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Analysis of thermometric parameters of Adsorption of Nickel (II) Ions onto a Low-Cost GSH adsorbent

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Abstract
Gram seed husk (GSH) reproduced by pulses forming industries has been demonstrated to be an effective adsorbent for the removal of metal ions like Ni (II) from aqueous solution. Batch adsorption techniques were conducted to study the effects of contact time, adsorbent dose , concentration of Ni (II) metal ion solution, and effect of pH of Ni (II) metal ion solution. Pseudo-first and pseudo- second order kinetics were tested to examine the adsorption mechanism. The adsorption kinetic data were described very well by the pseudo-second-order kinetics. Equilibrium data were more effectively fitted to the both Langmuir and Freundlich model. Adsorption equilibrium was obtained at 60 min. for 5 to 20 mg/L of Ni (II) concentrations. Adsorption of Ni(II) metal ions was fundamentally dependent on pH of solution, and the optimum adsorption of Ni (II) ion was observed at pH 7. The thermodynamic parameters like (ΔG°), (ΔH) and (ΔS) indicates that adsorption was spontaneous and exothermic in nature. From experimental results, conclude that gram seed husk (GSH) is recommended as least expensive and easily available bio adsorbent to removal of harmful heavy metal ions from industrial wastewater.
Keywords: *Adsorption isotherms, Gram seed husk, Kinetics*

I. INTRODUCTION

Water is essential for survival. However today, about 200 million people in India don't get pure drinking water due to contamination of water. Besides in the fast development of the total world population, industrialization, unplanned urbanization, farming activities, as well as the more utilization of chemicals has contributed to environmental pollution1. Organic and inorganic wastes produced by human exercises have brought about high amount of contaminated water which threatens human health as well as other living beings. Industries such as textile, mining, explosive manufacturing, ceramic, metal plating, glass, leather, paint and steel etc., are some of the sources for heavy metal effluents2. Exorbitant ingestion of hazardous metal ions by people can cause accumulative poisoning, cancer, nervous system damage and eventually demise3. Nickel is 24th most abundant metal in earth's crust. Its ores are fundamentally of two types' viz. sulfides and oxides4. Nickel metal ions are non- biodegradable and unsafe. It might cause dermatitis and allergic sensitization5. Nickel and nickel compounds are also well recognized as carcinogens6, therefore Ni(II) metal ion is more essential to expel from water. Various techniques which have been used for the removal of heavy metal ion from industrial effluents include ion exchange, reverse osmosis, ultra- filtration, chemical precipitation, electrode dialysis, membrane filtration, coagulation, floatation and adsorption. Out of these few procedures utilized for metal ion removal, the most experimental technique was found to be the use of adsorption to adsorb the metal ion from waste water7. Numerous horticultural by-products have been utilized as adsorbents for the removal of various dangerous metal ions as well as organic compounds. The most advantage of adsorption techniques for water pollution control are low investment in terms of cost, simple design, easy and cheap operation and absence of toxic harmful substances. Many low cost adsorbents have been used for removal of toxic metal ion such from wastewater Grape fruit peel8, rice husk9, jackfruit leaf powder10, ginger waste11, black gram seed husk12 etc. In the present work a low-cost material like gram seed (GSH) husk powder was used as an adsorbent for the removal of Ni(II) metal ion from aqueous solutions. The main cause of the research is to investigate the adsorption efficiency of gram seed husk powder (GSH) for Ni(II) metal ions. Gram seed husk is a low cost adsorbent, easily available and biodegradable. The effects of initial concentration of Ni(II) metal ion solution, effect of adsorbent dose, effect of contact time and effect of temperature on Ni(II) metal ion adsorption have been investigated. Adsorption kinetics, adsorption isotherms and thermodynamics were also evaluated and reported.

II. MATERIALS AND METHOD

Preparation of Adsorbent: The develop and crisp gram seeds were purchased from neighborhood market and washed thoroughly by utilizing distilled water to clean them from dirt. After that, seeds are keeping into distilled water up to 24 hours. Then husk was isolated from the pulses and washed with distilled water, after washing

husk was dried in shade region. After complete drying, the husk was ground by using processor. The homogeneous powder was obtained by passing through mesh of desired particles size (micron) of gram seed husk (GSH). The fine powder of GSH adsorbent was stored in an air tight glass bottle prepared for further experimental process.

