International Journal of Research and Review
Vol. 9; Issue: 9; September 2022
Website: www.ijrrjournal.com
E-ISSN: 2349-9788; P-ISSN: 2454-2237

Research Paper

Biosorption of Cd (II), Pb (II) and Zn (II) from Aqueous Solutions using Garden Egg *(Solanum Melongena)* Leaf: Equilibrium, Kinetics and Thermodynamics

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DOI: https://doi.org/10.52403/ijrr.20220901

ABSTRACT

The prevalent problems associated with heavy metal contamination of the environment particularly underground and surface wastewater compel the need to work on economical means of mitigating the accumulation of heavy metals in the environment by espousing the heavy metal biosorption option. The choice of biosorption in this regard is hinged on the overall cost control of the process and its relative effective outcome compared to other means of diminishing heavy metal accumulation in the environment. Thus, the potential of Garden Egg (*Solanum melongena*) Leaf (GEL) to remove Cd(II), Pb(II) and Zn(II) from aqueous solutions through biosorption was studied. The influence of various process parameters such as pH, contact time, biomass dose and initial metal ion concentration was investigated using batch experiments to determine the efficiency of the biosorbent. The presence of ionizable functional groups (-N-H,-C≡N, C=O and –C-O) in the GEL were analyzed using Fourier Transform Infrared (FT-IR) which are responsible for the binding of the cations. The heat of the sorption process was estimated from Temkin Isothermal model and mean free energy were estimated from Dubinin-Radushkevich (D-R) model. The biosorption process revealed that the best pH for Cd(II), Pb(II) and Zn(II) adsorption was at pH 7. The kinetic studies showed that the biosorption process was best represented by pseudo-second order kinetics having the highest correlation coefficient (R^2) of 0.9398, 0.9339 and 0.9398 for Cd(II), Pb(II) and Zn(II) respectively, among various kinetic models tested. Equilibrium data were better represented by Freundlich isotherm among Langmuir, Freundlich, Temkin and Dubinin-Radushkevich (D–R) absorption isotherms. The Freundlich model appeared to have better regression coefficients among four Equilibrium data tested. The study on the effect of dosage showed that the dosage of the biomass significantly affected the uptake of the metal ions from solution. Thermodynamically, the biosorption of each of the metal ions was endothermic and the order of spontaneity of the biosorption process being Zn > Pb > Cd. However, positive change in entropy was observed for each and the order of disorder is Zn > Cd > Pb. The results indicate that Garden egg (*Solanum melongena*) leaf has a potential for the adsorption of Cd(II), Pb(II) and Zn(II) and could be found useful in the treatment of industrial effluents.

Keywords: Biosorption; Heavy metals; Effluents; Garden egg; Thermodynamics; Kinetics.

INTRODUCTION

The discharge of toxic metals from industrial operations has led to Cd(II), Pb(II) and Zn(II) entering the environment. There is need to control their levels in the environment before they enter food chain. The conventional approach to their removal from effluents is rather expensive with associated demerits. Comparative study on the biosorption of Pb(II), Cd(II) and Zn(II) using Lemon grass (*Cymbopogon citratus*) was investigated under various physicochemical parameters **[1]**.

A wide range of conventional treatment techniques such as chemical precipitation, solvent extraction, electrolyte processes, ion-exchange, reverse osmosis, ultra-filtration, membrane separation, biological systems and adsorption have been reported to be used for removal of heavy metal ions from industrial effluents **[2]**. However, these processes are not economically feasible for small scale industries common in developing economies due to huge capital investment. These have made it imperative to search for alternative methods, which are low cost, simple and sludge free, and environmentally friendly. However, biosorption has been recognized as the alternative technology for efficient and effective removal of these metal ions from solutions. It has been employed using different materials of either animal or plant origin as biosorbents in the removal of toxic metals **[3-8]**.

Cadmium can be regarded as an element of high toxicity and mobility in the environment, and according to the World Health Organisation (WHO), Cd(II) is among the metals of most immediate concern **[9]**. Its harmful effect on man includes renal dysfunction, bone degeneration, lung insufficiency, liver damage and hypertension in humans **[9]**. Cadmium is released into the environment through wastewater disposed from electroplating, smelting alloy and plastic manufacturing, pigments, fertilizers, batteries, mining and metal refining process **[9-11]**.

Zinc is a common metal ion found in effluents of many industries. This metal is an essential element for life and is a micronutrient in trace amounts **[12]**. It is used in industries such as brass manufacture and galvanization etc. However, a chronic exposure to Zn^{2+} is detrimental for human health. At elevated level, zinc can cause damage to the pancreas, upset protein metabolism and cause arteriosclerosis. Consequently, there is need to reduce zinc concentrations in water and water waste to acceptable levels. The toxicological profile of this pollutant has been well documented in literature and its presence in water and waste waters is a potential risk for the environment and public health **[12-13]**.

