Kinetic, Equilibrium, and Thermodynamic Studies of the Biosorption of Cd(II) and Pb(II) from Aqueous Solutions by *Talinum triangulare* (Water Leaf).

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ABSTRACT

The biosorption of Cd(II) and Pb(II) by *Talinum triangulare* (waterleaf) has been studied. Experimental tests have been studied under different operation conditions. The highest adsorption was observed for lead biosorption at pH 4, while increase in the pH of the solution resulted in corresponding increase in cadmium uptake up to pH 6. The optimum time for the adsorption process was found to be 10 minutes for cadmium and 30 minutes for lead. Biomass dosage also had influence on the amount of metal adsorbed. Metal uptake increased with increase in the initial metal ion concentration up to 2 mM, which was the maximum concentration tested. The kinetics of the process can best be described by the pseudo-second order model, and the thermodynamic parameters showed that the biosorption process is feasible.

(Keywords: biosorption, *Talinum triangulare*, cadmium (II), lead (II), isotherm)

INTRODUCTION

Environmental pollution by toxic heavy metals occurs globally through military, industrial, and agricultural processes and waste disposal (Pagnanelli et al., 2000). Uncontrolled discharge of these heavy metal containing wastewaters to the environment can be detrimental to humans, animals and plants (Pamukoglu and Kargi, 2007; Fergusson, 1990).

The toxicity of cadmium and lead is extremely high, and these two heavy metals have been classified in the USEPA's Group B of pollutants (Evangelou, 1998; Volesky, 1990; Wase and Forster, 1997). "Itai Itai desease" is a well-known disease associated with exposure to cadmium. It is a problem of skeletal and renal system, and it was first diagnosed in Japanese individuals who consume rice and water contaminated with high level of cadmium (Kjellstroem, 1986).

Lead on its own may cause a range of health effects, from behavioral problems and learning disabilities, to seizures and death (Hoa et al., 2007; Schumann, 1990). This environmental and health concern have led to the development of various technologies for the removal of metals from wastewater generated from industries (Fergusson, 1990; Volesky, 1990; Kratochvil and Volesky, 1998; Aksu and Tunc, 2005). Conventional methods used for the removal of heavy metals from wastewater include precipitation, adsorption, ion-exchange, and solvent extraction. These methods require high capital and operating costs and may produce large volumes of solid wastes (Volesky, 1990).

Biosorption, a process whereby certain types of inactive, dead biomass may bind and concentrate heavy metals from aqueous solution is considered as alternative technology for the removal of toxic heavy metals from wastewater and industrial effluents (Naja et al., 2005; Volesky, 2003). The main advantage of using biosorption technology is the cost effectiveness of using the biosorbent, since it maybe derived from various cheap raw materials (Veglio et al., 2003; Davis et al, 2001). Numerous researchers have reported the biosorption of cadmium and lead onto different biomass including water hyacinth (Hasan et al, 2007; Wolverton and McDonald, 1978), coconut copra meal (Ofomaja and Ho, 2006), *Fucus spiralis* (Cordero et al, 2004), *Stereophyllum radiculosum* ((Babarinde et al., 2008a) and Calymperes erosum (*Babarinde et al., 2008b).

All the reports have shown that these bio-materials posses the ability for the sorption of these two metals. The purpose of this experiment was to determine the aqueous cadmium(II) and lead(II) removal potential of *Talinum triangulare*. This work was undertaken by studying the effect of pH, agitation time, biosorbent dose, and initial metal ion concentration on the biosorption process. The plant used for this process was *Talinum triangulare* (waterleaf). *Talinum triangulare* is a multi-ethnic weed common throughout the humid tropics (Fontem & Schippers, 2004). Several *Talinum* species including the closely related *Talinum portulacifolium* (Forssk.) Schweinf occur in Africa. The plant grows abundantly in the southern part of Nigeria and it is most times considered as weed and as a result, alternative use for it has to be sort. Since this plant has a high water content (i.e., 90.8 g per 100 g of edible leaf) it is speculated that the plant would be a good biosorbent in the removal of heavy metals, especially cadmium and lead from industrial wastewater.

MATERIALS AND METHODS

Biosorbent: Samples of *Talinum triangulare* were collected from a farm plot located within the campus of University of Ibadan, Ibadan, Nigeria prior to the experiment. The leaves were separated from the stems and washed in a running tap water. It was subsequently rinsed in de-ionized water and then oven dried. The dried sample was pulverize and then sifted through a 150 sieve mesh size. The prepared biosorbent was stored in air tight polythene bag.

Preparation of Stock Solution: Different solutions of cadmium and lead were also prepared by dissolving known masses of $Cd(NO_3)_2.4H_2O$ and $Pb(NO_3)_2$, respectively, in de-ionized water and then made up to the marks in appropriate standard volumetric flasks.

Biosorption Experiments: Batch studies carried out include pH dependence, biosorbent dose dependence, time dependence and concentration dependence.

