

# Adsorption Studies Of Lead (II) By Using Modified Agricultural Byproducts

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## I. INTRODUCTION

Excessive release of heavy metals into the environment due to industrialization and urbanization has passed a great problem worldwide, unlike organic pollutant, the majority of which are susceptible to biological degradation heavy metal ions do not degrade into harmless and products<sup>1</sup>Awareness encouragement of pollutant toxicity has forced industries and municipal authorities to treat wastewater before discharging to the natural water bodies Therefore, to remove toxic heavy metal from contaminated wastewater is one of the most important environmental and economic issues today. Lead is one of the most toxic heavy metal that is attracting wide attention of environmentalists. The sources of lead release into the environment by waste streams are acid metal plating, fishing, ammunition, battery manufacturing, tetraethyl lead manufacturing, glass industries, printing, painting, dyeing and other industries. In human, lead poisoning causes problems to the kidney, nervous system, reproductive system, liver and brain. Severe exposure to lead leads to sterility, abortion, stillbirths and neo natal deaths<sup>2</sup>Lead is known to cause mental retardations, it interferes with normal cellular metabolism and it reduce hemoglobin production which is necessary for oxygen transport<sup>3</sup>There are number of various method used to minimize for removal of metal ions from aqueous solutions, such as reduction, ion exchange, electro dialysis, electrochemical precipitation, evaporation, solvent extraction, reverse osmosis, adsorption & electro flotation<sup>4</sup> etcLead as Pb (II) is released into the environment from various industrial processes: industries engaged in lead acid batteries, pulp and paper, petrochemicals, refineries, printing, pigments, photographic materials, explosive manufacturing, ceramics, glass, paint, oil metal, phosphate fertilizer, electronics, wood production and also combustion of fossil fuel, forest fires, mining activity, automobile emissions, sewage wastewater, sea spray and many more<sup>5</sup>. Hashem<sup>6</sup> studied the sorption of Pb (II) using okra wastes, Singh et al.<sup>7</sup> used maize bran in the adsorption of lead using maize bran, while El-Ashtoukhy et al.<sup>8</sup> employed pomegranate peel as a adsorbent in the removal of lead (II) and copper (II) from aqueous solution. Yoshita et al.<sup>9</sup> carried out the study on the removal of lead by spent tea leaf residue after instant tea extraction.

Imamoglu and Tekir<sup>10</sup> have studied removal of copper (II) and lead (II) ions from aqueous solution by adsorption on activated carbon from a new precursor hazelnut husks. The thermodynamic study on the adsorption of Pb (II) and Zn (II) from aqueous solution by human hair was done by Ekop and Eddy<sup>11</sup>. Adie et al.<sup>12</sup> carried out the comparative analysis of the adsorption of Pb (II) and Cd(II) in wastewater using Borrassus aethiopicum and Cocos nucifera. The adsorption of lead from aqueous solution onto untreated orange barks was studied by Azouaou et al.<sup>13</sup>. This study was undertaken to explore the efficiency of bengal gram husk (Cicer arietinum) as adsorbent for the removal of lead from aqueous solutions. Batch experiment studies were carried out to investigate the effect of various parameters like weight of adsorbent, initial metal ion concentration & contact time. Equilibrium studies were performed by analyzing the Langmuir and Freundlich isotherms. The kinetics and thermodynamic parameters were evaluated.

## II. MATERIAL AND METHODS

### A. Materials:

**Chemicals:** Lead nitrate AR grades, Sulphuric acid AR grade, Formaldehyde AR grade, Sodium hydroxide AR grade, all chemicals used were purchased from Merck India.

**Instruments:** Atomic adsorption spectrophotometer, Rotary shaker, P<sub>H</sub> meter, electric oven and electric grinder etc.



Fig:(A) Atomic absorption spectrophotometer



Fig: (B) PH meter



Fig: (E) Bengal gram husk



Fig: (C) Mechanical shaker

#### Preparation of adsorbent:

50g of Bengal gram (*Cicer arietinum*) was soaked and the Bengal gram husks were peeled out. The Bengal gram husk was extensively washed in running tap water to remove dirt and other particulate matter. Washing and boiling in distilled water repeatedly to remove color followed this. The washed Bengal gram husk was oven dried at 105°C for 24 hrs. The oven dried Bengal gram husk was ground and sieved to get 0.25mm particle size. The stages of Bengal gram husk adsorbent preparation are shown in fig (F).



