Biosorption Characteristics of Zinc (II) from Aqueous Solution by Luffa Cylindrica

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Abstract

The biosorption of zinc ions from synthetic aqueous solutions using physico-chemically treated Luffa Cylindrica was considered, investigating the kinetics and equilibrium of the process batchwise. The biosorption characteristics of Zn (II) ions were studied with respect to well-established effective parameters including pH, temperature, biosorbent dosage, initial metal ion concentration and contact time. The uptake of Zn (II) increased with an increase of the pH and of the concentration. Biosorption equilibrium was established in about 90 min.

The Zn (II) biosorption data were analyzed using the first and the second order kinetic models as well as intra-particular rate expressions. The second-order equation was the most appropriate to predict the biosorption capacities of the fungal preparation.

The biosorption data obtained at pH =6 followed reasonably well both the Langmuir and the Freundlich isotherm models.

The thermodynamic parameters, Gibbs free energy $\Delta G°$, enthalpy $\Delta H°$ and entropy $\Delta S°$ changes, were also calculated, and the values indicated that the biosorption process was exothermic, spontaneous and feasible in the temperature range of 303–323 K.

It was concluded that Luffa Cylindrica powder can be used as an effective, low cost, and environmentally friendly biosorbent for the removal of Zn (II) ions from aqueous solution.

Keywords: Biosorption; Zinc; Isotherms; Luffa sponge.

I. Introduction

Industrial wastewaters generated from industrial activities may contain different toxic heavy metals [1] such as Zinc which is one of the most important metals often found in effluents discharged from industries involved in acid mine drainage, galvanizing plants and municipal wastewater treatment plants [2]. It is important for the physiological functions of living tissue and regulates many biochemical processes. However, too much zinc can cause eminent health problems, such as stomach cramps, skin irritations, vomiting and anemia [3].Therefore, selective separation of Zn (II) from aqueous solution, especially wastewater has drawn more and more attention in recent years. There are many techniques for Zn (II) removal, such as chemical precipitation, ion exchange, membrane filtration, electrolytic methods and reverse osmosis [4]. Among all of these methods biosorption has been preferred due to its low cost and the high-quality of the treated effluents especially for well-designed biosorption processes. Biosorption using agricultural by-products has proven to be an economical, efficient and realistic method for removal of different pollutants such as Zinc [5]. Luffa Cylindrica (LC), mainly consists of cellulose, hemicelluloses and lignin; of composition (60, 30 and 10% by weight, respectively) [6]. Cellulose structure consists of a monomer unit of a -d-glucopyranose linked through 1, 4-glucosidic linkage. Cellulose is renewable, cheap and with low density, it exhibits better processing flexibility and is a biodegradable material. Cellulose is a highly functionalized, linear stiff chain homopolymer, characterized by its hydrophobicity, chirality, biodegradability and broad chemical modifying capacity [7]. Because of its unique structure, LC has been used as an efficient adsorbent or as a carrier for immobilization of some microalgae cells for the removal of water pollutants [8, 9].

In this study, luffa Cylindrica, which was an agricultural waste product, was used as a biosorbent for the removal of Zn (II) ions. The luffa sponge was characterized by FTIR spectroscopy to identify the type of chemical bonds in the present molecules and the chemical composition and proximate analysis were determined using appropriate techniques. The uptake of Zn (II) ions by LC was investigated under different experimental conditions, including pH, temperature, biosorption time, and initial concentration of the Zn (II) ions. The experimental data were analyzed using Langmuir and Freundlich models. In addition, kinetic and thermodynamic parameters and maximum biosorption capability were calculated and compared with those of some current sorbents.

II. Experimental

A. Adsorbent

The Luffa sponges were obtained from matured dried fruit of Luffa Cylindrica. The sponge was cut into pieces, soaked in boiling water for 3 min, thoroughly washed under tap water, and left for 24 h in distilled water, changed 3-4 times. The sponge discs were oven dried at 70°C and stored in desiccators ready for further use.