Lead is currently used in several industries such as batteries, cables, pigments, paints, steels and alloys, metal and plastic industries. The discharge of these industries causes the contamination of the aquatic environment by lead.

MATERIALS & METHODS

Biomass Preparation

Solanum Melongena (Garden Egg Leaf) were collected from a farm located in Isale-Otta Idode, Ago-Iwoye Ogun State, Nigeria. The leaves were carefully collected inside a transparent nylon sun dried immediately and kept dry till time of usage.

Preparation of Solution

All chemicals used in this study were of analytical reagent grade and were used without further purification. Standard solutions of Cd(II), Pb (II) and Zn (II) used for the study were prepared from $Cd(NO_3)_2.4H_2O$, $Pb(NO_3)_2$ and $Zn(NO_3)_2\ 6H_2O$ respectively. The solutions with different concentrations of the metal ions were being worked out and prepared by appropriate dilutions of the stock solution immediately prior to their use with distilled water.

The initial pH of the solution was adjusted accordingly with a pocket-sized pH meter. The concentration before and after absorption of each metal ion was determined using a

BUCK SCIENTIFIC Atomic Absorption Spectrophotometer (AAS) MODEL 210/21/VGP with deuterium background corrector. Fourier transform infrared (FT – IR) spectra of dried unloaded biomass and metal loaded biomass were recorded at 400 – 4000cm^{-1} using a Shimadzu FT–IR model 8400s spectrophotometer.

Batch Biosorption Study

The biosorption study was conducted by batch experiments under different conditions for a period in a glass tube. The biosorption studies were conducted at 27 ^{0}C using thermostated water bath to determine the effect of pH, contact time, biosorbent dosage, initial metal ion concentration and temperature on the biosorption of each metal ion. The residual metal ions were analysed using a BUCK SCIENTIFIC Atomic Absorption Spectrophotometer (AAS) MODEL 210/211VGP. The amount of metal ion biosorbed from solution was determined by difference and the mean value calculated.

Effect of Initial Concentration on Biosorption

Batch biosorption study of metal ion was carried out using a concentration range of 10 - 100 mgL^{-1}. This was done by contacting 0.5g of the leaf with 25ml of each solution at optimal pH. Two glass tubes were used for each concentration. The tubes were left in a thermostated water bath maintained at 27^{0}C for the predetermined optimum time. The leaf was removed from the solution and the concentration of residual metal ion in each solution was determined.

Effect of pH on Biosorption

This was done by contacting 0.5 g of garden egg leaf into 25 ml of 100 mg/L of each of the metal ions solution in glass tubes. The pH of each solution was adjusted to the desired value by drop wise addition of 0.1 M HNO_3 to decrease the pH and 0.1 M NaOH to increase the pH using a pH meter. The glass tubes containing the mixture were suspended in a water bath for three hours. The biomass was removed from the solution by decantation. The residual metal ions concentration in the solution were analysed using Atomic Absorption Spectrophotometer (AAS).
The initial pH values of the metal solutions were adjusted before mixing with the biosorbent.

Effect of Temperature on Biosoprtion

The batch biosorption process was studied at different temperatures with the range 20-50^{0}C to investigate the effect of temperature on the biosorption process.
This was done by contacting 0.5g of *Solanum melongena* leaf with 25ml of 100mgL^{1} of metal ion solution at the optimal pH and time.

Statistical Analysis

The curve fittings of the data obtained were performed using Microcal Origin 8.0 software.

RESULT AND DISCUSSION

Physical Characterization of *Solanum Melongena* leaf

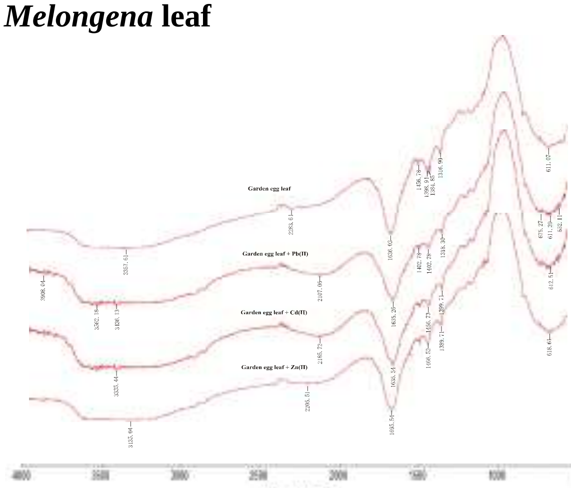

Figure. 1: FTIR Spectra of the free and metal bound garden egg leaf.