Fig: (D) Electric oven

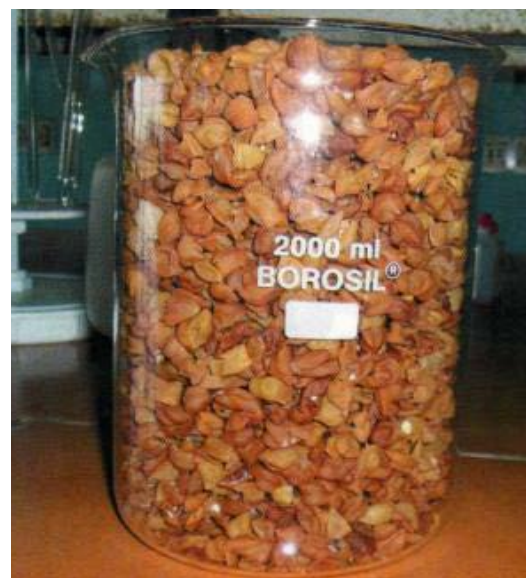


Fig: (F) Peeled Husk.

#### Activation of adsorbent (*Cicer arietinum* husk):

250 ml  $H_2SO_4$  and 62.5 ml of 39% HCHO was taken in a 500 ml beaker and 25g of finely ground powder was added to it. The solution was kept in a water bath at 60° for (6 hrs.) and the whole mixture was occasionally stirred. The mixture was then filtered by using whatman filter paper no.42. The residue was washed with demineralized water

Sample: Bengal gram husk(*Cicer arietinum* husk)

several times till the pH of filtrate was 4-5 (to remove  $H_2SO_4$ ). The residue was then dried for 24hrs at  $50^\circ C$  in an electric oven and used for removal of metal ions.



Fig: Bengal gram husk powder before activation and after activation.

#### Preparation of Adsorbate:

A stock solution of 1000mg/L Pb(II) was prepared by dissolving 0.3995g

$Pb(NO_3)_2$  in 250 ml volumetric flask by using distilled water. All the chemicals used were of analytical reagent grade.



Fig: stock solutions of different ppm

### III. METHODS

#### Batch Adsorption Studies:

##### Effect of adsorbent dose:

To study the effect of Bengal gram husk dose on the  $Pb^{2+}$  ions adsorption, different amounts of Bengal gram husk adsorbent (0.05, 0.2, 0.3) were added into a 250ml conical flask containing a definite volume (50 ml) of fixed initial concentration (100 mg/L) of  $Pb^{2+}$  ions solution at  $30^\circ C$ . The pH of the solution was 4.5. The mixtures were agitated at 100 rpm for 5 hr. followed by filtration using whatman filter paper no 42. The filtrate containing the residual concentration of lead was determined spectrophotometrically and the  $Pb^{2+}$  ions concentrations were measured<sup>15</sup>.

##### Effect of initial concentration:

To study the effect of initial ion concentration on the  $Pb^{2+}$  ions adsorption, different concentration of  $Pb^{2+}$  solution were taken as (100,250,500mg/l) from these solution definite volume of 50 ml solution from each  $Pb^{2+}$  solution were pour into a 250ml conical flask containing 0.3 g of Bengal gram husk adsorbent.(at  $30^\circ C$ .The pH of the solution was 4.5). The mixtures were agitated at 100 rpm for 5hr. followed by filtration using whatman filter paper no 42. The filtrate containing the residual concentration of  $Pb^{2+}$  was determined spectrophotometrically and the initial ion concentrations were measured.

##### Effect of temperature:

Adsorption Experiments were performed at different temperatures of 30,  $40^\circ C$  for the initial lead concentrations of 100, 250 and 500 mg/L. 50 ml of this initial concentration were poured in 250 ml conical flask containing 0.3 g of adsorbent pH 5. The mixtures were agitated at 100 rpm for 5 hrs.

##### Effect of contact time:

To study the effect of contact time on  $Pb^{2+}$  ions adsorption at different contact time (1, 2.5, 5 hrs.). 50 ml of stock solutions of lead (100, 250, 500) were pour into a 250ml conical flask containing 0.3 g of Bengal gram husk adsorbent .(at  $30^\circ C$ .The pH of the solution was 4.5). The mixtures were agitated at 100 rpm for different contact time. Filtration is done using whatman filter paper no.42. The filtrate containing the residual concentration of  $Pb^{2+}$  was determined spectrophotometrically and the initial ion concentrations were measured.