B. Adsorbate

All the chemicals used were of analytical reagent (AR) grade. Stock solution of 100mg/L of Zinc (II) was prepared from $Zn (NO3)_2/6H2O$ using double distilled water. Desired test solutions of zinc (II) ions were prepared using appropriate subsequent dilutions of the stock solution. The range of concentrations of zinc (II) ions prepared from standard solution varies between 10and 100mg/L. Before mixing the adsorbent, the pH of each test solution was adjusted to the required value with 0.1 M NaOH or 0.1 M H_2SO4.

C. Analysis

The concentrations of zinc in the solutions before and after equilibrium were determined by AAS6300Atomic absorption spectrometer (Shimadzu, Japan). The pH of the solution was measured with a WTW pH meter using a combined glass electrode. Fourier Transform Infrared Spectroscopy, FTIR (IRPrestige-21, Shimadzu, Japan) was used to identify the different chemical functional groups present in the LC and also to determine the functional groups which are responsible for the metal binding with the LC. The analysis was carried out using KBr and the spectral range varying from 400to 40cm. The FTIR spectra of the free unloaded LC were compared with those obtained for LC loaded with zinc. Also, MO (NOVEX, HOLLAND) was employed for the observation of the surface microporous structure of LC before and after loading with LC.

D. Biosorption experiments

The batch studies were performed to study the removal of zinc from aqueous solution. A predetermined amount of adsorbent was added to 100 mL solution of known concentration in 250 mL Erlenmeyer flasks at temperature 25 °C and agitated at 150 rpm on thermostatic orbital shaker (Scigenics Biotech ORBITEK) for 90 min. At predetermined time interval, the adsorbent was separated by centrifugation at 4000 rpm for 10 min. The residual zinc concentration in the supernatant was determined as stated before. The zinc removal percent was calculated for each run by the following equation 1:

$$R(\%) = \frac{(C_0 - C_e)*100}{C_0} \quad (1)$$

with C_0 and C_e are the initial and final concentrations of zinc in the solution, respectively, in mg/L.

The zinc uptake loading capacity (mg/g) of LC for each concentration of zinc at equilibrium was determined as:

$q_e = \frac{(C_0 - C_e).V}{m}$ (2)

Where Ce is the equilibrium concentrations of the zinc (mg/L) in solution, m is the dose of adsorbent (g/L).

III. Results and discussion

A. Luffa Cylindrica powder characterization

- *FTIR analysis*

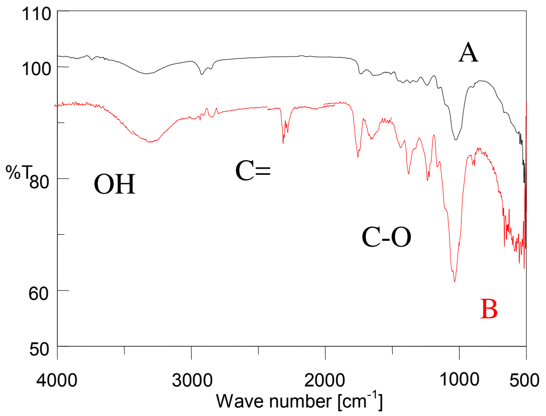

Figure 1. FTIR spectrum of the L. Cylindrica fibers (a) without adsorbed Zn(II),(b) with adsorbed Zn(II).

FTIR spectroscopy was applied to identify the functional groups of LC straw responsible for metal biosorption. To reveal the systematic changes in the spectral features upon reaction with metal ions, FTIR spectra were obtained for the sample after reacting with zinc (Figure1). By comparing the FTIR spectra of LC before and after biosorption, there were remarkable shifts in some bands (Table 1). These bands are the function groups of LC participate in Zn(II) biosorption.

Table 1:.FTIR spectral characteristics of luffa cylindrical before and after biosorption of Zn(II).