All the batch studies except biosorbent dose study were carried out to yield a biosorbent-metal ion solution concentration of approximately 2 mg/ml, that is, 30 mg of biosorbent suspended in 15 mL of metal ion solution. These suspensions were shaken in a 250 mL plastic bottle on an electric shaker at 200 rpm. Atomic Absorption Spectrometer (Thermo S Series) was employed in the analysis of the resulting supernatant obtained from centrifugation of the agitated metal ion suspension. Each experiment was done in duplicate.

In order to investigate the effect of pH on metal uptake, the biosorption experiment was carried out at different pH of the metal ion solution, experimental temperature of 28^{o}C and 2 hours of agitation time. The pHs investigated were pH 2, 3, 4, 5 and 6. The pH adjustment was done by using concentrated solutions of nitric acid and sodium hydroxide.

Influence of biosorbent dosage was investigated by varying the biosorbent dosage. Biosorbent masses used for the study were 10, 30, 50, 70, 90 and 110 g. The study was carried out at the temperature of 28^{o}C and at the optimum pH values earlier determined for each metal. The agitation time for this experiment was set at 2 hours.

To investigate the effect of agitation time, the experiment was performed by varying the agitation time for the biosorption process. The time intervals were 5, 10, 20, 30, 60 and 120 min. Experimental temperature was set at 28^{o}C. The effect of initial metal concentration on biosorption was determined by using the pre-determined optimum pH, and equilibration time for each metal ion at 25^{o}C. Initial metal ion concentrations, C_o investigated for each metal were 0.1 mM, 0.3 mM, 0.5 mM, 0.75 mM, 1.0 mM and 2.0 mM. data obtained from this investigation was used to fit the adsorption isotherm models.

The thermodynamics of the biosorption process was determined by carrying out batch study at an initial metal ion concentration of 0.3mM, with all other experimental conditions maintained. The temperatures chosen for this study were 15^{o}C, 25^{o}C, 37^{o}C and 50^{o}C.

RESULTS AND DISCUSSION

Effect of pH: The effects of pH on the percentage Cd^{2+} and Pb^{2+} removal by *Talinum triangulare* are shown in Figures 1 and 2, respectively.

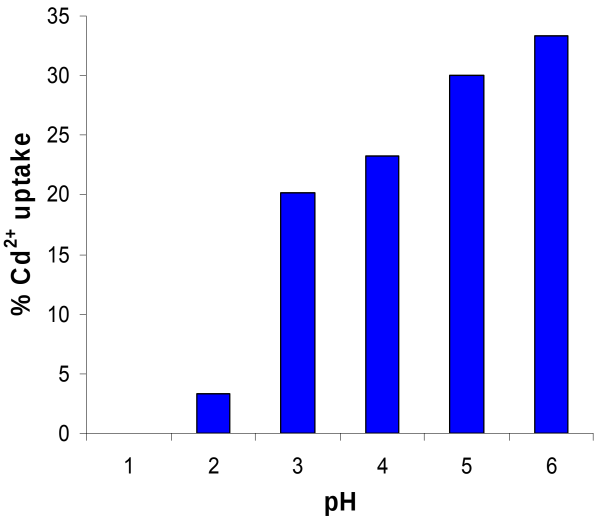


Figure 1: pH Dependent Profile of the Biosorption of Cadmium by *Talinum triangulare* using 30 mg Biomass Dose, 120 minute Agitation Time, 0.3 mM Initial Metal ion concentration and 28°C.

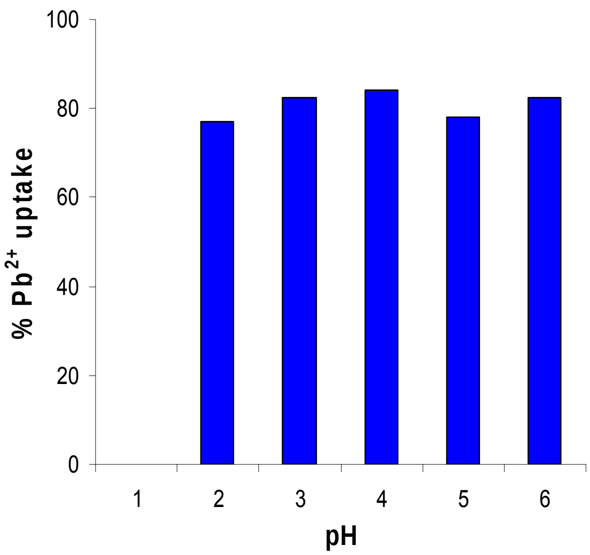


Figure 2: pH Dependent Profile of the Biosorption of Lead by *Talinum triangulare* using 30 mg Biomass Dose, 120 minute Agitation Time, 0.3 mM Initial Metal Ion Concentration and 28°C.