##### Equilibrium study:

Adsorption isotherms are mathematical models that describe the distribution of the adsorbate species among liquid and adsorbent, based on a set of assumptions that are

mainly related to the heterogeneity/homogeneity of adsorbents, the type of coverage and possibility of interaction between the adsorbate species. Adsorption data are usually described by adsorption isotherms, such as Langmuir, Freundlich isotherms. These isotherms relate metal uptake per unit mass of adsorbent,  $q_e$ , to the equilibrium adsorbate concentration in the bulk fluid phase  $C_e$ . The amount of metal adsorbed by activated Bengal gram husk was calculated from the difference between metal quantities added to the metal content of the supernatant using

$$q_e = \frac{(C_i - C_e)V}{M} \dots \dots \dots (1)$$

Where  $q_e$  is the metal uptake (mg metal adsorbed per g adsorbent),  $C_i$  and  $C_e$  are the initial and equilibrium metal concentration in solution (mg/L),  $V$  is the volume of the solution (mL), and  $M$  is the weight of activated Bengal gram husk (g). The percentage of removed Pb (II) ions (%) in solution was calculated using formula 2.

$$R\% = \frac{(C_i - C_e)}{C_i} \times 100 \dots \dots \dots (2).$$

**Langmuir isotherm:**

The Langmuir model <sup>16, 17</sup> is based on the assumption that the maximum

Adsorption occurs when a saturated monolayer of solute molecules is present on the adsorbent surface, the energy of adsorption is constant and there is no migration of adsorbate molecules in the surface plane. The Langmuir isotherm is given by:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \dots \dots \dots (3)$$

The constants in the Langmuir isotherm can be determined by plotting  $(1/q_e)$  versus  $(1/C_e)$  and making use of above equation written as:

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{q_m K_L C_e} \dots \dots \dots (4)$$

Where  $q_m$  and  $K_L$  are the Langmuir constants, representing the maximum adsorption capacity for the solid phase loading and the energy constant related to the heat of adsorption respectively.

**The Freundlich isotherm**

The Freundlich isotherm model <sup>16, 18</sup>, is an empirical relationship describing the adsorption of solutes from a liquid to a solid surface and assumes that different sites with several adsorption energies are involved. Freundlich adsorption isotherm is the relationship between the amounts of nickel adsorbed per unit mass of adsorbent,  $q_e$ , and the concentration of the nickel at equilibrium,  $C_e$ .

$$q_e = K_f C_e^{1/n} \dots \dots \dots (5)$$

The logarithmic form of the equation becomes,

$$\text{Log } q_e = \text{log } K_f + \frac{1}{n} \text{log } C_e \dots \dots \dots (6)$$

Where  $K_f$  and  $n$  are the Freundlich constants is the characteristics of the system.  $K_f$  and  $n$  are the indicators of the adsorption capacity and adsorption intensity, respectively. The ability of Freundlich model to fit the experimental data was examined.

**Thermodynamic parameters:**

The thermodynamic parameters for the adsorption of lead ions by Bengal gram husk such as the enthalpy change ( $\Delta H^\circ$ ), the Gibbs free energy change ( $\Delta G^\circ$ ) and the entropy change ( $\Delta S^\circ$ ) can be calculated from the variation of Langmuir constant ( $K_L$ ) with temperature (T) using the following basic thermodynamic relations <sup>19</sup>.

$$\Delta G = -RT \ln K_c \dots \dots \dots (7)$$

$$\Delta G = \Delta H - T \Delta S \dots \dots \dots (8)$$

$$\ln K_c = \frac{-\Delta H}{RT} + \frac{\Delta S}{R} \dots \dots (9)$$

The adsorption of lead ions by Bengal gram over the temperature range studied can be determined graphically by the linear plotting of  $\ln K_c$  against  $1/T$  using the least squares analysis, The mean enthalpy change can be determined from the slope of the straight line. The variation of Gibbs free energy and entropy change with temperature can be calculated using equations 7, 8 and 9, respectively.

**Adsorption Kinetics:**

**Pseudo first-order kinetic model:**

The Pseudo first-order kinetic model was proposed by Lagergren (Maniatis and Nurmala, 1992). The equation is generally expressed as

$$\frac{dq_t}{dt} = K_1 (q_e - q_t) \dots \dots \dots (10)$$

Where,  $q_e$  and  $q_t$  are the adsorption capacity at equilibrium and at time  $t$ , respectively ( $\text{mg g}^{-1}$ ),  $K_1$  is the rate constant of pseudo first -order adsorption ( $\text{L min}^{-1}$ ).

