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Kinetic and Isothermal Studies for Removal of Zn (II) Ions from Aqueous Solutions by Using *Lawsonia inermis* as a Novel Biosorbent

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Authors' contributions

This work was carried out in collaboration between both authors. Both authors read and approved the final manuscript.

Original Research Article

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ABSTRACT

In this work, the use of a cost-effective biosorbent *Lawsonia inermis* was investigated for removal of Zn (II) ions from aqueous solutions. Different operational parameters such as the effect of pH, biomass dose, equilibrium time, temperature and initial metal ion concentrations were studied. Maximum adsorption of Zn (II) took place at optimum conditions of pH 5.0 and biomass dose of 0.2 g/L. It was found that the interactions between zinc ions and *Lawsonia inermis* were complicated, and the pH of solution was a key governing factor of such interactions. Biosorption equilibrium was achieved in 60 minutes. The adsorption kinetics followed Pseudo second order kinetic model and the value of rate constant was found to be $1.23 \times 10^{-2} \text{ g mg}^{-1} \text{ min}^{-1}$. Fourier transform infrared (FTIR) spectroscopy was used to characterize the surface functional groups of the biosorbent. FTIR analysis was used to reveal the involvement of hydroxyl and carboxyl groups in the removal of Zn (II) from aqueous streams. Linear Langmuir and Freundlich models were applied to describe the equilibrium isotherms, and both of the two models were synchronic. The Langmuir adsorption capacity (q_m) was found to be 76.92 mg g^{-1} . Desorption experiments indicated that 0.5 M HCl was efficient desorbent for the recovery of Zn (II) from biomass. All the experimental facts reveal the efficiency of *Lawsonia inermis* based Zn (II) removal technology.

Keywords: Zn (II); *Lawsonia inermis*; biosorption; isotherm; kinetic.

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1. INTRODUCTION

Heavy metals have been excessively released into the environment due to rapid industrialization and have created a major global concern [1]. They can be accumulated in living tissues, causing various diseases and disorders [2]. They exist in aqueous streams from many industries such as metal plating, mining, tanneries, painting, car radiator manufacturing, as well as in agricultural runoff [3]. Zinc can be considered as a raw material for corrosion-resistant alloys and brass, for galvanizing steel and iron products. Zinc oxide is a white pigment for rubber and paper. On the other hand, zinc is an essential element for human health, but free zinc ions in solution are highly toxic to plants and fishes. Trace amounts of free zinc ions can cause heavy damage to the environment and can kill some organisms [4,5]. Excessive intake of zinc by human can cause eminent health problems, such as stomach cramps, skin irritations, vomiting, nausea and anemia [6]. According to guidelines for Drinking Water Quality of the World Health Organization (GDWQ-WHO), the daily requirement of zinc of an adult human is 15–22 mg/day (WHO) [7]. The National Institutes of Health, Office of Dietary Supplements recommended 40mg/day as a tolerable upper intake level for adults from foods and supplements [8]. So it is essential to remove zinc from aqueous streams.

The main techniques currently used for metal removal include chemical precipitation, electrochemical deposition, evaporation, cementation, membrane process, ion-exchange and activated carbon adsorption [9-12]. However, the application of these conventional techniques is often limited due to their inefficiency, high capital investment or operational costs. For example, ion-exchange resins and activated carbons are efficiently used in the removal of a range of metals and exhibit high uptake capacities, but their utilisation may be prohibitively costly for treating huge volume of water. Consequently, there is an emerging requirement for novel, efficient and cost-effective techniques for the remediation of metal bearing waters before their discharge into the environment.

Biosorption is one of the potentially promising technology for the removal and recovery of heavy metal ions from aqueous streams. It can be an alternative to the above conventional processes. Several types of biosorbent such as Leaf Powder of *Caesalpinia bonducella* [13], crab carapace [14], bael leaves (*Aegle marmelos*) [15], *Momordica charantia* [16], *Moringa oleifera* bark [17], wine processing Sludge [18], grain crops [19], apple wastes [20], mushroom biomass (*Agaricus bisporus*) [21] have been used to remove metal ions from water.