The removal of Cd(II) increased with increasing pH with the optimum uptake occurring at pH 6. Yin et al, (1999) has also reported increase in metal removal with increasing pH. For the biosorption of Pb(II), the percentage removal showed little variation with pH: significant amount of Pb(II) ion was removed at all pHs studied. Nevertheless, the highest adsorption was observed at pH 4. Experiments were not performed beyond pH 6 because at higher pH precipitation of metals is said to occur which may therefore interfere with the biosorption process (Parvathi et al, 2006).

Effect of Biosorbent Dose: Figures 3 and 4 show the effect of biosorbent dose on biosorption of Cd (II) and Pb(II) ions by *Talinum triangulare*.

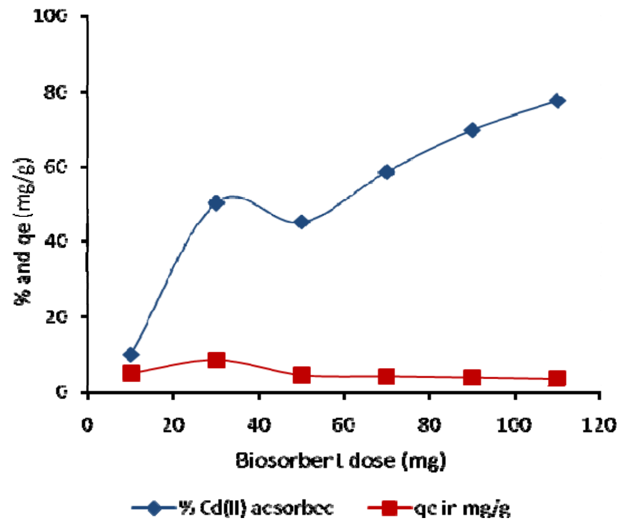


Figure 3: Effect of Biosorbent Dose on Biosorption of Cd(II) ion by *Talinum triangulare* using pH 6, 120 minute Agitation Time, 28° C, and 0.3 mM Initial Metal Concentration.

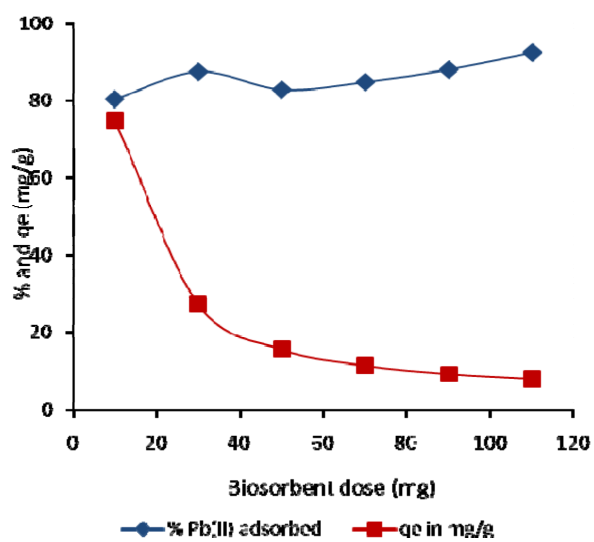


Figure 4: Effect of Biosorbent Dose on Biosorption of Pb(II) ion by *Talinum triangulare* using pH 4, 120 minute Agitation Time, 28° C, and 0.3 mM Initial Metal Concentration.

For Cd(II) ion, an increase in percentage removal of 10% to 50.3% was observed as the biosorbent dose increased from 10 to 30 mg. the % removal then decreased as the dose increased to 50 mg, and later increased steadily until it reached 77.5% at 110 mg biomass dose. For the metal uptake capacity, q_e, the highest Cd(II) uptake was observed at biomass dosage of 30 mg. A decrease in cadmium uptake was then observed with further increase in dosage.

A steady decrease in adsorption capacity, q_e with increasing mass dose was observed for Pb(II) ion as shown in Figure 4. The percentage removal of lead increased from 80.32 % to 87.5 % for a mass increase from 10g to 30 g, it subsequently decreased to 82.83 % and steadily increased thereafter with increasing mass dose. Reduction in metal uptake, q_e, by *Talinum triangulare* with increasing biomass concentration was attributed to an insufficiency of metal ions in solution with respect to available binding sites (Fourest & Roux, 1992; De Rome & Gadd, 1987). Other studies having similar result have been reported (Al-Asheh & Duvnjak, 1995; Sampedro et al., 1995). Higher specific uptake at lower dry mass concentrations has also been attributed to an increased metal-to-biosorbent ratio, which decreases upon an increase in dry mass concentration (Puranik & Pakniker, 1999).

To maintain evenness and have reason for comparison in the present study, a biomass dose of 30 mg was chosen for all the batch equilibrium studies carried out.

Effect of Agitation Time: The rate of Pb (II) ion removal was rapid in the first 30 min and decreased significantly until it attained equilibrium (Figure 5). For an initial metal ion concentration of 0.3 mM, it was observed that rapid metal uptake occurred within the first 10 minutes for Cd (II) (Figure 6).