Preparation of Ni (II) Solution: Ammonium nickel sulphate [$NiSO_4$ $(NH_4)2.6H_2O$] was purchased from Sd. Fine Chemicals Pvt. Ltd. Mumbai (India). Stock solution of nickel metal ion (100 ppm) was prepared by weighing and dissolving 0.675 g. of ammonium nickel sulfate [$NiSO_4$ (NH_4)2.6H2O] in 1000 ml volumetric flask and fill it up to the mark with double distilled water. All the necessary solutions were prepared by diluting the stock solution with double distilled water. By using UV-Visible single beam Spectrophotometer, the concentration of Ni (II) metal ion was determined. The absorbance was determined at 366 nm against blank was determined Ni (II) ions. Batch adsorption technique was performed at room temperature.

Batch Experiments: Batch equilibrium adsorption techniques were conducted by adding known amount of GSH powder to Erlenmeyer flasks containing 50 mL of different concentration of Ni (II) ions solution at pH 7. The mixture was shaken for various intervals of time. The solutions were then filtered through filter paper and the absorbance of Ni (II) metal ions was determined by spectrophotometer. The amount of Ni (II) ions adsorbed (mg/g) was calculated using the formulae reported by Vanderborght and Van Griekenm and then the kinetic adsorption parameters were determined.

$$q = \frac{v\,(c_0 - c_t)}{m} \qquad (1)$$

Where, q is the amount of Ni (II) ion adsorbed from the solution (mg/g), C_o is the concentration of Ni (II) metal ion solution before adsorption (mg/L), and Ct is concentration after adsorption. V is the volume of Ni (II) ion solution mg/L and M is the weight of the GSH (g).

The percentage adsorption of Ni (II) ions was calculated by following equation;

$$\text{Percentage adsorption} = \frac{(c_0 - c_\theta)}{c_0} \times 100 \quad (2)$$

Where, c_0 and c_θ are the initial and equilibrium concentrations respectively.

III. Results And Discussion

Effect of Contact Time and Initial Concentration: The relationship between Ni (II) metal ion adsorption and contact time onto GSH at various initial concentration of Ni (II) ion desired pH, and room temperature is shown in "Fig;1". It is clearly seen from the figure that the amount of Ni (II) metal ions adsorption increased with increase in contact time. This is result of the increase in initial concentration of metal ion prompts to an increase in mass gradient between the Ni.

(II) solution and GSH adsorbent and thus acts as a driving force for the transfer of Ni(II) ions from the bulk solution to the adsorbent surface. As increase the initial concentration of Ni (II) ions, increase the percentage adsorption capacity due to higher probabilities of collusion between Ni (II) ions and GSH adsorbent. Similar observation was reported in literature13.

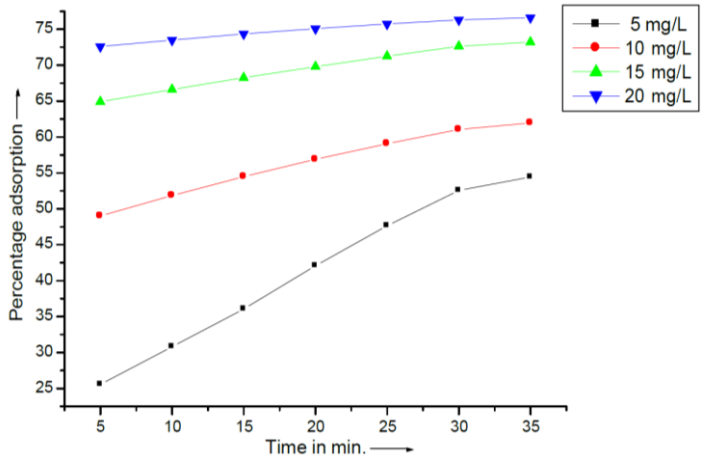

Fig.1:Effect of contact time and initial concentration of Ni(II)ion on adsorption of GSH adsorbent. [Adsorbent dose=0.5 gm., Volume=50 ml., Temp.=304.5 K, pH=5.7]

Effect of Adsorbent Dose: It is a significant parameter that strongly influences the adsorption technique by influencing adsorption capacity of the adsorbent. The experiments were carried out following general procedure for adsorption studies at the various contact time for each adsorbent. The dependence of Ni (II) ions adsorption on adsorbent dose was studied by varying the amount of adsorbents from 0.25 to 1.0 g. per 50 mL volume by

keeping the other parameters (pH, contact time) constant. The plots of percentage adsorption of Ni (II) ions against contact time of various dose of adsorbent. The results are shown in "Fig. 2".