Peak	Transmission band (cm^{-1})		Assignment
	Before biosorption	After biosorption	
1 2 3 4	3340 2921 1650 1000	3326 2923 2348 1100	Stretching vibration of OH Aliphatic C-H group C=C C-O

A weak band at 2920cm^{-1} refers to the deformation in an axial CH methylene (CH_2) and peak axial deformation of the carboxyl C=O bond, which may be amide groups or carboxylic groups, at 1650cm-[1], acetal group (carbonyl of aldoses). The CO stretching peak at 1743 cm-1-suggests the angular deformation in the plane, a characteristic of compounds with ester groups and carboxyl. The intensity of the band at 1743 cm^{-1}-(C=O acetal) was different in the zinc sponge with and without zinc adsorbed on its surface. The band in 1000 cm^{-1} refers to C-OH stretching of the primary alcohol in typical polysaccharides. It is of great importance to identify functional groups present in the adsorptive material to observe the formation of complexes between the metal ions and the functional groups contained in the material by means of electrostatic interaction.

- *Results of Point of Zero Charge (pH_{PZC}) Determination*

The point of intersection of ΔpH versus pH_0 showed that the pH_{ZPC} of LFC occurred at pH 5.2 as shown in Figure 2. This showed that at pH less than 5.2 the surface of LFC is predominated by positive charges while at pH greater than 5.2, the surface is predominated by negative charges.

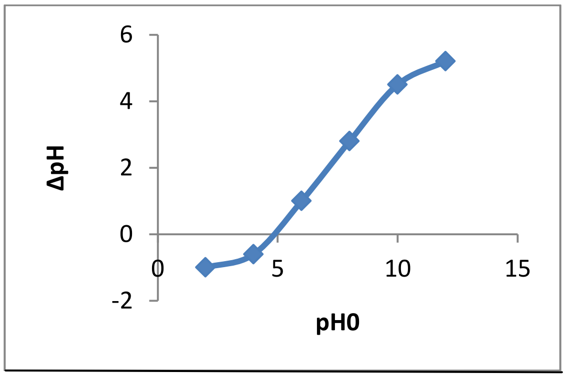

Figure 2: Results of the determination of the pH_{ZPC}.

B. Effect of initial concentration

To evaluate the biosorption characteristics of LC for zinc ions, the change of biosorption capacity with time for different initial solution concentrations has been investigated. A series of experiments were undertaken by varying the initial Zn concentration in the range 10 –60 mg/L on removal kinetics of Zn^{2+} from the solution. The result showed that biosorption of Zn (II) by luffa increases as the initial Zn (II) concentration increased. The contact time required to reach the equilibrium of Zn(II) solution within 90 min. However, the experimental data were measured at 120 min to confirm the complete equilibrium was reached.

C. Effect of pH

The pH is an important process parameter on biosorption of metal ions from aqueous solutions since it is responsible for protonation of metal binding sites. It was found that Zn (II) uptake by LC was a function of solution pH. The uptake of zinc increased with the increase in pH from 2.to 6.0. At lower pH values zinc removal was inhibited, possibly as a result of the competition between hydrogen and zinc ions on the biosorption sites, with an apparent preponderance of hydrogen ions, which restricts the approach of metal cations as in consequence of the repulsive force. As the pH increased, the active sites in (LC) would be exposed, increasing the negative charge density on the LC surface, increasing the attraction of metallic ions with positive charge and allowing the biosorption onto the LC surface. In this study, these Zinc cations at around pH 6 would be expected to interact more strongly with the negatively charged binding sites in the biosorbent. As a result, the optimum pH for zinc biosorption was found as 6 (figure 3) and the other biosorption experiments were performed at this pH value.

