0.9998) indicates that 0.0002 % of the total variations are not satisfactorily explained by the model. The ANOVA Table can be used to test the statistical significance of the ratio of mean square due to regression and mean square due to residual error. From that table, it is evident that, the F-statistics value for entire model is higher. This large value implies that % removal can be adequately explained by the model equation. Generally P values lower than 0.05 indicates that the model is considered to be statistical insignificance at the 95% confidence level. The % biosorption prediction from the model is shown in Table-4. From Table-5, it is known that all the squared terms and the linear terms of all the variables are significant (P < 0.05)

Table-4

Levels of different process variables in coded and un-coded form for % biosorption of nickel using rice husk ash

Variable	Name	Ranges and levels		
		-1	0	1
X ₁	pH of aqueous solution	4	5	6

X_2	Initial concentration, C_0 , mg/L	15	20	25
X_3	Biosorbent dosage, W , grams	1	1.25	1.5
X_4	Temperature, T °K	293	303	313

Table-5 Results from BBD for Nickel biosorption by rice husk ash

Runs	pH(X_1)	Conc(X_2)	Dosage(X_3)	Temp(X_4)	pH	Conc	Dosage	Temp	% removal	Predicted
1	-1	-1	0	0	4	15	1.25	30	84.66	84.49
2	1	-1	0	0	6	15	1.25	30	84.78	84.37
3	-1	1	0	0	4	25	1.25	30	83.24	83.93
4	1	1	0	0	6	25	1.25	30	83.44	83.8
5	0	0	-1	-1	5	20	1	20	85.72	86.2
6	0	0	1	-1	5	20	1.5	20	86.24	86.12
7	0	0	-1	1	5	20	1	40	86.12	86.84
8	0	0	1	1	5	20	1.5	40	87.38	86.77
9	0	0	0	0	5	20	1.25	30	96.46	96.46
10	-1	0	0	-1	4	20	1.25	20	84.58	84.7
11	1	0	0	-1	6	20	1.25	20	84.76	84.58
12	-1	0	0	1	4	20	1.25	40	85.24	85.35
13	1	0	0	1	6	20	1.25	40	85.51	85.22
14	0	-1	-1	0	5	15	1	30	86.43	85.99
15	0	1	-1	0	5	25	1	30	85.49	85.42
16	0	-1	1	0	5	15	1.5	30	85.16	85.91
17	0	1	1	0	5	25	1.5	30	85.82	85.34
18	0	0	0	0	5	20	1.25	30	96.46	96.46
19	-1	0	-1	0	4	20	1	30	85.68	84.72
20	1	0	-1	0	6	20	1	30	84.36	84.6
21	-1	0	1	0	4	20	1.5	30	84.47	84.65
22	1	0	1	0	6	20	1.5	30	84.28	84.53

23	0	-1	0	-1	5	15	1.25	20	85.91	85.96
24	0	1	0	-1	5	25	1.25	20	85.78	85.4
25	0	-1	0	1	5	15	1.25	40	86.42	86.61
26	0	1	0	1	5	25	1.25	40	86.19	86.04
27	0	0	0	0	5	20	1.25	30	96.46	96.46
28	0	0	0	0	5	20	1.25	30	96.46	96.46
29	0	0	0	0	5	20	1.25	30	96.46	96.46
30	0	0	0	0	5	20	1.25	30	96.46	96.46

Table-6

ANOVA of Nickel biosorption for entire quadratic model

Source of variation	SS	df	Mean square(MS)	F value	P > F
Model	613.7276	8	152.7468	739.133	0
Error	4.3398	21	0.2067		
Total SS	618.0674	29			

Df- degree of freedom, SS- sum of squares, F- factor F and P- probability.

$R^2=0.9896$; R^2 (adj):0.9799

Table-7 Estimated regression coefficients for the nickel biosorption onto rice husk ash

Terms	Regression coefficient	Standard error of coefficient	t(21)	P
Mean/Interc.	-332.126	7.997247	-41.5300	0
(1)pH (L)	66.680	1.740967	38.3006	0
pH (Q)	-6.674	0.173601	-38.44	0

			54	0
(2)Concentration (L)	8.958	0.278999	32.10 76	0 . 0 0
Concentration (Q)	-0.225	0.006944	- 32.45 46	0 . 0 0
(3)Dosage (L)	206.067	6.963867	29.59 08	0 . 0 0
Dosage (Q)	-82.487	2.777622	- 29.69 69	0 . 0 0
(4)Temperature (L)	2.923	0.104984	27.84 23	0 . 0 0
Temperature (Q)	-0.048	0.001736	- 27.75 28	0 . 0 0