It was seen that percentage adsorption capacity of Ni (II) ions generally increased with increasing GSH dose. This was expected due to the fact that the higher GSH dose in the solution, the availability of exchangeable sites for the metal ions is greater. At 20 mg/L concentration of Ni (II) ion, the maximum Ni (II) ions adsorption capacity was about 94.48% at the dosage of 1.0 g. per 50 mL at time of equilibrium. From observation after a certain dose of GSH, the maximum adsorption sets and thus the amount of NI (II) ions bound to the GSH and the amount of free ions remains constant even with further addition of GSH dose.

Effect of pH of Metal Ions: In order to establish the effect of pH on the adsorption of Ni (II) ions onto GSH adsorbent, the batch adsorption studies at different pH value was carried out in the range of 2 - 11. The plot of percentage adsorption of Ni (II) ions against various pH of solution, the results are shown in "Fig.3"

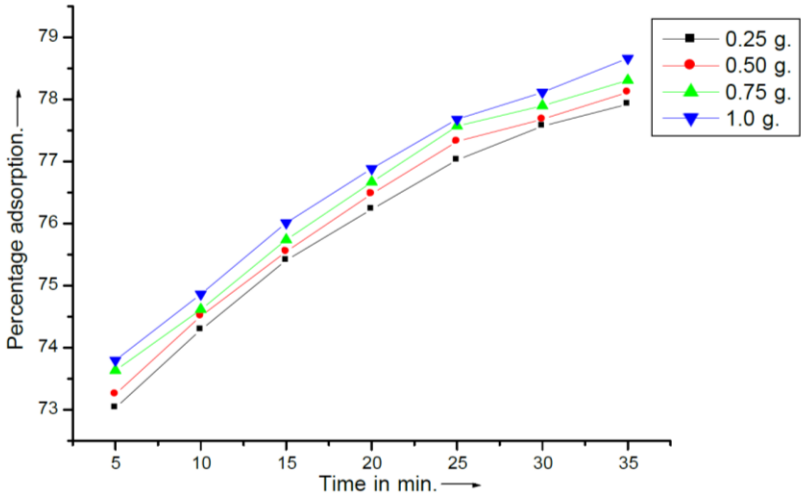

Fig.2:Effect of adsorbent dose of GSH on adsorption of Ni(II) ion. [Initial conc.= 20 mg/L,Volume of adsorbate=50 ml,Temp.=304.5K, pH=5.7]

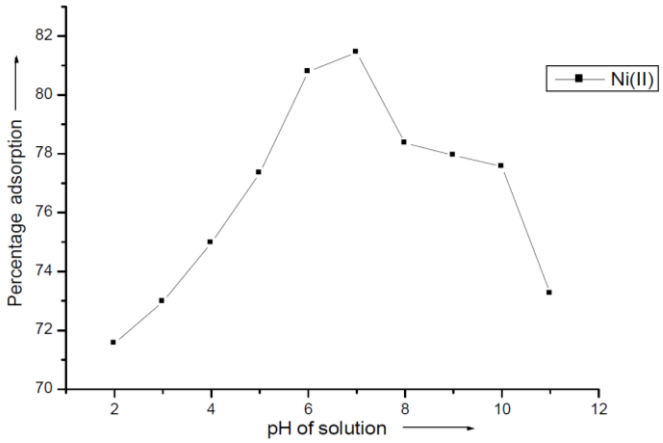

Fig.3:Effect of pH of GSH on adsorption of Ni (II). [Initial conc.= 20 mg/L,Volume of adsorbate=25 ml, Adsorbent dose=0.5 gm, Temp.=304.5K.]