The observed fast biosorption kinetics is consistent with biosorption of metal involving non-energy mediated reactions, where metal removal from solutions is due to purely physico-chemical interaction between biomass and metal solution (Aksu, 2001). This fast metal uptake from solution indicates that binding might have resulted from interaction with functional groups on the cell wall of the biosorbent rather than diffusion through the cell wall of the biomass.

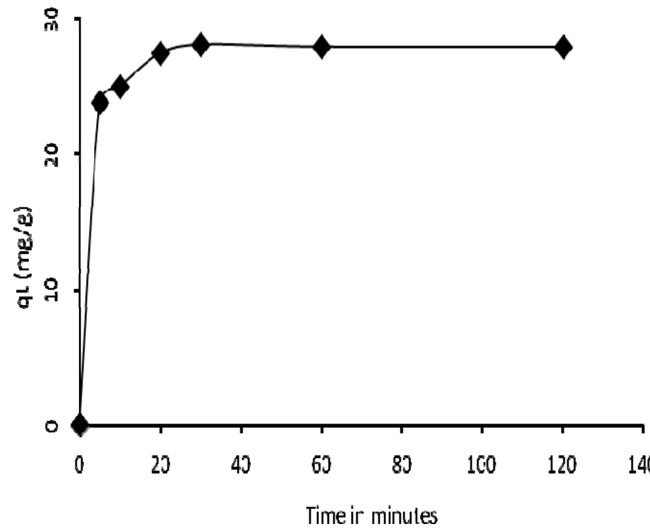
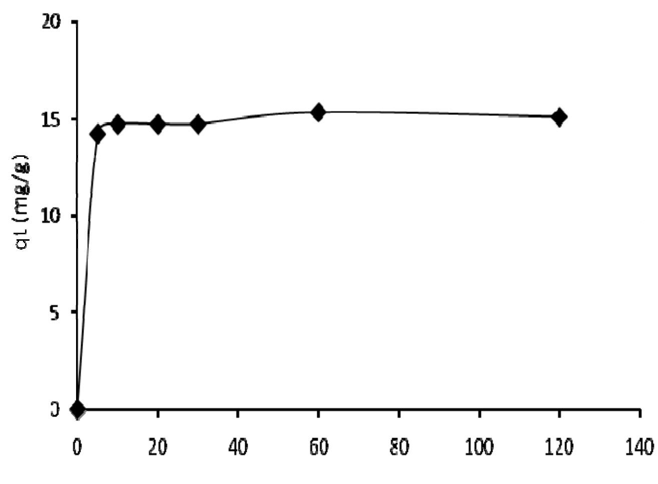


Figure 5: Time Dependent Study of the Biosorption of Lead by *Talinum triangulare* using 30 mg Biomass Dose, 28^{o} C, 0.3 mM Initial Metal concentration and pH 4.



Time in minutes
Figure 6: Time Dependent Study of the Sorption of Cadmium by *Talinum triangulare* using 30 mg Biomass Dose, 28^{o} C, 0.3 mM Initial Metal Concentration, and pH 6.

Kinetic Study Profile: In order to evaluate the kinetics of the sorption process, data from the time study were fitted into the pseudo-first-order and pseudo-second-order models. Kinetic sorption of heavy metals from wastewater has been studied using mostly pseudo-first-order (Langergren, 1898) and pseudo-second-order models (Ho and McKay, 2000). The linearized form of pseudo-first-order and pseudo-second-order models are giving by Equations 1 and 2, respectively.

$$\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \qquad (1)$$

where, k_1 is the rate constant of pseudo-first-order biosorption; q_e is the metal uptake in mg/g at equilibrium; and q_t is the metal uptake in mg/g at time, t.

A plot of $\log(q_e\text{-}q_t)$ against t was made and values of k_1 and q_e were obtained from the slope and intercept respectively. q_e is pre-estimated by making a plot of q_t against time, t , which gives a parabolic curve whose q_t value plateau to q_e at infinite time t.

$$t/q_t = \left(\frac{1}{k_2 q_e^2}\right) + \left(t/q_e\right) \qquad (2)$$

K_2 is the rate constant of pseudo-second order biosorption.

A plot of (t/q_t) against t gives $(1/q_e)$ as slope and $(1/\ k_2\ q_e^2)$ as intercept from which k_2 can be obtained. Both models are tested for suitability using their correlation coefficient, R^2 (Ho and McKay, 2000).

Figure 7 shows the linear plot of pseudo-second-order kinetics for the biosorption of Cd(II) by *Talinum triangulare.* Also, Figures 8 and 9 show the pseudo-first-order and pseudo-second-order kinetics for the biosorption of Pb(II) by *Talinum triangulare,* respectively.