After integration and applying boundary conditions,

$t = 0$  to  $t = t$  and  $q_t = 0$  to  $q_t = q_t$ , the integrated form of equation (10) becomes.

$$\text{log}(q_e - q_t) = \text{log}(q_e) - \frac{K_1}{2.303} t \dots \dots \dots (11)$$

The values of  $\text{log}(q_e - q_t)$  are linearly correlated with  $t$ . The slope and intercept of plots of  $\text{log}(q_e - q_t)$  versus  $t$  were used to determine the pseudo-first order rate constants,  $K_1$  and  $q_e$  at different temperatures.

**Pseudo second- order model:**

The adsorption kinetics may also be described by a pseudo second-order equation (Chiou and Li, 2002; Nwabanne and Igboke, 2008; Ho and Chiang, 2001).

$$\frac{dq_t}{dt} = K_2(q_e - q_t)^2 \dots \quad (12)$$

Equation (13) can be rearranged to obtain a linear form

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

Integrating equation (12) and applying the boundary condition,

$t = 0$  to  $t = t$  and  $q_t = 0$  to  $q_t = q_t$  gives.

$$\frac{t}{q_e - q_t} = \frac{1}{q_e} + K_2 t \dots (13)$$

Where,  $K_2$  is the rate constant,  $t$  of pseudo second-order adsorption ( $\text{gmg}^{-1} \text{min}^{-1}$ ). The slope and intercept of plot of  $t/q_t$  versus  $t$  were used to calculate the pseudo second-order rate constant, at different temperatures.

Results and Discussion;

Table: 1

Ca, mol/L (30°C)	Sample No. and time (min)	Ce, mol/L	Qe mol/g	Langmuir Isotherm		Freundlich Isotherm	
				1/Ce L/mol	1/Qe g/mol	Log Ce	Log Qe
0.000482625	-	0.000444862	0.0000377630	2247.888	26480.94	-3.317746	-4.422933
0.00120656	-	0.00069947	0.00050709	1429.653	1972.0365	-3.1552309	-3.2949110
0.00241312	-	0.00104406	0.00136906	957.7993	730.4281	-2.981274	-2.863576

Table: 2

Ca, mol/L (40°C)	Sample No. and time (min)	Ce, mol/L	Qe mol/g	Langmuir Isotherm		Freundlich Isotherm	
				1/Ce L/mol	1/Qe g/mol	Log Ce	Log Qe
0.000482625	X1:60	0.000475626	0.00000699	2102.49229	142896.5619	-3.322734	-5.15502178
	X2:150	0.0004749995	0.000007625	2105.26537	131139.2409	-3.3233068	-5.1177326
	X3:300	0.0004731172	0.000009507	2113.64118	105177.6872	-3.3250312	-5.02192361
0.00120656	Y1:60	0.001006545	0.000200014	993.49755	4999.640026	-2.9971668	-3.6989387
	Y2:150	0.000999032	0.00020752	1000.97094	4818.626884	-3.00042	-3.6829232
	Y3:300	0.000970074	0.000236486	1030.84919	4228.580127	-3.13319	-3.626194
0.00241312	Z1:60	0.001574	0.000838127	635.324015	1193.136601	-2.802995	-3.07669016
	Z2:150	0.001447725	0.000965395	690.738918	1035.845431	-2.8393139	-3.01529495
	Z3:300	0.00088788	0.00152524	1126.27832	655.6345231	-3.05164572	-2.81666181

*Effect of amount of adsorbent:*

Adsorption dosage is an important parameter because this factor determines the capacity of an adsorbent for a given initial concentration of the adsorbate.

The effect of adsorbent concentration on the percentage removal of Pb(II) using an initial Pb concentration of 100 ppm is examined. The studied adsorbent dosages were 0.05, 0.2, 0.3 g as shown in fig 1.

The results showed that as the adsorbent (natural Bengal gram) dosage increased, the percentage of adsorbed Pb (II) also increased.

*Effect of initial concentration:*

The effect of initial concentration on the percentage removal of Pb(II) using an adsorbent concentration of 0.3 g is examined. The studied initial concentrations were 100, 250 and 500 ppm. As observed from the figure (fig. 2), the

removal of Pb(II) ion is found to increase with decrease in initial concentration.

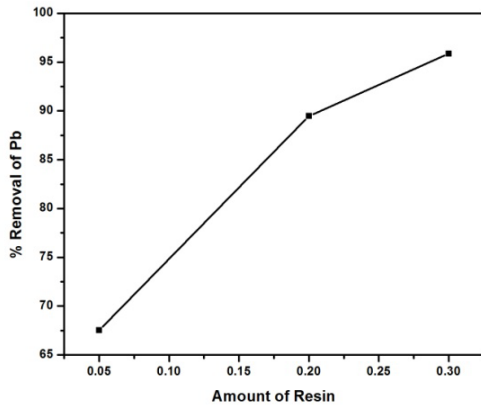


Fig 1: Effect of amount of adsorbent.