It reveals that the percentage adsorption of Ni (II) ions increased with increasing pH due to lesser number of H+ and greater number of surface ligands with negative charges. The best pH for removal of Ni(II) ion is fixed as 7.0 for all adsorbents. The neutral pH is found to be favourable14.

Effect of Zero Point pH: The point of zero charge of adsorbent is the pH value at which the concentrations of acid and base species are equal15. After the batch equilibrium method, the pHpzc values of GSH determinate were found to 6.2, indicating that the adsorbent is slightly acidic. The difference in pHpzc values could be attributed to the structural change during the modification process, the results are shown in "Fig. 4."

At low pH, metal cations and protons compete for binding sites on a GSH surface which results in lower adsorption of Ni (II) metal ions. The overall surface charge on GSH became negative and at higher pH, lower

adsorption is attributed due to reduced solubility of the ions and their precipitation as hydroxides anions forming nickel hydroxide precipitate.

Effect of Addition of Salt (KCl): The ionic strength on the adsorption of Ni (II) ions was studied by adding different amount of KCl in the range of 0.25 to 1.0 g. The plots of percentage adsorption of Ni (II) ions against contact time with different amount of KCl, the results are shown in "Fig. 5".

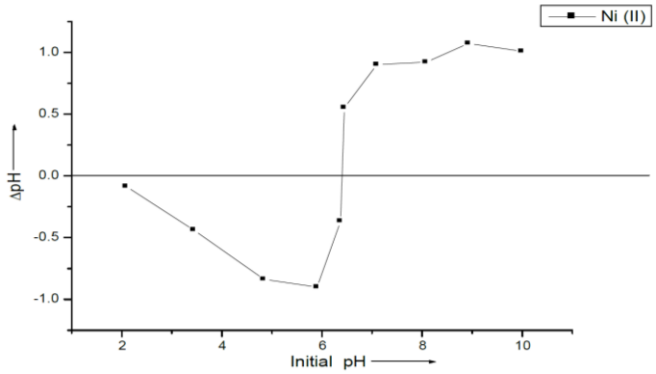

Fig:4. Effect of zero point pH on adsorption of Ni (II) with GSH adsorbents.

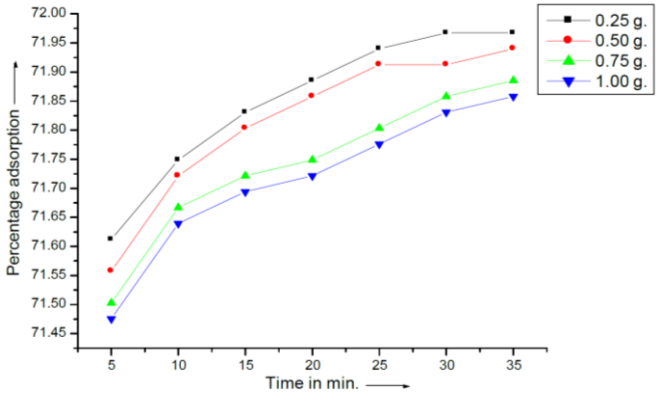

Fig.5:Effect of addition of salt (KCl) on adsorption of Ni(II) ion on GSH adsorbent. [Initial conc.= 20 mg/L,Volume of adsorbate=50 ml,Temp.=304.5K, pH=5.7]

The "Fig.5" illustrates the various shapes of curves obtained when the amount of KCl in the mixture was increased, the percentage adsorption of Ni (II) ions was decreased, it may be due to competition of K+ to change the ionic strength of Ni (II)solution with positive charges for the same binding sites on the adsorbent surface. Additionally, ionic atmosphere may be progressively formed around nickel molecules with increased KCl concentration. When the electrostatic attraction is repulsive, the increase in the ionic force or strength increases the adsorption capacity16.

Effect of Temperature: Temperature has a pronounced effect on the adsorption capacity of Ni (II) ions on GSH adsorbent. The temperature effect was investigated for temperature ranging from 306.5 to 326.5 K. The results are shown in "Fig.6".