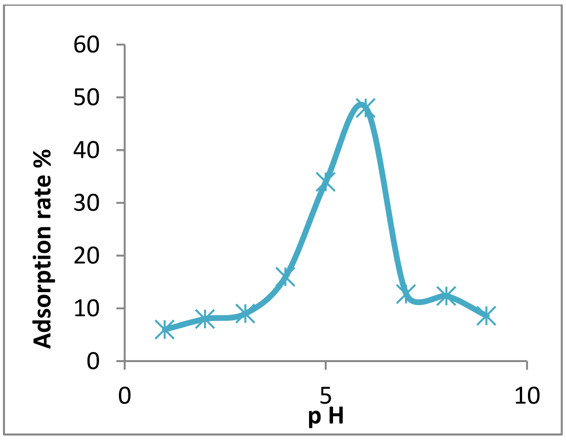

Figure.3: Effect of pH on the biosorption of Zn(II) onto LC biosorbent dose10g/L; temperature25°C; initial zinc concentration 10 mg/L.

D. Biosorption Kinetics

The biosorption kinetic data of zinc are analyzed using three kinetics models were applied mainly, pseudo-first order, pseudo-second order and, intra-particle diffusion model rate equations Lagergren suggests the pseudo-first order kinetics rate equation [10], which is expressed as follows:

$$\frac{dqt}{dt} = k_1(\text{qe} - \text{qt}) \quad (3)$$

Where k_1 is the pseudo-first order rate constant, qe represents biosorption capacity. The integrating rate law by applying the initial condition of t = 0 to t and qt = 0 to qt, Eq.() becomes:

$$\log(q_e - q_t) = \log q_e - K_I.t/2.303 \quad (4)$$

Where, qe and qt both (mg/g) are the amount of Zn(II) adsorbed per unit of mass of LC at equilibrium and time t. respectively, and K_1 the rate constant (1/min). The value for the K_1 was calculated from the slope of the linear plot of log (qe - qt) versus t Figure 5. . The K_1 values and correlation coefficients R^2 are given in Table 3. The pseudo-second order reaction rate equation used by Ho et al [11] to study the kinetics of biosorption of heavy metals on peat. This model was also applied to assess the kinetics of biosorption of Zn (II) on LC. The equation is as follows:

$$\frac{dqt}{dt} = K_2(qe - qt)^2 \quad (5)$$

where k_2 is the rate constant of pseudo second-order biosorption. The integrating rate law Eq.(6), after applying the initial conditions [12], and rearranging it gives as linearized form of pseudo second-order rate kinetics expressed as follows:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (6)$$

Where, qe and qt both (mg/g) are the amount of Zn(II) adsorbed per unit of mass of LC at equilibrium and time t. respectively, and K_2 is the rate constant of pseudo second order biosorption (g/mg min). The kinetics plots between t/qt versus t were plotted for the different initial concentrations Figure 6. Slope and intercept values were solved to give the value of pseudo-second order rate constant (Table 2). Figure 9 and Table 3 shows that highly significant regression line (R^2>99) and the data were well fitted only to the pseudo second-order rate equation. The straight line was obtained indicating that the process follow a pseudo second order kinetics for various concentration of Zn(II). While the initial Zn(II) concentration increases from 10 mg/L to 60mg/L, the biosorption capacity, q_{exp}, increase from 0.97to 3.06 mg/g. This indicates that the initial Zn(II) concentration plays a key role in determining the biosorption capacity of Zn(II) on Luffa Cylindrica . It is also observed in Table 3 that when initial Zn(II) concentration increase from 10 mg/L to 60 mg/L, the rate constant, k_2 decrease from 0.05 to 0.0099 g/mg min. The pseudo-second-order and Elovich kinetic models could not identify the diffusion mechanism and the kinetic results were then analyzed by using the intra-particle diffusion model. In the model developed by Weber and Morris [13], McKay and Poots, the initial rate of intra-particle diffusion is calculated by linearization of Equation 7:

$$qt = K_i t^{1/2} + C \qquad (7)$$

Where, q is the amount of Zn (II) adsorbed (mg/g) at time t, ki the intra-particle diffusion constant (mg/g min$^{1/2}$), and C is the intercept.