As can be seen from Table 1, the q_e (calculated) determined from the plot of the pseudo-first-order model for each metal, differs from that obtain experimentally, q_e (experimental). This implies that the model is not very good in explaining the kinetics of the adsorption of the metals. On the other hand, the pseudo-second-order model as shown in Table 2 better fits the kinetics as their correlation coefficient is close to 1 (i.e. 0.999 for both Cd(II) and Pd(II)). The estimated values of q_e are also close to the q_e, experimental. All these point to the fact that second order kinetic best explain the observed rate, suggesting that biosorption is the rate-limiting step; and that sorption of the metal ions involves two species, in this case, the metal ion and the biomass (Wallace, et al. 2003).

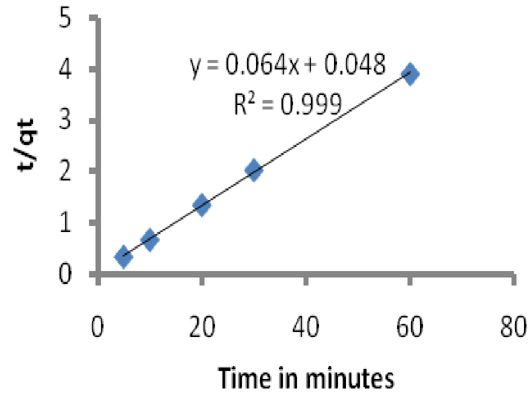


Figure 7: Pseudo-Second Order Graph for Sorption of Cd(II) Ions by *Talinum triangulare.*

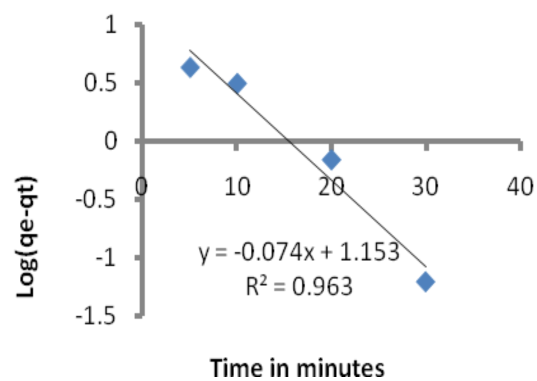


Figure 8: Pseudo-First Order Graph for Sorption of Pd(II) Ions by *Talinum triangulare.*

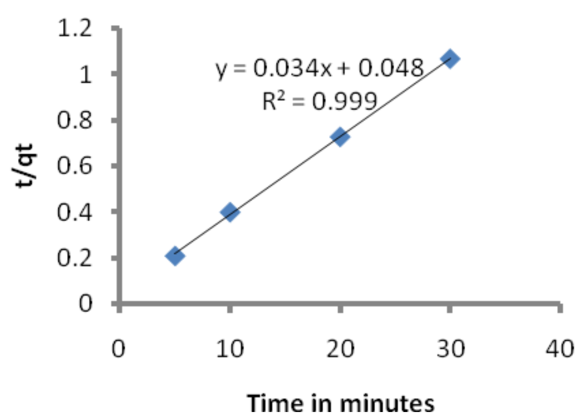


Figure 9: Pseudo-Second Order Graph for Sorption of Pd(II) Ions by *Talinum triangulare.*

Table 1: Pseudo-First Order Constants for the Biosorption of Pb(II) and Cd(II) by *Talinum triangulare.*

Metal	K_1	q_e(calc)	q_e(expt)	R^2
Pb	0.170	14.22	28.20	0.963
Cd	0.067	2.02	15.38	0.871

Table 2: Pseudo-Second Order Constants for the Biosorption of Pb(II) and Cd(II) by *Talinum triangulare.*

Metal	K_2	q_e(calc)	q_e(expt)	R^2
Pb	0.024	29.41	28.20	0.999
Cd	0.085	15.63	15.38	0.999

Thermodynamic Studies: The thermodynamic study was done by carrying out equilibrium study at various temperatures to obtain corresponding C_a's and C_e's (Mckay and Ho, 1999). From these values, equilibrium constant, K is calculated using the relationship

$$K = \frac{C_a}{C_e} \text{.....................................} \quad (3)$$

where, C_a represents adsorption in mg/L at equilibrium; Ce is the equilibrium concentration of the metal in mg/L; K represents the thermodynamic equilibrium constant (Sarin et al., 2006).

The Gibb's free energy ΔG was calculated using the following relationship:

$$\Delta G = -RT \ln K \text{.........................} \quad (4)$$

where, ΔG is the Gibb's free energy in J/mole; R is the ideal gas constant whose value is 8.314 $Jmol^{-1}K^{-1}$; K represents the thermodynamic equilibrium constant; T is temperature in Kelvin.