In the present work adsorption of Ni (II) ions decreased from 80.96 % to 75.66 %, by increasing temperature from 306.4K to 326.5K. The percentage adsorption of Ni(II) ions decreases with increase in temperature. Since adsorption is an exothermic process. Thus the removal of Ni (II) ions are leading to a decrease in the residual forces on the surface of the GSH adsorbent and hence causing a decrease in the surface energy of the adsorbent17.

The Gibb's free energy (ΔG^c), enthalpy (ΔH^c), and entropy (ΔS^c) changes for the adsorption were determined by using equation.

$$\Delta G^o = \Delta H^o - T\Delta S^c \qquad (3)$$

$$\text{Log}\ \frac{qe^m}{Ce} = \frac{\Delta S^c}{2.303} + \frac{-\Delta H^o}{2.303RT} \qquad (4)$$

For the adsorbent concentration is unity (m= 1.0 g) equation (4) becomes

$$\text{Log}\ \frac{qe}{Ce} = \frac{\Delta S^c}{2.303} + \frac{-\Delta H^o}{2.303RT} \qquad (5)$$

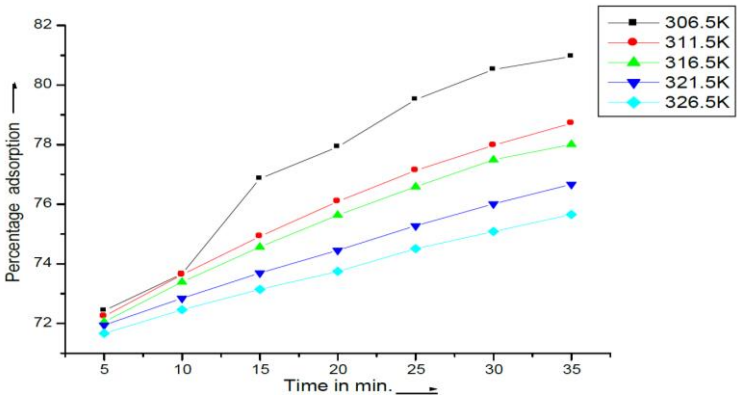

Fig. 6. Effect of temperature on percentage adsorption of Ni (II) on GSH adsorbent. [Initial conc.= 20 mg/L,Adsorbent dose = 0.5 gm.,pH=5.7]

q_e is the amount of MB dye adsorbed perunite mass of BGSH (mg/g), Ce is the equilibrium concentration (mg/L) and T is the temperature in k. q_e/C_e is called adsorption affinity. The value of Gibb's free energy (ΔG^c) has been calculated by knowing the enthalpy of adsorption (ΔH^c) and entropy of adsorption (ΔG^c) and (ΔH^c) was obtained from a plot of log q_e/C_e versus 1/T from equation (4) and (5). Once these two parameters were obtained, (ΔG^c) is determined from equation (3) Thermodynamic parameters are given in "Table: 1".
The values obtained in this study for the Ni (II) ions are < -10 KJ /mole, it indicates that physical adsorption was the predominant mechanism in the adsorption process. The Gibb's free energy indicates the degree of spontaneity of the adsorption process, where more negative value reflects a more energetically favourable adsorption process. The negative value of "Table.1. indicates that the adsorption is favourable and spontaneous18. The negative values of and suggests that the decreased disorder and randomness at the Ni (II) metal ion solution interface with exothermic adsorption.

ANALYSIS OF ADSORPTION ISOTHERM:

Langmuir Isotherm: Langmuir adsorption isotherm describes quantitatively the formation of a monolayer adsorbate on the outer surface of the adsorbent and after that no further adsorption takes place. The Langmuir adsorption isotherm is valid for monolayer adsorption onto the surface containing a finite number of identical sites. The linear form of Langmuir adsorption isotherm equation is given by,

$$\frac{1}{q_e} = \left(\frac{1}{Q_o}\right) + \frac{1}{bQ_oC_e} \qquad (6)$$

Table.1: Thermodynamic parameter values of GSH adsorbent with Ni (II) ion solution at different temperatures

Temperature (K)	ΔG^o KJ/mole	ΔH KJ/mole	ΔS J/mole
304.2	- 4.170	- 17.998	- 45.455
309.2	-3.943		
314.2	-3.716		
319.2	-3.489		
324.2	-3.262		