Biomass depends upon the chemical reactivity of functional groups at the surface. The FTIR spectra of dried unloaded and metal-loaded leaf were taken to obtain information on the

nature of possible interactions between the functional groups of *Solanum Melongena* leaf biomass and the metal ions as presented in Figure 1. Several peaks were observed from the spectra indicating that *Solanum Melongena* leaf is composed of various functional groups which are responsible for binding of the cations. The IR spectra pattern absorptions indicative of the existence of the -NH, -C≡N, C=O and -C-O at 3357cm^{-1}, 2283cm^{-1}, 1636cm^{-1} and 1398cm^{-1} respectively as shown in Figure 1. These bands are due to the functional groups of *Solanum Melongena* leaf that participate in the biosorption of Cd(II), Pb(II) and Zn(II).
On comparison, there are clear band shifts and decrease in intensity between the of bands of the unloaded biomass and the metal-bound biomass as presented in Table 1. The FTIR spectra of the *Solanum Melongena* leaf biomass indicated slight changes in the absorption peak frequencies since the binding of the metal ions causes reduction in absorption frequencies. These observed shifts in absorbance implies that there were metal binding processes taking place on the active sites of the biomass. Analysis of the FTIR spectra showed the presence of ionizable functional groups (C =O, O-H, NH_2) which are able to interact with cations **[6, 14-15]**. This implies that these functional groups would serve in the removal of positively charged ions from solution.

Effect of Solution pH on Metal ion Biosorption

The pH of the solution usually plays an important role in the biosorption of the metal ion **[7]**. It is an important parameter governing the uptake of heavy metals by biosorption process as it not only affects metal species in solution, but also influences the surface properties of biosorbents in terms of dissociation of binding sites and surface charge.

Table 1: FT–IR Spectral characteristics of *Solanum Melongena* leaf before and after Biosorption of Cd(II), Pb(II) and Zn(II).

Metal ion	FTIR absorption peaks (cm^{-1})			
	Before	**After**	**Difference**	**Assignment**
Pb(II)	3357.61	3136.31	221.30	N-H Stretching
Cd(II)		3335.44	22.17	N-H Stretching
Zn(II)		3135.04	222.57	N-H Stretching
Pb(II)	2283.61	2107.06	176.55	C≡N Stretching
Cd(II)		2185.72	97.89	C≡N Stretching
Zn(II)		2205.57	78.04	C≡N Stretching
Pb(II)	1636.02	1635.26	0.76	C=O Stretching
Cd(II)		1635.57	0.45	C=O Stretching
Zn(II)		1695.51	-59.49	C=O Stretching
Pb(II)	1398.91	1402.78	-3.87	C-O Stretching
Cd(II)		1456.73	-57.82	C-O Stretching
Zn(II)		1466.52	-67.61	C-O Stretching

Figure 2 shows the variation of the metal ions biosorbed on *Solanun Melongena* leaf at various solution pH values. For the three metal ions, the biosorption increased as the pH increased from pH 1 to pH 4. The increase observed in the biosorption with increase in pH implies that ion-exchange process is involved.

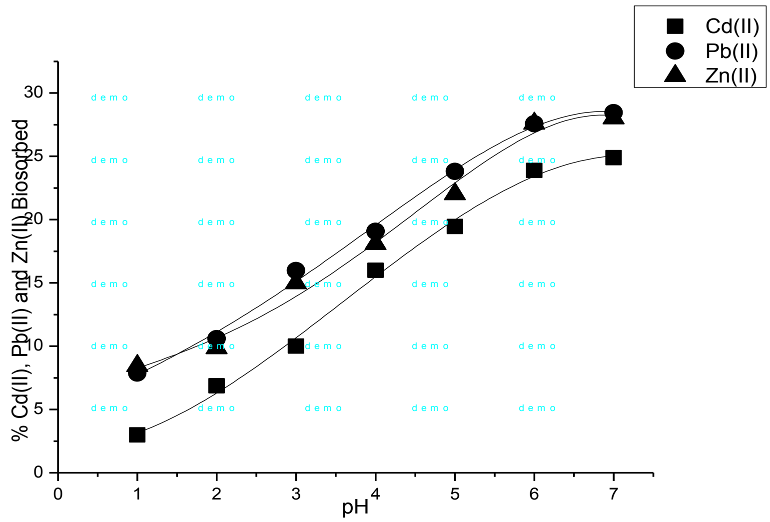

Figure 2: pH dependence profile for the biosorption of Cd(II), Pb(II) and Zn(II) ions using Garden egg leaf (GEL).