Lawsonia inermis is a much-branched glabrous shrub or small tree, cultivated for its leaves although stem bark, roots, flowers and seeds have also been used in traditional medicine. This plant is commonly known as Henna or Mhendi and abundantly available in tropical and subtropical areas. Its leaves are small, opposite in arrangement along the branches, subsessile, about 1.5 to 5 cm long, 0.5 to 2 cm wide, greenish brown to dull green, elliptic to broadly lanceolate with entire margin, petiole short and glabrous and acute apex with tapering base [22].

In the present study, the potential of *Lawsonia inermis* leaf powder as novel biosorbent for removal of Zn (II) ions from aqueous streams was investigated. The influence of various factors such as pH, biosorbent dose, equilibrium time, temperature and initial metal ion concentration were also studied. Equilibrium and kinetic parameters were studied to determine the biosorption mechanism. Desorption studies were conducted to find out reusability of biosorbent.

2. MATERIALS AND METHODS

2.1 Plant Collection and Preparation of Biomass

The *Lawsonia inermis* leaves were collected from the different sites located in the region of Raipur, Chhattisgarh, India. The leaves were washed with Milli-Q ultrapure water several times to remove surface impurities, sun-dried and subsequently dried in an electric oven at 60°C for 3 days. The dried biomass was grounded and sieved through 250- μm mesh Tyler screen and stored in an air-tight container in order to avoid moisture. The fine biomass obtained was used for further adsorption studies.

2.2 Preparation of Zinc Solution

All analytical reagents grade chemicals used in this work were obtained from MERCK (India/Germany). All the solutions were prepared with Milli-Q ultrapure water. All the glassware's used were washed with 10% (v/v) HNO_3 and rinsed with water. A Zinc metal stock solution of 1000 mg L^{-1} was prepared by dissolving the required amount of $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (purity >99.99%) in Milli-Q ultrapure water and making the volume up to 1000 ml in a volumetric flask. To the stock solution 3–6 drops of concentrated HNO_3 were added to prevent hydroxide formation. Fresh working solutions were prepared by appropriate dilution of stock solution prior to use.

2.3 Instrumentation

The Zn (II) ions in the solution were analyzed using Atomic adsorption spectrophotometer AAS4129D, ECIL India which operates in flame mode. A high precision electronic balance was used for weighing and a digital pH meter was used for the measurements of pH of solutions. An orbital shaker was used for shaking solutions.

2.4 Batch Adsorption Study

Batch adsorption experiments of the *Lawsonia inermis* were carried out in 250 ml Erlenmeyer flasks at 25°C. The effect of pH on metal uptake by *Lawsonia inermis* was studied by varying the pH in the range 2.0-8.0 with either 0.1 M NaOH or 0.1 M HCl, fixed quantity of biomass (0.2 g) was added to each flask containing 100 ml of 50 mg L^{-1} of zinc solution and the mixtures were thoroughly stirred for 60 min at 200 rpm, which was the enough duration to achieve equilibrium. The effect of initial metal ion concentration was studied in the range 10-500 mg L^{-1} and was treated with a 0.2 g of biomass at pH 5.0 for 60 min. The effect of biosorbent dose was carried out by taking known weight of the *Lawsonia inermis* ranging from 0.1-0.8 g into 250 mL Erlenmeyer flasks containing 100 mL of 50 mg L^{-1} of zinc metal solution and was agitated for 60 min at 200 rpm at 25°C. The effect of temperature was studied by the procedure for initial concentration at 25°C, 30°C, 40°C and 50°C. The effect of contact time was investigated in the range 15-180 min at pH 5.0 at 25°C.

Finally the *Lawsonia inermis* biomass was separated from the metal solution by passing through a whatmanm filter paper no. 42 and the filtrate was analyzed by using AAS. The amount of metal bound by the biosorbent was calculated from the difference between the initial and equilibrium concentrations of the metal ions in solution. All the experiments were

performed in triplicate. The blank which was free from metal and biomass was used as a control.