Where, C_e (mg/L) is the equilibrium concentration of the adsorbate, q_e (mg/g) is the amount of adsorbate adsorbed per unit mass of adsorbent, at equilibrium, Q_o(mg/g) and b(L/m) are Langmuir constants are related to maximum monolayer adsorption capacity and energy of adsorption respectively. The values of and b are calculated from the slope and intercept of plot of $1/q_e$ against $1/C_e$ respectively. The essential features of the Langmuir adsorption isotherm may be expressed in terms of equilibrium parameter R_L . Which is a dimensionless constant called as separation factor?

$$R_L = \frac{1}{1+bC_o} \qquad (7)$$

Where, C_o is initial concentration in ppm and b is Langmuir constant related to the energy of adsorption. R_L Value indicates the adsorption nature to be either unfavourable if $R_L > 1$, linear if $R_L = 1$, favourable if $0 < R_L < 1$ and irreversible if, $R_L = 0$.

Freundlich Isotherm: Freundlich adsorption isotherm for non-ideal adsorption on heterogeneous surfaces as well as multilayer adsorption expressed as

$$\frac{x}{m} = K_f\, C_e^{1/n} \qquad (8)$$

Where, x is the quantity adsorbed, m is the mass of the adsorbent, C_e is the equilibrium concentration of adsorbate (mg/L), The constants K_f and n can be obtained by taking log on both sides of equation "(8)" as follows,

$$\text{Log}\,\frac{x}{m} = \frac{1}{n}\log C_e + \log K_f \qquad (9)$$

The constant K_f is an approximate indicator of adsorption capacity, while 1/n is a function of the strength of adsorption in the adsorption process19. When n= 1 then the partitions between the two phases are independent of the concentration. If value of 1/n is below one, it indicates a normal adsorption, on the other hand 1/n being above one indicates co-operative adsorption20. A plot of log x/m against K_f gives a straight line with an intercept log C_e on the ordinate axis. The value of n and K_f can be obtained from the slope and the intercept of the linear plot. The Isotherm parameters are given in "Table. 2"

Table.2: Isotherm parameter values of the adsorption of Ni (II) ions on GSH adsorbent.

Langmuir isotherm				Freundlich isotherm		
Q_0 (mg/g)	b*10^{-3} (L/mg)	R_L	R^2	K_f (mg/L)	n	R^2
851.49	0.009	0.99	0.99	17.41	1.14	0.99

The R_L value was found to be between 0 and 1 for Ni (II) ions studies, it confirmed that the on-going adsorption of Ni (II) ions is favourable. The data reveal that the both Langmuir and Freundlich model yields better fit. The value of n is greater than unity, (1< n <10), that means favourable adsorption21. If value of > 1 indicates the adsorption is favoured and new adsorption sites are generated 22-25. The value of n was found to be in between 1 to 10, indicates that adsorption is favourable. The value of n presented in "Table. 2".

Morris-Weber Relationship: According to this model, the plot of amount of substance adsorbed per unit mass of adsorbent against square root of time gives a curve, which can be divided in to three segments,

i. The initial portion representing rapid internal diffusion or boundary layer diffusion and surface adsorption.

ii. The linear portion including a gradual adsorption state due to intra particle diffusion, and

iii. A plateau to equilibrium where the intra particle diffusion starts to decrease due to the low concentration in solution as well as small number of available adsorption sites.

$$q_t = K_{id}\, t^{1/2} + C \qquad (10)$$

Where, q_t is amount of Ni (II) ions adsorbed per unit mass of GSH; is the intra particle diffusion coefficients mg/(gm.min½). The plot of q_t against $t^{1/2}$ does not pass through origin. This indicates that intra particle diffusion is not the rate limiting step. Straight line with slope K_{id} and intercept is zero, if the adsorption is limited by the internal diffusion process are shown in "Fig.7".