The values of ki indicate an enhancement in the rate of biosorption. It is observed in Table 3 that when initial Zn(II) concentration increase from 10 mg/L to 60 mg/L, the intra-particle diffusion constant, ki increase from 1.109 to0.305 g/mg min respectively.

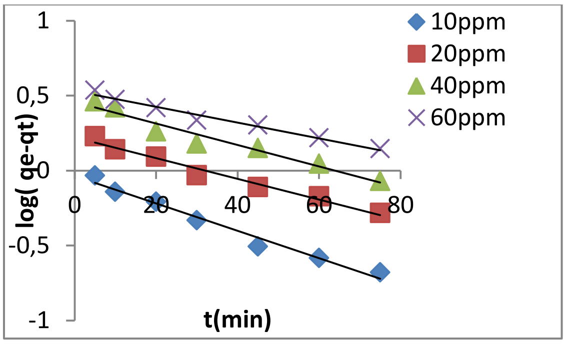

Figure.4: Pseudo-first order kinetic model for Zn (II) biosorption onto LC

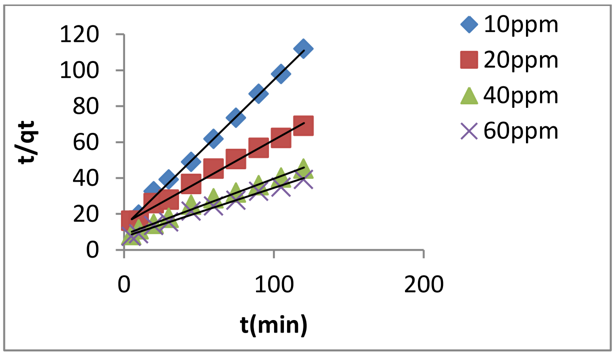

Figure.5: Pseudo-second order kinetic model for Zn (II) biosorption onto LC

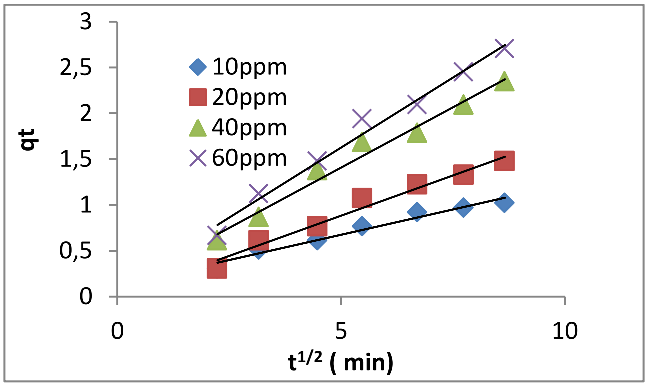

Figure.6: intra-particle diffusion kinetic model for Zn(II) biosorption onto LC

Table 2: Biosorption kinetic model parameters for zinc (II) by **LC.**

Zinc concentration. (mg/L)	q_{exp}	***Pseudo-first order***			**Pseudo-second**			**Intraparticle diffusion**	
		qe (mg/g)	k_1 (min^{-1})	R^2	qe (mg/g)	k_2 (g/(mg min))	R^2	ki (mg/(gmin))	R^2
10	0.97	0.92	0.0207	0.976	1.226	0.05	0.998	0.109	0.968
20	1.63	1.05	0.0161	0.974	2.15	0.014	0.991	0.176	0.973
40	2,65	2.81	0.0133	0.954	3.225	0.0112	0.99	0.262	0.976
60	3.06	3.57	0.0011	0.976	3.69	0.0099	0.99	0.305	0.984