According to thermodynamics the Gibb's free energy is related to the enthalpy change (ΔH) and entropy change (ΔS) at constant temperature by the following van't Hoff equation:

$$\log K = -\left(\frac{\Delta H}{2.303RT}\right) + \left(\frac{\Delta S}{2.303T}\right) \text{..........} \quad (5)$$

where, ΔH stands for enthalpy change in J/mol; ΔS represents entropy change in $Jmol^{-1}K^{-1}$;

The values of ΔH and ΔS were obtained from the slope and intercepts respectively of the plot of logK against 1/T (Mckay and Ho, 1999). The results of the thermodynamic studies of the biosorption process are presented in Tables 3 and in Figures 10 and 11. Pb(II) gave negative values for ΔH and ΔS. A negative enthalpy (ΔH) indicates that the adsorption process is exothermic. This is consistent with the result obtained from ΔG which decreased in magnitude with increasing temperature. ΔG decreases in magnitude from -5463.92 through -5297.54 to -4883.30 J/mol for the temperatures 298 K, 310 K and 323 K.

The decrease in adsorption with rise in temperature may be due to weakening of adsorptive forces between the active sites of adsorbent and adsorbate species and also between the adjacent molecules of the adsorbed phase (Pandey et al., 1986). A negative change in entropy, ΔS, indicates that the biosorption is stable, as there is increase in orderliness of the system, which is at the aqueous metal ion- solid biosorbent interface.

An opposite trend was observed for cadmium. Positive values of entropy (ΔS) and enthalpy (ΔH) were noticed (Table 3). From thermodynamics, a positive ΔH indicates endothermic reaction. A positive change in entropy is an indication of increased in randomness at the solid-liquid interface during the adsorption of nickel and cadmium (Kobya, 2003). As expected, ΔG increased in magnitude with increasing temperature. In general, negative values of ΔG show the feasibility and spontaneous nature of the sorption of Cd(II) and Pb(II) on *Talinum triangulare*.

Effect of Initial Metal-Ion Concentration: The effect of initial metal ion concentration on the biosorption of Cd(II) and Pb(II) indicated that sorption capacity increased with increase in initial metal ion concentration.

Table 3: Thermodynamic Parameters for the biosorption of Cd^{2+} and Pb^{2+} by *Talinum triangulare.*

Metal ion	ΔH^o ($kJmol^{-1}$)	ΔS^o ($kJK^{-1}mol^{-1}$)	ΔG^o ($kJmol^{-1}$)			
			T_1=288K	T_2=298K	T_3=310K	T_4=323K
Cd(II)	5.33	27.36	-2548.19	-2818.69	-3149.44	-1074.87
Pb(II)	-10.52	-16.91	-3975.87	-5463.92	-5297.54	-4883.30

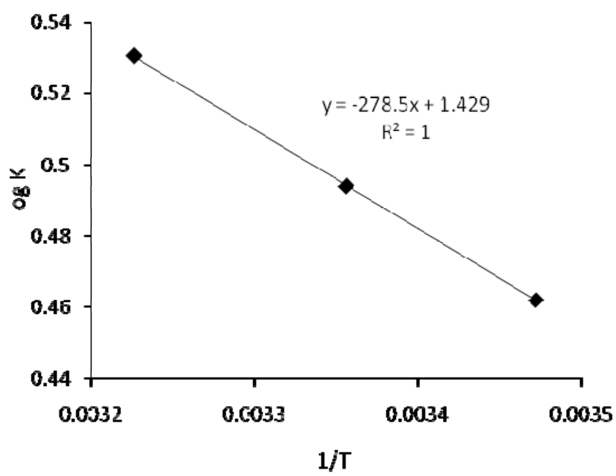


Figure 10: Thermodynamic Profile of Sorption of Cd(II) Ions by *Talinum triangulare* at 30 mg Dose, pH 6, 10 minute Agitation Time, and 0.3 mM Initial Metal Ion Concentration.

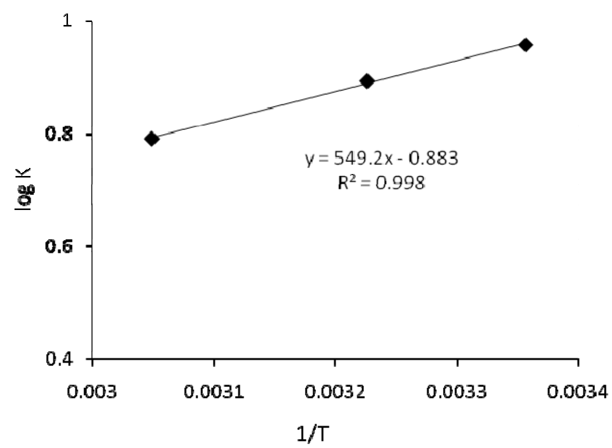


Figure 11: Thermodynamic Profile of Sorption of Pb(II) Ions by *Talinum triangulare* at 30 mg Dose, pH 4, 30 minute Agitation time, and 0.3 mM Initial Metal Ion Concentration.

The profile is shown in Figure 12. This sorption characteristic indicated that surface saturation was dependent on the initial metal ion concentrations. At low concentrations, adsorption sites took up the available metal more quickly. When the concentrations became high the rate of diffusion became slow. This is because metal needed to diffuse to the biomass surface by intraparticle diffusion and greatly hydrolyzed ions will diffuse at a slower rate (Zafar et al., 2006).