The reaction involved the biosorption of metal ion (represented as M^{x+}) from the liquid phase to the solid phase, the biosorbent with lone pair of electron (represented as A), and can be considered as a reversible reaction with an equilibrium being made between the two phases as schematically shown below for a divalent metal ion in solution:

$$\ddot{A} + M^{2+} \rightleftharpoons A - M \qquad (1)$$

Biosorption Kinetics

The kinetics of sorption is probably the most important factors in predicting the rate at which sorption takes place for a given system. However, sorption kinetics shows a large dependence on the physical and/or chemical characteristics of the sorbent material, which also influences the sorption process and the mechanism. The study of biosorption kinetics describes the solute uptake rate and evidently these rate controls the residence time of biosorbate uptake at the solid- solution surface.

Figure 3 shows the effect of contact time of Cd(II), Pb(II) and Zn(II) on garden egg leaf. The data obtained from the adsorption of the three metal ions onto garden egg showed that the biosorption increased with increasing contact time. It can be seen that the biosorption process occurred slowly in 5 mins and the metal ion uptake capacity value reach equilibrium at 160 mins for both Cd(II) and Zn(II), 180 mins for Pb(II). Therefore, 160 mins was selected as equilibrium time for biosorption process for Cd(II), Zn(II) and 180 mins for Pb(II).

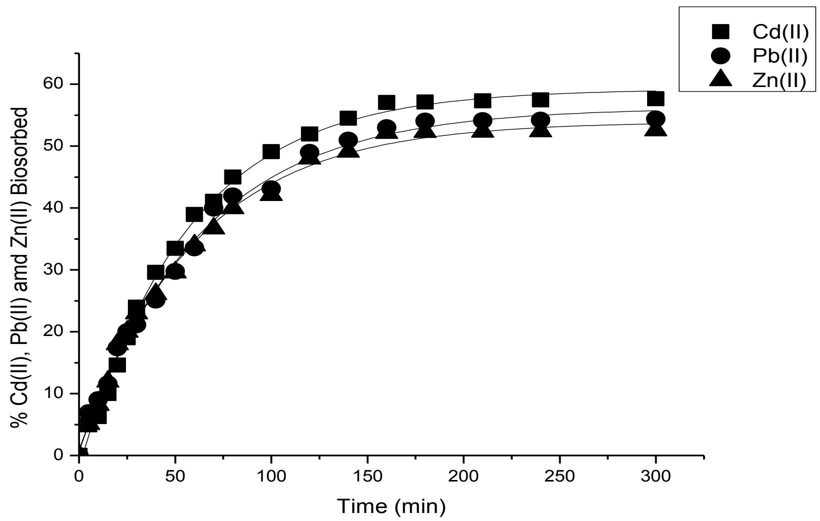

Figure 3: Contact Time dependence profile for the biosorption of Cd(II), Pb(II) and Zn(II) using garden egg leaf

Several kinetic models are needed to establish the mechanism of a biosorption process. To investigate the kinetics of the biosoprtion of these metal ions on *Solanum Melongena* leaf, four kinetic models were employed. These are the pseudo–first order, pseudo-second order, elovich and intraparticle diffusion kinetic models.

The pseudo-first order was studied with Lagergren equation which considers that the rate of occupation of the biosorption site is proportional to the number of the occupied sites.

$$rate = -d[A]/dt = k[A]^n \quad (2)$$

This can also be written as

$$d/dt\, q_t = k_1(q_e - q_t) \quad (3)$$

Integrating between the limits q_t = 0 at t =0 and q_t= q_t at t =t, we obtain

$$\log[q_e/(q_e - q_t)] = (k_1/2.303)t \quad (4)$$

The linearized form is:

$$\log(q_e - q_t) = \log q_e - (k_1/2.303)t \quad (5)$$

Where k_1 (min^{-1}) is the rate constant of pseudo- first order biosorption rate constant, q_e and q_t are amounts of metal ions biosorbed (mgg^{-1}) on biosorption at equilibrium and at time t(s) respectively. The rate constants, K_1 were calculated from the plots of log (q_e – q_t) versus t for different concentrations of Cd(II), Pb(II) and Zn(II) . Since the correlation coefficient was lower than 0.99 and disagreement between experimental and calculated values of equilibrium biosorption capacity (q_e) was in question, the biosorption of Cd(II), Pb(II) and Zn(II) in garden egg leaf was not well fitted by pseudo-first order kinetic model (Figure 4.4). The kinetic parameters are presented as:

$$(d/d_t)q_t = k_2(q_e - q_t)^2 \quad (6)$$

On integrating between boundary conditions, we have

$$1/q_e - q_t = 1/q_e + k_2 t \quad (7)$$

The linearized form is:

$$t/q_t = 1/k_2 q_e^2 + (1/q_e)t \quad (8)$$

Where k_2 is the equilibrium rate constant of pseudo–second-order rate constant ($gmg^{-1}min^{-}$

[1]). The experimental and calculated values of q_e showed a good agreement. The correlative coefficients for the pseudo– second order kinetic model were higher than the pseudo–first order, and therefore good agreement between experimental and calculated values of equilibrium biosorption capacity (q_e) indicating the applicability of this kinetic model for the biosorption process of Cd(II), Pb(II) and Zn(II) on garden egg leaf.