E. Biosorption Isotherms

The analysis of equilibrium data to monitor the biosorption process is quite important for design purposes. Biosorption isotherms express the mathematical relationship between the quantity of adsorbate and equilibrium concentration of adsorbate remaining in the solution at a constant temperature. Biosorption data for wide range of adsorbate concentration are most conveniently described by biosorption isotherms. The biosorption studies were carried out at 303, 313 and 323 K to determine the biosorption isotherms and the isotherm parameters were evaluated using non-linear Langmuir and Freundlich models. Langmuir biosorption isotherm is often used to describe the maximum biosorption capacity of biosorbent, which is a most important parameter for a biosorption system. The Langmuir equation [14] is written as follows:

$$\frac{C_e}{q_e} = \frac{q_{max}K_lC_0}{1+K_LC_0} \quad (8)$$

The above can be rewritten to the following linear form:

$$\frac{Ce}{q_e} = \frac{1}{K_Lq_{max}} + \frac{Ce}{q_{max}} \quad (9)$$

where, q_{max} (mg/g) is the maximum biosorption capacity. K_L (L/mg) is a constant related to the affinity of binding sites or bonding energy. q_{max} denotes a practical limiting biosorption capacity when the surface of biosorbent is completely covered with adsorbate. The maximum biosorption capacity is compared in Table 3 with the data reported by other authors for zinc biosorption. As can be seen, the maximum zinc biosorption value of LC is higher than those reported in the literature.

This comparison indicates the great potential of LC for the removal of zinc from waste water .The empirical Freundlich isotherm is based on biosorption on a heterogeneous surface. It is expressed by the following equation [15]:

$$q_e = K_FC_e^{1/n} \quad 10)$$

Where K_F [(mg/l).(l/mg)$^{1/n}$] and n (dimensionless) are the Freundlich constant and exponent related to the biosorption capacity and intensity, respectively. Eq. (12) is generally used at the linear form represented by:

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (11)$$

The Langmuir and Freundlich isotherms for the biosorption of zinc on the luffa Cylindrica powder at different temperatures are presented in Figures 7 and 8. The corresponding Langmuir and Freundlich parameters along with correlation coefficients are given in Table 4. The experimental results are well fitted by the Langmuir model then the Freundlich model considering the values of regression coefficients presented in Table 3which are higher than 0.99. The values of q_{max} and K_L calculated from Langmuir plots were found to be 4.69 mg/g and 0.0940 L/mg for the experiments carried out at 303K.. The values of q_{max} increased from 4.5 to 4.29 mg/g, when the solution temperature increased from 313 to 323 K. The increasing trend was observed for the values of K_L depending upon the temperature of solution. The increase in the values of q_{max} and K_L with temperature indicates that the zinc ions are favorably adsorbed by LC powder at less temperature, which shows that the zinc biosorption phenomenon is exothermic. The essential feature of the Langmuir isotherm can be expressed in terms of dimensionless separation constant or equilibrium parameter (R_L), which is defined as:

$$R_L = \frac{1}{(1+K_L\ C_0\ )} \quad (12)$$

Where K_L is the Langmuir constant and Co is the initial concentration of zinc ions. The value of separation parameter R_L provides important information about the nature of biosorption. The value of RL indicated the type of Langmuir isotherm to be irreversible (R_L =0), favorable (0< R_L < 1), linear (R_L = 1) or unfavorable (R_L >1). The R_L was found to be 0.150-0081for concentration of 10-120 mg/L of zinc. They are in the range of 0-1 which indicates the favorable biosorption.