Insignificant ($P \geq 0.05$)

The model is reduced to the following form by excluding undistinguished terms in eq.5.2

$$Y = -332.126 + 66.68X_1 + 8.958 X_2 + 206.067X_3 + 2.923X_4 - 6.674 X_1^2 - 0.225X_2^2 - 82.487X_3^2 - 0.048X_4^2$$

→(1)

3.11 Interpretation of residual graphs:

Normal probability plot (NPP) is a graphical technique used for analyzing whether or not a data set is normally distributed to greater extent. The difference between the observed and predicted values from the regression is termed as residual. fig. 3.12 exhibits normal probability plot for the present data. It is evident that the experimental data are reasonably aligned implying normal distribution.

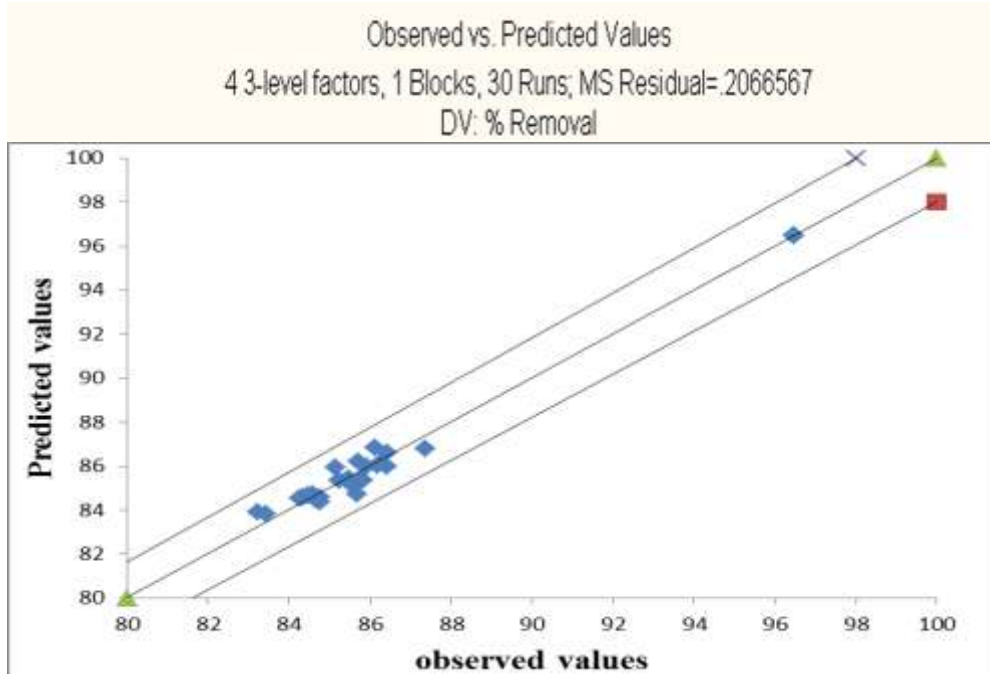


Fig. 3.12 Normal probability plot for % biosorption of nickel

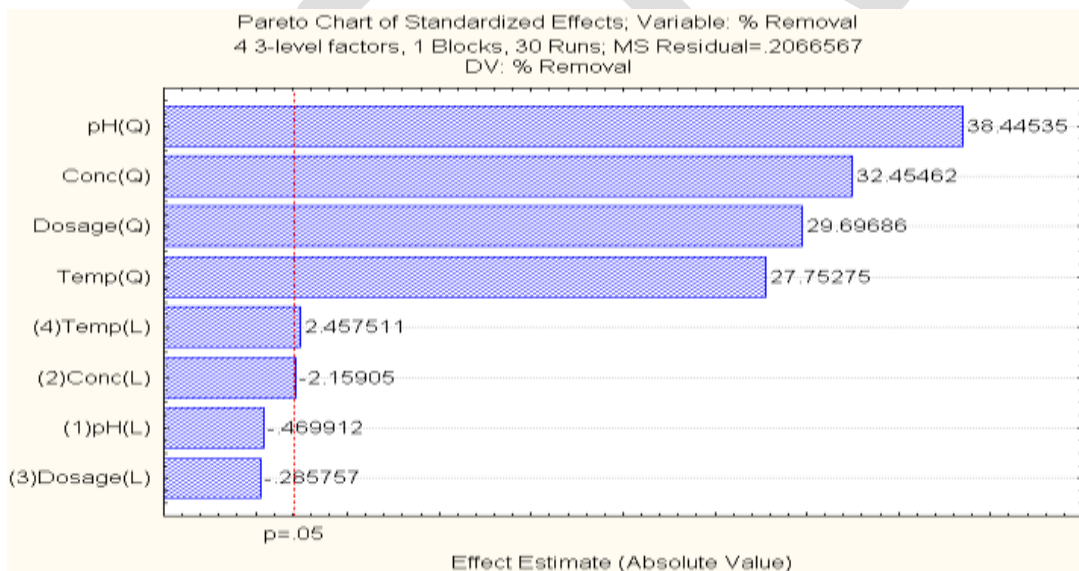


Fig. 3.13 Pareto Chart for % biosorption of nickel

Pareto chart

From the Pareto chart can be explained in such a way that $P > 0.05$ are significant and is presented in Fig. 3.13. The red line in the x-axis is $P=0.05$.

Interaction effects of biosorption variables:

Three dimensional view of response surface contour plots [Fig. 3.14(a)& 3.14(b)] exhibit, % biosorption of the nickel using *rice husk ash* for different combinations of dependent variables. All the plots are delineated