The amount of zinc ions adsorbed by the biomass at equilibrium was calculated using the equation 1.

$$q_e = \frac{(C_o - C_e)V}{w} \quad (1)$$

Where q_e (mg g^{-1}) is the adsorption capacity at equilibrium,

C_o (mg L^{-1}) is the initial and C_e (mg L^{-1}) is the equilibrium concentration of metal ions,

W (g) is the weight of adsorbent used and V (ml) is the volume of sample.

Zinc ions removal efficiency (% R) was calculated using the equation 2.

$$R = \frac{C_o - C_e}{C_o} \times 100\% \quad (2)$$

Where C_o (mg L^{-1}) is the initial and C_e (mg L^{-1}) is the equilibrium concentration of metal ions, respectively.

2.5 Regeneration of Biosorbent

To find out the reusability of the biosorbent, same *Lawsonia inermis* biomass was used four times after regeneration. About 0.5 g of the *Lawsonia inermis* biomass was brought in contact with 100 ml of 50 mg L^{-1} of zinc solution at 25°C for 60 min with shaking by the orbital shaker and then separated by filtering through whatmanm filter paper no. 42. This separated biomass was washed with Milli-Q-ultra pure water and was left in 15 ml of eluting agent i.e. 0.1-0.7 HCl for 60 min at 25°C in beaker. The biomass was separated from solution by filtration and supernatant was analyzed for Zn (II) concentration. After that regenerated *Lawsonia inermis* biomass was dried in an oven at 60°C for 24 hr and then reused. Desorption efficiency was calculated by using following equation 3

$$\text{Desorption Efficiency} = \frac{\text{Amount of Zn (II) desorbed}}{\text{Amount of Zn (II) adsorbed}} \times 100 \quad (3)$$

2.6 FTIR Analysis

To find out possible active centers responsible for adsorption, FTIR spectrums of raw and metal loaded biomass were recorded with a Thermo Nicolet, Avatar 370 Model FTIR spectrometer in the range 4000-400 cm^{-1} having resolution 4 cm^{-1} at the SAIF KOCHI, Cochin, Kerala, India.

3. RESULTS AND DISCUSSION

3.1 Effect of pH on Biosorption

pH of the aqueous solution is a key factor influencing the adsorption process. It not only affects the states of the functional groups on the surface of the biomass, but also the existing form of the metal ions presented in solution [23]. To investigate the effect of the pH of solutions on the biosorption performance, experiments were carried out.

At pH less than 2 due to the presence of higher concentration of H^+ in the solution the removal efficiency is low, this is because higher concentration of H^+ competes with the Zn (II) ions for the active sites on the biosorbent surface. With the increase in the pH of the solution, the electrostatic repulsion decreases due to reduction of the positive charge density of protons on the sorption sites, thus resulting in an increase of Zn (II) biosorption. The maximum percent removal of Zn (II) was found to be 79% at pH 5.0, and it is shown in Fig. 1. With the further increase in pH beyond 5.0 caused a decrease in percent removal of Zn (II) ions due to higher pH precipitate formation Zn (II) ions as $Zn(OH)_2$ was observed in Zn (II) solution. The optimum pH value of 5.0 was maintained for all subsequent experiments.