However, plots of t versus t/q_t showed good fitness of experimental data with the pseudo–second order kinetic model as presented in Figure 4.

The data were also subjected to the Elovish Kinetic model given by:

$$q_t = A + B \ln t \qquad (9)$$

The Intra particle diffusion model was equally used to analyse the data. The correlation coefficients obtained were found to be highest for the pseudo-second-order kinetics as it was found to be in excess of 0.93 as presented in Table 2

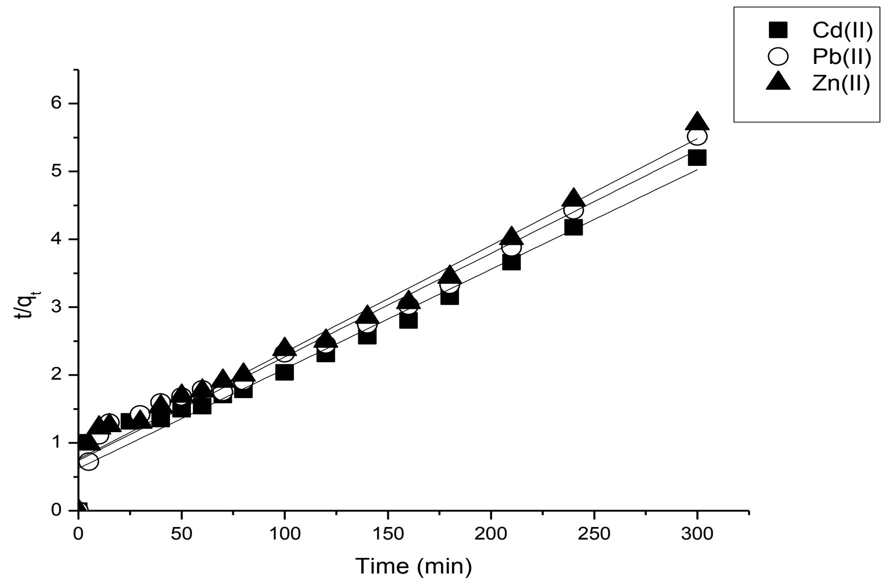

Figure 4: Pseudo-second order kinetic plot for the biosorption of Cd(II), Pb(II) and Zn(II) ions using *Solanum Melongena* leaf

On comparison of the values of R^2 for the experimental points, the pseudo-second-order kinetic model is the best kinetic model to predict the dynamic biosorption of Cd(II), Pb(II) and Zn(II) on *Solanum Melongena* leaf. The result shows that the rate of biosorption of the metal ions is of the order Cd(II), Pb(II) and Zn(II). The differences observed in the rate of biosorption as well as in the biosorption capacity may be accounted for in terms of differences in ionic charges and hydrated ionic sizes of the ions in solution.

Biosorption Isotherm

Figure 5 reports the biosorption isotherms of Cd(II), Pb(II) and Zn(II) on *Solanum Melongena* leaf. The equilibrium biosorption capacity increases with increase in metal ion concentration.