as a function of two factors at a time, imposing other factors fixed at zero level. It is evident from response surface contour plots that the % biosorption is minimal at low and high levels of the variables. This behavior conforms that there is a presence of optimum for the input variables in order to maximize % biosorption. The role played by all the variables is so vital in % biosorption of nickel and seen clearly from the plots. The predicted optimal set of conditions for maximum % biosorption of nickel is

- Biosorbent dosage = 1.24909 gram
- Initial nickel ion concentration = 19.87428 mg/L
- pH of aqueous solution = 4.99538
- % biosorption of nickel = 96.42

The experimental optimum values are compared with those predicted by BBD in table-5. The experimental values are in close agreement with those from and BBD

Table-7 Comparison between optimum values from BBD and experimentation

Critical values; Variable: % Removal

Predicted value at solution: 96.46917

Table 8

	Observed	Critical	Observed
pH	4.00000	4.99538	6.00000
Concentration	15.00000	19.87428	25.00000
Dosage	1.00000	1.24909	1.50000
Temp	20.00000	30.33469	40.00000

Surface contour plots.

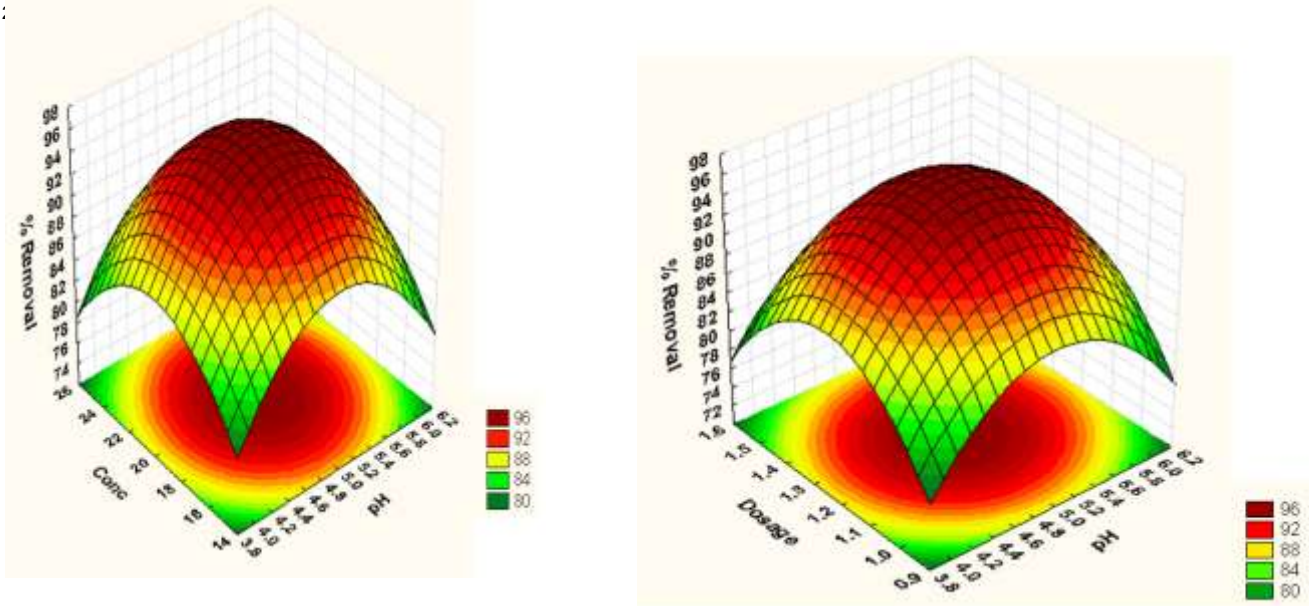


Fig. 3.14(a) Surface contour plot for the effects of pH and initial concentration, Dosage of nickel on % Removal