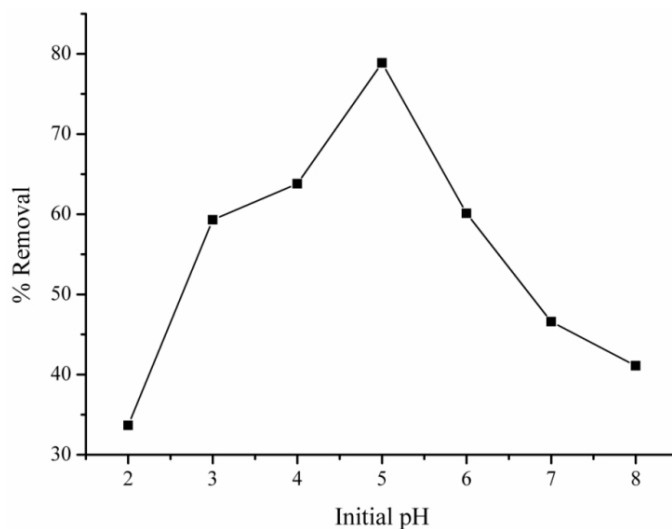


Fig. 1. Effect of pH on biosorption of Zn (II) onto the *L. Inermis*: Biosorption conditions: $C_0 = 50 \text{ mg L}^{-1}$; biosorbent dosage = 0.2 g; temperature = 25°C; rotational speed = 200 rpm; contact time = 60 min

3.2 Effect of Biosorbent Dose

The quantity of biosorbent can influence the extent of metal uptake from the solution. It was found that there was marginal increase in percent removal of Zn (II) ions from 69% to 88% with increasing the biosorbent dose from 0.1 to 0.8 g L^{-1} (Fig. 2).

3.3 Effect of Initial Metal Concentration

The effect of initial metal concentration on Zn (II) removal by using *Lawsonia inermis* is shown in Fig. 3. The result indicates that when the initial metal concentration increases, Zn (II) biosorption increases until the desorption sites are not saturated. This indicates that surface saturation is dependent on the initial metal ion concentrations [13]. The percent removal of metal was 85- 26% for 10-500 mg L⁻¹ of Zn (II) solution.

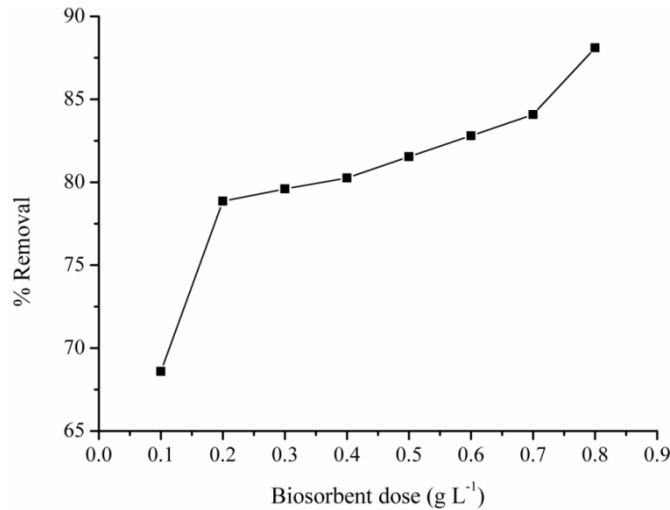


Fig. 2. Effect of biosorbent dose on biosorption of Zn (II) onto the *L. Inermis*: Biosorption conditions: C₀ = 50 mg L⁻¹; pH = 5.0; temperature = 25°C; rotational speed = 200 rpm; contact time = 60 min

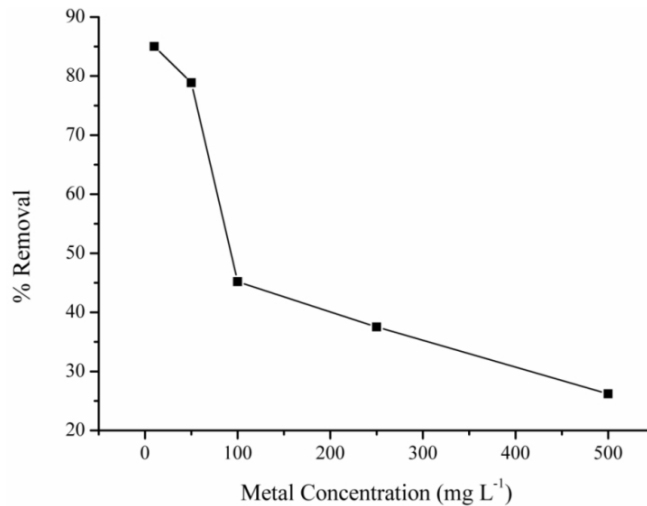


Fig. 3. Effect of initial metal concentration on biosorption of Zn (II) onto the *L. Inermis*: Biosorption conditions: biosorbent dosage = 0.2 g; pH = 5.0; temperature = 25 °C; rotational speed = 200 rpm; contact time = 60 min