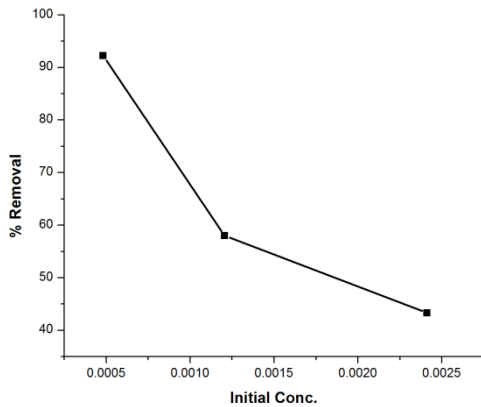


Fig: 2 Effect of initial concentration

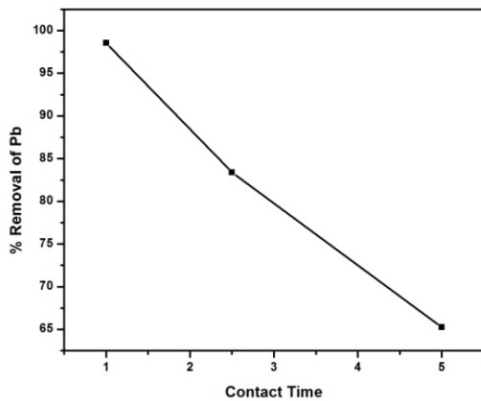


Fig 3: Effect of contact time at 60 min.

Effect of contact time:

From Fig. 3, 4 and 5 it reveals that the rate of percent lead removal is higher at the beginning. This is probably due to larger surface area of the Bengal gram husk being available at beginning for the adsorption of Pb<sup>2+</sup> ions. As the surface adsorption sites become exhausted, the uptake rate is controlled by the rate at which the adsorbate is transported from the exterior to the interior sites of the adsorbent particles.

300 min.

Most of the maximum percent lead removal was attained after about 150 min of shaking time at different initial concentrations. The increasing contact time increased the Pb<sup>2+</sup> adsorption and it remains constant after equilibrium reached in 60 min for different initial concentrations

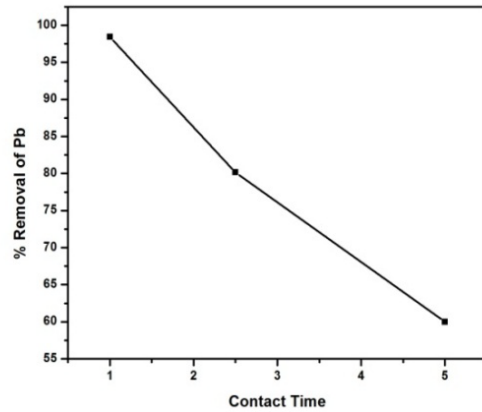


Fig 4: Effect of contact time on lead at 150 min.

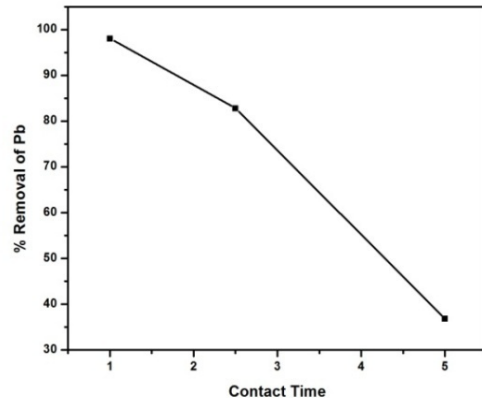
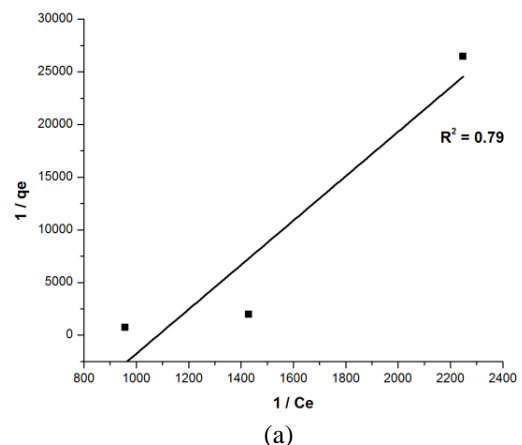
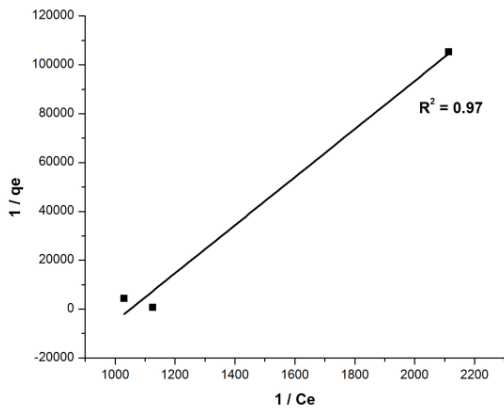