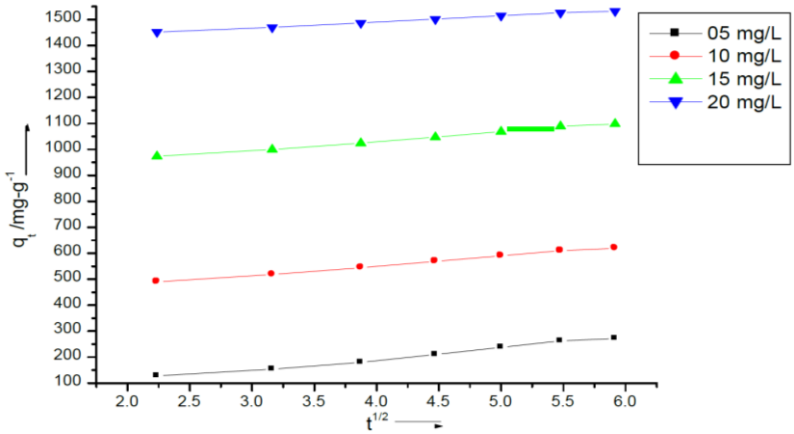

Fig 7. : Plot of q against t1/2 in internal diffusion model with Initial concentration of Ni (II) metal ion.

ANALYSIS OF ADSORPTION KINETICS

The kinetic study of adsorption was carried out to calculate the rate adsorption. Mainly two kinetic models can be suggested for an adsorption including the Lagergren pseudo-first order and pseudo- second order kinetics.

Pseudo-first order kinetics is present to describe the rate of adsorption process in liquid-solid phase. The Lagergren pseudo-first order kinetic rate equation is given as,

$$\frac{dq}{dt} = K_1 (q_e - q_t) \qquad (11)$$

After definite integration by applications of the conditions t= 0 to t=t and to Equation "(11)" becomes,

$$\text{Log}\,(q_e - q_t) = \log q_e - \frac{K_1}{2.303}t \qquad (12)$$

Where, q_e (mg/g) is the amount of adsorption of Ni (II) ion at equilibrium, q_t (mg/g) denotes the amount of adsorption of Ni (II) ion at time t (min.) and K_1 (min^{-1}) is the pseudo-first order rate constant. Based on experimental results, linear graph was plotted between Log (q_e - q_t) versus t, to calculate K1, q_e and R^2. It is shown in "Fig.8" (a).

The pseudo-second order kinetic equation can be written as

$$\frac{dq}{dt} = K_2 (q_e - q_t)^2 \qquad (13)$$

Where, K_2 ($g.mg^{-1}min^{-1}$) is the pseudo- second order rate constant. Integrating equation (10) for the boundary conditions t=0 to t=t and q= 0 to q = q_e gives the linear form of equation is

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e}t \qquad (14)$$

K_2 and q_e can be obtained from the intercept and slope of plotting t/q_t against t . It is shown in "Fig.8" (b).

Adsorption rates were calculated from the slope and intercept the calculated values of k_1 , k_2 and q_e values are given in "Table.3".

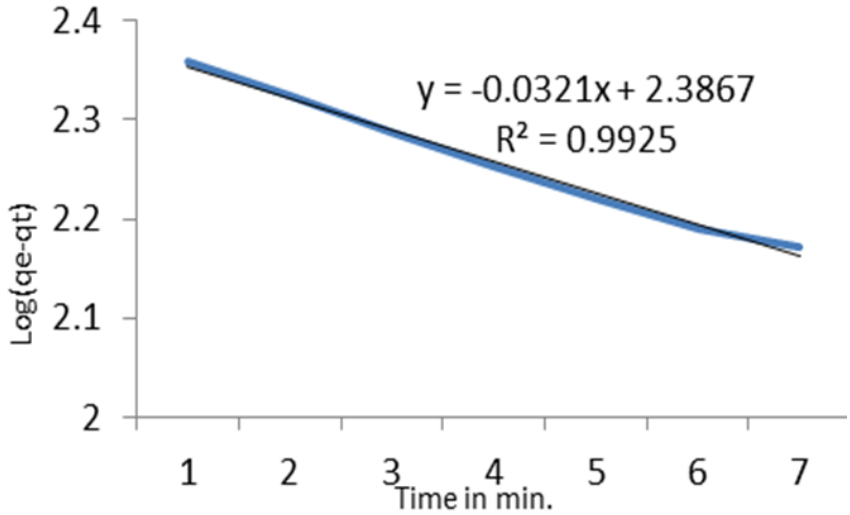

Figure 8 (a): Pseudo First order Kinetics.