3.4 Kinetic Study of Biosorption

The effect of contact time (15-180 min) on Zn (II) removal was investigated for an initial concentration of 50 mg L^{-1} of Zn (II) solution. It was observed (Fig. 4.) that the percentage removal of Zn (II) initially increased with contact time and the equilibrium was attained within 60 minutes. After this time there was decrease in percentage removal of Zn (II) biosorption. Therefore, the optimum contact time was selected as 60 min for further experimental studies.

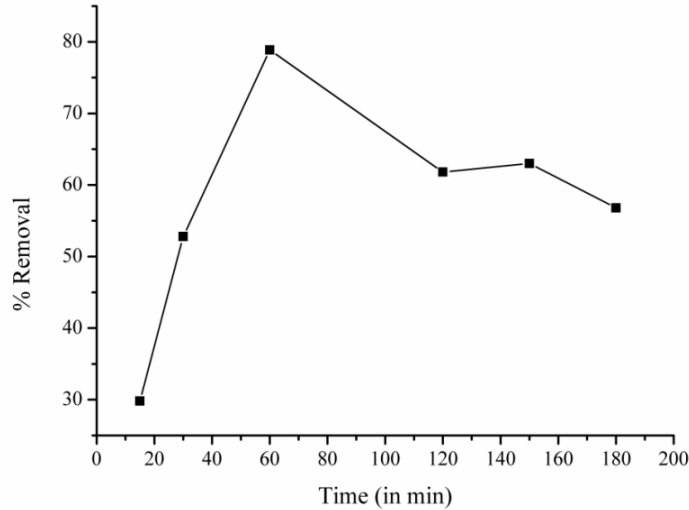


Fig. 4. Effect of contact time on biosorption of Zn (II) onto the *L. Inermis*: Biosorption conditions: $C_0 = 50 \text{ mg L}^{-1}$; biosorbent dosage = 0.2g; pH = 5.0; temperature = 25 °C; rotational speed = 200 rpm.

To analyze the adsorption kinetics of Zn (II) by the *Lawsonia inermis*, the pseudo-second-order was applied to the experimental data.

The pseudo-second-order (Eq. 4) kinetic rate equation [24] is expressed as

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (4)$$

Where, q_t is the amount of metal ion adsorbed at time t (mg g^{-1})

q_e is the amount of metal ions adsorbed at equilibrium (mg g^{-1}).

k_2 is the rate constant of second-order adsorption. ($\text{g mg}^{-1} \text{min}^{-1}$)

The pseudo-second-order rate constant (k_2) is obtained from a linear plot of t/q versus t (Fig. not shown), and the values are shown in Table 1.

Table 1. Pseudo second order kinetic parameters for the biosorption of Zn (II) onto the *L. Inermis*

Experimental	Pseudo second order kinetic model		
q_e (mg g ⁻¹)	k_2 (g mg ⁻¹ min ⁻¹)	q_e (mg g ⁻¹)	R ²
19.71	0.0123	15.62	0.972

Based on correlation ($R^2 = 0.972$) as given in Table 1, it can be concluded that Pseudo second order kinetic model is appropriate to describe this adsorption.

3.5 Effect of Temperature

Biosorption experiments were performed at different temperatures (25°, 30°, 40° & 50°C) for 50 mg L⁻¹ of Zn (II) ion at constant pH 5.0 and a biosorbent dose of 0.2 g L⁻¹. It was found that a temperature range has insignificant influence on adsorption behavior of biomass and there was an increase in the percentage removal of Zn (II) ions with rise in temperature (Fig. 5). Since adsorption is an energy-independent phenomenon, it is less likely to be affected by temperature [23].