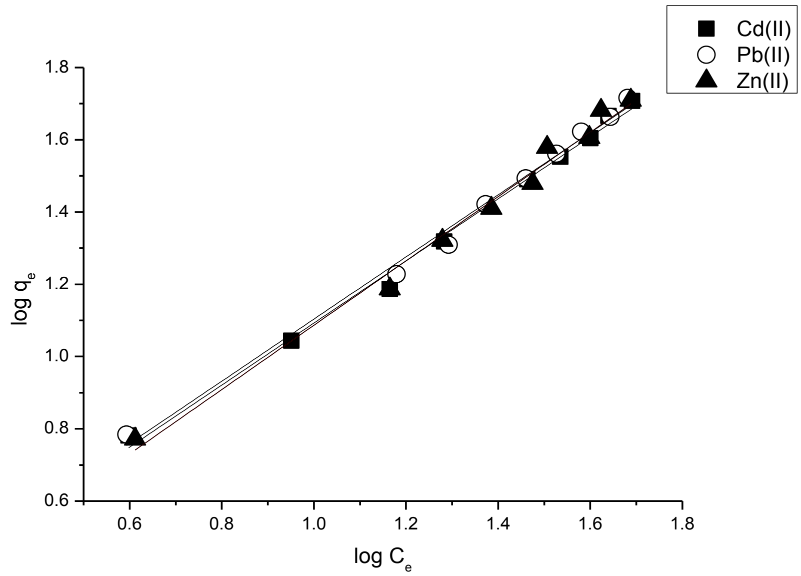

Figure 5: Freundlich isothermal plot for the biosorption of Cd(II), Pb(II) and Zn(II) using *Solanum Melongena* leaf.

The Freundlich and Langmuir isotherms were employed to calculate the biosorption capacity. The Freundlich isotherm is an empirical equation describing adsorption onto a heterogeneous surface. The Freundlich isotherm is expressed as:

$$\log\Gamma = \frac{1}{n}\log C_e + \log K_f \qquad (10)$$

Where K and $\frac{1}{n}$ are the Freundlich constants related to the biosorption capacity and biosorption intensity of the biosorbent respectively.

The linear form of the Langmuir equation is expressed as:

$$\frac{1}{\Gamma} = \frac{1}{b_m}\frac{1}{C_e} + \frac{1}{\Gamma_m} \qquad (11)$$

Where Γ, Γ_m and b_m are the Langmuir parameter. The Freundlich isothermal parameters for the biosorption are presented in Table 3.

Table 2: Parameters of the Pseudo-Second-Oder Kinetic Model for the Biosorption of Cd(II), Pb(II) and Zn(II) using *Solanun Melongena* leaf

Metal ion	K_2 (g. mg^{-1} .min^{-1})	q_e (mg. g^{-1})	R^2	S.D.
Cd(II)	3.46 x 10^{-4}	68.12	0.9398	0.09
Pb(II)	3.14 x 10^{-4}	65.53	0.9339	0.09
Zn(II)	3.26 x 10^{-4}	63.49	0.9398	0.09

Table 3: Freundlich Isothermal Parameters the Biosorption of Cd(II), Pb(II) and Zn(II) using *Solanum Melongena* leaf

Metal ion	1/n	K_f	R^2	S.D.
Cd(II)	0.8929	1.57	0.9738	0.080
Pb(II)	0.9009	1.57	0.9738	0.080
Zn(II)	0.8621	2.09	0.9827	0.051

The results show that the regression coefficients obtained for Freundlich isotherm are higher than for Langmuir isotherm. This implies that the biosorption is assumed to be a monolayer sorption with a heterogenous energetic distribution of active sites, accompanied by interactions between biosorbed molecules.

Biosorption Efficiency

The result of the study on the effect of initial metal ion concentration on biosorption efficiency is shown in Figure 6. The plots show that the biosorption efficiency of the biomass reduces with increase in the initial metal ion concentration of Cd(II) which might be due to the fixed number of binding sites in the biosorbent having more ions than at lower concentration. On the other hand, the biosorpotion efficiency increased with increase in initial metal ion concentration for Cd(II) and Zn (II). The biosorption efficiency (E) for each metal ion was calculated as

$$E = 100\frac{(C_i - C_e)}{C_i} \quad (12)$$

Effect of Biomass Dosage on Biosorption

The effect of biomass dosage on biosorption capacity is reported in Figure 7. The general trend of increase in metal ion absorbed with increase in biomass dosage indicates an increase uptake due to more binding sites on the biomass available for biosorption. It was found that biosorption capacity increases with increase in dosage of the biosorption. This is due to the fact that increase in biomass dosage leads to increase in the number of active sites available for biosorption. Hence, biosorbent will be less when amount of biosorbent is increased. The difference in absorption capacity q (mg g-1) at the same initial metal ion concentration and contact time may also be attributed to the difference in their chemical affinities and ion exchange capacity, with respect to the chemical functional group on the surface of the biosorbent.

Biosorption Thermodynamics

The biosorption of metal ions may involve chemical bond formation and ion exchange since the temperature is a main parameter affecting them. The variation of temperature affects the biosorption of metal ions onto solid surfaces of biomass since the biosorption process is a reversible one. The nature of each side of the equilibrium determines the effect temperature has on the position of equilibrium. The side that is endothermic is favoured by increase in temperature while the contrary holds for the exothermic side. The corresponding free energy change was calculated from the relation **[15]**.