The Temkin isotherm model assumes the biosorption energy decreases linearly with the surface coverage due to adsorbent adsorbate interactions. The linear form of Temkin isotherm model [16] and [17] is defined by:

$$q_e = RTln\ K_T + \frac{RT}{b_T}\ In\ C_e \quad (13)$$

Where b_T is the Temkin constant related to heat of biosorption (J/mol) and KT is the Temkin isotherm constant (L/g). These constants n were obtained from plotting qe versus ln Ce. Values of b_T and K_T are listed in Table 4

The D-R isotherm model is a semi-empirical Eq. (14), where biosorption follows a pore filling mechanism. It assumes that the biosorption has a multilayer character, involves van der Waals forces and is applicable for physical biosorption processes. The linear form of D-R isotherm model [18] is expressed as:

$$ln\ q_e = In\ q_d - \beta\varepsilon^2 \quad (14)$$

Where qd is the D-R constant (mg/g), b is the constant related to free energy and e is the Polanyi potential which is defined as:

$$\varepsilon = RTln\left(1 + \frac{1}{C_e}\right) \quad (15)$$

The Polanyi potential e was determined from Eq. (15). The calculated q_d and b constants in Eq. (14), Table 4 were determined from intercept and slope of a plot of ln qe versus ε^2. From the data in Table 3, it was observed that Langmuir model showed a better fit than the Freundlich isotherm, Temkin equation, and Dubinin Radushkevich (D-R) equation, thus indicating the applicability of monolayer coverage of zinc (ions on the Luffa Cylindrica (LC) surface. Also the equilibrium data were also well described by Temkin equation, Table3.

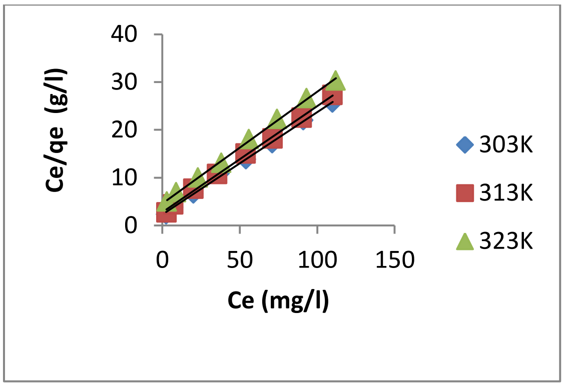

Figure.7: Langmuir isotherm for Zn(II) biosorption onto LC:. at pH 6.

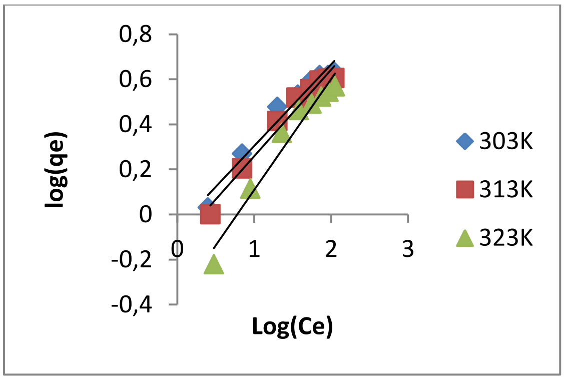

Figure.8 Freundlich isotherm for Zn(II) biosorption onto LC:. at pH 6.

Table 3: Langmuir, Freundlich, Temkin and Dubinin–Radushkevich constants for Zn(II) biosorption by LC.

Langmuir	T°(C)	q_{max} (mg/g)	k_l (L/mg)	R^2
	303	4.67	0.0944	0.998
	313	4.5	0.081	0.998
	323	4.29	0.05	0.998
Freundlich		K_L(mg/l)	1/n	R^2
	303	0.872	0.362	0.962
	313	0.766	0.384	0.972
	323	0.412	0.493	0.958
Temkin	T°(C)	**b_T (J/mol)**	**K_T (L/g)**	R^2
	303	2849.7	1.000098	0.880
	313	2957.1	1.000014	0.880
	323	3048.05	1.00016	0.810
D-R		**qd(mg/g)**	B	R^2
	303	3.57	2E-06	0.812
	313	3.33	2E-06	0.821
	323	2.88	3E-à-	0.812