Fig 5: Effect of contact time on lead at 300 min.

*The Langmuir isotherm:*

The Langmuir adsorption equilibrium isotherm of lead onto activated Bengal gram husk at 30<sup>o</sup> and 40<sup>o</sup>C is presented in Fig. 6. Regression analysis reveals that the Langmuir model fits the experimental data well with correlation factor higher than 0.98.





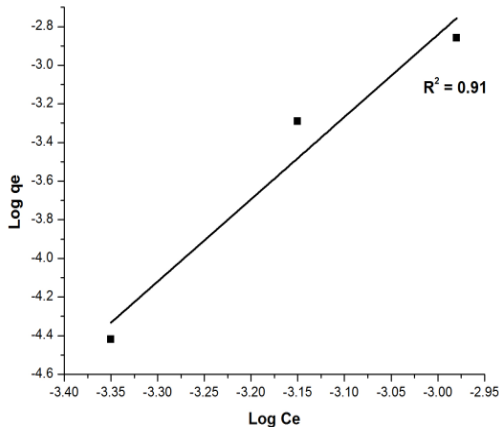
(b)

Fig 6:Langmuir adsorption isotherm for lead adsorbed on Bengal gram husk

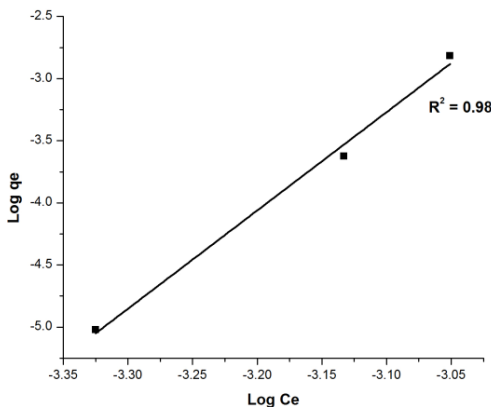
A plot of  $1/q_e$  versus  $1/C_e$  was found to be a straight line with  $1/q_m K_L$  as intercept and slope, and hence  $q_m$  and  $K_L$  can be calculated. Figure 6, shows the Langmuir adsorption isotherm. Langmuir constants  $q_m$  and  $K_L$ , and the correlation coefficient  $R^2$  are given in Table 4.

Fig: (a) at 30°C and (b) at 40°C

The Freundlich isotherm:



(a)



(b)

Fig 7: Freundlich adsorption isotherm for lead adsorbed on Bengal gram husk.

The Freundlich adsorption equilibrium isotherm of lead onto activated Bengal gram husk at 30°C and 40°C [Fig: 7 (a) and (b)] is studied. Plot of  $\text{Log } q_e$  versus  $\text{Log } C_e$  is found to be a straight line. Values of  $K_F$  and  $n$  (obtained from intercept and slope) indicate that both adsorption systems at 30°C and 40°C were favorable and had a higher adsorption capacity.

Fig: (a) at 30°C and (b) at 40°C

Both the Langmuir and Freundlich isotherms parameters for the adsorption of Lead onto activated Bengal gram husk and the correlation coefficients were

Shown (Table 4). The correlation coefficients of the Freundlich model were higher than that of Langmuir model suggesting towards monolayer adsorption process.

Table 4. Isotherm Model Constant and Correlation Coefficient for Adsorption of Lead.

Langmuir Isotherm		Freundlich Isotherm	
$K_L$	$R^2$	$K_f$	$R^2$
-0.11029	0.79	-7.345	0.91
-0.379	0.97	-7.854	0.98

Since the value of  $R^2$  is nearer to 1 which indicates that the respective equation better fits the experimental data. Freundlich isotherm was concluded to be preferred model for the adsorption process. The observations confirm the capacity of Bengal gram husk to adsorb lead. The Freundlich model fits well with  $R^2$  value of 0.98.