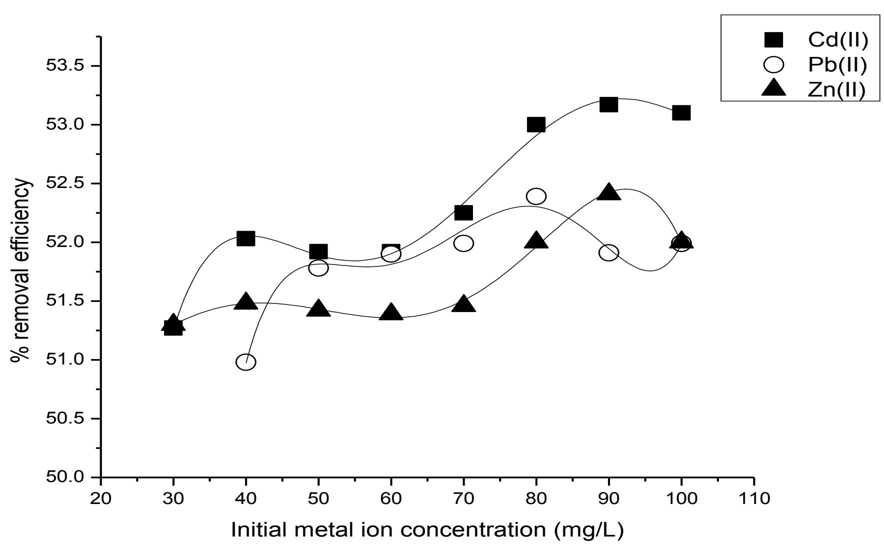

Figure 6: **Efficiency Plot for the Biosorption of Cd(II), Pb(II) and Zn(II) ions using *Solanum Melongena* leaf.**

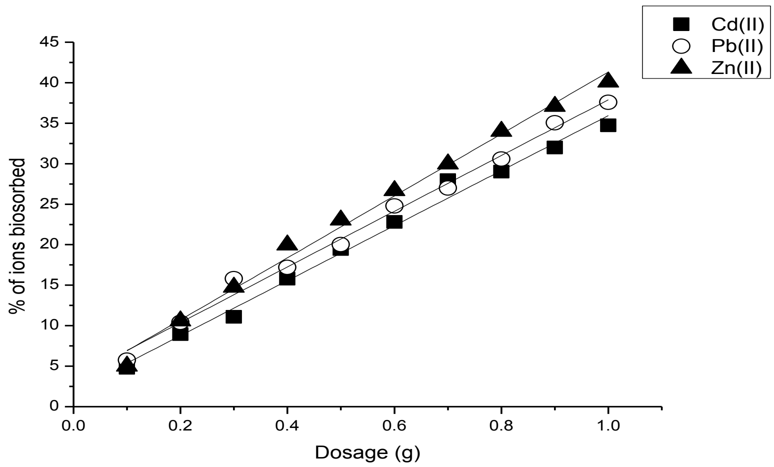

Figure 7: Effect of Biomass Dosage on the biosorption of Cd(II), Pb(II) and Zn(II) using S*olanum melongena* leaf

$$\Delta G^{\circ} = -RT \ln K_c \qquad (13)$$

Where T (K) is the absolute temperature. The expression for the equilibrium constant (K_c) was calculated from the following relationship:

$$K_c = C_{ad} / C_e \qquad (14)$$

Where C_e and C_{ad} are the equilibrium concentrations of metal ions (mgL^{-1}) in solution and on biosorption respectively, consequently the thermodynamic behaviour of the biosorption of Cd(II), Pb(II) and Zn(II) onto S*olanum melongena* leaf was evaluated through the change in free energy (ΔG^0), enthalphy (ΔH^O) and entropy (ΔS^O). The thermodynamic parameters like enthalpy and entropy were calculated using van't Hoff equation **[6]**. The change in free energy is related to other thermodynamic properties as:

$$\Delta G^{\circ} = \Delta H^{\circ} - T\Delta S^{\circ} \qquad (15)$$

$$\ln K_c = \Delta S^o / R - \Delta H^o / RT \qquad (16)$$

Where T is the absolute temperature (K); R is the gas constant (8.314 J $mol^{-1}K^{-1}$). The changes in enthalpy and entropy calculated from the intercept and slope of the plot of T verses ΔG^0 as presented in Figure 4.15 while the thermodynamic parameters are presented in Table 4.

The thermodynamic parameters (free energy (ΔG°), enthalphy (ΔH^o) and entropy (ΔS^o) for the biosorption of the metal ions were determined by the application of Equations 15 and 16 as presented in Table 4.