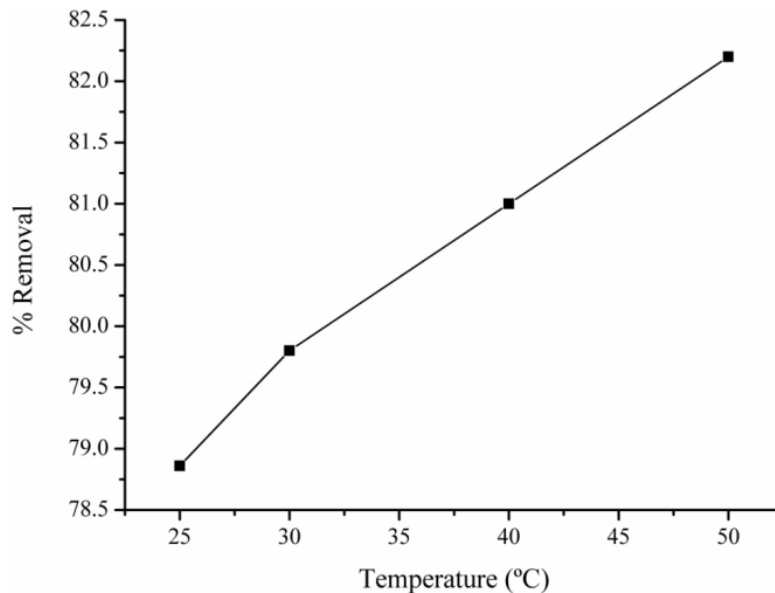


Fig. 5. Effect of temperature on biosorption of Zn (II) onto the *L. Inermis*: Biosorption conditions: $C_0 = 50$ mg L⁻¹; biosorbent dosage = 0.2 g; pH: 8.0; rotational speed = 120 rpm; contact time = 60 min

3.6 Isotherm Studies

The biosorption capacity of *Lawsonia inermis* was predicted by using the isotherms which explain the distribution of adsorbate molecules (Zn) between liquid (metal salt solution) and solid phase (biomass) when the adsorption process reaches equilibrium state. The isotherms were investigated using two models: Langmuir and Freundlich models [25].

3.6.1 Langmuir isotherm

The Langmuir isotherm model assumes that uptake of metal ions occurs on a homogeneous surface by monolayer adsorption and there is no interaction between adsorbed species. The Langmuir equation can be represented as follows:

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m}$$

Where, C_e is the concentration of the adsorbate at equilibrium in the solution (mg L^{-1}), q_e is the amount adsorbed at equilibrium on the adsorbent (mg g^{-1}), q_m is the monolayer adsorption capacity of the adsorbent (mg g^{-1}) and K_L = energy of adsorption constant (L mg^{-1}) or Langmuir constant.

A plot of C_e/q_e Vs C_e gives a straight line. The value q_m and K_L were determined from the slopes and intercept of the graph (Fig. not shown) and are given in Table 2.

The essential characteristics of the Langmuir isotherms can be expressed in terms of a dimensionless constant separation factor or equilibrium parameter, R_L , which is defined as:

$$R_L = \frac{1}{1 + K_L C_0}$$

Where, C_0 is the initial concentration of Zn (II) in the solution (mg L^{-1}).

The value of R_L indicates the shape of isotherm to be either unfavorable ($R_L > 1$) or linear ($R_L = 1$) or favorable ($0 < R_L < 1$) or irreversible ($R_L = 0$). The R_L was found to be 0.562 indicating favorable adsorption.