Adsorption Kinetics:

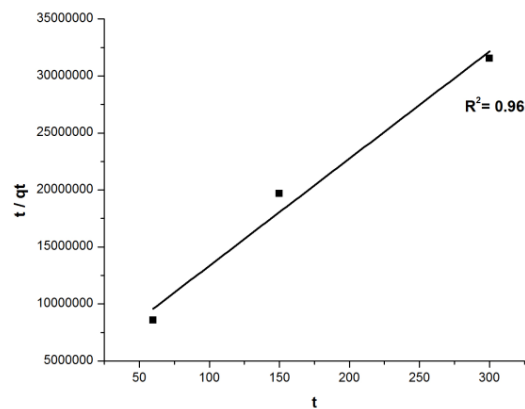


Fig 8: Adsorption kinetics of Pb(II) by linear plots of Pseudo second-order rate equation in 100 ppm.

The kinetic studies of Pb(II) adsorption on activated Bengal gram husk was carried out using the pseudo-first order and pseudo-second-order models on experimental data. The effect of initial lead concentrations was investigated to find the best fit kinetic model. The kinetic constants and correlation coefficients of pseudo first-order

kinetic model fail to give straight line. Therefore, Pseudo second-order kinetic model is preferred.

The pseudo-second-order kinetic model was applied by plotting  $t/qt$  versus  $t$ , and this model gave high values of regression correlation coefficient as seen in Figure: 8, 9, and 10. This implies that the mechanism of adsorption of Pb(II) ion on the activated Bengal gram husk follows the pseudo-second-order kinetics, as shown in Figure: 8, 9 and 10.

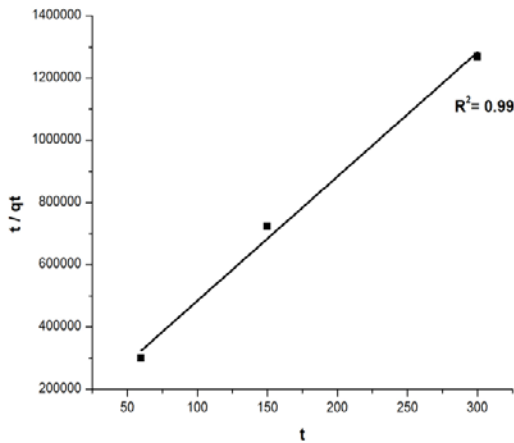


Fig 9: Adsorption kinetics of Pb (II) by linear plots of Pseudo second-order rate equation in 250 ppm.

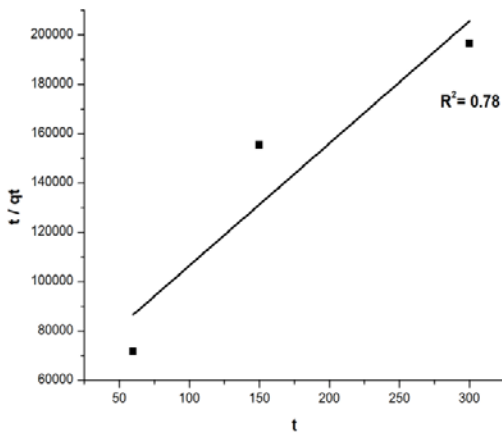


Fig 10: Adsorption kinetics of Pb (II) by linear plots of Pseudo second-order rate equation in 500 ppm.

This is compared with the kinetic studies of Ni (II) adsorption of coconut husk conducted by Kehind et. al. (20) where regression coefficients showed high values for the pseudo-second-order kinetic model ( $R^2 = 0.96, 0.99$  and  $0.78$ ) indicating its applicability to adsorption. Both factors indicate that the adsorption of metal ions followed the second-order kinetic model, indicating that the rate-limiting step was a chemical adsorption process between the metal ion and activated Bengal gram husk (21). It was clear that the pseudo second-order rate constant  $k^2$  decreased with the increase in the initial lead concentrations.

*Thermodynamic Studies:*

A plot of  $\ln K_c$  versus  $1/T$  was found to be linear (Fig.11 (A), (B) and 12),  $\Delta H$  and  $\Delta S$  determined from the slope and intercept of the plot, respectively.