Equilibrium Modeling**:** The equilibrium of the biosorption process is often described by fitting the experimental points with models, which are used to represent the equilibrium adsorption isotherm (Gadd, et al.1988).

The simplest forms of these terms are Langmuir and Freundlich isotherms. Both isotherms are described based on physical interactions of adsorption and desorption. Other models are more empirical in form, only formulated to give a fitting for experimental data (Muhamad, 1998).

The linearized form of Langmuir, (1918) model is represented by the expression below:

$$\frac{1}{q_e} = \frac{1}{q_{\max} K_L}\left(\frac{1}{C_e}\right) + \frac{1}{q_{\max}} \quad \text{.............} \quad (6)$$

where, C_e is the equilibrium solute concentration in the fluid (mg/L or mM); K_L represents Langmuir equilibrium adsorption constant (L/mg or mM^{-1});

q_{max} is the Langmuir maximum metal uptake in (mg/g); q_e is metal uptake in (mg/g)

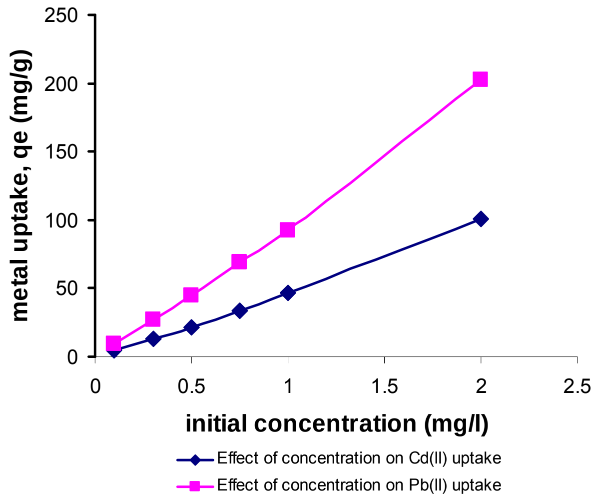


Figure 12: Effect of Cadmium and Lead Concentration on Biosorption by *Talinum triangulare* .

A plot of $1/q_e$ against $1/C_e$ gives a straight line plot with a slope of $(1/q_{max}K_L)$ and an intercept of $1/q_{max}$.

Langmuir model essentially describes monolayer kind of adsorption. It's advantages hinges on availability of interpretable parameters. However it is limited in application as structured and can only be applied only for monolayer type adsorption (Langmuir, 1918).

Also, the linearized form of Freundlich, (1907) model is represented by the following expression:

$$\log q_e = \left(\frac{1}{n}\right)\log C_e + \log K_F \quad (7)$$

where, q_e is metal uptake in (mg/g); C_e is the equilibrium solute concentration in the fluid (mg/L or mM); n represents Freundlich constant (dimensionless).Its related to adsorption intensity; K_F is Freundlich adsorption constant related to adsorption capacity.

A plot of $\log q_e$ against $\log C_e$ a straight line graph with (1/n) as slope and log K_F as intercept. The corresponding constants and coefficients of correlation (R^2) associated with each linearized models of Langmuir and Freundlich adsorption isotherms at 25°C for each of Cd(II) and Pb(II) ions are presented in Table 4. Figures 13 to 16 show the graphs of the linearised Langmuir and Freundlich adsorption isotherms at 28°C.

Table 4: Langmuir and Freundlich Adsorption Constants Associated with the adsorption of Pb(II) and Cd(II) by *Talinum triangulare* at 28°C.

Metal	q_m (mg/g)	K_L (L/g)	R^2	K_f	N	R^2
Cd(II)	100.0	0.028	0.971	2.0606	0.9533	0.874
Pb(II)	142.9	0.041	0.987	5.4828	1.1557	0.979

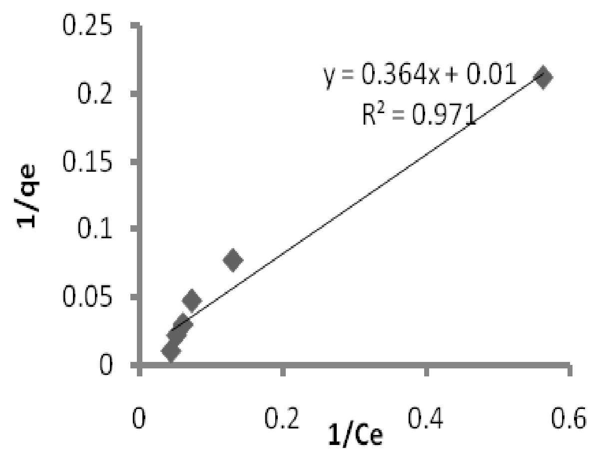


Figure 13: Langmuir Isotherm for Sorption of Cd(II) Ions by *Talinum triangulare.*