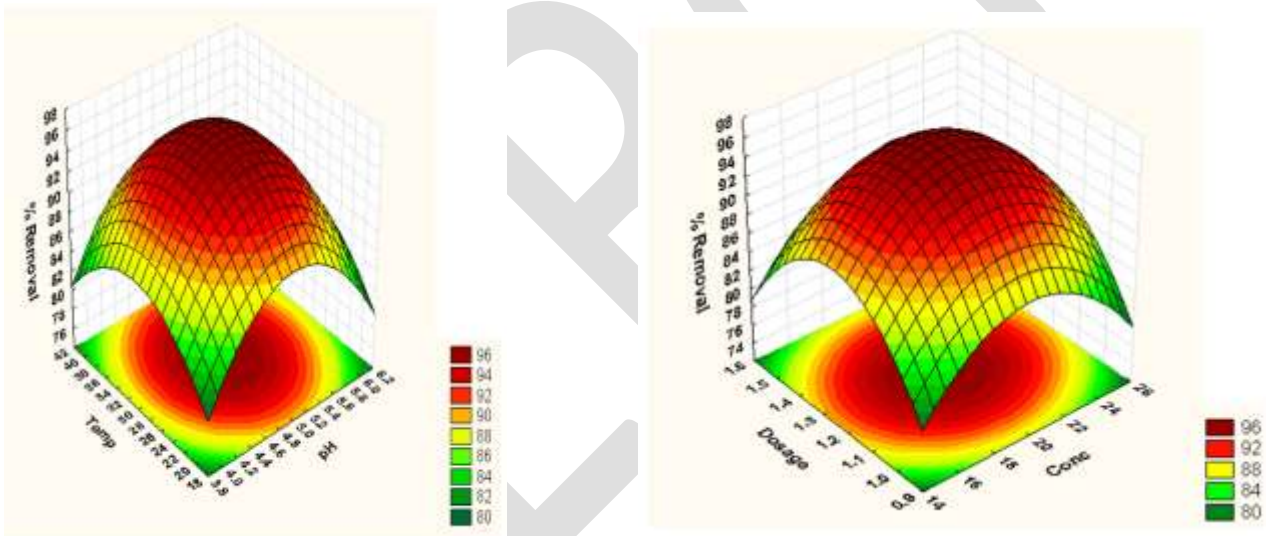


Fig. 3.14(b) Surface contour plot for the effects of Temperature and pH, dosage and concentration, of nickel on % Removal.

CONCLUSIONS

Rice Husk Ash is used as biosorbent for the removal nickel ions from aqueous solution. In the biosorption of nickel, effect of pH, biosorbent dosage, metal ion concentration, contact time, biosorption isotherm and kinetics studies were carried out. The metal ion concentrations were analyzed using Atomic absorption spectroscopy.

1. The equilibrium agitation time for nickel biosorption is 90 min.

2. The % biosorption of nickel is increased from 28.19 % (2.27 mg/g) to 52.28 % (4.39 mg/g) as pH is increased from 2 to 5 and decreased beyond the pH value of 6. % biosorption is decreased from pH 5 to 6 reaching 47.01 % (4.28 mg/g) from 52.25 % (4.39 mg/g). Thus indicating the optimum pH is 5 for the biosorbent rice husk ash.
3. With an increase in the initial concentration of nickel in the aqueous solution, the percentage biosorption of nickel from the aqueous solution is decreased.
4. The percentage biosorption of nickel is increased significantly with increase in biosorbent dosage up to 1.25 grams and there by remained constant. The biosorption of nickel increased from 34.23 % (0.41 mg/g) to 92.03 % (0.18 mg/g) with an increase in biosorbent dosage from 0.5 gram to 1.25gram.
5. The percentage biosorption of nickel is increased significantly with increase in the temperature.
6. The kinetic studies show that the biosorption of nickel is better described by pseudo second order kinetics. ($K_2 = 1.97$), $R^2 = 0.9984$

The present study involves the use of statistical design to optimize process conditions for maximal biosorption of nickel from aqueous solution using BBD involving RSM

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