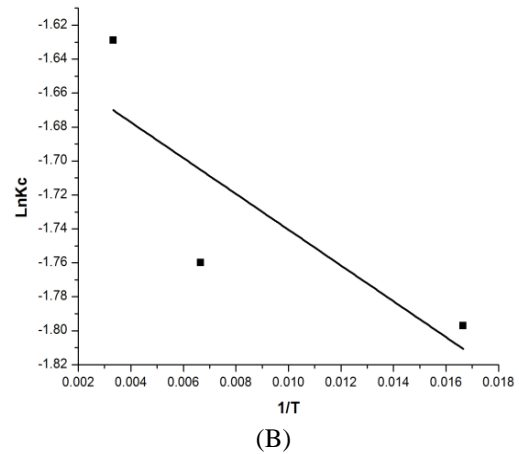
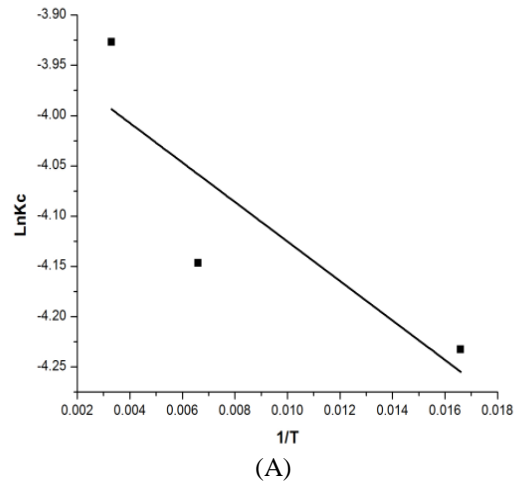


Fig 11: The estimation of thermodynamic parameter for adsorption of Pb(II) onto Bengal gram husk (A) for 100 ppm. (B) for 250 ppm.

An important result can be obtained from table 5. Is that the Gibbs free energy change ( $\Delta G$ ) is positive with its value decreases with increasing the initial concentration. This indicates that the adsorption process of lead ions by activated Bengal gram husk can be enhanced by increasing concentrations (at 400 C). The negative values of change in enthalpy ( $\Delta H$ ) suggest the exothermic nature of adsorption and the negative values of change in entropy ( $\Delta S$ ) can be used to describe the randomness at the solid solution interface during the adsorption of Pb(II) ion on Bengal gram husk (Table 5).



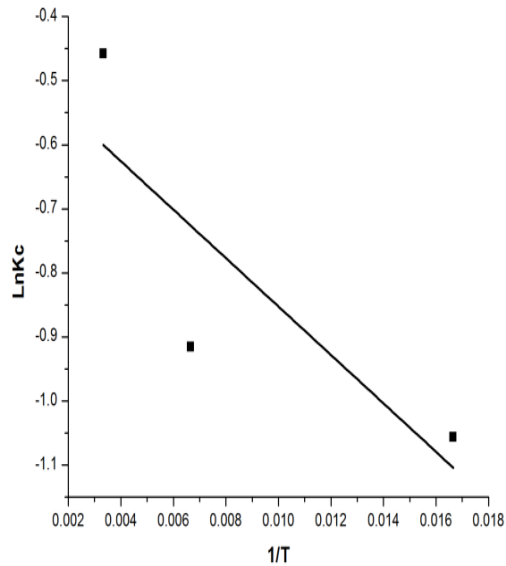


Fig 12: The estimation of thermodynamic parameter for adsorption of Pb(II) onto Bengal gram husk for 500 ppm

### I. CONCLUSIONS

Equilibrium, kinetic and thermodynamic studies were made for the adsorption of Pb<sup>2+</sup> ions from aqueous solution onto activated Bengal gram husk. The equilibrium data have been analyzed using Langmuir, Freundlich isotherms. The characteristic parameters for each isotherm and related correlation coefficients have been determined. The Freundlich isotherm was demonstrated to provide the best correlation for the sorption of Pb<sup>2+</sup> ions onto activated Bengal gram husk. The suitability of the second-order equations kinetic model for the sorption of Pb<sup>2+</sup> ions onto activated Bengal gram husk is also discussed. The pseudo second-order kinetic model agrees very well with the dynamical behavior for the adsorption of Pb<sup>2+</sup> ions onto Bengal gram husk for different initial Pb<sup>2+</sup> ions concentrations over the whole range studied. It may be concluded that activated Bengal gram husk may be used as a low-cost, natural and abundant source for the removal of Pb<sup>2+</sup> ions from the wastewater.

Table 5: Thermodynamic parameters calculated for the adsorption of Pb(II) ions onto activated Bengal gram husk.

Graph No.	Initial Pb(II) conc.(mg/L)	ΔG(kJ/mol)	ΔH(kJ/mol)	ΔS(kJ/K.mol)	Temp.(k)
11(A)	0.000482625	1207.31	-19.65	-3.92	313 k
11(B)	0.00120656	499.65	-10.54	-1.63	
12	0.00241312	110.55	-37.81	-0.474	

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