3.6.2 Freundlich isotherm

The Freundlich Isotherm model assumes that that uptake of metal ions occurs on a heterogeneous adsorbent surface. The Freundlich equation can be represented as follows:

$$\log q_e = \log K_F + \frac{1}{n} \log C_e$$

Where, K_F is the constant related to adsorbent capacity (mg g^{-1}) and $1/n$ is an empirical parameter related to sorption intensity of the adsorbent ($n = 1-10$).

A plot of $\log q_e$ Vs $\log C_e$ gives a straight line. The value K_F and n were determined from the slopes and intercept of the graph (Fig. not shown) and are given in Table 2.

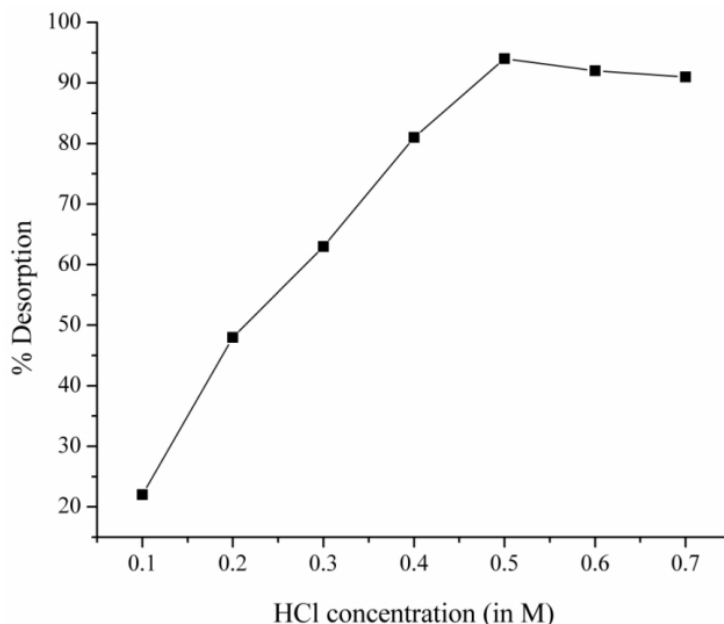
The high regression coefficients ($R^2 > 0.92$) suggested that both Langmuir and Freundlich models were suitable for describing the adsorption behavior of Zn (II) by *Lawsonia inermis* adsorbent.

Table 2. Values of parameters and correlation coefficient of Langmuir and Freundlich equation

Langmuir constants			Freundlich constants		
q_m (mg g^{-1})	K_L (L g^{-1})	R^2	K_F (mg g^{-1})	n	R^2
76.92	0.0156	0.929	4.3052	2.145	0.940

3.7 Desorption of Zn (II) by HCl

The result of the desorption study of Zn (II) by the HCl is shown in Fig. 6. It was observed that desorption efficiencies of Zn (II) by biomass increases with increase in HCl concentration because a high concentration of HCl desorbs the Zn (II) from adsorbed biomass. The maximum desorption (94%) of Zn (II) was achieved with 0.5 M HCl. The result shows that, beyond 0.5 M HCl concentration, the desorption capacity of biomass remained constant. The *Lawsonia inermis* biomass could be repeatedly used in Zn (II) ion biosorption studies without significant losses in their initial adsorption capacities. Hence 0.5 M HCl is selected as the desorbing agent in the present study.

**Fig. 6. Effect of HCl concentration on desorption of Zn (II) onto the *L. Inermis***

3.8 FTIR Studies

The FTIR spectra of pure and Zn (II)-loaded biomass were taken to determine functional groups involved in the process of metal binding, Fig. 7. Shows the FTIR Spectra of pure and Zn (II) loaded biomass. FTIR spectra of the pure biomass show the change in the intensity of the bands after binding with Zn (II). The broad peak at 3376 cm^{-1} is the characterization of –OH stretching vibrations. The peak at 2933 cm^{-1} is related to the aromatic C–H stretching vibrations [26]. The peak at 1725 cm^{-1} and 1661 cm^{-1} was attributed to C=O stretching vibration [27]. Those peaks at 1626 cm^{-1} are related to the aromatic –C=C– bonds [28].