F. Thermodynamic study

The thermodynamic parameters can be determined from following equations [19, 20]:

$$\Delta G^0 = -RTlnKc \quad (16)$$

$$In\ K_c = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \quad (17)$$

$$\Delta G^0 = \Delta H^0 - T\Delta S^0 \quad (18)$$

where kc can be defined as:

$$K_c = \frac{as}{ae} = \frac{Ys}{Ye} = \frac{Cs}{Ce} \quad (19)$$

where Kc is the distribution constant, as is activity of adsorbed Zn(II), ae is the activity of Zn(II) in solution at equilibrium, Cs is activity coefficient of adsorbed Zn(II), Ce is activity coefficient of Zn (II) in equilibrium solution, Cs is Zn(II) adsorbed on LC(mg/g), and Ce is Zn(II) concentration in equilibrium solution (mg/L).**ΔG°** , **ΔH°** and **ΔS°** are changes in Gibbs free energy (kJ/mol), enthalpy (kJ/mol) and entropy (J/mol K), respectively. R is the gas constant (8.314 J/mol K) and T is the temperature (K). The values of**ΔH°** and **ΔS°** were determined from the slope and the intercept from the plot of lnKc versus 1/T.

The thermodynamic parameters of the zinc biosorption onto LC are given in Table 5. The standard Gibbs free energies **ΔG°** of biosorption were negative at all investigated temperatures. The negative values of **ΔG°** of the biosorption confirmed that the biosorption of zinc onto LC was feasible and spontaneous [21]. In addition, the**ΔG°** values increased as the temperature increased, suggesting that biosorption might be more spontaneous at lower temperature. Generally, the range of free energy values **ΔG°** for physisorption is between

-20 and 0 kJ/mol, while chemisorption is between -80 and -400 kJ/mol [22]. This further indicated that the biosorption of the zinc onto LC was by physisorption. The change in biosorption standard enthalpy **ΔH°** for zinc adsorbed onto LC was 0.463 kJ/mol, indicating a weak bonding between the zinc and L C since zinc was adsorbed and penetrated into the interlayer space of adsorbent, indicating that the biosorption process was exothermic [23]. The biosorption of zinc onto modified LC can be concluded to be via physisorption since the change in the standard enthalpy is less than 40.0 kJ/mol [24]. The standard entropy change **ΔS°** for zinc adsorbed onto LC was -1.455 J/mol. The negative value of $\Delta S°$ suggested a decrease in degree of freedom of the adsorbed zinc.

Table 4: Thermodynamic parameters of Zn(II) biosorption onto LC at different T.

T (K)	$\Delta G°$ (kJ/mol)	$\Delta H°$ (kJ/mol)	$\Delta S°$ (J/mol K)
303	-1.53		
313	-0.627	-0.463	-1.455
323	-0.48		

IV. Conclusion

The potential of Luffa Cylindrica (LC) for the removal of Zn(II) ions from aqueous solutions was dependent on biosorption process such as pH, initial Zn(II) ions concentration, biosorbent dose, contact time, and temperature. The data obtained from thermodynamic studies were used to calculate the thermodynamic quantities such as $\Delta G°$, $\Delta H°$ and $\Delta S°$ biosorption. The results indicated that zinc (II) biosorption onto LC was found to be spontaneous and exothermic. The equilibrium data have been analyzed using Langmuir, Freundlich isotherms, Temkin equation, and D-R. The characteristic parameters for each isotherm and related correlation coefficients, R^2 were determined. The Langmuir biosorption isotherm was demonstrated to provide the best correlation for the biosorption of zinc (II) ions onto LC. The maximum monolayer biosorption capacity of LC was found to be 4.67 mg/g of zinc (II)/g of LC. The kinetic results provided the best correlation of the experimental data of biosorption of zinc (II) onto LC by pseudo second-order equation. It can be concluded that since the LC is an easily, locally available, low-cost adsorbent and has a considerable high biosorption capacity, it may be treated as an alternative adsorbent for treatment of wastewater containing zinc (II) ions.

Acknowledgments

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