In general, the change of standard free energy for physisorption is in the range of -20 to 0 kJ mol^{-1} and for chemisorption varies between -80 and -400 $kJmol^{-1}$**[16]**. In the present study, the overall (as shown in Figure 8) has values ranging from -0.1 to -2.9 kJ mol^{-1}. These results correspond to a spontaneous physical adsorption of the metal ions, indicating that this system does not gain energy from external resource **[16-17]**.

Table 4: Thermodynamic Parameters for Biosorption of Cd(II), Pb(II) and Zn(II) onto S*olanum melongena* lea

Metal ion	ΔH^o **(kJ mol-1)**	ΔS^o **(J $mol^{-1}k^{-1}$)**	A **(kJ mol^{-1}) (303K)**	A **(kJ mol^{-1}) (318K)**
Cd(II)	+25.37	+87.08	27.89	28.02
Pb(II)	+24.22	+82.74	26.74	26.86
Zn(II)	+26.16	+89.19	28.68	28.80

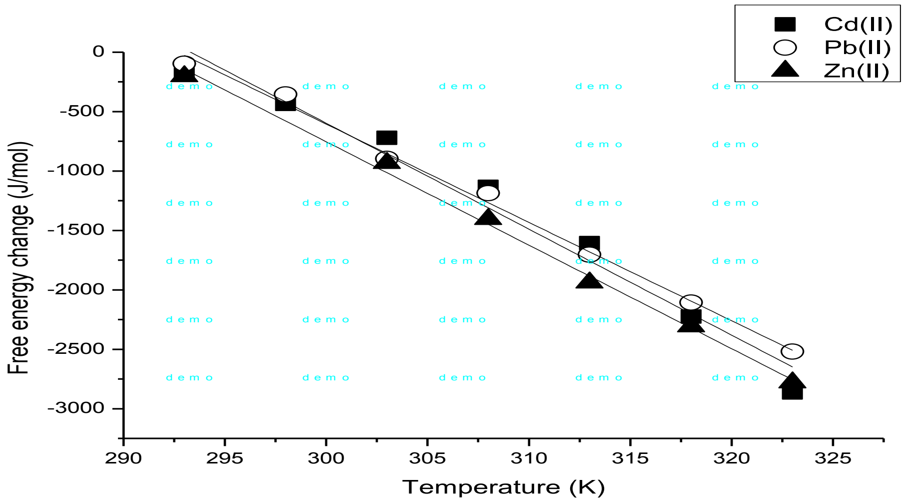

Figure 8: Thermodynamic plots for the biosorption of Cd(II), Pb(II) and Zn(II) using garden egg leaf

The decrease in with increase in temperature indicates more efficient biosorption at higher temperature. The order of spontaneity of the biosorption process was found to be Cd(II), Pb(II) and Zn(II) similar to the order reported for the biosorption of these metal ions with banana leaf **[8]**. The positive value to enthalpy change indicates the endothermic nature of the biosorption process while negative value implies exothermic process. The positive values of for the biosorption of Cd(II) and Zn(II) suggest an endothermic nature of each biosorption process. This is also supported by the increase in the value of biosorption capacity of the biosorbent with rise in temperature. The positive value of indicate the presence of an energy barrier in the biosorption process.

Energies of activation, A below 42 kJ/mol indicate diffusion-controlled processes, and higher values give chemical reaction-based processes. Therefore, energy of activation, A, has been calculated as per the following reaction:

$$A = \Delta H^{o} + RT \qquad (17)$$

The values of A at two different temperatures have been tabulated in Table 4. In this study, the activation energy (A) values were less than 42 $kJmol^{-1}$ indicating diffusion-controlled adsorption processes.

CONCLUSION

While a number of works on heavy metals exist in literature including ameliorative measures **[18-20]**, we have studied the biosorption of Cd(II), Pb(II) and Zn(II) by S*olanum melongena* leaf under the various conditions. The biosorption of each was influenced by each of the parameters investigated. The pH has much effect on the biosorption of these metal ions from aqueous solutions. The rate of the biosorption of these metal ions followed pseudo-second-order kinetic model. The sorption isotherms of these metal ions onto the biosorbent are better represented by the Freundlich isotherm model. The thermodynamic study shows that the biosorption of each of Cd(II), Pb(II) and Zn(II) was spontaneous in the order Cd(II) > Pb(II) > > Zn(II). The order of disorderliness being Zn(II) > Cd(II) > Pb (II).

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How to cite this article: Ahmed Adebanjo, Adesola Babarinde, K. Moberuagba et.al. Biosorption of Cd (II), Pb (II) and Zn (II) from aqueous solutions using garden egg (Solanum Melongena) leaf: equilibrium, kinetics and thermodynamics. *International Journal of Research and Review*. 2022; 9(9): 1-12. DOI: *https://doi.org/10.52403/ijrr.20220901*

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