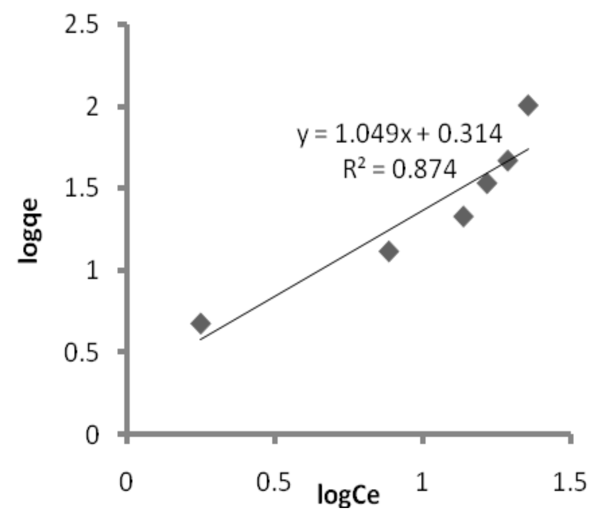


Figure 14: Freundlich Isotherm for Sorption of Cd(II) Ions by *Talinum triangulare.*

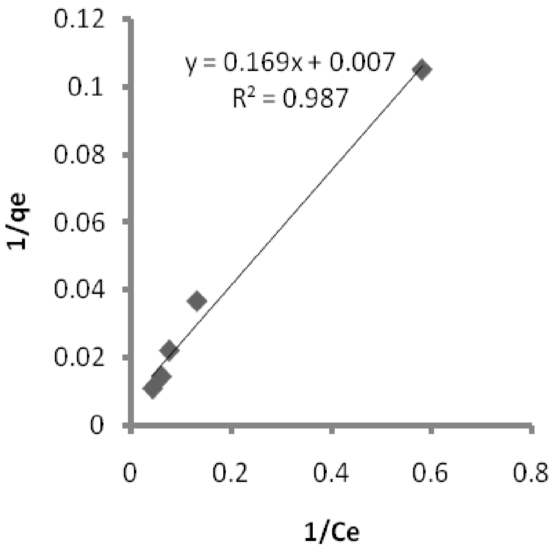


Figure 15: Langmuir Isotherm for Sorption of Pb(II) Ions by *Talinum triangulare.*

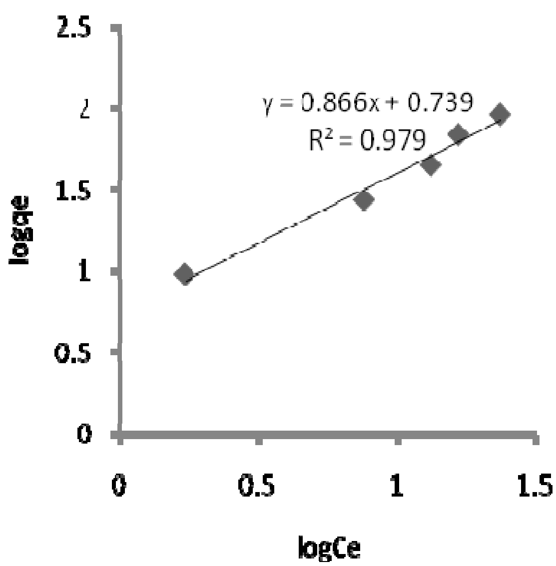


Figure 16: Freundlich Isotherm for Sorption of Pb(II) Ions by *Talinum triangulare.*

The result showed that Langmuir isotherm better fits Cd(II) and Pb(II) ions biosorption equilibrium data. Comparing the maximum value of metal uptake, it was found that lead had the highest value, with q_m of 142.86 mg/g compared to cadmium 100 mg/g for an initial metal ion concentration of 0.3mM.

CONCLUSIONS

In this study, *Talinum triangulare* biomass was used for the biosorption of the aqueous solutions containing cadmium or lead ion. Batch experiments showed that the solution pH strongly influenced the biosorptive capacity of the biomass. As the solution pH increased, the removal efficiency of Cd(II) increased, whereas the influence of pH was not all that significant in the biosorption of Pb(II) ion. However, greatest metal uptake was observed at pH 4. Metal uptake decreased with increase in metal dose for Pb(II). For Cd(II), the highest metal uptake was observed at biomass dose of 30 mg, after which it then decreased with increase in biomass dose.

Maximum sorption occurred within the first 10 minutes for cadmium, while equilibrium was reached for lead after 30 minutes of agitation. Moreover, a pseudo-second-order model has been successfully fitted to cadmium and lead-specific uptake versus time profiles, and the effect of the main operating conditions on the model parameters has been evidenced. Initial metal concentration also affected the overall metal uptake capacity of *Talinum triangulare.* Metal uptake increased as the initial concentration of the metal ions was increased. Biosorption equilibrium data for cadmium fit well to Langmuir model, while those of lead fit well to both Langmuir and Freundlich model, although it fits Langmuir model better. The thermodynamic study shows that the reaction is feasible and spontaneous.

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