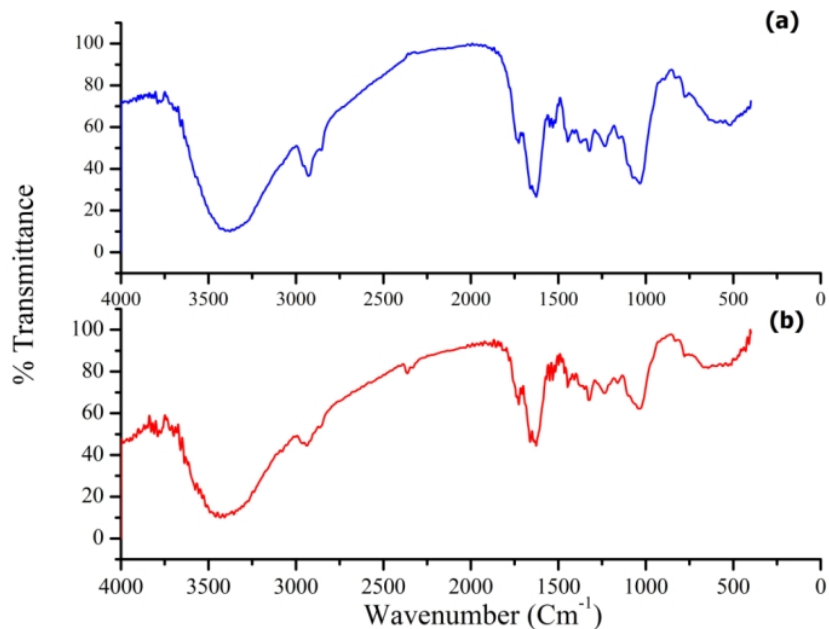


Fig. 7. FTIR Spectra of (a) Pure biomass (b) Zn (II) loaded biomass

After Zn (II) adsorption -OH stretching vibration was shifted to 3416 cm^{-1} , respectively. This shift of peak to the higher wave number revealed that chemical interaction takes place between the metal ions and the hydroxyl (-OH) groups on the biomass surface. In addition after metal adsorption the peaks of C=O stretching vibration shifted to 1726 and 1666 cm^{-1} , respectively. These results indicated that the -OH and C=O were mainly involved in the biosorption of Zn (II) by *Lawsonia inermis* biomass.

4. COMPARISON OF *Lawsonia inermis* WITH OTHER BIOSORBENTS

Zn (II) uptake by *Lawsonia inermis* was compared with the literature values of other biosorbents as shown in Table 3. The biosorption capacity of present biosorbents was much higher than most of the other biosorbents reported in literature. Hence, it can be used on large scale application due to the abundant availability of the biomass.

Table 3. Comparison of Zn (II) adsorption capacity with some reported biosorbent materials

Biosorbents	Adsorption capacity (mg g^{-1})	Reference
<i>Penicillium chrysogenum</i>	13	[29]
African Alfalfa shoots	4.9	[30]
Rice straw	13.2	[31]
Corncoobs	1.96	[32]
Sugar beet pulp	17.78	[32]
<i>Penicillium spinulosum</i>	0.2	[33]
Blast furnace slag	17.65–98.08	[34]
Lignite	22.83	[34]
<i>Lawsonia inermis</i>	76.92	Present work

5. CONCLUSION

The present study shows that the *Lawsonia inermis* was an efficient biosorbent for Zn (II) removal from aqueous streams in a very rapid adsorption process. Batch adsorption studies concluded that all parameters such as pH, biomass dose, equilibrium time, temperature and initial metal ion concentrations had effect on the removal of Zn (II). The phenomenon was explained with the Langmuir and Freundlich isotherms. The maximum biosorption capacity of the biomass was 76.92 mg g⁻¹. The kinetics of adsorption followed Pseudo second order kinetics. The result of this study reveals that such *Lawsonia inermis* biomass can very well be applied for water treatments and control of environmental pollution.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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