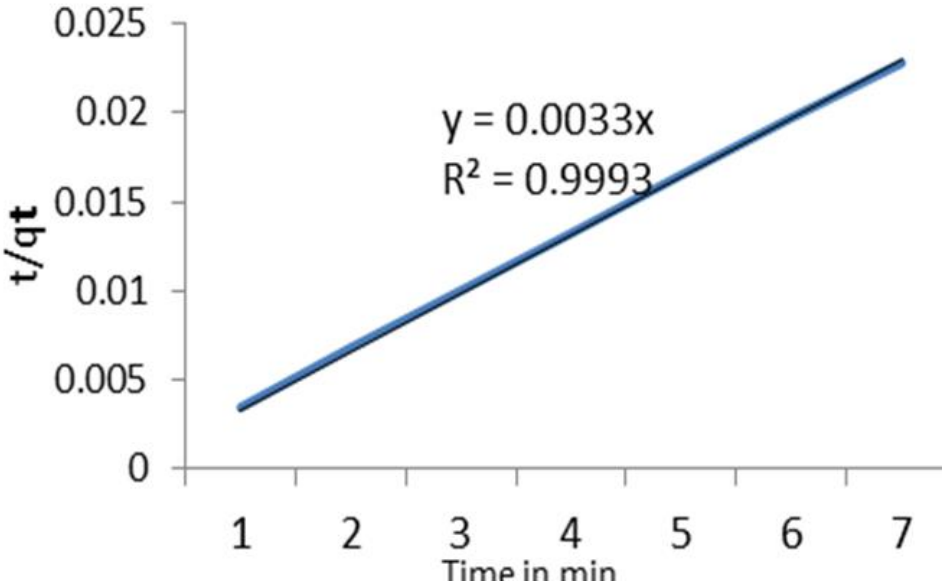

Figure 8 (b): Pseudo Second order Kinetics.

Table. 3: Pseudo first order and Pseudo second order kinetic models for the adsorption of Ni (II) ions on Gram seed husk (GSH) adsorbent.

Initial Conc. of Ni (II) ions (mg/L)	q_e. *expt*.(mg/g)	Pseudo first order Kinetic model			Pseudo second order kinetic model		
		q_e. *cal*.(mg/g)	k_1 (min^{-1})	R^2	q_e. *cal*.(mg/g)	k_2 (g/mg.min)	R^2
20	349.18	243.78	0.0739	0.992	303.03	$1.09*10^{-5}$	0.999

The value of R^2 with first order kinetics was 0.992, while for second order is 0.999 for GSH adsorbent. It is clear that the adsorption of Ni (II) ions on GSH adsorbent was better represented by pseudo second order kinetics. This suggests that the adsorption system belongs to the second order kinetic model.

IV. CONCLUSION

1. In adsorption study the metal, namely, Nickel (II) was selected for removal from aqueous solutions using adsorption technique.
2. Batch experimental techniques were conducted to study the impacts of contact time, initial metal ion concentrations, pH, and adsorbent dose.
3. The adsorption increase with increase of initial Ni (II) metal ion concentration.
4. Maximum adsorption was recorded at pH 7.0 for Ni (II) ions.
5. The negative value of ΔG confirms that the feasibility and spontaneous nature of the adsorption.
6. The negative value of ΔS and ΔH suggests that the decreased disorder and randomness at the solid solution interface with exothermic adsorption.
7. The adsorbent GSH selected for the present study proved to be good adsorbent which was evident with the adsorption data obeying the Langmuir and Freundlich isotherms.
8. GSH adsorbent was better represented by pseudo second order kinetics. This indicates that the adsorption system belongs to the second order kinetic model.
9. The investigation showed that GSH adsorbent as agricultural waste is abundant, cheap, readily available and environment-friendly effective adsorbent, which could be used as potential aqueous removal of Nickel(II) ions from aqueous solution and polluted water.
10. To improve profit in industries, they can be adopted such low cost adsorbents.

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